Food and Agriculture Organization of the United Nations

World Health Organization

EVALUATION 2022

PART I - RESIDUES

Pesticide Residues in Food

Joint FAO/WHO Meeting on Pesticide Residues

Pesticide Residues in Food 2022

Joint FAO/WHO Meeting on Pesticide Residues

Evaluation
Part I - Residues

Sponsored jointly by FAO and WHO Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group, Rome, Italy 13–22 September 2022

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Monographs containing summaries or residue data and toxicological data considered at the 2022 JMPR, together with recommendations, are available upon request from FAO or WHO under the title:
Pesticide residues in food 2022 Evaluations Part I: Residues

DISCLAIMER OF THE JMPR

This monograph was written by experts based on the evaluation of the original studies and the dossier provided by the sponsor of the compound, of the relevant published scientific literature and the data submitted by Codex members. When found consistent with the data of the original study, the monograph may contain parts of the text and tables of the dossier submitted by the sponsor, but not the sponsor's conclusions. This represents an appropriate use of such material and does not represent an endorsement of the sponsors' position.

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* New compound

** Evaluated within the periodic review programme of the Codex Committee on Pesticide Residues

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Abbreviations

ADI	acceptable daily intake
ADME	absorption, distribution, metabolism, excretion
AGF	aspirated grain fractions
AR	administered radioactivity
ARfD	acute reference dose
as	as received
BBCH	Biologische Bundesanstalt, Bundessortenamt und Chemische Industrie
bw	body weight
CAC	Codex Alimentarius Commission
CAS	Chemical Abstracts Service
CCPR	Codex Committee on Pesticide Residues
cGAP	critical GAP
ChE	cholinesterase
DAA	days after application
DALA	days after last application
DAT	days after treatment
DM	dry matter
DT_{50}	time required for 50% dissipation of the initial concentration
dw	dry weight
EFSA	European Food Safety Authority
eq	equivalent(s)
ESI	electrospray ionization
FAO	Food and Agriculture Organization of the United Nations
GAP	good agricultural practice
GC-ECD	gas chromatography – electron capture detector
GC-MS	gas chromatography – mass spectrometry
GC-NPD	nitrogen-phosphorus detectors
GECDE	global estimate of chronic dietary exposure
GEMS	Global Environment Monitoring System – Food Contamination Monitoring and Assessment Programme
GI	gastrointestinal tract
GLP	good laboratory practice

HBGV	health-based guidance values
HPLC	high performance liquid chromatography
HR	highest residue level in the edible portion of a commodity
HR-P	highest residue level in a processed commodity
IEDI	International Estimated Daily Intake
IESTI	International Estimate of Short-Term Dietary Intake
ILV	independent laboratory validation
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
JECFA	Joint FAO/WHO Expert Committee on Food Additives
JMPR	Joint FAO/WHO Meeting on Pesticide Residues
LC-MS/MS	liquid chromatography-tandem mass spectrometry
LOAEL	lowest-observed-adverse-effect level
LOD	limit of detection
LOQ	limit of quantification
LP	large portion
MOA	mode of action
MRL	maximum residue limit
NOAEC	no-observed-adverse-effect concentration
NOAEL	no-observed-adverse-effect level
NTE	neuropathy target esterase
OECD	Organisation for Economic Co-operation and Development
PBI	plant-back interval
PF	processing factor
PHI	pre-harvest interval
Po	post-harvest
ppm	parts per million
PXR	pregnane X receptor
QuEChERS	quick, easy, cheap, effective, rugged and safe
QSAR	quantitative structure–activity relationship
RAC	raw agricultural commodity
RSD	relative standard deviation
RTI	re-treatment interval
SC	suspension concentrate

SD	standard deviation
SDHI	succinate dehydrogenase inhibitor
SPE	solid phase extraction
STMR	supervised trials median residue
STMR-P	supervised trials median residue in a processed commodity
TLC	thin layer chromatography
TRR	total radioactive residues
TTC	threshold of toxicological concern
UK	United Kingdom of Great Britain and Northern Ireland
USA	United States of America
v/v	volume for volume
WHO	World Health Organization
w/v	weight for volume
w/w	weight for weight

Use of JMPR reports and evaluations by registration authorities

Most of the summaries and evaluations contained in this report are based on unpublished proprietary data provided for use by JMPR in making its assessments. A registration authority should not grant a registration on the basis of an evaluation unless it has first received authorisation for such use from the owner of the data provided for the JMPR review or has received the data on which the summaries are based, either from the owner of the data or from a second party that has obtained permission from the owner of the data for this purpose.

1. Introduction

A Joint Meeting of the Food and Agriculture Organization of the United Nations (FAO) Panel of Experts on Pesticide Residues in Food and the Environment and the World Health Organization (WHO) Core Assessment Group on Pesticide Residues (JMPR) was held at FAO Head-quarters, Rome (Italy), from 13 to 22 September 2022. The FAO Panel Members met in preparatory sessions from 8–12 September.

The Meeting was opened by Dr Jingyuan Xia, Director, Plant Production and Protection Division (NSP), FAO. On behalf of FAO and WHO, Dr Xia welcomed and thanked the participants for providing their expertise and for devoting significant time and effort to the work of the JMPR, noting that this was the first physical JMPR meeting since 2019 due to the impact of the COVID-19 pandemic, with 45 participants from 15 countries.

Dr Xia highlighted food safety is fundamental to healthy and sustainable food systems. The establishment of pesticide residue standards is a key and critical element in the global effort to improve food safety and agricultural development in the world. The unique role of the JMPRs work in establishing internationally acceptable MRLs for pesticide residues in food and feed which acted as global benchmarks in trade facilitation, as well as providing authoritative assessments, important in consumer protection. Dr Xia then outlined how the JMPRs efforts aligned with the Divisions strategic objectives of ensuring food security and nutrition; enhancing food quality and safety; supporting farmers' livelihoods; protecting the environment and biodiversity; and facilitating safe trade and economic growth. As the establishment of global standards were a key and critical element in the global efforts to improve food safety and agricultural development in the world.

Dr Xia also took the opportunity to express his and called on the meeting participants toexpress their appreciation to Madam YongZhen Yang, retiring FAO JMPR Secretariat, for her dedicated commitment and outstanding contribution in fulfilling the secretariat role over the past 16 years.

Dr Soren Madsen, WHO JMPR Secretariat, took the opportunity to thank the FAO for giving priority to JMPR to alow the meeting to occur at the FAO.

During the meeting, the FAO Panel of Experts was responsible for reviewing residue and analytical aspects of the pesticides under consideration, including data on their metabolism, fate in the environment and use patterns, and for estimating the maximum levels of residues that might occur as a result of use of the pesticides according to good agricultural practice (GAP). Maximum residue levels and supervised trials median residue (STMR) values were estimated for commodities of animal origin. The WHO Core Assessment Group was responsible for reviewing toxicological and related data in order to establish acceptable daily intakes (ADIs) and acute reference doses (ARfDs), where necessary.

The Meeting evaluated 34 pesticides, including seven new compounds and four compounds that were re-evaluated within the periodic review programme of the CCPR, for toxicity or residues, or both.

The Meeting established ADIs and ARfDs, estimated maximum residue levels and recommended them for use by CCPR, and estimated STMR and highest residue (HR) levels as a basis for estimating dietary intake.

The Meeting also estimated the dietary exposures (both short-term and long-term) of the pesticides reviewed and, on this basis, performed a dietary risk assessment in relation to the relevant ADI and where necessary ARfD. Cases in which ADIs or ARfDs may be exceeded were clearly indicated in order to facilitate the decision-making process by CCPR.

The Meeting considered a number of current issues related to the risk assessment of chemicals, the evaluation of pesticide residues and the procedures used to recommend maximum residue levels.

AFIDOPYROPEN (312)

First draft prepared by Mr C Sieke, Federal Institute for Risk Assessment, Berlin, Germany

EXPLANATION

Afidopyropen is an insecticide developed for control of piercing and sucking insects. Afidopyropen disrupts the gating of TRPV (Transient Receptor Potential Vanilloid) channel complexes in chordotonal stretch receptor organs of insects. This disrupts feeding and other behaviour in target insects leading to death by starvation.

Afidopyropen was first evaluated by the 2019 JMPR when an ADI of 0–0.08 mg/kg bw was established. The Meeting also established an ARfD of 0.2 mg/kg bw for women in childbearing age and an ARfD of 0.3 mg/kg for the general population. In addition, it was concluded that the metabolites M440I007 and CPCA are likely to be of similar toxicity to its parent. The 2021 JMPR reconsidered the wording of the residue definition. The residue definitions are:

Definition of the residue for compliance with the MRL for plant and animal commodities: *afidopyropen*

Definition of the residue for dietary risk assessment for plant commodities: sum of afidopyropen + dimer of [(3R,6R,6aR,12S,12bR)-3-[(cyclopropanecarbonyl)oxy]-6,12-dihydroxy-4,6a,12b-trimethyl-11-oxo-9-(pyridin-3-yl)-1,3,4,4a,5,6,6a,12,12a,12b-decahydro-2H,11H-naphtho[2,1-b]pyrano[3,4-e]pyran-4-yl]methyl rac-cyclopropanecarboxylate (M007)

Definition of the residue for dietary risk assessment for animal commodities, except liver: afidopyropen + M001 + CPCA and its carnitine conjugate, expressed as afidopyropen

Definition of the residue fir dietary risk assessment for liver: afidopyropen + M001 + M017 + CPCA and its carnitine conjugate, expressed as afidopyropen

The residue is not fat-soluble.

At the Fifty-first Session of the CCPR (2019), afidopyropen was scheduled for toxicology and residue evaluation by the 2022 JMPR.

The Meeting received information from the manufacturer on aerobic soil metabolism, use patterns and residues resulting from supervised trials on strawberries, sorghum and alfalfa/clover.

ENVIRONMENTAL FATE IN SOIL

The Meeting received information on environmental fate in soil.

Aerobic metabolism and degradation in soil with afidopyropen

In an aerobic soil metabolism study (Nejad, H., 2020, 2018/7007218), a silt loam from New Jersey (NJ), a loamy sand from California (CA), loamy sand (LUFA 2.2 or L2) and sandy loam (LUFA 5M or L5) soils from Germany were dosed with pyranone-6-^{14}C-afidopyropen at approximately 0.2 mg/kg (~10.3 µg/vessel). This concentration was selected to represent an application rate of 50 g/ha considering uniform distribution to 2.5 cm depth and a soil bulk density of 1.0 g/cm^3. Following dosing, the soil sample vessels were incubated aerobically in the dark at approximately 50 percent of maximum water holding capacity moisture and 20±2 °C over 120 days. Additional samples of each soil were dosed with solvent blank acetonitrile and incubated similarly for determination of biomass. High dosed samples were prepared at

10 times rate with mixture of ^{12}C and ^{14}C-afidopyropen (approximately 50:50 ratio) at approximately 0.102 mg/vessel.

Table 1 Soil characteristics (pyranone label)

Soil name	LUFA 2.2 (L2)	LUFA 5M (L5)	California (CA)	New Jersey (NJ)
Location, Coordinates	Hanhofen, Rheinland-Pfalz, Germany 49.31308, 8.32697	Rheinland-Pfalz / Mechtersheim Germany 49.27203, 8.40464	Guadalupe, San Luis Obispo, CA 34.98405, -120.53395	Baptistown, Hunterdon, NJ 40.54563, -74.99312
Soil texture (USDA)	Loamy sand	Sandy loam	Sandy loam	Silt loam
-- Sand (percent)	83	59	77	15
-- Silt (percent)	14	28	14	60
-- Clay (percent)	3	13	9	25
Organic Carbon (percent)	1.5	1.2	0.42	1.2
Organic Matter (percent)	2.5	2.0	0.72	2.0
CEC (meq/100 g)	7.9	9.3	7.2	7.7
pH (1`:1 soil:water)	6.0	7.7	7.9	7.1
Moisture holding capacity (MWHC) (g/100 g dry soil)	32.9	40.0	20.0	44.4
Moisture holding capacity at 1/3 bar (percent moisture)	10.7	14.5	5.8	27.4
Moisture holding capacity at 15 bar (percent)	7.3	8.2	3.4	9.3
Microbial biomass (mg microbial carbon/100 g dry soil)	171.6 (0 DAT) 179.0 (120 DAT)	112.2 (0 DAT) 133.0 (120 DAT)	38.6 (0 DAT) 20.9 (120 DAT)	115.7 (0 DAT) 126.2 (120 DAT)

Notes:

MWHC = Maximum Water Holding Capacity.

Duplicate samples for each label were removed at 0, 5, 12, 29, 62, and 120 days after treatment (DAT). The soil moisture of each vessel was adjusted periodically to maintain approximately 50 percent MWHC.

Immediately after removal from the incubator system, the soil samples were extracted with solvents (as outlined below) by shaking for approximately 30 minutes at 300 rpm followed by centrifugation at 4000 rpm for another 15 minutes. The supernatant was decanted into a graduated cylinder, the volume adjusted, and an aliquot assay by LSC.

Sample concentration:

For 0–5 DAT, equal aliquots from each extract were pooled together. For 12 DAT and on, equal aliquots of each extract were pooled together, concentrated by rotary evaporator at 30 °C to small volume then, the rotavap flask was rinsed with ACN, followed by water. The combined concentrate was further concentrated to smaller volume and adjusted to final volume, vortexed and centrifuged prior to liquid scintillation counting (LSC) and high-pressure liquid chromatography (HPLC) analysis.

Volatiles Analysis:

The aqueous sodium hydroxide traps were assayed at all sampling times (except for 0 DAT) by directly adding aliquots of the trapping solution into liquid scintillation cocktail and counting by LSC. The traps were replaced with fresh aqueous sodium hydroxide (1N), at each sampling time.

Bound Residues Analysis:

Non-extractable radioactive residues (NER) were characterized by destructive NaOH treatment with 0.5 M NaOH. The radioactivity was determined by LSC analysis.

The remaining soil was allowed to dry at room temperature. Triplicate aliquots of samples were combusted and the resulting $^{14}CO_2$ was trapped and measured by LSC in order to determine the ^{14}C residues in the humin fraction.

To fractionate the fulvic acids and the humic acids, the fractions were adjusted to pH 1–2 by adding concentrated HCI and storing in the refrigerator. After precipitation, the suspension was centrifuged. The supernatant containing the fulvic acids was decanted and analysed by LSC. The radioactivity found in the humic acid was calculated based on the following equation: Humic Acid = [Sum(NaOH Ext 1-3 and water wash)-Fulvic Acid]. The fulvic acid fraction was further analysed by partitioning two times with ethyl acetate. Each ethyl acetate phase and the aqueous phase were measured by LSC. The ethyl acetate extracts were combined and reduced to dryness using a rotary evaporator, the remainder was taken up using an appropriate solvent and measured by LSC. An aliquot was subjected to HPLC analysis.

Table 2 Distribution of radioactivity (percent TAR) in soils treated with 0.2 mg ai/kg (50 g ai/ha) [6-PYRA-^{14}C]-labelled afidopyropen in German and United States soils at 20 °C and 50 percent MWHC (values are means of duplicate analyses)

DAT	0	5	12	29	62	120
Characterisation	percent TAR	percent TAR	percent TAR	percent TAR	percent TAR	percent TAR
CA Soil						
total extracted (ERR)	98.1	95.7	94.2	87.6	69.6	50.8
ACN	91.3	77.6	70.3	60.8	41.3	27.5
ACN:water (3:7, v/v)	6.8	18.1	22.8	25.7	27.1	22.3
EtoAc	n.a.	n.a.	1.2	1.1	1.2	1.0
Hexane	n.a.	n.a.	<0.5 percent	<0.5 percent	<0.5 percent	<0.5 percent
NER	1.9	5.2	4.3	9.1	16.0	17.8
CO_2	n.a.	1.3	1.6	3.0	10.0	25.1
Total	100	102.2	100.1	99.7	95.5	93.7
LUFA 2.2 (L2) soil						
total extracted (ERR)	97.1	94.1	88.9	74.7	65.2	47.6
ACN	88.6	77.6	60.9	42.3	33.9	20.3
ACN:water (3:7, v/v)	8.6	16.3	27.1	31.8	30.8	26.8
EtoAc	n.a.	n.a.	0.9	0.6	0.5	0.6
Hexane	n.a.	n.a.	<0.5 percent	<0.5 percent	<0.5 percent	<0.5 percent
NER	2.9	6.1	10.3	22.4	30.1	41.8
CO_2	n.a.	0.9	1.4	2.3	4.1	7.0
Total	100	101.1	100.6	99.4	99.3	96.4
NJ soil						
total extracted (ERR)	97.8	89.4	84.8	67.8	48.4	36.6
CAN	91.0	74.4	60.5	39.6	23.6	16.9
ACN:water (3:7, v/v)	6.8	15.0	22.2	27.3	24.1	19.0
EtoAc	n.a.	n.a.	2.1	0.9	0.7	0.6
Hexane	n.a.	n.a.	<0.5 percent	<0.5 percent	<0.5 percent	<0.5 percent
NER	2.2	9.9	12.0	26.9	37.5	41.2
CO_2	n.a.	1.2	1.9	4.3	10.6	17.6
Total	100	100.5	98.7	99.0	96.4	95.4
LUFA 5M (L5) soil						
total extracted (ERR)	100	94.4	02.0	76.8	54.0	39.6

DAT	0	5	12	29	62	120
Characterisation	percent TAR	percent TAR	percent TAR	percent TAR	percent TAR	percent TAR
ACN	90.2	76.4	66.9	49.1	28.7	18.6
ACN:water (3:7, v/v)	9.7	18.1	20.8	24.0	21.3	17.6
EtoAc	n.a.	n.a.	1.4	0.7	0.7	0.6
Hexane	n.a.	n.a.	<0.5 percent	<0.5 percent	<0.5 percent	<0.5 percent
NER	2.8	6.9	7.7	16.7	25.5	29.2
CO_2	n.a.	1.5	2.6	7.1	15.4	25.1
Total	102.9	102.8	102.3	100.6	94.8	93.9

Notes:

n.a. = Not analysed; ERR: extracted radioactive residue; NER = non-extracted residue; –= not detected (<0.1 percent TAR).

Table 3 Identification and characterization of radioactivity (percent TAR) in soils treated with 0.2 mg ai/kg (50 g ai/ha) [6-PYRA-^{14}C]-labelled afidopyropen in German and United States soils at 20 °C and 50 percent MWHC (values are means of duplicate analyses)

DAT	0	5	12	29	62	120
Characterisation	percent TAR	percent TAR	percent TAR	percent TAR	percent TAR	percent TAR
CA Soil						
Afidopyropen (RT 49.1-49.2 min)	98.2	82.4	56.0	33.9	17.0	13.5
Unknown (RT 3.9-4.1 min)	<0.02	1.3	<0.02	<0.02	3.0	3.8
Unknown (RT 18.3-18.7 min)	<0.02	<0.02	<0.02	<0.02	3.7	3.5
Unknown (RT 19.9-20.3 min)	<0.02	<0.02	0.3	3.2	2.2	1.5
Unknown (RT 20.9-21.2 min)	<0.02	<0.02	2.0	7.4	4.7	2.5
Unknown (RT 25.0-25.1 min)	<0.02	<0.02	<0.02	<0.02	1.2	0.7
Unknown (RT 26.7-26.8 min)	<0.02	<0.02	0.8	2.7	4.2	2.8
Unknown (RT 27.6-28.1 min)	<0.02	<0.02	<0.02	1.1	1.7	1.3
M02 (RT 29.4-29.9 min)	<0.02	3.0	6.5	5.1	2.1	1.8
Unknown (RT 33.4-33.5 min)	<0.02	<0.02	0.4	1.7	3.1	2.3
M57 (RT 34.8-35.2 min)	<0.02	4.7	20.0	25.7	20.8	10.9
M03 (RT 37.9-38.2 min)	<0.02	<0.02	3.0	2.1	1.1	1.2
M24 (RT 46.2-46.6 min)	<0.02	<0.02	0.6	1.1	<0.02	<0.02
Unknown (RT 50-52 min)	<0.02	0.7	1.0	0.7	<0.02	<0.02
Others*	<0.02	3.1	0.6	3.1	5.2	4.9
LUFA 2.2 (L2) soil						
Afidopyropen (RT 49.1-49.2 min)	97.1	70.2	53.3	33.9	24.9	15.6
Unknown (RT 3.9-4.1 min)	<0.02	2.9	2.4	4.8	2.7	5.0
Unknown (RT 19.4-20.4 min)	<0.02	<0.02	<0.02	1.6	1.5	1.2
Unknown (RT 20.7-21.2 min)	<0.02	<0.02	1.3	1.6	2.8	1.2
Unknown (RT 23.4-23.6 min)	<0.02	<0.02	<0.02	<0.02	3.3	3.1
Unknown (RT 26.5-26.8 min)	<0.02	<0.02	<0.02	<0.02	1.2	1.2
Unknown (RT 27.0-27.3 min)	<0.02	<0.02	<0.02	<0.02	1.7	1.8
Unknown (RT 27.6-28.1 min)	<0.02	<0.02	1.5	1.4	<0.02	<0.02
M02 (RT 29.3-29.9 min)	<0.02	3.7	3.2	1.0	<0.02	<0.02
Unknown (RT 33.4-33.5 min)	<0.02	<0.02	1.3	2.6	1.5	0.6
M57 (RT 34.8-35.2 min)	<0.02	<0.02	2.1	1.4	0.8	<0.02
Unknown (RT 36.0-36.2 min)	<0.02	<0.02	<0.02	0.7	0.8	1.0
M16 (RT 37.5-37.6 min)	<0.02	<0.02	5.5	5.4	5.9	3.4
M03 (RT 38.0-38.5 min)	<0.02	13.1	11.7	7.8	8.9	2.7
M24 (RT 46.2-46.6 min)	<0.02	4.3	5.7	4.7	2.9	2.1
Unknown (RT 50-52 min)	<0.02	<0.02	1.2	0.7	0.5	<0.02
Others*	<0.02	<0.02	<0.02	7.6	6.2	9.1
NJ soil						

DAT	0	5	12	29	62	120
Characterisation	percent TAR	percent TAR	percent TAR	percent TAR	percent TAR	percent TAR
Afidopyropen (RT 49.1-49.2 min)	97.8	70.2	40.0	25.7	11.8	9.9
Unknown (RT 3.9-4.1 min)	<0.02	<0.02	1.5	2.2	3.7	0.9
Unknown (RT 18.3-19.0 min)	<0.02	<0.02	<0.02	1.6	1.2	1.0
Unknown (RT 19.8-20.4 min)	<0.02	<0.02	<0.02	1.3	1.2	1.6
Unknown (RT 20.8-21.2 min)	<0.02	<0.02	1.5	1.0	0.9	1.2
Unknown (RT 25.0-25.1 min)	<0.02	<0.02	<0.02	0.4	0.5	<0.02
Unknown (RT 26.5-26.8 min)	<0.02	<0.02	<0.02	0.9	1.2	1.0
Unknown (RT 27.6-28.1 min)	<0.02	<0.02	0.5	2.5	2.7	1.6
M02 (RT 29.4-29.9 min)	<0.02	<0.02	6.0	3.2	1.3	0.5
Unknown (RT 33.4-33.5 min)	<0.02	<0.02	0.5	1.3	1.3	0.9
M57 (RT 34.8-35.2 min)	<0.02	<0.02	3.0	2.3	1.1	0.3
M16 (RT 37.5-37.6 min)	<0.02	<0.02	2.5	5.5	6.4	4.5
M03 (RT 38.0-38.2 min)	<0.02	11.8	10.0	7.7	4.2	1.9
M24 (RT 46.2-46.6 min)	<0.02	7.4	6.5	9.5	9.0	5.7
Unknown (RT 50-52 min)	<0.02	<0.02	1.0	0.6	0.3	0.3
Others*	<0.02	<0.02	<0.02	2.6	2.1	4.9
LUFA 5 (L5) soil						
Afidopyropen (RT 49.1-49.2 min)	100	76.6	50.0	38.4	22.0	15.1
Unknown (RT 3.3-3.4 min)	<0.02	<0.02	<0.02	1.0	1.8	1.4
Unknown (RT 3.9-4.1 min)	<0.02	<0.02	2.5	3.4	2.4	3.2
Unknown (RT 18.2-18.7 min)	<0.02	<0.02	<0.02	1.0	3.0	1.9
Unknown (RT 19.4-20.4 min)	<0.02	<0.02	<0.02	0.9	1.2	1.1
Unknown (RT 20.7-21.2 min)	<0.02	<0.02	1.5	2.5	2.2	0.7
Unknown (RT 26.3-26.8 min)	<0.02	<0.02	0.7	2.5	2.2	1.8
Unknown (RT 27.6-28.1 min)	<0.02	<0.02	<0.02	0.6	0.7	<0.02
M02 (RT 29.3-29.9 min)	<0.02	4.3	8.0	4.8	4.5	1.2
M57 (RT 34.8-35.2 min)	<0.02	<0.02	8.0	9.4	6.1	3.5
M03 (RT 37.9-38.5 min)	<0.02	8.4	9.0	9.1	3.5	2.6
M24 (RT 46.2-46.6 min)	<0.02	<0.02	<0.02	0.3	0.2	<0.02
Unknown (RT 50-52 min)	<0.02	<0.02	1.0	0.8	0.5	0.4
Others*	<0.02	3.2	0.8	2.4	4.2	7.0

Notes:

n.a. = Not analysed; ERR: extracted radioactive residue; NER = non-extracted residue;–= not detected (<0.1 percent TAR).

*Others reflects a combined value of two or more peaks. No individual peak in "Others" exceeded 2 percent TAR.

Characterization of post extraction solids (PES) was performed with selected samples from both soils and both label treatments by NaOH extraction and subsequent fractionation into fulvic acids, humic acids and humins.

Table 4 Characterisation of none-extracted residues in soils treated with 0.2 g ai/kg (50 g ai/ha) [4-PYR-^{14}C]- labelled afidopyropen or [6-PYRA-^{14}C/2,6-PYRI-^{14}C]-labelled afidopyropen in German and US soils (values are means of replicate analyses)

DAT	62	120
Characterisation	percent TAR	percent TAR
CA soil		
NER	16.0	17.8
NaOH	13.0	14.3
humic acids	1.8	2.4
fulvic acids	11.2	11.9
humins	3.8	4.2
LUFA 2.2 soil		

DAT	62	120
Characterisation	percent TAR	percent TAR
NER	30.1	41.8
NaOH	27.2	37.2
humic acids	7.8	13.4
fulvic acids	19.4	23.8
humins	3.3	4.8
NJ soil		
NER	37.4	41.2
NaOH	32.3	34.3
humic acids	4.8	5.8
fulvic acids	27.5	28.4
humins	6.6	8.3
LUFA 5 soil		
NER	25.4	29.2
NaOH	21.4	23.7
humic acids	5.2	6.8
fulvic acids	16.2	17.0
humins	5.3	7.0

Notes:

DAT = Days after treatment; PES = post extraction solids; n.d. = not determined.

A kinetic analysis for afidopyropen and metabolites M002, M003, M016, M024 and M057 was performed using KinGUI (version 2.2014.224.1704).

Table 5 Degradation endpoints ($DegT_{50}$, $DegT_{90}$) for afidopyropen and metabolites derived from best-fit models in a pathway fitting scheme

Compound	Soil	Best-fit model	chi^2 error [percent]	$DegT_{50}$ [d]	$DegT_{90}$ [d]
afidopyropen	CA soil	FOMC	6.90	14.4	120.6
	NJ soil	FOMC	5.77	9.52	82.3
	LUFA 2.2	FOMC	1.57	13.6	268.9
	LUFA 5	FOMC	4.37	13.6	208.7
M002	LUFA 2.2	SFO	6.45	4.30	14.3
M057	CA soil	SFO	4.37	39.9	132.7
	NJ soil	SFO	29.7	7.73	25.7
	LUFA 2.2	SFO	37.8	10.6	35.1
	LUFA 5	SFO	14.6	19.5	64.9
M003	LUFA 2.2	SFO	24.8	23.8	79.1
	LUFA 5	SFO	12.0	17.5	58.2
M024	NJ soil	SFO	12.6	78.2	259.7
	LUFA 2.2	SFO	11.9	27.7	92.1
M016	NJ soil	SFO	6.43	32.0	106.3

Notes:

FOMC = Gustafson-Holden model; DFOP = bi-exponential kinetics model; SFO = single first order kinetics.

USE PATTERN

For the purpose of estimating new MRLs, use pattern information for afidopyropen from the United States were submitted.

Table 6 List of uses of afidopyropen considered by the current Meeting

Crop or crop group	Country	Rate	Number of treatments (minimum interval)	Pre-harvest interval (PHI)
Strawberries	United States	0.05 kg ai/ha (max. 0.1 kg ai/ha and crop and max. 0.3 kg ai/ha per year)	7 days	0 days
Sorghum	United States	0.02 kg ai/ha (max. 0.044 kg ai/ha and year)	14 days	7 days (forage) 14 days (grain and stover)
Grass forage, fodder and hay group	United States	0.036 kg ai/ha (max. 0.058 kg ai/ha and year)	7 days	0 days
Non-grass animal feed group (forage, fodder, hay, seed and straw) [a]	United States	0.036 kg ai/ha (max. 0.058 kg ai/ha and year)	7 days	0 days

Notes:

[a] alfalfa, bean velvet, clover, kudzu, lasperdeza, lupin, sainfoin, trefoil, vetch, vetch crown, vetch milk.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised residue trials for strawberries, sorghum, alfalfa and clover.

Crop	Commodity	Treatment	Table no.
Strawberries	Fruits	Foliar	Table 7
Alfalfa	Seeds	Foliar	Table 8
Sorghum	Grain	Foliar	Table 9
Alfalfa	Forage	Foliar	Table 10
Clover	Forage	Foliar	Table 11
Grass	Forage	Foliar	Table 12
Sorghum	Forage	Foliar	Table 13
Alfalfa	Hay	Foliar	Table 14
Clover	Hay	Foliar	Table 15
Grass	Hay	Foliar	Table 16
Alfalfa	Straw	Foliar	Table 17
Sorghum	Straw	Foliar	Table 18

All trials have been conducted with a Dispersible Concentrate (DC) formulation. Application rates, spray concentrations and residues have been rounded to two figures. Residue data are recorded un-adjusted for percentage recoveries or for residue values in control samples unless otherwise stated. Soil characteristics were not included in the tables as all applications are foliar applications.

The limit of quantification (LOQ) for the method is 0.01 mg/kg for parent afidopyropen and plant metabolite M007. Non-quantifiable residues are listed as <0.01 mg/kg. For calculation of total residues, residues below LOQ are considered as <0.01 mg/kg.

Residues are expressed in their respective analytes. The totals are summed and expressed in parent equivalents, without correction.

Where multiple analyses were conducted on a single sample, the average value is reported. Where multiple samples were taken from a single plot, the individual and average values are reported. Results are therefore sometimes presented as single values or as duplicate/triplicate single values with the (mean) value between brackets. Where results from separate plots with distinguishing characteristics such as different formulations, crop varieties or treatment schedules were reported, results are listed separately for each plot.

Residues from the trials conducted according to the critical GAP, which have been used for the estimation of maximum residue levels (mean values), STMR and HR (based on the individual samples) values are underlined.

Strawberries

A total of five greenhouse trials were conducted on strawberries during the 2016 and 2017 growing seasons in the United States.

Each trial consisted of one untreated plot (Treatment 01) and one treated plot (Treatment 02). At each trial, four foliar applications of afidopyropen (100.0 g ai/L DC formulation) were made to the treated plots targeting 0.010 kg ai/ha/application for the first and second application and 0.050 kg ai/ha/application for the third and forth application with 7-day retreatment intervals each, totaling 0.120 kg ai/ha per season. The applications were made with 315–567 L/ha of spray volume with an adjuvant added to the spray mixture for all applications. All applications were made using appropriate spray equipment, and the spray volume was sufficient to provide adequate dispersal of the test substance.

At all the greenhouse trials, fresh strawberries were harvested on the day of the last application (0-day PHI). In decline trial, 11680.16-FL127, samples were collected at 0, 1, 3, 7 and 14 days after the last application.

A minimum sample size of 60 fruits respective of >1kg of fruit/sample were harvested for all trials. Frozen samples were delivered from the field sites via freezer truck to IR-4 North Central Research Center in Lansing, Michigan. Fruit samples were stored frozen until processed. Raw agricultural commodity (RAC) samples were ground using a Robot Coupe RSI 10B blender with dry ice. The entire sample was chopped and homogenized. Sample analysis for residues of afidopyropen was conducted by the IR-4 North Central Research Center, Lansing, MI, United States.

All samples were analysed for residues of afidopyropen and metabolite M440I007 using BASF Method D1103/01, which quantifies residues by LC-MS/MS and was already reviewed by the 2019 JMPR. Additional concurrent recoveries of afidopyropen and M440I007 in strawberry samples ranged from 78 to 114 percent and 70 to 115 percent for M440I007 with RSDs of 1–16 percent. The limit of quantification (LOQ) was 0.01 mg/kg.

The maximum freezer storage interval between sampling and analysis was 289 days for all analytes. Extracts were analysed within 24 hours.

Table 7 Residues of afidopyropen in strawberries (greenhouse) after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location	Application					Residue mg/kg [a]			
	no. (RTI)	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	DAT	parent	M007	Total	Study Reference, Trial No.
United States, 2018, Citra, Florida	4 (7)	10.3 10.0 49.7 48.7	324 315 324 318	3.2 3.2 15.4 15.3	0	0.0422, 0.0528 (0.0475)	<0.01, <0.01 (<0.01)	0.0522, 0.0628 (0.0575)	2018/7007938, 16-FL127
					1	0.0309, 0.0403 (0.0356)	<0.01, <0.01 (<0.01)	0.0409, 0.0503 (0.0456)	
					3	0.034, 0.036 (0.035)	<0.01, <0.01 (<0.01)	0.044, 0.046 (0.045)	
					7	0.0222, <0.01 (0.0161)	<0.01, <0.01 (<0.01)	0.0322, <0.02 (0.0262)	
					14	0.0181, <0.01 (0.0140)	<0.01, <0.01 (<0.01)	0.0281, <0.02 (0.0240)	
Canada, 2017, Kentville, Nova Scotia	4 (6-7)	10.4 10.7 50.5 51.1	417 427 404 409	2.5 2.5 12.5 12.5	0	0.0378, 0.050 (0.0439)	<0.01, <0.01 (<0.01)	0.0378, 0.060 (0.0539)	2018/7007938, 16-NS527
Canada, 2017, Agassiz, British Columbia	4 (7-8)	10.2 10.5 49.9 50.0	421 431 426 426	2.5 2.5 11.7 11.7	0	0.0448, 0.0265 (0.0356)	<0.01, <0.01 (<0.01)	0.0548, 0.0365 (0.0456)	2018/7007938, 17-BC6
United States, 2017, Parlier, California	4 (7)	10.1 17.0 51.3 51.4	567 561 434 435	1.8 1.8 11.8 11.8	0	0.0523, 0.0678 (0.060)	<0.01, <0.01 (<0.01)	0.0623, 0.0778 (0.070)	2018/7007938, 17-CA1
United States, 2017, Chuckey, Tennessee	4 (6-7)	10.3 10.1 49.4 49.7	343 338 330 332	2.9 2.9 15.0 15.0	0	0.0351, 0.0302 (0.0326)	<0.01, <0.01 (<0.01)	0.0451, 0.0402 (0.0426)	2018/7007938, 17-TN472

Notes:

RTI = retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Alfalfa (seeds)

See alfalfa forage for study description.

Table 8 Residues of afidopyropen in alfalfa seeds after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location	Application						Residue mg/kg [a]			
	no.	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT	parent	M007	Total	Study Reference, Trial No.
United States, 2017, Northwood, North Dakota	2 (7)	22 34	140 178	15.7 19.1	Foliar, BBCH 89	0	0.076, 0.064 (0.07)	<0.01, <0.01 (<0.01)	0.086 0.074 (0.080)	2018/7005908, R170080
						7	0.041, 0.046 (0.0435)	<0.01, <0.01 (<0.01)	0.051 0.056 (0.0535)	
United States, 2017, San Joaquin, California	2 (7)	22 34	139 139	15.8 24.44	Foliar, BBCH 97	0	0.049, 0.066 (0.0575)	<0.01, <0.01 (<0.01)	0.059 0.076 (0.0675)	2018/7005908, R170081
						7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Malad City, Idaho	2 (6)	22 34	181 179	12.1 19.0	Foliar, BBCH 87–89	0	0.081, 0.096 (0.0875)	0.028, 0.036 (0.032)	0.109 0.132 (0.1195)	2018/7005908, R170082
						7	*0.033, 0.014 (0.0235)*	*<0.01, <0.01 (<0.01)*	*<0.02, <0.02 (<0.02)*	

Notes:

RTI = retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Sorghum (grain)

A set of twelve supervised field trials was conducted in the United States, each field trial included three plots, an application-free control plot and two treated plots. The first treated plot was for the collection of sorghum forage and the second treated plot was the collection of sorghum grain and stover. The test item (100.0 g ai/L DC formulation) was applied as two broadcast foliar applications at the maximum label nominal rate of 0.019–0.021 kg ai/ha with a 13–15 day retreatment interval, resulting in a total rate of 0.039–0.041 kg ai/ha. Applications were made to the first treated plot at target 21- and 7-days before normal commercial harvest. Applications were made to the second treated plot at target 28- and 14-days before normal commercial harvest. The applications were made using a spray volume of 193–278 L/ha. A commercially available adjuvant was added to the spray mixture.

Samples of forage, grain and stover were collected 0 to 21 days after the last application. A simulated aspirated grain fraction (AGF) was collected by sieving the frozen sorghum grain stored prior to homogenization. One composite simulated AGF sample was generated by combining the grain from

multiple trials. Forage samples were frozen within 4 hours of collection and weighed at least 1 kg. Grain and stover samples were frozen within 4 hours of collection and weighed at least 1 kg or 0.5 kg, respectively.

All RAC samples were maintained frozen at the field facilities and were kept on dry until homogenization and analysis. The residues of afidopyropen and M440I007 in sorghum samples were quantitated by LC-MS/MS using BASF Method No. D1103/01, which was already reviewed by the 2019 JMPR. Additional concurrent recoveries of afidopyropen and M440I007 in sorghum forage, grain and stover ranged from 72.0 to 108 percent (RSDs 9–11 percent), based on 4-5 replicate samples each at fortification levels of 0.01 and 1.0 mg/kg. For aspirated grain fractions, single concurrent recovery samples at 0.01 and 5 mg/kg showed recoveries of 76 to 95 percent. The maximum storage period for sorghum-based based samples in this study was 306 days (~10 months). Extracts were stored for a maximum of three days under refrigerator conditions, accompanied by concurrent standards.

Table 9 Residues of afidopyropen in sorghum grain after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location	Application						Residue mg/kg [a]			
	no.	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT	parent	M007	Total	Study Reference, Trial No.
United States, 2016, Plains, Georgia	2 (14)	20.1 19.6	212 211	9.5 9.3	Foliar, BBCH 91 + 95	14	0.097, 0.11 (0.1035)	0.011, <0.01 (0.01)	0.108 0.12 (0.1135)	2017/7016329, R161032
United States, 2016, Proctor, Arizona	2 (14)	19.8 19.8	196 196	10.1 10.1	Foliar, BBCH 86 + 88	14	0.035, 0.033 (0.034)	<0.01, <0.01 (<0.01)	0.045 0.043 (0.044)	2017/7016329, R161033
United States, 2016, Atlantic, Iowa	2 (15)	20.0 21.0	236 214	8.5 9.8	Foliar, BBCH 85 + 87	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	2017/7016329, R161034
United States, 2016, Leonard, Montana	2 (14)	20.7 20.3	220 215	9.4 9.4	Foliar, BBCH 85–89	14	0.042, 0.039 (0.0405)	<0.01, <0.01 (<0.01)	0.052, 0.049 (0.0505)	2017/7016329, R161035
United States, 2016, Zearing, Iowa	2 (14)	19.9 20.9	203 215	9.8 9.7	Foliar, BBCH 75–85	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	2017/7016329, R161036
United States, 2016, Stilwell, Kansas	2 (14)	20.3 19.8	278 267	7.3 7.4	Foliar, BBCH 85–87	0	0.052, 0.060 (0.056)	<0.01, <0.01 (<0.01)		2017/7016329, R161037
						9	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						14	<0.01, 0.011 (0.0105)	<0.01, <0.01 (<0.01)	<0.02, 0.021 (0.0205)	

Country, Year, Location	Application						Residue mg/kg [a]			
	no.	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT	parent	M007	Total	Study Reference, Trial No.
						16	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						20	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2016, Madill, Oklahoma	2 (14)	20.3 20.4	225 244	9.0 8.4	Foliar, BBCH 85	14	0.015, 0.016 (0.0155)	<0.01, <0.01 (<0.01)	0.025, 0.026 (0.0255)	2017/7016329, R1610348
United States, 2016, Hinton, Oklahoma	2 (13)	20.6 20.2	212 223	9.7 9.1	Foliar, BBCH 87–89	13	0.046, 0.039 (0.0425)	<0.01, <0.01 (<0.01)	0.056, 0.049 (0.0525)	2017/7016329, R161039
United States, 2016, Prosser, Nebraska	2 (15)	20.9 20.0	219 204	9.5 9.8	Foliar, BBCH 85–89	14	0.065, 0.069 (0.067)	<0.01, <0.01 (<0.01)	0.075, 0.079 (0.077)	2017/7016329, R161040
United States, 2016, Wall, Texas	2 (14)	20.6 20.3	276 274	7.5 7.4	Foliar, BBCH 87–89	12	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	2017/7016329, R161041
United States, 2016, Claude, Texas	2 (14)	20.1 20.6	224 231	9.0 8.9	Foliar, BBCH 85–88	14	0.017, 0.021 (0.019)	<0.01, <0.01 (<0.01)	0.027, 0.031 (0.029)	2017/7016329, R161042
United States, 2016, Dill City, Oklahoma	2 (13)	20.5 20.4	224 211	9.2 9.7	Foliar, BBCH 85–87	15	0.065, 0.077 (0.071)	<0.01, <0.01 (<0.01)	0.075, 0.087 (0.081)	2017/7016329, R161043

Notes:

RTI = retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Alfalfa (forage)

A total of 21 field trials were conducted during the 2017 and 2018 growing seasons, twelve on alfalfa and nine on clover. Each field trial included two plots, an application-free control plot and one treated plot. In the treated plot, DC test formulations were applied as two broadcast foliar applications. The first application was applied at a target rate of 0.022 kg ai/ha. The second application was applied with a 6 to 8 day retreatment interval (RTI) at a target rate of 0.034 kg ai/ha. The spray volume ranged from 131 to 187 L/ha. A commercially available adjuvant was added to the spray mixture.

The plots were harvested for either forage and hay (alfalfa and clover trials) or seed and straw (select alfalfa trials) at 0 and 7 DALA, targeting crop maturity. For the alfalfa trials, additional samples were taken at the second and third cuttings. For the two alfalfa decline trials, samples were harvested, targeting maturity, at 0, 3, 7, 14, and 21 days after last application. A sample size for alfalfa of 0.4–1.3 kg (hay), 1.0–2.9 kg (forage), 1.0–2.7 kg (seed) and 1.0–1.4 kg (straw) and for clover of 1.1–1.9 kg (forage) and 0.5–1.3 kg (hay) 1 to 2 kg was collected for all trials. All RAC samples were shipped frozen to the laboratory where samples were homogenized to a consistency appropriate for analysis using commercial equipment and stored in freezers. The frozen pre-homogenized samples were shipped from BASF to EAG Laboratories (Columbia, MO) for analysis.

The residues of afidypyropen and M440I007 in alfalfa forage, hay, seed, and straw, as well as clover forage and hay samples were quantitated by LC-MS/MS using BASF Method No. D1103/01. The concurrent recoveries for afidypyropen were 87 to 107 percent (95±6.5, n=12) in alfalfa forage, 83 to 110 percent (97±8.4, n=12) in alfalfa hay, 88 to 104 percent (94±7.8, n=4) in alfalfa seed, 98 to 111 percent (104±6.4, n=4) in alfalfa straw, 85 to 104 percent (97±7.3, n=9) in clover forage and 83 to 95 percent (90±4.9, n=9) in clover hay. M440I007 recoveries were 78 to 103 percent (90±9.2, n=12) in alfalfa forage, 83 to 121 percent (101±9.6, n=12) in alfalfa hay, 73 to 100 percent (88±14.7, n=4) in alfalfa seed, 84 to 105 percent (95±9.2, n=4) in alfalfa straw, 89 to 104 percent (98±5.7, n=9) in clover forage and 92 to 106 percent (97±4.6, n=9) in clover hay.

The non-grass animal feed RAC samples were stored frozen, from collection to extraction, for a maximum of 420 days, and were analysed within 9 days of extraction.

Table 10 Residues of afidopyropen in alfalfa forage after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location	Application						Residue mg/kg [a]			
	no.	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT	parent	M007	Total	Study Reference, Trial No.
United States, 2017, Germans-ville Pennsylvania	2 (6)	22 34	158 163	13.9 20.9	Foliar, BBCH 51-55	0	0.816, 0.773 (0.7945)	0.359, 0.319 (0.339)	1.176 1.092 (1.1335)	2018/7005908, R170070
						7	0.33, 0.389 (0.3595)	0.064, 0.063 (0.0635)	0.394 0.452 (0.423)	
						42 (2nd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						67 (3rd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Northwood, North Dakota	2 (7)	22 34	139 142	15.8 23.9	Foliar, BBCH 63	0	0.779, 0.683 (0.731)	0.522, 0.395 (0.4585)	1.301 1.078 (1.1895)	2018/7005908, R170072
						7	0.151, 0.151 (0.151)	0.046, 0.044 (0.045)	0.197 0.195 (0.196)	
						36 (2nd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	

Country, Year, Location	Application						Residue mg/kg [a]			
	no.	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT	parent	M007	Total	Study Reference, Trial No.
						82 (3rd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Shelbyville, Indiana	2 (7)	22 34	141 145	15.9 23.4	Foliar, BBCH 63	0	0.61, 0.603 (0.6065)	0.175, 0.178 (0.1765)	0.785 0.781 (<u>0.783</u>)	2018/7005908, R170073
						7	0.197, 0.171 (0.184)	0.032, 0.025 (0.0285)	0.229 0.196 (0.2125)	
						48 (2nd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						106 (3rd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Brunswick, Nebraska	2 (7)	22 34	141 138	15.6 24.6	Foliar, BBCH 65	0	1.619, 1.532 (1.5755)	0.729, 0.743 (0.736)	2.348 2.275 (<u>2.3115</u>)	2018/7005908, R170074
						7	0.26, 0.172 (0.216)	0.056, 0.043 (0.0495)	0.316 0.215 (0.2655)	
						29 (2nd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						55 (3rd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Aurora, South Dakota	2 (7)	22 34	141 138	15.6 24.6	Foliar, BBCH 61	0	1.274, 1.015 (1.1445)	0.999, 0.852 (0.9255)	2.243 1.867 (<u>2.07</u>)	2018/7005908, R170075
						7	0.298, 0.236 (0.267)	0.097, 0.076 (0.0865)	0.395 0.312 (0.3535)	
						27 (2nd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						67 (3rd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Montpelier, North Dakota	2 (7)	22 34	185 185	11.9 18.4	Foliar, BBCH 61	0	2.434, 2.374 (2.404)	0.046, 0.054 (0.050)	2.48 2.428 (<u>2.454</u>)	2018/7005908, R170076
						7	0.486, 0.501 (0.4935)	0.023, 0.022 (0.0225)	0.509 0.523 (0.516)	
						42 (2nd	<0.01, <0.01	<0.01, <0.01	<0.02, <0.02	

Country, Year, Location	Application						Residue mg/kg [a]			
	no.	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT	parent	M007	Total	Study Reference, Trial No.
						cut.)	(<0.01)	(<0.01)	(<0.02)	
						67 (3rd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Paso Robles, California	2 (7)	22 34	162 160	13.6 21.3	Foliar, BBCH 49	0	0.632, 0.886 (0.759)	0.275, 0.359 (0.317)	0.907 1.245 (1.076)	2018/7005908, R170077
						7	0.564, 0.60 (0.582)	0.168, 0.182 (0.175)	0.732 0.782 0.757	
						39 (2nd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						71 (3rd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Monte Vista, Colorado	2 (8)	22 34	184 176	12.0 19.3	Foliar, BBCH 61-63	0	0.785, 0.816 (0.8015)	0.191, 0.202 (0.1975)	0.976 1.028 (0.999)	2018/7005908, R170078
						3	0.368, 0.323 (0.346)	0.017, 0.016 (0.0165)	0.385 0.339 (0.3625)	
						7	0.192, 0.176 (0.184)	<0.01, <0.01 (<0.01)	0.202 0.186 (0.194)	
						14	0.060, 0.074 (0.067)	<0.01, <0.01 (<0.01)	0.07 0.084 (0.077)	
						21	0.025, 0.020 (0.0225)	<0.01, <0.01 (<0.01)	0.035 0.020 (0.0325)	
						49 (2nd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						76 (3rd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, American Falls, Idaho	2 (8)	22 34	163 166	13.5 20.5	Foliar, not reported	0	0.703, 0.703 (0.703)	0.52, 0.557 (0.5385)	1.223 1.26 (1.2415)	2018/7005908, R170079
						3	0.60, 0.578 (0.589)	0.257, 0.235 (0.246)	0.857 0.813 (0.835	
						7	0.424, 0.424 (0.424)	0.116, 0.113 (0.1145)	0.54 0.537 (0.5385)	
						14	0.227, 0.233	0.070, 0.069	0.297 0.302	

Country, Year, Location	Application					DAT	Residue mg/kg [a]			Study Reference, Trial No.
	no.	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage		parent	M007	Total	
							(0.23)	(0.0695)	(0.2995)	
						21	0.147, 0.169 (0.158)	0.061, 0.058 (0.0595)	0.208 0.217 (0.2175)	
						48 (2nd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						88 (3rd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	

Notes:

RTI = Retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Clover (forage)

See alfalfa forage for study description.

Table 11 Residues of afidopyropen in clover forage after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location	Application					DAT cut	Residue mg/kg [a]			Study Reference, Trial No.
	no. RTI	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage		parent	M007	Total	
United States, 2017, Frenchtown New Jersey	2 (8)	22 34	168 170	13.1 20.0	Foliar, BBCH 61-65	0	0.578, 0.524 (0.551)	0.121, 0.10 (0.1105)	0.699, 0.624 (0.6615)	2018/7005908, R170071
						7	0.245, 0.284 (0.2645)	0.028, 0.034 (0.031)	0.273 0.314 (0.2955)	
United States, 2017, Chula, Georgia	2 (7)	22 34	182 170	12.1 20.0	Foliar, BBCH 60	0	1.136, 0.534 (0.835)	0.257, 0.090 (0.1735)	1.393 0.624 (1.0085)	2018/7005908, R170083
						7	1.149, 0.538 (0.8435)	0.135, 0.106 (0.1205)	1.284 0.644 (0.964)	
United States, 2017, Washington Louisiana	2 (7)	22 34	163 181	13.5 18.8	Foliar, BBCH 60	0	1.602, 1.683 (1.6425)	0.573, 0.572 (0.5725)	2.175 2.255 (2.215)	2018/7005908, R170084
						7	0.17, 0.162 (0.166)	0.034, 0.032 (0.033)	0.204, 0.194 (0.199)	

Country, Year, Location	Application						Residue mg/kg [a]			
	no. RTI	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT cut	parent	M007	Total	Study Reference, Trial No.
United States, 2017, Northwood, North Dakota	2 (7)	22 34	141 141	15.6 24.1	Foliar, BBCH 61	0	0.526, 0.549 (0.5385)	0.383, 0.394 (0.3885)	0.909, 0.943 (0.926)	2018/7005908, R170085
						7	0.287, 0.278 (0.2825)	0.080, 0.076 (0.078)	0.367, 0.364 (0.3655)	
United States, 2017, Gardner, North Dakota	2 (7)	22 34	177 186	14.5 18.3	Foliar, BBCH 61-65	0	1.905, 1.837 (1.871)	0.733, 0.711 (0.722)	2.638 2.548 (2.593)	2018/7005908, R170086
						7	0.446, 0.526 (0.486)	0.064, 0.064 (0.064)	0.510, 0.590 (0.550)	
United States, 2017, Lebanon, Oklahoma	2 (8)	22 34	152 145	14.5 23.4	Foliar, BBCH 49-61	0	1.611, 1.594 (1.6025)	0.539, 0.433 (0.486)	2.150, 2.027 (2.0885)	2018/7005908, R170087
						7	0.206, 0.230 (0.218)	0.035, 0.035 (0.035)	0.231, 0.265 (0.248)	
United States, 2017, Montpelier, North Dakota	2 (7)	22 34	139 176	15.8 19.1	Foliar, BBCH 61-64	0	1.507, 1.587 (1.547)	0.130, 0.131 (0.1305)	1.637, 1.718 (1.6775)	2018/7005908, R170088
						7	0.483, 0.431 (0.457)	0.042, 0.040 (0.041)	0.525, 0.471 (0.498)	
United States, 2017, Claude, Texas	2 (7)	22 34	160 149	13.8 22.8	Foliar, BBCH 61-63	0	1.391, 1.362 (1.3765)	<.0.01, 0.011 (0.0105)	1.401, 1.373 (1.387)	2018/7005908, R170089
						7	0.421, 0.408 (0.4145)	0.014, 0.013 (0.0135)	0.435, 0.421 (0.428)	
United States, 2017, American Falls, Idaho	2 (8)	22 34	163 165	13.5 20.6	Foliar, BBCH 61-63	0	0.372, 0.430 (0.401)	0.190, 0.210 (0.20)	0.562, 0.640 (0.601)	2018/7005908, R170090
						7	0.106, 0.123 (0.115)	0.054, 0.055 (0.0545)	0.160, 0.178 (0.166)	

Notes:

RTI = retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Grasses (forage)

A total of twelve field trials were conducted during the 2017 growing season, four each on Bermuda grass and bluegrass and two each on fescue and brome grass, respectively across the United States.

Each field trial included two plots, an application-free control plot and one treated plot. In the treated plot, ta DC formulation was applied as two broadcast foliar applications. The first application was applied at a rate of 0.0196 to 0.0204 kg ai/ha. The second application was applied with a 6 to 7-day retreatment interval (RTI) at a rate of 0.0346 to 0.0359 kg ai/ha. The spray volume ranged from 140 to 187 L/ha using ground equipment, with a commercially available adjuvant added to the spray mixture for all applications.

For each trial, a forage sample were collected at normal crop maturity (~BBCH 38-60) 0 and 7 days after the last application (DALA). For the decline trials (R170029 for Bermuda grass and R170035 for fescue), forage samples were collected 3, 14, and 21 days after the last application. Forage samples weighed at least 1 kg each.

In addition, hay samples were collected at normal crop maturity (~BBCH 38-60) 0 and 7 days after the last application (DALA). The samples were allowed to dry for 1 to 10 days to a typical moisture level (10–20 percent) for hay. Hay samples weighed at least 0.4 kg each.

Frozen samples were shipped to the laboratory, homogenized using commercial processing equipment (vertical cutter and mill with dry ice) and stored in freezers. The frozen subsamples were then shipped for analysis. Samples were stored for a maximum of 396 days and analysed within 36 days of extraction. Stored extract were confirmed to be stable by concurrent recovery samples extracted and stored in parallel.

The residues of afidopyropen and M440I007 in grass forage and hay samples were quantitated by LC-MS/MS using BASF Method No. D1103/01. Concurrent recoveries of afidopyropen for forage matrices (bermuda grass, bluegrass, bromegrass, and fescue) ranged from 86 to 96 percent (mean=92 ± 2.9 percent, n=11, RSD=3.2). Concurrent recoveries of M440I007 for forage samples ranged from 83 to 107 percent (mean=96 ± 7.5 percent, n=11, RSD=7.8). Concurrent recoveries of afidopyropen for hay samples ranged from 84 to 100 percent (mean=93 ± 4.0 percent, n=12, RSD=4.3). Concurrent recoveries of M440I007 for hay samples ranged from 88 to 117 percent (mean=101 ± 8.8 percent, n=12, RSD=8.7).

All residues were observed to decline between the day 0 PHI and day 7 PHI. This was also observed in the decline trials for bermuda grass (forage and hay) and fescue (forage and hay) were residues decreaed with increasing pre-harvest intervals.

Table 12 Residues of afidopyropen in grasses forage after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location, Crop	Application					DAT	Residue mg/kg [a]			Study Reference, Trial No.
	no. (RTI)	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage		parent	M007	Total	
United States, 2017,	2 (6)	0.20 0.36	168 159	11.9 22.6	Foliar BBCH 41-43	0	1.1, 1.4 (1.25)	0.090, 0.13	1.19, 1.413	2018/7004965, R170026

Country, Year, Location, Crop	Application					DAT	Residue mg/kg [a]			Study Reference, Trial No.
	no. (RTI)	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage		parent	M007	Total	
Abbeville, Georgia, Bermuda grass								(0.11)	(1.36)	
						7	0.42, 0.40 (0.41)	<0.01, <0.01 (<0.01)	0.43, 0.41 (0.42)	
United States, 2017, Lecompte, Louisiana, Bermuda grass	2 (7)	0.20 0.35	150 150	13.3 23.3	Foliar BBCH 31-49	0	2.0, 1.6 (1.8)	0.27, 0.20 (0.235)	2.27, 1.8 (2.035)	2018/7004965, R170027
						7	0.077, 0.085 (0.081)	0.011, 0.012 (0.0115)	0.088, 0.097 (0.0925)	
United States, 2017, Uvalde, Texas, Bermuda grass	2 (7)	0.20 0.35	140 178	13.5 19.7	Foliar BBCH 25-41	0	4.1, 3.4 (3.75)	0.29, 0.37 (0.33)	4.29, 3.77 (4.12)	2018/7004965, R170028
						6	1.5, 1.4 (1.45)	0.20, 0.18 (0.19)	1.7, 1.58 (1.64)	
United States, 2017, Porterville, California, Bermuda grass	2 (7)	0.20 0.35	140 140	14.3 25	Foliar BBCH 31-38	0	1.5, 1.6 (1.55)	0.62, 0.66 (0.64)	2.12, 2.26 (2.19)	2018/7004965, R170029
						3	0.39, 0.44 (0.415)	0.046, 0.054 (0.0405)	0.436, 0.494 (0.4555)	
						7	0.33, 0.30 (0.315)	0.031, 0.031 (0.031)	0.361, 0.331 (0.346)	
						14	0.18, 0.20 (0.19)	0.016, 0.016 (0.016)	0.196, 0.216 (0.206)	
						21	0.070, 0.099 (0.0845)	<0.01, <0.01 (<0.01)	0.08, 0.109 (0.0945)	
United States, 2017, Germansville, Pennsylvania, Bluegrass	2 (7)	0.20 0.35	178 178	11.2 19.7	Foliar BBCH 39-60	0	1.3, 1.4 (1.35)	0.50, 0.64 (0.57)	1.8, 2.04 (1.92)	2018/7004965, R170030
						8	0.19, 0.24 (0.215)	0.034, 0.037 (0.0355)	0.224, 0.277 (0.2505)	
United States, 2017,	2 (7)	0.20 0.35	178 178	11.2 19.7	Foliar BBCH 41-43	0	1.1, 1.4 (1.25)	0.20, 0.27	1.3, 1.67	2018/7004965, R170031

Country, Year, Location, Crop	Application					DAT	Residue mg/kg [a]			Study Reference, Trial No.
	no. (RTI)	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage		parent	M007	Total	
Delavan, Wisconsin, Bluegrass								(0.235)	(1.485)	
						7	0.061, 0.071 (0.066)	0.014, 0.013 (0.0135)	0.075, 0.084 (0.0795)	
United States, 2017, Carlyle, Illinois, Bluegrass	2 (7)	0.20 0.36	187 140	10.7 25.7	Foliar BBCH 30-49	0	2.0, 2.0 (2.0)	0.85, 0.79 (0.82)	2.85, 2.79 (2.82)	2018/7004965, R170032
						7	0.26, 0.27 (0.265)	0.042, 0.040 (0.041)	0.302, 0.31 (0.305)	
United States, 2017, Marysville, Ohio, Bluegrass	2 (7)	0.20 0.35	140 140	14.3 25	Foliar BBCH 45	0	1.0, 1.1 (1.05)	1.0, 1.1 (1.05)	2.0, 2.2 (2.1)	2018/7004965, R170033
						7	0.16, 0.17 (0.165)	0.066, 0.078 (0.072)	0.226, 0.248 (0.237)	
United States, 2017, York, Nebraska, Fescue	2 (6)	0.20 0.35	178 168	11.2 20.8	Foliar BBCH 36-43	0	1.2, 1.0 (1.1)	2.3, 1.9 (2.1)	3.5, 2.9 (3.2)	2018/7004965, R170034
						7	0.14, 0.15 (0.145)	0.042, 0.043 (0.0425)	0.182, 0.193 (0.1875)	
United States, 2017, Richland, Indiana, Fescue	2 (7)	0.20 0.35	168 168	11.9 20.8	Foliar BBCH 40-45	0	0.69, 0.76 (0.725)	0.37, 0.36 (0.365)	1.06, 1.12 (1.09)	2018/7004965, R170035
						3	0.29, 0.34 (0.315)	0.21, 0.28 (0.245)	0.50, 0.62 (0.56)	
						7	0.18, 0.15 (0.165)	0.072, 0.058 (0.065)	0.252, 0.208 (0.23)	
						14	0.099, 0.097 (0.098)	0.030, 0.027 (0.0285)	0.129, 0.124 (0.1265)	
						20	0.087, 0.092 (0.0895)	0.018, 0.019 (0.0185)	0.105, 0.112 (0.1085)	
United States, 2017, Ephrata, Washington, Brome grass	2 (7)	0.20 0.35	140 140	14.3 25	Foliar BBCH 41-45	0	0.62, 0.56 (0.59)	0.22, 0.21 (0.215)	0.84, 0.77 (0.805)	2018/7004965, R170036
						7	0.032,	0.019,	0.041,	

Country, Year, Location, Crop	Application					DAT	Residue mg/kg [a]			Study Reference, Trial No.
	no. (RTI)	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage		parent	M007	Total	
							0.036 (0.034)	0.021 (0.205)	0.057 (0.0545)	
United States, 2017, Jerome, Idaho, Brome grass	2 (7)	0.20 0.35	168 159	11.9 22	Foliar BBCH 30-41	0	0.68, 0.69 (0.685)	0.68, 0.68 (0.68)	1.36, 1.37 (1.365)	2018/7004965, R170037
						7	0.075, 0.063 (0.069)	0.015, 0.012 (0.0135)	0.09, 0.075 (0.0825)	

Notes:

RTI = Retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Sorghum (forage)

See sorghum grain for study description.

Table 13 Residues of afidopyropen in sorghum forage after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location	Application					DAT	Residue mg/kg [a]			Study Reference, Trial No.
	no.	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage		parent	M007	Total	
United States, 2016, Plains, Georgia	2 (14)	20.0 19.6	212 211	9.4 9.3	Foliar, BBCH 53 + 58	7	0.23, 0.17 (0.2)	0.055, 0.045 (0.05)	0.285, 0.215 (0.25)	2017/7016329, R161032
United States, 2016, Proctor, Arizona	2 (14)	19.8 19.8	196 196	10.1 10.1	Foliar, BBCH 51 + 77	7	0.017, 0.018 (0.0175)	<0.01, <0.01 (<0.01)	0.027, 0.028 (0.0275)	2017/7016329, R161033
United States, 2016, Atlantic, Iowa	2 (15)	19.7 20.0	202 216	9.8 9.3	Foliar, BBCH 37 + 77	6	0.015, <0.01 (0.0125)	<0.01, <0.01 (<0.01)	0.025, <0.02 (0.0225)	2017/7016329, R161034
United States, 2016, Leonard, Montana	2 (14)	20.0 19.5	209 220	9.6 8.8	Foliar, BBCH 69 + 84	7	0.022, 0.032 (0.027)	<0.01, <0.01 (<0.01)	0.032, 0.042 (0.037)	2017/7016329, R161035
United States, 2016, Zearing, Iowa	2 (14)	20.0 20.7	193 210	10.4 9.9	Foliar, BBCH 73 + 75	6	0.038, 0.050 (0.044)	0.012, 0.017 (0.0145)	0.050, 0.067 (0.0485)	2017/7016329, R161036

Country, Year, Location	Application						Residue mg/kg [a]			
	no.	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT	parent	M007	Total	Study Reference, Trial No.
United States, 2016, Stilwell, Kansas	2 (13)	20.3 19.9	267 263	7.6 7.6	Foliar, BBCH 73 + 85	0	0.26, 0.23 (0.245)	<0.01, <0.01 (<0.01)	0.27, 0.24 (0.255)	2017/7016329, R161037
						3	0.017, 0.016 (0.0165)	<0.01, <0.01 (<0.01)	0.027, 0.026 (0.0265)	
						6	0.011, <0.01 (0.0105)	<0.01, <0.01 (<0.01)	021, <0.02 (0.0205)	
						9	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2016, Madill, Oklahoma	2 (15)	20.3 20.3	225 225	9.0 9.0	Foliar, BBCH 85	7	0.045, 0.038 (0.041)	0.011, <0.01 (0.0105)	0.055, 0.048 (0.0515)	2017/7016329, R1610348
United States, 2016, Hinton, Oklahoma	2 (14)	20.4 19.7	230 216	8.9 9.1	Foliar, BBCH 87–89	7	0.052, 0.048 (0.050)	0013, 0.013 (0.013)	0.065, 0.061 (0.063)	2017/7016329, R161039
United States, 2016, Prosser, Nebraska	2 (14)	18.9 20.2	204 210	9.3 9.6	Foliar, BBCH 85–89	8	0.032, 0.026 (0.029)	<0.01, <0.01 (<0.01)	0.042, 0.036 (0.039)	2017/7016329, R161040
United States, 2016, Wall, Texas	2 (15)	20.0 19.8	268 261	7.5 7.6	Foliar, BBCH 87–89	7	0.025, 0.029 (0.027)	<0.01, 0.011 (0.0105)	0.035, 0.039 (0.0375)	2017/7016329, R161041
United States, 2016, Claude, Texas	2 (15)	20.1 20.4	228 226	8.8 9.0	Foliar, BBCH 85–88	7	0.040, 0.053 (0.0465)	0.013, 0.014 (0.0135)	0.053, 0.067 (0.06)	2017/7016329, R161042
United States, 2016, Dill City, Oklahoma	2 (13)	20.2 20.2	215 234	9.4 8.6	Foliar, BBCH 85–87	7	0.090, 0.074 (0.082)	0.021, 0.021 (0.021)	0.111, 0.095 (0.103)	2017/7016329, R161043

Notes:

RTI = Retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Alfalfa (hay)

See alfalfa forage for study description.

Table 14: Residues of afidopyropen in alfalfa hay after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location	Application						Residue mg/kg [a]			
	no. RTI	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT cut (+dry)	parent	M007	Total	Study Reference, Trial No.
United States, 2017, Germans-ville Penn-sylvania	2 (6)	22 34	158 163	13.9 20.9	Foliar, BBCH 51-55	0 (+7)	1.392, 1.242 (1.319)	0.539, 0.443 (0.491)	1.931 1.685 (1.810)	2018/7005908, R170070
						7 (+3)	0.672, 0.695 (0.6835)	0.108, 0.095 (0.1015)	0.78 0.79 (0.785)	
						42 (2nd cut.) (+5)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						67 (3rd cut.) (+1)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Northwood, North Dakota	2 (7)	22 34	139 142	15.8 23.9	Foliar, BBCH 63	0 (+7)	1.902, 1.764 (1.833)	1.279, 1.257 (1.268)	3.181 3.021 (3.101)	2018/7005908, R170072
						7 (+8)	0.462, 0.418 (0.440)	0.132, 0.106 (0.119)	0.594 0.524 (0.559)	
						36 (2nd cut.) (+3)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						82 (3rd cut.) (+4)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Shelbyville, Indiana	2 (7)	22 34	141 145	15.9 23.4	Foliar, BBCH 63	0 (+1)	1.247, 1.456 (1.3515)	0.456, 0.470 (0.463)	1.703 1.926 (1.8145)	2018/7005908, R170073
						7 (+1)	0.391, 0.343 (0.367)	0.072, 0.054 (0.063)	0.463 0.397 (0.43)	
						48 (2nd cut.) (+2)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						106 (3rd cut.) (+1)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017,	2 (7)	22 34	141 138	15.6 24.6	Foliar, BBCH 65	0 (+4)	3.323, 2.919 (3.121)	1.771, 1.513 (1.642)	5.094 4.432 (4.763)	2018/7005908, R170074

Country, Year, Location	Application						Residue mg/kg [a]			
	no. RTI	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT cut (+dry)	parent	M007	Total	Study Reference, Trial No.
Brunswick, Nebraska						7 (+7)	0.702, 0.533 (0.6175)	0.157, 0.127 (0.142)	0.859 0.66 (0.7595)	
						29 (2nd cut.) (+12)	0.01, <0.01 (0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						55 (3rd cut.) (+4)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Aurora, South Dakota	2 (7)	22 34	141 138	15.6 24.6	Foliar, BBCH 61	0 (+5)	2.853, 2.969 (2.911)	2.379, 2.376 (2.375)	5.232 5.345 (5.286)	2018/7005908, R170075
						7 (+6)	0.801, 0.880 (0.8405)	0.268, 0.280 (0.274)	1.069 1.16 (1.145)	
						27 (2nd cut.) (+5)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						67 (3rd cut.) (+6)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Montpelier, North Dakota	2 (7)	22 34	185 185	11.9 18.4	Foliar, BBCH 61	0 (+1)	5.039, 4.787 (4.913)	0.226, 0.199 (0.2125)	5.265 4.986 (5.1255)	2018/7005908, R170076
						7 (+3)	1.007, 0.913 (0.960)	0.044, 0.053 (0.0485)	1.051 0.966 (1.0085)	
						42 (2nd cut.) (+5)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						67 (3rd cut.) (+1)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, Paso Robles, California	2 (7)	22 34	162 160	13.6 21.3	Foliar, BBCH 49	0 (+4)	2.475, 3.132 (2.8035)	1.216, 1.439 (1.328)	3.691 4.571 (4.1315)	2018/7005908, R170077
						7 (+5)	0.944, 0.822 (0.883)	0.278, 0.237 (0.258)	1.222 1.059 (1.141)	
						39 (2nd cut.) (+6)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						71 (3rd cut.)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	

Country, Year, Location	Application						Residue mg/kg [a]			
	no. RTI	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT cut (+dry)	parent	M007	Total	Study Reference, Trial No.
						(+10)				
United States, 2017, Monte Vista, Colorado	2 (8)	22 34	184 176	12.0 19.3	Foliar, BBCH 61-63	0 (+10)	0.257, 0.466 (0.3615)	0.018, 0.022 (0.020)	0.275 0.488 (0.3815)	2018/7005908, R170078
						3 (+7)	0.161, 0.203 (0.182)	<0.01, <0.01 (<0.01)	0.171 0.213 (0.192)	
						7 (+4)	0.105, 0.052 (0.0775)	<0.01, <0.01 (<0.01)	0.115 0.062 (0.0875)	
						14 (+3)	0.17, 0.154 (0.162)	<0.01, <0.01 (<0.01)	0.18 0.164 (0.172)	
						21 (+6)	0.044, 0.037 (0.0405)	<0.01, <0.01 (<0.01)	0.054 0.047 (0.0505)	
						49 (2nd cut.) (+7)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						76 (3rd cut.) (+4)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
United States, 2017, American Falls, Idaho	2 (8)	22 34	163 166	13.5 20.5	Foliar, not reported	0 (+6)	2.733, 2.68 (2.7065)	2.838, 2.674 (2.756)	5.571 5.354 (5.4625)	2018/7005908, R170079
						3 (+5)	1.561, 1.647 (1.604)	0.696, 0.724 (0.71)	2.257 2.471 (2.314)	
						7 (+6)	1.521, 1.611 (1.566)	0.404, 0.432 (0.418)	1.925 2.042 (1.984)	
						14 (+6)	0.903, 0.872 (0.8875)	0.293, 0.283 (0..288)	1.196 1.155 (1.1755)	
						21 (+6)	0.43, 0.44 (0.435)	0.191, 0.159 (0.175)	0.621 0.599 (0.61)	
						48 (2nd cut.) (+7)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	
						88 (3rd cut.) (+7)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	

Notes:

RTI = Retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Clover (hay)

See alfalfa forage for study description.

Table 15 Residues of afidopyropen in clover hay after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location	Application						Residue mg/kg [a]			
	no. RTI	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT cut (+dry)	parent	M007	Total	Study Reference, Trial No.
United States, 2017, Frenchtown New Jersey	2 (8)	22 34	168 170	13.1 20.0	Foliar, BBCH 61-65	0 (+2)	1.811, 1.373 (1.592)	0.369, 0.282 (0.3255)	2.180, 1.655 (1.9175)	2018/7005908, R170071
						7 (+1)	0.711, 0.666 (0.6885)	0.074, 0.067 (0.0705)	0.785, 0.733 (0.759)	
United States, 2017, Chula, Georgia	2 (7)	22 34	182 170	12.1 20.0	Foliar, BBCH 60	0 (+4)	2.671, 2.583 (2.627)	0.611, 0.598 (0.6045)	3.282, 3.181 (3.2315)	2018/7005908, R170083
						7 (+3)	1.153, 0.813 (0.983)	0.135, 0.106 (0.1205)	1.288, 0.919 (1.1035)	
United States, 2017, Washington Louisiana	2 (7)	22 34	163 181	13.5 18.8	Foliar, BBCH 60	0 (+3)	6.225, 5.637 (5.931)	2.717, 2.516 (2.6165)	8.942, 8.153 (8.5475)	2018/7005908, R170084
						7 (+3)	0.563, 0.701 (0.632)	0.095, 0.115 (0.105)	0.658, 0.826 (0.737	
United States, 2017, Northwood, North Dakota	2 (7)	22 34	141 141	15.6 24.1	Foliar, BBCH 61	0 (+6)	1.276, 1.211 (1.2435)	1.004, 0.959 (0.9815)	2.280, 2.170 (2.225)	2018/7005908, R170085
						7 (+6)	0.287, 0.278 (0.2825)	0.131, 0.127 (0.129)	0.418, 0.405 (0.4115)	
United States, 2017, Gardner, North Dakota	2 (7)	22 34	177 186	14.5 18.3	Foliar, BBCH 61-65	0 (+3)	4.207, 4.351 (4.279)	0.143, 0.129 (0.136)	4.350, 4.480 (4.415)	2018/7005908, R170086
						7 (+6)	1.050, 1.082 (1.066)	0.034, 0.037 (0.0355)	1.084, 1.119 (1.1015)	
United States,	2 (8)	22 34	152 145	14.5 23.4	Foliar,	0 (+3)	2.638, 2.365	1.084, 1.080	3.722, 3.445	2018/7005908, R170087

Country, Year, Location	Application						Residue mg/kg [a]			
	no. RTI	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT cut (+dry)	parent	M007	Total	Study Reference, Trial No.
2017, Lebanon, Oklahoma					BBCH 49-61		(2.501)	(1.082)	(3.5835)	
						7 (+3)	0.538, 0.573 (0.5555)	0.089, 0.095 (0.092)	0.628, 0.668 (0.6475	
United States, 2017, Montpelier, North Dakota	2 (7)	22 34	139 176	15.8 19.1	Foliar, BBCH 61-64	0 (+5)	4.321, 3.570 (3.9455)	0.462, 0.380 (0.421)	4.783, 3.950 (4.3665)	2018/7005908, R170088
						7 (+5)	1.196, 1.432 (1.314)	0.109, 0.095 (0.102)	1.305, 1.527 (1.416)	
United States, 2017, Claude, Texas	2 (7)	22 34	160 149	13.8 22.8	Foliar, BBCH 61-63	0 (+15)	2.454, 2.407 (2.4305)	1.058, 1.072 (1.065)	3.512, 3.479 (3.4955)	2018/7005908, R170089
						7 (+8)	1.362, 1.375 (1.3685)	0.170, 0.150 (0.160)	1.532, 1.535 (1.5285)	
United States, 2017, American Falls, Idaho	2 (8)	22 34	163 165	13.5 20.6	Foliar, BBCH 61-63	0 (+6)	1.589, 1.347 (1.468)	0.947, 0.839 (0.893)	2.536, 2.186 (2.261)	2018/7005908, R170090
						7 (+5)	0.460, 0.380 (0.420)	0.184, 0.167 (0.1755)	0.644, 0.547 (0.5955)	

Notes:

RTI = Retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Grasses (hay)

See grasses forage for study description.

Table 16 Residues of afidopyropen in grasses hay after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location, Crop	Application						Residue mg/kg [a]			
	no. (RTI)	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT (+dry)	parent	M007	Total	Study Reference, Trial No.
United States, 2017, Abbeville,	2 (6)	0.20 0.36	168 159	11.9 22.6	Foliar BBCH 41-43	0 (+4)	2.4, 2.4 (2.4)	0.31, 0.30 (0.305)	2.71, 2.70 (2.705)	2018/7004965, R170026

Country, Year, Location, Crop	Application						Residue mg/kg [a]			
	no. (RTI)	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT (+dry)	parent	M007	Total	Study Reference, Trial No.
Georgia, Bermuda grass							DM based: 3.27		DM based: 3.69	Moisture: 27.7 percent
						7 (+2)	0.71, 0.71 (0.71) DM based: 1.0	0.015, 0.014 (0.0145)	0.725, 0.724 (0.7245) DM based: 1.02	Moisture: 28.7 percent
United States, 2017, Lecompte, Lousianna, Bermuda grass	2 (7)	0.20 0.35	150 150	13.3 23.3	Foliar BBCH 31-49	0 (+4)	4.5, 4.3 (4.4) DM based: 5.76	0.83, 0.75 (0.79)	5.33, 5.05 (5.19) DM based: 6.79	2018/7004965, R170027 Moisture: 23.6 percent
						7 (+2)	0.22, 20 (0.21) DM based: 0.25	0.025, 0.027 (0.026)	0.245, 0.227 (0.236) DM based: 0.282	Moisture: 16.4 percent
United States, 2017, Uvalde, Texas, Bermuda grass	2 (7)	0.20 0.35	140 178	13.5 19.7	Foliar BBCH 25-41	0 (+2)	3.3, 4.0 (3.65) DM based: 4.12	0.27, 0.45 (0.36)	3.57, 4.45 (4.01) DM based: 4.53	2018/7004965, R170028 Moisture: 11.5 percent
						6 (+1)	2.0, 1.9 (1.95) DM based: 2.34	0.27, 0.25 (0.26)	2.27, 2.15 (2.21) DM based: 2.65	Moisture: 16.7 percent
United States, 2017, Porterville, California, Bermuda grass	2 (7)	0.20 0.35	140 140	14.3 25	Foliar BBCH 31-38	0 (+2)	2.3, 2.2 (2.25) DM based: 2.39	1.0, 0.82 (0.91)	3.3, 3.02 (3.16) DM based: 3.36	2018/7004965, R170029 Moisture: 5.9 percent
						3 (+2)	1.2, 1.5 (1.35)	0.14, 0.19 (0.165)	1.34, 1.69 (1.515)	
						7 (+2)	0.47, 0.54 (0.505) DM based: 0.54	0.043, 0.049 (0.046)	0.513, 0.589 (0.551) DM based: 5.88	Moisture: 6.3 percent
						14 (+2)	0.42, 0.41 (0.415)	0.036, 0.037 (0.0365)	0.456, 0.447 (0.4515)	
						21 (+2)	0.27, 0.28 (0.275)	0.026, 0.020 (0.023)	0.296, 0.30 (0.298)	
United States, 2017,	2 (7)	0.20 0.35	178 178	11.2 19.7	Foliar BBCH 39-60	0 (+3)	3.7, 3.8 (3.75)	2.0, 2.0 (2.0)	5.7, 5.8 (5.75)	2018/7004965, R170030

Country, Year, Location, Crop	Application						Residue mg/kg [a]			
	no. (RTI)	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT (+dry)	parent	M007	Total	Study Reference, Trial No.
Germansville, Pennsylvania, Bluegrass							DM based: 4.64		DM based: 7.13	Moisture: 19.3 percent
						8 (+1)	0.66, 0.63 (0.645) DM based: 0.84	0.12, 0.11 (0.115)	0.78, 0.75 (0.765) DM based: 1.0	Moisture: 23.4 percent
United States, 2017, Delavan, Wisconsin, Bluegrass	2 (7)	0.20 0.35	178 178	11.2 19.7	Foliar BBCH 41-43	0 (+8)	2.6, 2.8 (2.7) DM based: 4.46	0.76, 0.93 (0.845)	3.36, 3.73 (3.545) DM based: 5.85	2018/7004965, R170031 Moisture: 39.4 percent
						7 (+4)	0.14, 0.13 (0.135) DM based: 0.313	0.035, 0.039 (0.037)	0.175, 0.169 (0.172) DM based: 0.398	Moisture: 56.8 percent
United States, 2017, Carlyle, Illinois, Bluegrass	2 (7)	0.20 0.36	187 140	10.7 25.7	Foliar BBCH 30-49	0 (+4)	5.0, 4.7 (4.85) DM based: 8.38	3.5, 3.0 (3.25)	8.5, 7.7 (8.1) DM based: 14.0	2018/7004965, R170032 Moisture: 42.1 percent
						7 (+4)	0.43, 0.44 (0.435) DM based: 0.518	0.072, 0.088 (0.080)	0.502, 0.528 (0.515) DM based: 0.60	Moisture: 16.1 percent
United States, 2017, Marysville, Ohio, Bluegrass	2 (7)	0.20 0.35	140 140	14.3 25	Foliar BBCH 45	0 (+2)	2.9, 2.5 (2.7) DM based: 3.48	4.5, 3.9 (4.2)	7.4, 6.4 (6.9) DM based: 8.9	2018/7004965, R170033 Moisture: 22.5 percent
						7 (+5)	0.49, 0.52 (0.505) DM based: 0.639	0.23, 0.25 (0.24)	0.72, 0.77 (0.745) DM based: 0.943	Moisture: 21.0 percent
United States, 2017, York, Nebraska, Fescue	2 (6)	0.20 0.35	178 168	11.2 20.8	Foliar BBCH 36-43	0 (+4)	3.2, 3.0 (3.1) DM based: 4.82	6.3, 6.7 (6.5)	9.5, 9.7 (9.6) DM based: 14.9	2018/7004965, R170034 Moisture: 35.7 percent
						7 (+4)	0.30, 0.19 (0.245) DM based: 0.308	0.088, 0.055 (0.715)	0.388, 0.245 (0.96) DM based: 1.21	Moisture: 20.4 percent
United States, 2017, Richland,	2 (7)	0.20 0.35	168 168	11.9 20.8	Foliar BBCH 40-45	0 (+2)	2.1, 2.4 (2.25)	1.9, 2.1 (2.0)	4.0, 4.5 (4.25)	2018/7004965, R170035

Country, Year, Location, Crop	Application						Residue mg/kg [a]			
	no. (RTI)	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT (+dry)	parent	M007	Total	Study Reference, Trial No.
Indiana, Fescue							DM based: 2.77		DM based: 5.24	Moisture: 18.9 percent
						3 (+1)	0.98, 0.79 (0.885)	1.1, 1.0 (1.05)	2.08, 1.79 (1.935)	
						7 (+1)	0.54, 0.57 (0.555) DM based: 0.850	0.23, 0.23 (0.23)	0.77, 0.80 (0.785) DM based: 1.20	Moisture: 34.7 percent
						14 (+1)	0.25, 0.26 (0.255)	0.082, 0.081 (0.0815)	0.323, 0.341 (0.3365)	
						20 (+1)	0.19, 0.16 (0.175)	0.051, 0.036 (0.0435)	0.241, 0.196 (0.2185)	
United States, 2017, Ephrata, Washington, Brome grass	2 (7)	0.20 0.35	140 140	14.3 25	Foliar BBCH 41-45	0 (+10)	2.1, 2.3 (2.2) DM based: 2.59	1.0, 1.1 (1.05)	3.1, 3.4 (3.25) DM based: 3.83	2018/7004965, R170036 Moisture: 15.1 percent
						7 (+6)	0.19, 0.17 (0.18) DM based: 0.226	0.12, 0.11 (0.115)	0.31, 0.28 (0.295) DM based: 0.370	Moisture: 20.3 percent
United States, 2017, Jerome, Idaho, Brome grass	2 (7)	0.20 0.35	168 159	11.9 22	Foliar BBCH 30-41	0 (+6)	3.0, 2.7 (2.85) DM based: 3.15	3.7, 3.4 (3.55)	6.7, 6.1 (6.4) DM based: 7.06	2018/7004965, R170037 Moisture: 9.4 percent
						7 (+6)	0.27, 0.29 (0.28) DM based: 0.399	0.066, 0.058 (0.062)	0.336, 0.348 (0.342) DM based: 0.487	Moisture: 29.8 percent

Notes:

RTI = Retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Alfalfa (straw)

See alfalfa forage for study description.

Table 17 Residues of afidopyropen in alfalfa straw after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location	Application						Residue mg/kg [a]			
	no.	rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT	parent	M007	Total	Study Reference, Trial No.
United States, 2017, Northwood, North Dakota	2 (7)	22 34	140 178	15.7 19.1	Foliar, BBCH 89	0	2.08, 2.144 (2.112)	0.286, 0.281 (0.283)	2.366 2.425 (2.395)	2018/7005908, R170080
						7	1.378, 1.00 (1.189)	0.162, 0.135 (0.1435)	1.540 1.135 (1.3325)	
United States, 2017, San Joaquin, California	2 (7)	22 34	139 139	15.8 24.44	Foliar, BBCH 97	0	0.715, 0.837 (0.776)	0.086, 0.068 (0.077)	0.801 0.905 (0.853)	2018/7005908, R170081
						7	0.143, 0.157 (0.15)	0.042, 0.046 (0.044)	0.185 0.203 (0.194)	
United States, 2017, Malad City, Idaho	2 (6)	22 34	181 179	12.1 19.0	Foliar, BBCH 87–89	0	0.803, 0.651 (0.727)	0.363, 0.246 (0.3045)	1.166 0.897 (1.0315)	2018/7005908, R170082
						7	0.151, 0.151 (0.151)	0.020, 0.022 (0.021)	0.171 0.173 (0.172)	

Notes:

RTI = Retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

Sorghum (stover)

See sorghum grain for study description.

Table 18 Residues of afidopyropen in sorghum stover after foliar treatment using a DC formulation with 100 g/L afidopyropen

Country, Year, Location	Application						Residue mg/kg [a]			
	no.	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage	DAT	parent	M007	Total	Study Reference, Trial No.
United States, 2016, Plains, Georgia	2 (14)	20.1 19.6	212 211	9.5 9.3	Foliar, BBCH 91 + 95	14	0.13, 0.15 (0.14)	0.015, 0.015 (0.015)	0.145, 0.165 (0.155)	2017/7016329, R161032
United	2	19.8	196	10.1	Foliar,	14	0.012,	<0.01,	0.022,	2017/7016329,

Country, Year, Location	Application					DAT	Residue mg/kg [a]			Study Reference, Trial No.
	no.	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage		parent	M007	Total	
States, 2016, Proctor, Arizona	(14)	19.8	196	10.1	BBCH 86 + 88		0.011 (0.0105)	<0.01 (<0.01)	0.021 (0.0205)	R161033
United States, 2016, Atlantic, Iowa	2 (15)	20.0 21.0	236 214	8.5 9.8	Foliar, BBCH 85 + 87	14	0.020, 0.021 (0.0205)	<0.01, <0.01 (<0.01)	0.030, 0.031 (0.0305)	2017/7016329, R161034
United States, 2016, Leonard, Montana	2 (14)	20.7 20.3	220 215	9.4 9.4	Foliar, BBCH 85–89	14	0.17, 0.068 (0.119)	0.02, <0.01 (0.015)	0.19, 0.078 (0.134)	2017/7016329, R161035
United States, 2016, Zearing, Iowa	2 (14)	19.9 20.9	203 215	9.8 9.7	Foliar, BBCH 75–85	14	0.012, <0.01 (0.011)	<0.01, <0.01 (<0.01)	0.022, <0.02 (0.021)	2017/7016329, R161036
United States, 2016, Stilwell, Kansas	2 (14)	20.3 19.8	278 267	7.3 7.4	Foliar, BBCH 85–87	0	0.26, 0.22 (0.24)	0.085, 0.067 (0.076)	0.345, 0.287 (0.316)	2017/7016329, R161037
						9	0.018, 0.015 (0.0165)	<0.01, <0.01 (<0.01)	0.028, 0.025 (0.0265)	
						14	0.014, 0.017 (0.0155)	<0.01, <0.01 (<0.01)	0.024, 0.027 (0.0255)	
						16	0.012, 0.014 (0.013)	<0.01, <0.01 (<0.01)	0.021, 0.024 (0.023)	
						20	0.014, 0.010 (0.012)	<0.01, <0.01 (<0.01)	0.024, 0.02 (0.022)	
United States, 2016, Madill, Oklahoma	2 (14)	20.3 20.4	225 244	9.0 8.4	Foliar, BBCH 85	14	0.10, 0.062 (0.081)	0.01, <0.01 (0.01)	0.11, 0.072 (0.091)	2017/7016329, R1610348
United States, 2016, Hinton, Oklahoma	2 (13)	20.6 20.2	212 223	9.7 9.1	Foliar, BBCH 87–89	13	0.028, 0.033 (0.0305)	<0.01, <0.01 (<0.01)	0.038, 0.043 (0.0405)	2017/7016329, R161039
United States, 2016, Prosser, Nebraska	2 (15)	20.9 20.0	219 204	9.5 9.8	Foliar, BBCH 85–89	14	0.035, 0.061 (0.048)	0.012, 0.019 (0.0155)	0.047, 0.080 (0.0635)	2017/7016329, R161040
United States, 2016, Wall,	2 (14)	20.6 20.3	276 274	7.5 7.4	Foliar, BBCH 87–89	12	0.014, 0.017 (0.0155)	<0.01, <0.01 (<0.01)	0.024, 0.027 (0.0255)	2017/7016329, R161041

Country, Year, Location	Application					DAT	Residue mg/kg [a]			
	no.	Rate g ai/ha	Spray volume L/ha	Rate g ai/hL	Method, timing, growth stage		parent	M007	Total	Study Reference, Trial No.
Texas										
United States, 2016, Claude, Texas	2 (14)	20.1 20.6	224 231	9.0 8.9	Foliar, BBCH 85–88	14	0.064, 0.047 (0.0505)	<0.01, <0.01 (<0.01)	0.074, 0.057 (0.0605)	2017/7016329, R161042
United States, 2016, Dill City, Oklahoma	2 (13)	20.5 20.4	224 211	9.2 9.7	Foliar, BBCH 85–87	15	0.083, 0.087 (0.085)	0.013, 0.013 (0.013)	0.096, 0.10 (0.098)	2017/7016329, R161043

Notes:

RTI = Retreatment interval (days).

[a] Residues are expressed in their respective analytes. The totals expressed the sum of both analytes, without molar correction.

APPRAISAL

Afidopyropen is an insecticide developed for control of piercing and sucking insects. Afidopyropen disrupts the gating of TRPV (Transient Receptor Potential Vanilloid) channel complexes in chordotonal stretch receptor organs of insects. This disrupts feeding and other behaviour in target insects leading to death by starvation.

Afidopyropen was first evaluated by the 2019 JMPR when an ADI of 0–0.08 mg/kg bw was established. The Meeting also established an ARfD of 0.2 mg/kg bw for women in childbearing age and an ARfD of 0.3 mg/kg for the general population. In addition, it was concluded that the metabolites M440I007 and CPCA are likely to be of similar toxicity to its parent. The 2021 JMPR reconsidered the wording of the residue definition. The residue definitions are:

Definition of the residue for compliance with the MRL for plant and animal commodities: *afidopyropen*

Definition of the residue for dietary risk assessment for plant commodities: *sum of afidopyropen + dimer of [(3R,6R,6aR,12S,12bR)-3-[(cyclopropanecarbonyl)oxy]-6,12-dihydroxy-4,6a,12b-trimethyl-11-oxo-9-(pyridin-3-yl)-1,3,4,4a,5,6,6a,12,12a,12b-decahydro-2H,11H-naphtho[2,1-b]pyrano[3,4-e]pyran-4-yl]methyl rac-cyclopropanecarboxylate (M007).*

Definition of the residue for dietary risk assessment for animal commodities, except liver: *afidopyropen + M001 + CPCA and its carnitine conjugate, expressed as afidopyropen.*

Definition of the residue fir dietary risk assessment for liver: *afidopyropen + M001 + M017 + CPCA and its carnitine conjugate, expressed as afidopyropen.*

The residue is not fat-soluble.

At the Fifty-first Session of the CCPR (2019), afidopyropen was scheduled for toxicology and residue evaluation by the 2022 JMPR.

The Meeting received information from the manufacturer on aerobic soil metabolism, use patterns and residues resulting from supervised trials on strawberries, sorghum and alfalfa/clover.

Environmental fate in soil

The Meeting received information on environmental fate in soil.

Aerobic soil metabolism

The aerobic metabolism and degradation in soil was tested on four soil types from the United States, including silt loam, loamy sand and sandy loam soils. Pyranone-6-^{14}C-afidopyropen was applied at approximately 0.2 mg/kg and soil samples were incubated for up to 120 days at 20 °C with 50 percent maximum water holding capacity. Major degradation products exceeding 10 percent of the applied radioactivity were M057 (max. 25.7 percent AR after 29 days) and M003 (max. 11.8 percent after 5 days). Modelled DT_{50} and DT_{90} values for parent afidopyropen were 9.5–14 days and 82–268 days, respectively, following Gustafson-Holden model kinetics (FOMC). The major metabolites followed single 1st order kinetics with estimated DT_{50}/DT_{90} values of 17.5–23.8/58–79 days for M003 and 7.7-39/26–132 days for M057.

The current Meeting noted that both the metabolic pattern following aerobic soil degradation observed in the newly provided study and the estimated half-life times are similar to the conclusion drawn by the 2019 Meeting. The Meeting confirms its previous conclusion, that afidopyropen and its soil metabolites are not persistent in soil.

Methods of analysis

The current Meeting did not receive additional analytical methods for afidopyropen. All supervised field trials were analysed with analytical methods already evaluated and described by the 2019 Meeting.

Stability of pesticide residues in stored analytical samples

The current Meeting did not receive additional information on the storage stability of afidopyropen and its metabolite M007.

The 2019 Meeting evaluated the storage stability of these analytes and concluded that they are stable for at least 24 months in all plant commodity categories. Field trial samples were analysed within this interval.

Results of supervised residue trials on crops

The Meeting received information on use patterns and supervised residue trials for strawberries, sorghum, alfalfa and clover from the United States.

Strawberries

Afidopyropen is registered in the United States for the use on strawberries with a maximum GAP involving two foliar spraying of 0.05 kg ai/ha each (7 day retreatment interval (RTI)) and a PHI of 0 days. The maximum rate is 0.1 kg ai/ha per crop and a maximum of 0.3 kg ai/ha and year.

Supervised field trials conducted in Canada and the United States on strawberries were provided approximating the cGAP both with two additional initial treatments at ~ 0.01 kg ai/ha at 7 day RTI (treatment regime: 0.01 kg ai/ha + 0.01 kg ai/ha + 0.05 kg ai/ha + 0.05 kg ai/ha, total crop rate:

0.12 kg ai/ha). The Meeting concluded that the two first treatments in addition to the cGAP do not affect residue concentrations at harvest.

Residues of afidopyropen in strawberries were (n=5): 0.0326, 0.0356, 0.0439, 0.0475, 0.06 mg/kg.

Residues of afidopyropen plus M007 in strawberries were (n=5): 0.0426, 0.0456, 0.0539, 0.0575, 0.07 mg/kg. Highest residues in a single sample were 0.0778 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg for afidopyropen and STMR and HR values of 0.0539 mg/kg and 0.0778 mg/kg, respectively, for afidopyropen plus M007 in strawberries.

Sorghum

Afidopyropen is registered in the United States for the use on sorghum with a maximum GAP involving two foliar spraying of 0.02 kg ai/ha each (14 day RTI) and a PHI of 14 days for grain. The maximum rate is 0.044 kg ai/ha per year.

Supervised field trials conducted in the United States on sorghum were provided approximating the cGAP.

Residues of afidopyropen in sorghum grain were (n=12): < 0.01(3), 0.01, 0.016, 0.019, 0.034, 0.041, 0.042, 0.067, 0.071, 0.104 mg/kg.

Residues of afidopyropen plus M007 in sorghum grain were (n=12): < 0.02(3), 0.02, 0.026, 0.029, 0.044, 0.051, 0.052, 0.077, 0.081, 0.114 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg for afidopyropen and an STMR value of 0.0365 mg/kg for afidopyropen plus M007 in sorghum grain.

Alfalfa forage

Afidopyropen is registered in the United States for the use on non-grass animal feed crops with a maximum GAP involving two foliar spraying of 0.022 kg ai/ha followed by 0.036 kg ai/ha (7 day RTI) and a PHI of 0 days for forage. The maximum rate is 0.058 kg ai/ha per year.

Supervised field trials conducted in the United States on alfalfa were provided approximating the cGAP.

Residues of afidopyropen plus M007 in alfalfa forage were (n=9): 0.783, 0.999, 1.08, 1.13, 1.19, 1.24, 2.07, 2.31, 2.45 mg/kg (as received).

The Meeting estimated a median residue level of 1.19 mg/kg and highest residue level of 2.45 mg/kg for afidopyropen plus M007 in alfalfa forage (as received).

Clover forage

Afidopyropen is registered in the United States for the use on non-grass animal feed crops with a maximum GAP involving two foliar spraying of 0.022 kg ai/ha followed by 0.036 kg ai/ha (7 day RTI) and a PHI of 0 days for forage. The maximum rate is 0.058 kg ai/ha per year.

Supervised field trials conducted in the United States on clover were provided approximating the cGAP.

Residues of afidopyropen plus M007 in clover forage were (n=9): 0.601, 0.662, 0.926, 1.01, 1.39, 1.68, 2.09, 2.22, 2.59 mg/kg (as received).

The Meeting estimated a median residue level of 1.39 mg/kg and highest residue level of 2.59 mg/kg for afidopyropen plus M007 in clover forage (as received).

Grass forage

Afidopyropen is registered in the United States for the use on grass animal feed crops with a maximum GAP involving two foliar spraying of 0.022 kg ai/ha followed by 0.036 kg ai/ha (7 day RTI) and a PHI of 0 days for forage. The maximum rate is 0.058 kg ai/ha per year.

Supervised field trials conducted in the United States on grass were provided approximating the cGAP.

Residues of afidopyropen plus M007 in grass forage were (n=12): 0.805, 1.09, 1.36, 1.36, 1.48, 1.92, 2.04, 2.1, 2.19, 2.82, 3.2 and 4.12 mg/kg (as received).

The Meeting estimated a median residue level of 1.98 mg/kg and highest residue level of 4.12 mg/kg for afidopyropen plus M007 in grass forage (as received).

Sorghum forage

Afidopyropen is registered in the United States for the use on sorghum with a maximum GAP involving two foliar spraying of 0.02 kg ai/ha each (14 day RTI) and a PHI of 7 days for forage. The maximum rate is 0.044 kg ai/ha per year.

Supervised field trials conducted in the United States on sorghum were provided approximating the cGAP.

Residues of afidopyropen plus M007 in sorghum forage were (n=12): 0.0225, 0.0275, 0.037, 0.0375, 0.039, 0.0485, 0.0515, 0.06, 0.063, 0.103, 0.25 and 0.255 mg/kg (as received).

The Meeting estimated a median residue level of 0.05 mg/kg and highest residue level of 0.255 mg/kg for afidopyropen plus M007 in sorghum forage (as received).

Alfalfa fodder

Afidopyropen is registered in the United States for the use on non-grass animal feed crops with a maximum GAP involving two foliar spraying of 0.022 kg ai/ha followed by 0.036 kg ai/ha (7 day RTI) and a PHI of 0 days for cutting. The maximum rate is 0.058 kg ai/ha per year.

Supervised field trials conducted in the United States on alfalfa were provided approximating the cGAP. Alfalfa was cut according to GAP treatment and left in the field to dry until commercial dryness.

Residues of afidopyropen in alfalfa hay were (n=9): 0.362, 1.32, 1.35, 1.83, 2.71, 2.8, 2.91, 3.12 and 4.19 mg/kg (fresh).

Residues of afidopyropen plus M007 in alfalfa hay were (n=9): 0.382, 1.81, 1.81, 3.10, 4.13, 4.76, 5.13, 5.29 and 5.46 mg/kg (as received).

The Meeting estimated a maximum residue level of 8 mg/kg (dry matter, based on 89 percent dry-weight) for afidopyropen and a median and highest residue for afidopyropen plus M007 in alfalfa fodder (as received) of 4.13 mg/kg and 5.46 mg/kg, respectively.

Clover fodder

Afidopyropen is registered in the United States for the use on non-grass animal feed crops with a maximum GAP involving two foliar spraying of 0.022 kg ai/ha followed by 0.036 kg ai/ha (7 day RTI) and a PHI of 0 days for cutting. The maximum rate is 0.058 kg ai/ha per year.

Supervised field trials conducted in the United States on clover were provided approximating the cGAP. Clover was cut according to GAP treatment and left in the field to dry until commercial dryness.

Residues of afidopyropen in clover hay were (n=9): 1.24, 1.47, 1.59, 2.43, 2.5, 2.64, 3.95, 4.28 and 5.93 mg/kg (fresh).

Residues of afidopyropen plus M007 in clover hay were (n=9): 1.92. 2.23, 2.26, 3.23, 3.50, 3.58, 4.37, 4.42 and 8.55 mg/kg (as received).

The Meeting estimated a maximum residue level of 10 mg/kg (dry matter, based on 89 percent dry-weight) for afidopyropen and a median and highest residue for afidopyropen plus M007 in clover fodder (as received) of 3.5 mg/kg and 8.55 mg/kg, respectively.

Grass hay

Afidopyropen is registered in the United States for the use on grass animal feed crops with a maximum GAP involving two foliar spraying of 0.022 kg ai/ha followed by 0.036 kg ai/ha (7 day RTI) and a PHI of 0 days for cutting. The maximum rate is 0.058 kg ai/ha per year.

Supervised field trials conducted in the United States on grass were provided approximating the cGAP. Grass was cut according to GAP treatment and left in the field to dry until commercial dryness.

Residues of afidopyropen in grass hay were (n=12): 2.39, 2.59, 2.77, 3.15, 3.27, 3.48, 4.12, 4.46, 4.64, 4.82, 5.76 and 8.38 mg/kg (DM based).

Residues of afidopyropen plus M007 in grass hay were (n=12): 3.36, 3.69, 3.83, 4.53, 5.24, 5.85, 6.79, 7.06, 7.13, 8.9, 14.0 and 14.9 mg/kg (DM based).

The Meeting estimated a maximum residue level of 15 mg/kg (dry-matter, trial specific dry-weight basis) for afidopyropen and a median and highest residue for afidopyropen plus M007 in grass hay (dry-matter) of 6.32 mg/kg and 14.9 mg/kg, respectively.

Sorghum stover

Afidopyropen is registered in the United States for the use on sorghum with a maximum GAP involving two foliar spraying of 0.02 kg ai/ha each (14 day RTI) and a PHI of 14 days for stover. The maximum rate is 0.044 kg ai/ha per year.

Supervised field trials conducted in the United States on sorghum were provided approximating the cGAP.

Residues of afidopyropen in sorghum stover were (n=12): 0.0105, 0.011, 0.0155, 0.0155, 0.0205, 0.0305, 0.048, 0.0505, 0.081, 0.085, 0.119, 0.14 mg/kg (fresh).

Residues of afidopyropen plus M007 in sorghum stover were (n=12): 0.0205, 0.021, 0.0255, 0.0255, 0.0305, 0.0405, 0.0605, 0.0635, 0.091, 0.098, 0.134 and 0.155 mg/kg (as received).

The Meeting estimated a maximum residue level of 0.3 mg/kg (dry matter, based on 88 percent dry-weight) for afidopyropen and a median and highest residue for afidopyropen plus M007 in sorghum stover (as received) of 0.0505 mg/kg and 0.155 mg/kg, respectively.

Residues in animal commodities

Farm animal feeding studies

The 2019 Meeting evaluated farm animal feeding studies with afidopyropen on lactating cows and laying hens.

In the following tables, residues in bovine tissues and milk and in poultry tissues and eggs according to the residue definitions for MRL setting (afidopyropen) and for exposure estimation

(*afidopyropen + M001 + CPCA and its carnitine conjugate, expressed as afidopyropen* for animal commodities except liver and *afidopyropen + M001 + M017 + CPCA and its carnitine conjugate, expressed as afidopyropen* for liver) are summarised:

Table 19 Overview of mean and highest residue levels observed in the dietary feeding study with lactating cows (based on 2019 JMPR Report)

	1.54 ppm		4.61 ppm		15.3 ppm	
	parent	Total [a]	parent	Total [a]	parent	Total [a]
Liver	0.017 (0.019)	0.15 (0.15)	0.046 (0.056)	0.18 (0.19)	0.19 (0.20)	0.36 (0.37)
Kidney	< 0.01 (< 0.01)	< 0.13 (< 0.13)	< 0.01 (< 0.01)	< 0.13 (< 0.13)	< 0.01 (< 0.01)	< 0.13 (< 0.13)
Muscle	< 0.01 (< 0.01)	< 0.13 (< 0.13)	< 0.01 (< 0.01)	0.15 (0.17)	< 0.01 (< 0.01)	0.29 (0.29)
Fat	< 0.01 (< 0.01)	< 0.13 (< 0.13)	< 0.01 (< 0.01)	< 0.13 (< 0.13)	< 0.01 (< 0.01)	< 0.13 (< 0.13)
Milk	< 0.001 (< 0.001	< 0.013 (< 0.013)	< 0.001 (< 0.001	0.016 (0.020)	< 0.001 (< 0.001	0.035 (0.044)

Notes:

[a] Total residues include parent+M001+CPCA-carnitine, corrected for their respective molecular weights in g/mol (parent = 593.67, M001 = 457.52, CPCA-carnitine =265.74). In liver residue levels of parent were corrected for M017 using a correction factor of 1.2.

Table 20 Overview of mean (and highest) residue levels observed in the dietary feeding study with laying hens (based on 2019 JMPR Report)

	0.20 ppm		0.62 ppm		2.0 ppm	
	parent	Total [a]	parent	Total [a]	parent	Total [a]
Liver	0.010 (0.011)	0.14 (0.15)	0.025 (0.027)	0.16 (0.17)	0.085 (0.095)	0.24 (0.28)
Muscle	< 0.01 (< 0.01)	< 0.13 (< 0.13)	< 0.01 (< 0.01)	< 0.13 (< 0.13)	0.011 (0.012)	0.14 (0.13)
Fat	< 0.01 (< 0.01)	< 0.13 (< 0.13)	0.011 (0.012)	0.14 (0.14)	0.036 (0.042)	0.16 (0.17)
Eggs	< 0.01 (< 0.01)	< 0.13 (< 0.13)	0.011 (0.018) [b]	0.14 (0.14)	0.026 (0.036)	0.15 (0.16)

Notes:

[a] Total residues include parent+ M001+CPCA-carnitine (+ M017 in liver) corrected for molecular weight differences in g/mol (parent = 593.67, M001 = 457.52, M017 = 609.7; CPCA-carnitine =265.74).

[b] Results from day 28 and 32 only.

Farm animal dietary burden

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the JMPR in 2019 and the current recommendations. The dietary burdens, estimated using the most recent version of the OECD livestock dietary burden calculator diets are presented in Annex 6 and summarised below. As Australia does not allow the importation of fodders due to biosecurity concerns, forage and fodder commodities were excluded from the dietary burden calculation for Australia.

Table 21 Estimated maximum and mean dietary burdens of farm animals (sum of afidopyropen and M007)

	Animal dietary burden: sum of afidopyropen and M007, ppm of dry matter diet							
	United States-Canada		European Union		Australia /[a]		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	3.8	1.7	17.5	8.7	0.09	0.09	6.67	3.1
Dairy cattle	9.4	4.53	18.2 ❶	9.0 ❷	0.082	0.082	12.2	5.8
Broilers	0.04	0.04	0.036	0.036	0.035	0.035	0.035	0.035
Layers	0.037	0.037	1.2 ❸	0.54 ❹	0.035	0.035	0.03	0.03

Notes:

[a/] Excluding forage/fodder due to import restrictions.

❶ Highest maximum beef or dairy cattle dietary burden suitable for HR estimates for mammalian tissues and milk.

❷ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues and milk.

❸ Highest maximum poultry dietary burden suitable for HR estimates for poultry tissues and eggs.

❹ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Animal commodity maximum residue levels

Cattle

The calculations used to estimate maximum residue levels, STMR and HR values for cattle matrices are shown below. For cattle, the maximum dietary burden of 18.2 ppm represents 119 percent of the highest dose administered in the provided feeding study, which is at the upper end of acceptable under dosing for the estimation of residues in animal commodities.

Table 22 Anticipated residues of afidopyropen in cattle commodities

	Feed Level (ppm) for milk residues	Total residues (mg eq/kg) in milk	Feed Level (ppm) for tissue residues	Total residues (mg eq/kg)			
				Muscle	Liver	Kidney	Fat
HR Determination (beef or dairy cattle)–Parent + M001+ CPCA-carnitine + M017 (liver only)							
Feeding Study	15.3	0.035	15.3	0.29	0.38	< 0.13	< 0.13
Dietary burden and estimate of highest residue	18.2	0.042	18.2	0.34	0.45	< 0.15	< 0.15
STMR Determination (beef or dairy cattle)–Parent + M001 + CPCA-carnitine + M017 (liver only)							
Feeding Study	4.61 15.3	0.016 0.035	4.61 15.3	0.15 0.29	0.18 0.36	< 0.13 < 0.13	< 0.13 < 0.13
Dietary burden and estimate of median residue	9.0	0.024	9.0	0.21	0.25	< 0.13	< 0.13
MRL Determination (beef or dairy cattle)–Parent							
Feeding Study	15.3	< 0.001	15.3	< 0.01	0.20	< 0.01	< 0.01
Dietary burden and estimate of highest residue	18.2	< 0.001	18.2	< 0.01	0.24	< 0.01	< 0.01

The Meeting confirms its previous recommendation on maximum residue levels of 0.001(*) mg/kg in milk, 0.3 mg/kg in edible offal (based on liver), 0.01(*) mg/kg in meat (mammalian except marine mammals) and 0.01(*) mg/kg in mammalian fats.

For estimating dietary exposure, calculated HR values are: 0.45 mg/kg for edible offal (based on liver), 0.34 mg/kg for muscle, and 0.15 mg/kg for kidney and fat and 0.042 mg/kg in milk. Calculated STMRs are: 0.25 mg/kg edible offal (based on liver), 0.21 mg/kg for muscle, 0.13 mg/kg for kidney and fat, and 0.024 mg/kg for milk.

Poultry

The calculations used to estimate maximum residue levels, STMR and HR values for poultry matrices are shown below.

Table 23 Anticipated residues of afidopyropen in poultry commodities

	Feed Level (ppm) for egg residues	Total residues (mg eq/kg) in egg	Feed Level (ppm) for tissue residues	Total residues (mg eq/kg)		
				Muscle	Liver	Fat
HR Determination (poultry broiler or layer)–Parent + M001+ CPCA-carnitine + M017 (liver only)						
Feeding Study	0.62	0.14	0.62	< 0.13	0.17	0.14
	2.0	0.16	2.0	0.14	0.28	0.17
Dietary burden and estimate of highest residue	1.2	0.149	1:2	0.134	0.22	0.16
STMR Determination (poultry broiler or layer) – Parent + M001+ CPCA-carnitine + M017 (liver only)						
Feeding Study	0.20	< 0.13	0.20	< 0.13	0.14	< 0.13
	0.62	0.14	0.62	< 0.13	0.16	0.14
Dietary burden and estimate of median residue	0.54	0.138	0.54	< 0.13	0.156	0.138
MRL Estimation (poultry broiler of layer) – Parent only						
Feeding Study	0.62	0.018	0.62	< 0.01	0.011	< 0.01
	2.0	0.036	2.0	< 0.01	0.027	0.012
Dietary burden and estimate of highest residue	1.2	0.027	1.2	< 0.01	0.019	0.011

The Meeting confirms its previous recommendation on a maximum residue level of 0.01(*) mg/kg in poultry meat (muscle).

The Meeting estimated maximum residue levels of 0.015 mg/kg in poultry fat, 0.02 mg/kg in poultry edible offal and 0.03 mg/kg in eggs to replace its previous recommendation.

For estimating dietary exposure calculated HR values are: 0.22 mg/kg for poultry edible offal (based on liver), 0.134 mg/kg for muscle, 0.16 mg/kg for fat and 0.149 mg/kg for eggs. Calculated STMRs are: 0.156 mg/kg for liver and poultry edible offal (based on liver), 0.13 mg/kg for muscle, 0.138 mg/kg for fat and 0.138 mg/kg for eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with the MRL for plant and animal commodities: *afidopyropen*

Definition of the residue for dietary risk assessment for plant commodities: *sum of afidopyropen + dimer of [(3R,6R,6aR,12S,12bR)-3-[(cyclopropanecarbonyl)oxy]-6,12-dihydroxy-4,6a,12b-trimethyl-11-oxo-9-(pyridin-3-yl)-1,3,4,4a,5,6,6a,12,12a,12b-decahydro-2H,11H-naphtho[2,1-b]pyrano[3,4-e]pyran-4-yl]methyl rac-cyclopropanecarboxylate (M007).*

Definition of the residue for dietary risk assessment for animal commodities, except liver: *afidopyropen + M001 + CPCA and its carnitine conjugate, expressed as afidopyropen.*

Definition of the residue fir dietary risk assessment for liver: *afidopyropen + M001 + M017 + CPCA and its carnitine conjugate, expressed as afidopyropen.*

The residue is not fat-soluble.

Table 24 Recommendations for residues of afidopyropen from the 2022 JMPR

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New	Previous		
AL 1020	Alfalfa, fodder	8 (dw)	-	Median: 4.13 (ar)	Highest: 5.46 (ar)
AL 1031	Clover, fodder	10 (dw)	-	Median: 3.5 (ar)	Highest: 8.55 (ar)
AS 0162	Grass, hay	15 (dw)	-	Median: 6.32 (dw)	Highest: 14.9 (dw)
MO 0096	Edible offal (mammalian)	0.3	0.3	liver: 0.25 kidney: 0.13	liver: 0.45 kidney:0.15
PE 0112	Eggs	0.03	0.01(*)	0.138	0.149
MF 0100	Mammalian fats (except milk fats)	0.01(*)	0.01(*)	0.13	0.15
MM 0095	Meat (from mammals other than marine mammals)	0.01(*)	0.01(*)	muscle: 0.21 fat: 0.13	muscle: 0.34 fat: 0.15
ML 0106	Milks	0.001(*)	0.001(*)	0.024	
PO 0111	Poultry, edible offal of	0.02	0.01(*)	0.156 (liver)	0.22 (liver)
PF 0111	Poultry, fats	0.015	0.01(*)	0.138	0.16
PM 0110	Poultry, meat	0.01(*)	0.01(*)	0.13	0.134
GC 0651	Sorghum	0.2	-	0.0365	
AS 0651	Sorghum, stover	0.3 (DM)	-	Median: 0.0505 (ar)	Highest: 0.155 (ar)
FB 0275	Strawberries	0.15	-	0.0539	0.0778
AL 1021	Alfalfa, forage			Median: 1.19 (ar)	Highest: 2.45 (ar)
AL 1023	Clover, forage			Median: 1.39 (ar)	Highest: 2.59 (ar)
	Grass, forage			Median: 1.98 (ar)	Highest: 4.12 (ar)
AF 1053	Sorghum, forage			Median: 0.05 (ar)	Highest: 0.255 (ar)

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for afidopyropen is 0–0.08 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for afidopyropen were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 0–4 percentof the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of afidopyropen from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for afidopyropen is 0.2 mg/kg bw for women of child bearing age and 0.3 mg/kg bw for adults and children. The International Estimate of Short Term Intakes (IESTIs) for afidopyropen were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs were 0–1 percent of the ARfD for women of childbearing age and 0–2 percent of the ARfD for children and 0–1 percent of the ARfD for adults. The Meeting concluded that acute dietary exposure to residues of afidopyropen from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

References submitted and used.

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
2018/7007218	Nejad, H.	2020	Aerobic soil Metabolism of the ^{14}C- BAS 440 I (Afidopyropen), BASF Agricultural Solutions, Research Triangle Park NC, United States of America, 2018/7007218 GLP: yes Unpublished
2018/7007938	Lennon, G.	2018	Afidopyropen: Magnitude of the residue on strawberry (greenhouse (GH)), IR-4 Project Headquarters, Princeton NJ, United States of America, 2018/7007938 GLP: yes Unpublished
2017/7016329	Brungardt, J.	2017	Magnitude of the residue of Afidopyropen (BAS 440 I) after applications of BAS 440 01 I to grain sorghum, SynTech Research Laboratory Services LLC, Stilwell KS, United States of America, 2017/7016329 GLP: yes Unpublished
2018/7005908	Csinos, A.	2018	Magnitude of the residue of BAS 440 I in/on non-grass animal feed raw agricultural commodities, Precision Study Management LLC, Amarillo TX, United States of America, 2018/7005908 GLPL: yes

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			Unpublished
2018/7004965	Wyatt, D.	2018	Magnitude of the residues of BAS 440 I and its metabolite in or on pasture and rangeland grasses raw agricultural commodities following two applications of BAS 440 01 I, The Carringers Inc., Apex NC, United States of America, 2018/7004965, GLP: yes Unpublished

AZOXYSTROBIN (229)

First draft prepared by Dr A Leahigh, Environmental Protection Agency, Washington, United States of America

EXPLANATION

Azoxystrobin was first evaluated for toxicology and residues by JMPR in 2008. It was evaluated for residues by the JMPR in 2011, 2012, 2013, 2017, and 2019. An ADI of 0–0.2 mg/kg bw was established and an ARfD was unnecessary.

The residue definition for compliance with MRL and for dietary intake for plant and animal commodities is parent azoxystrobin. The residue is fat soluble.

Azoxystrobin was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional MRLs in 2021 and rescheduled to the 2022 JMPR. The current Meeting received additional analytical methods, storage stability data, GAP information, and residue trial data from uses on mango, papaya, sugar beet, and sugar beet processed commodities.

METHODS OF RESIDUE ANALYSIS

Residue analytical method RAM 305/03 was used for the analysis of azoxystrobin and R230310 residues in the supervised residue trials on mango, papaya, sugar beet, and processing studies on sugar beet. The method RAM 305, was previously validated as version RAM 305/01 in a wide range of crops and crop types by the 2008 JMPR. These data were considered sufficient. The version RAM 305/03 used in the residue studies in this submission is procedurally the same as the version RAM 305/01 and validation data evaluated for RAM 305/01 are applicable to RAM 305/03.

Briefly, residues of azoxystrobin and R230310 were extracted by homogenizing samples with a mixture of acetonitrile and water (90/10, v/v). Final determination was achieved by high performance liquid chromatography using triple quadrupole mass spectrometric detection (LC-MS/MS), monitoring for the primary transition (m/z 404 → 372) and the confirmatory transitions (m/z 404 → 344 and m/z 404 → 329).

RAM 305/03 was validated for the determination of residues of azoxystrobin in mango pulp and peel, papaya pulp and peel, sugar beet, and processed sugar beet commodities (juice, sugar, molasses, pulp, and press water). Method validation recoveries were all within the acceptable range and with acceptable relative standard deviations.

The limit of quantification for azoxystrobin and R230310 residues was established at 0.01 mg/kg for mango and papaya peel and pulp, and sugar beet roots and its processed products.

The Meeting concluded that Residue analytical method RAM 305/03 has been successfully validated for the analysis of azoxystrobin and R230310 residues in mango peel and pulp, papaya peel and pulp, and sugar beet and its processed commodities at an LOQ of 0.01 mg/kg.

Table 1 Method validation data relevant to mango, papaya, sugar beet, and processed commodities of sugar beet.

Matrix	Fortification level (mg/kg)	Individual recoveries (percent)	Range of recoveries (percent)	Mean recovery (percent)	RSD (percent)	Reference
Mango pulp	0.01	104, 109, 101, 102, 109	101-109	109	6	VR-016/20
	1.0	109, 109, 114, 113, 121	109-121			
Mango peel	0.01	101, 104, 105, 107, 114	101-114	102	7	

Matrix	Fortification level (mg/kg)	Individual recoveries (percent)	Range of recoveries (percent)	Mean recovery (percent)	RSD (percent)	Reference
	1.0	107, 112, 97, 108, 101	97-112			
	20	93, 91, 99, 91, 98	91-99			
Papaya pulp	0.01	105, 101, 107, 105, 104	101-107	102	4	VR-015-20
	1.0	100, 102, 104, 102, 92	92-104			
Papaya peel	0.01	103, 100, 104, 104, 104	100-104	106	4	
	1.0	103, 105, 108, 106, 113	103-113			
	20	115, 109, 105, 106, 105	105-115			
Sugar beet root (RAC)	0.01	92, 92	92	94	1.9	TK0044248
	0.10	95, 95	95			
Refined sugar	0.01	99, 95	95-99	96	2.3	
	0.10	96, 94	94-96			
Molasses	0.01	110, 112	110-112	105	8.6	
	0.10	92, 104	92-104			
Dried pulp	0.01	98, 98	98	104	9.6	
	0.10	102, 119	102-119			

USE PATTERN

The registered use patterns for azoxystrobin on mango, papaya, and sugar beet are summarised in Table 2. For mango and papaya, both pre-harvest foliar applications and post-harvest dip or spray applications may be made.

Table 2 Registered uses of azoxystrobin using dip and/or foliar application

Crop	Country	Formulation Content[1]	Formulation Type	Application Method	Application Rate	Conc. (g ai/hL)	No. of Apps	Timing/ PHI	Remarks
Mango	Brazil	239 g ai/L	SC	Dip or spray	NA	60-120	1	Post-harvest	Contact time of 2 minutes for dip application
Mango	Brazil	200 g ai/L	SC	Foliar spray	60-120 g ai/ha	6-20[2]	4	After pre-flowering/ 7 days	RTI = 14 days; spray volume = 600-1000 L/ha
Papaya	Brazil	239 g ai/L	SC	Dip or spray	NA	60-120	1	Post-harvest	Contact time of 2 minutes for dip application

Crop	Country	Formulation Content[1]	Formulation Type	Application Method	Application Rate	Conc. (g ai/hL)	No. of Apps	Timing/ PHI	Remarks
Papaya	Brazil	200 g ai/L	SC	Foliar spray	60-100 g ai/ha	6-17[2]	4	Fruit development / 3 days	RTI = 14 days; spray volume = 600-1000 L/ha
Sugar beet	United States	238 g ai/L	SC	Spray	4.7 g ai/t roots[3]	NA	1	Post-harvest	Roots must be tumbling

Notes:

NA: Not applicable.

RTI: Re-treatment interval.

[1] Content of azoxystrobin only.

[2] Calculated based on the GAP spray volume range of 600-1000 L/ha.

[3] Metric conversion from 0.0093 lb ai/2000 lb roots.

[4] Metric conversion from 0.5 gallons water/tonne.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The field trial reports included method validation data, as recoveries from spiked samples at levels reflecting those observed in the field trial samples; dates from critical events during the study, including application, harvest, storage, and analysis; as well as detailed information on the field site and treatment parameters. Analytical reports were sufficiently detailed and included example chromatograms and example calculations.

Mango

Four supervised trials with azoxystrobin on mango were conducted in 2020. Trials were conducted in Brazil using a suspension concentrate formulation nominally containing 200 g azoxystrobin/L and 125 g difenoconazole/L which was applied four times as a broadcast foliar spray at 120 g ai/ha with a 14 day treatment interval.

For some trials, foliar applications were followed by one post-harvest immersion or spray application of a suspension concentrate formulation nominally containing 239 g azoxystrobin/L and 239 g fludioxonil/L, which was applied 7 days after the last foliar application. Fruits were washed before being immersed for approximately 2 minutes in the treatment solution in a stainless steel tank, or sprayed with the formulation at a nominal concentration of 120 g ai/hL. Following the post-harvest treatment, fruits were left to dry before being sprayed with carnauba storage wax (diluted with water 1/4, v/v) before being left to dry and placed in storage.

Samples of treated and untreated whole fruits (at least 12 per sample) were collected at 7 days after the last foliar application, and at 0, 21, and 42 days after the post-harvest treatment. The whole samples were separated into peel and flesh fractions, and the stones were also removed. Peel and flesh samples were frozen (nominally -20 °C) and maintained in frozen storage for periods of up to 2.7 months prior to analysis, or placed in storage under conditions that simulate commercial practice: 1 day at 18–20 °C, 5 weeks at 11 °C, and 1 week at 18–20 °C.

Residues of azoxystrobin in mango were determined using the analytical method RAM 305/03. This method is validated for use on mango flesh and peel with an LOQ of 0.01 mg/kg.

The results of the trials are summarised Table 3, where residues relevant to the setting of an MRL for mango are underlined. Residues in control samples were <LOQ.

Table 3 Residues of azoxystrobin on mango in Brazil.[1]

Trial Information	Crop (Variety)	Country (Region)	Method	Application Rate (g ai/hL)	Water volume (L/ha)	BBCH Growth Stage at Application	DALA	Crop Part[2]	Azoxystrobin Residue mg/kg (mean)
GAP 1	Mango	Brazil	Foliar spray	4 x 120 g ai/ha 14-day RTI	600-1000 L/ha	From pre-flowering	7		
GAP 2	Mango	Brazil	Post-harvest Dip or Spray	1 x 120 g ai/hL 14-day RTI	Not specified	Not specified	0		
LBS19053-01 2020	Mango (Palmer)	Brazil (Pernambuco)	Foliar spray	120 120 120 120	1000 1000 1000 1000	65 72 80 82	7	Whole fruit Peel Flesh	0.14 0.53 <0.01
			Foliar followed by post-harvest dip	120 g ai/hL	100	79-82	0	Whole fruit Peel Flesh	1.85, 2.01 (1.93) 6.84, 7.50 (7.17) 0.01, 0.03 (0.02)
							21	Whole fruit Peel Flesh	1.62, 1.78 (1.70) 5.82, 7.13 (6.48) 0.01, 0.02 (0.02)
							42	Whole fruit Peel Flesh	1.64, 3.69 (<u>2.67</u>) 6.43, 7.29 (6.86) 0.01, 0.01 (0.01)
			Foliar followed by post-harvest spray	120 g ai/hL	42	79-82	0	Whole fruit Peel Flesh	1.55, 1.76 (<u>1.66</u>) 5.89, 6.99 (6.44) <0.01, 0.01 (0.01)
							21	Whole fruit Peel Flesh	1.45, 1.87 (1.66) 5.54, 7.40 (6.47) 0.01, 0.02 (0.02)
							42	Whole fruit Peel Flesh	1.53, 1.48 (1.51) 5.67, 5.70 (5.69) 0.01, 0.01 (0.01)
LBS19053-02 2020 Mango	(Keit) Brazil	(Bahia)	Foliar spray	120 120 120 120	1000 1000 1000 1000	65 72 80 82	7	Whole fruit Peel Flesh	0.18 0.73 <0.01
			Foliar followed by post-harvest dip	120 g ai/hL	100	79-81	0	Whole fruit Peel Flesh	2.01, 2.21 (<u>2.11</u>) 7.48, 8.32 (7.90) 0.01, 0.01 (0.01)
							21	Whole fruit Peel Flesh	1.67, 1.45 (1.56) 7.77, 6.92 (7.35) 0.02, 0.02 (0.02)

Trial Information	Crop (Variety)	Country (Region)	Method	Application Rate (g ai/hL)	Water volume (L/ha)	BBCH Growth Stage at Application	DALA	Crop Part[2]	Azoxystrobin Residue mg/kg (mean)
GAP 1	Mango	Brazil	Foliar spray	4 x 120 g ai/ha 14-day RTI	600-1000 L/ha	From pre-flowering	7		
GAP 2	Mango	Brazil	Post-harvest Dip or Spray	1 x 120 g ai/hL 14-day RTI	Not specified	Not specified	0		
							42	Whole fruit Peel Flesh	1.75, 1.86 (1.81) 7.29, 7.14 (7.22) 0.01, 0.02 (0.02)
			Foliar followed by post-harvest spray	120 g ai/hL	42	79-81	0	Whole fruit Peel Flesh	2.17, 2.45 (2.31) 7.80, 9.88 (8.84) 0.01, 0.01 (0.01)
							21	Whole fruit Peel Flesh	2.25, 2.29 (2.27) 10.75, 10.48 (10.62) 0.02, 0.08 (0.05)
							42	Whole fruit Peel Flesh	1.96, 2.19 (2.08) 7.90, 9.09 (8.50) 0.02, 0.03, (0.03)
LBS19053-03 2020 Mango	(Kent) Brazil	(Bahia)	Foliar spray	120 120 120 120	1000 1000 1000 1000	65 72 80 82	7	Whole fruit Peel Flesh	0.06 0.28 <0.01
			Foliar followed by post-harvest dip	120 g ai/hL	100	79-81	0	Whole fruit Peel Flesh	1.45, 1.84 (1.65) 6.54, 7.08 (6.81) 0.05, 0.07 (0.06)
							21	Whole fruit Peel Flesh	1.71, 1.62 (1.67) 6.63, 6.46 (6.55) 0.04, 0.07 (0.06)
							42	Whole fruit Peel Flesh	1.19, 1.40 (1.30) 4.93, 5.84 (5.39) 0.02, 0.03 (0.03)
			Foliar followed by post-harvest spray	120 g ai/hL	42	79-81	0	Whole fruit Peel Flesh	2.34, 1.98 (2.16) 8.91, 7.17 (8.04) 0.03, 0.05 (0.04)
							21	Whole fruit Peel Flesh	2.32, 1.73 (2.03) 9.47, 7.16 (8.32) 0.05, 0.06 (0.06)
							42	Whole fruit Peel Flesh	1.77, 1.51 (1.64) 6.70, 6.46 (6.58) 0.03, 0.04 (0.04)
LBS19053-04 2020 Mango	(Tommy) Brazil	(Pernambuco)	Foliar spray	120 120 120 120	1000 1000 1000 1000	72 75 78 80	7	Whole fruit Peel Flesh	0.19 0.85 <0.01

Trial Information	Crop (Variety)	Country (Region)	Method	Application Rate (g ai/hL)	Water volume (L/ha)	BBCH Growth Stage at Application	DALA	Crop Part[2]	Azoxystrobin Residue mg/kg (mean)
GAP 1	Mango	Brazil	Foliar spray	4 x 120 g ai/ha 14-day RTI	600-1000 L/ha	From pre-flowering	7		
GAP 2	Mango	Brazil	Post-harvest Dip or Spray	1 x 120 g ai/hL 14-day RTI	Not specified	Not specified	0		
			Foliar followed by post-harvest dip	120 g ai/hL	100	79-81	0	Whole fruit Peel Flesh	1.55, 2.31 (1.93) 8.24, 8.03 (8.14) 0.10, 0.04 (0.07)
							21	Whole fruit Peel Flesh	1.70, 1.87 (1.79) 8.03, 8.53 (8.28) 0.03, 0.03 (0.03)
							42	Whole fruit Peel Flesh	1.49, 1.47 (1.48) 6.45, 6.12 (6.29) 0.03, 0.05 (0.04)
			Foliar followed by post-harvest spray	120 g ai/hL	42	79-81	0	Whole fruit Peel Flesh	2.65, 2.12 (2.39) 11.49, 9.56 (10.53) 0.05, 0.02 (0.04)
							21	Whole fruit Peel Flesh	2.30, 2.77 (2.54) 10.63, 11.85 (11.24) 0.02, 0.02 (0.02)
							42	Whole fruit Peel Flesh	1.91, 1.92 (1.92) 8.08, 8.07 (8.08) 0.02, 0.02 (0.02)

Notes:

[1] Reference: LBS19053

[2]The amount of residues in whole fruit was calculated as follows: [(weight of the pulp sample x residue found in the pulp sample) + (weight of peel sample x residue found in the peel sample)] / weight of the whole fruit sample. The weight of the whole fruit sample was calculated as follows: weight of the pulp sample + weight of peel sample + weight of seeds.

Papaya

Four supervised trials with azoxystrobin on papaya were conducted in 2020 in Brazil. Trials were conducted using a suspension concentrate formulation nominally containing 200 g azoxystrobin/L and 125 g difenoconazole/L which was applied four times as a broadcast foliar spray at rates of 100 g ai/ha with a 14 day RTI.

For some trials, foliar applications were followed by one post-harvest immersion or spray application of a suspension concentrate formulation nominally containing 239 g azoxystrobin/L and 239 g fludioxonil/L, which was applied 3 days after the last foliar application. Fruits were washed before being immersed for approximately 2 minutes in the treatment solution in a stainless steel tank, or sprayed with the formulation at a nominal concentration of 120 g ai/hL. Following the post-harvest treatment, fruits

were left to dry before being sprayed with carnauba storage wax (diluted with water 1:4, v/v) before being left to dry and placed in storage.

Samples of treated and untreated whole fruits (at least 12 per sample) were collected 3 days after the last foliar application, and at 0, 6, and 13 days after the post-harvest treatment. The whole samples were separated into peel and flesh fractions, and the seeds removed.

Peel and flesh samples were frozen (nominally -20 °C) and maintained in frozen storage for periods of up to 2.9 months prior to analysis, or placed in storage under conditions that simulate commercial practice: 1 day at 20 °C, 5 days at 10 °C, and 7 days at 18–20 °C.

Residues of azoxystrobin in papaya were determined using the analytical method RAM 305/03. This method is validated for use on papaya flesh and peel in with an LOQ of 0.01 mg/kg.

The results of the trials are summarised in Table 4, where residues relevant to the setting of an MRL for papaya are underlined. Residues in control samples were generally <LOQ, with the exception of three peel samples where residues >LOQ were observed (corresponding to whole fruit control residues of 0.02 mg/kg, 0.09 mg/kg, and 0.12 mg/kg). When compared to the whole fruit residue values used for MRL calculation, the values in the corresponding control samples are considered negligible and to have no impact on the study or the derived MRL and STMR values.

Table 4 Residues of azoxystrobin on papaya in Brazil[1]

Trial Information	Crop (Variety)	Country (Region)	Application Method	Application Rate (g ai/hL)	Water Volume (L/ha)	BBCH Growth Stage at Application	DALA	Crop Part[2]	Azoxystrobin Residue mg/kg (mean)
GAP 1	Papaya	Brazil	Foliar spray	4× 100 g ai/ha 14-day RTI	600-1000 L/ha	From pre-flowering	3		
GAP 2	Papaya	Brazil	Post-harvest Dip or Spray	1× 120 g ai/hL 14-day RTI	Not specified	Not specified	0		
LBS19052-01 2020	(Aliança) Brazil	(Espirito Santo)	Foliar spray	100 100 100 100	1000 1000 1000 1000	71-79 71-79 71-79 71-79	3	Whole fruit Peel Flesh	0.29 1.20[3] <0.01
			Foliar followed by post-harvest dip	120 g ai/hL	100	79-81	0	Whole fruit Peel Flesh	1.57, 0.88 (<u>1.22</u>) 7.36, 3.95 (5.66)[3] 0.02, 0.01 (0.02)
							6	Whole fruit Peel Flesh	1.21, 1.10 (1.15) 5.34, 5.04 (5.19)[3] 0.04, 0.06 (0.05)
							13	Whole fruit Peel Flesh	0.98, 1.02 (1.00) 4.26, 4.67 (4.47)[3] 0.02, 0.06 (0.04)

Trial Information	Crop (Variety)	Country (Region)	Application Method	Application Rate (g ai/hL)	Water Volume (L/ha)	BBCH Growth Stage at Application	DALA	Crop Part[2]	Azoxystrobin Residue mg/kg (mean)
GAP 1	Papaya	Brazil	Foliar spray	4× 100 g ai/ha 14-day RTI	600-1000 L/ha	From pre-flowering	3		
GAP 2	Papaya	Brazil	Post-harvest Dip or Spray	1× 120 g ai/hL 14-day RTI	Not specified	Not specified	0		
			Foliar followed by post-harvest spray	120 g ai/hL	42	79-81	0	Whole fruit Peel Flesh	1.28, 1.72 (1.50) 5.67, 8.16 (6.92)[3] 0.02, 0.01 (0.02)
							6	Whole fruit Peel Flesh	1.36, 1.30 (1.33) 6.24, 5.48 (5.86)[3] 0.11, 0.12 (0.12)
							13	Whole fruit Peel Flesh	1.24, 1.54 (1.39) 5.64, 6.87 (6.26)[3] 0.06, 0.08 (0.07)
LBS19052-02 2020	(Golden/ THB) Brazil	(Bahia)	Foliar spray	100 100 100 100	1000 1000 1000 1000	71-79 71-79 71-79 71-79	3	Whole fruit Peel Flesh	0.27 1.19[4] <0.01
			Foliar followed by post-harvest dip	120 g ai/hL	100	79-81	0	Whole fruit Peel Flesh	1.10, 3.69 (2.40) 4.60, 7.32 (5.96)[4] 0.03, 0.02 (0.03)
							6	Whole fruit Peel Flesh	1.32, 1.44 (1.38) 5.43, 5.66 (5.55)[4] 0.06, 0.14 (0.10)
							13	Whole fruit Peel Flesh	1.95, 1.29 (1.62) 6.30, 5.63 (5.97)[4] 0.06, 0.12 (0.09)
			Foliar followed by post-harvest spray	120 g ai/hL	42	79-81	0	Whole fruit Peel Flesh	0.98, 1.15 (1.06) 4.50, 5.14 (4.82)[4] 0.01, 0.02 (0.02)

Trial Information	Crop (Variety)	Country (Region)	Application Method	Application Rate (g ai/hL)	Water Volume (L/ha)	BBCH Growth Stage at Application	DALA	Crop Part[2]	Azoxystrobin Residue mg/kg (mean)
GAP 1	Papaya	Brazil	Foliar spray	4× 100 g ai/ha 14-day RTI	600-1000 L/ha	From pre-flowering	3		
GAP 2	Papaya	Brazil	Post-harvest Dip or Spray	1× 120 g ai/hL 14-day RTI	Not specified	Not specified	0		
							6	Whole fruit Peel Flesh	1.31, 1.35 (1.33) 5.19, 5.64 (5.42)[4] 0.19, 0.15 (0.17)
							13	Whole fruit Peel Flesh	1.24, 1.35 (1.30) 5.42, 5.78 (5.60)[4] 0.13, 0.09 (0.11)
LBS19052-03 2020	Papaya (Sunrise/ BS)	Brazil (Bahia)	Foliar spray	100 100 100 100	1000 1000 1000 1000	71-79 71-79 71-79 71-79	3	Whole fruit Peel Flesh	0.34 1.26[5] <0.01
			Foliar followed by post-harvest dip	120 g ai/hL	100	79-81	0	Whole fruit Peel Flesh	0.82, 0.69 (0.76) 3.69, 2.67 (3.18)[5] 0.02, 0.01 (0.02)
							6	Whole fruit Peel Flesh	1.16, 1.10 (1.13) 4.85, 4.80 (4.83)[5] 0.03, 0.06 (0.05)
							13	Whole fruit Peel Flesh	0.92, 0.78 (0.85) 3.72, 3.11 (3.42)[5] 0.01, 0.03 (0.02)
			Foliar followed by post-harvest spray	120 g ai/hL	42	79-81	0	Whole fruit Peel Flesh	0.89, 0.96 (0.93) 3.64, 3.69 (3.67)[5] 0.01, 0.03 (0.02)
							6	Whole fruit Peel Flesh	0.48, 0.55 (0.52) 1.83, 2.32 (2.08)[5] 0.11, 0.09 (0.10)

Trial Information	Crop (Variety)	Country (Region)	Application Method	Application Rate (g ai/hL)	Water Volume (L/ha)	BBCH Growth Stage at Application	DALA	Crop Part[2]	Azoxystrobin Residue mg/kg (mean)
GAP 1	Papaya	Brazil	Foliar spray	4× 100 g ai/ha 14-day RTI	600-1000 L/ha	From pre-flowering	3		
GAP 2	Papaya	Brazil	Post-harvest Dip or Spray	1× 120 g ai/hL 14-day RTI	Not specified	Not specified	0		
							13	Whole fruit Peel Flesh	0.33, 0.39 (0.36) 1.51, 1.55 (1.53)[5] 0.01, 0.01 (0.01)
LBS19052-04 2020	Papaya (Tainung)	Brazil (Ceará)	Foliar spray	100 100 100 100	1000 1000 1000 1000	71-76 76-79 79-83 79-83	3	Whole fruit Peel Flesh	0.034 0.13 <0.01
			Foliar followed by post-harvest dip	120 g ai/hL	100	79-83	0	Whole fruit Peel Flesh	0.94, 1.30 (1.12) 5.06, 7.06 (6.06) <0.01, <0.01 (<0.01)
							6	Whole fruit Peel Flesh	0.94, 0.59 (0.76) 3.12, 2.22 (2.67) <0.01, <0.01 (<0.01)
							13	Whole fruit Peel Flesh	0.70, 0.63 (0.67) 3.73, 3.53 (3.63) <0.01, 0.01 (0.01)
			Foliar followed by post-harvest spray	120 g ai/hL	42	79-83	0	Whole fruit Peel Flesh	1.37, 0.99 (1.18) 6.67, 5.41 (6.04) <0.01, <0.01 (<0.01)
							6	Whole fruit Peel Flesh	1.24, 1.34 (1.29) 4.81, 5.55 (5.18) 0.08, 0.08 (0.08)
							13	Whole fruit Peel Flesh	1.48, 1.14 (1.31) 6.57, 5.93 (6.25) 0.11, 0.14 (0.13)

Notes:

[1] Reference: LBS19052.

[2] The amount of residues in whole fruit was calculated as follows: [(weight of the pulp sample x residue found in the pulp sample) + (weight of peel sample x residue found in the peel sample)] / weight of the whole fruit sample. The weight of the whole fruit sample was calculated as follows: weight of the pulp sample + weight of peel sample + weight of seeds.

[3] Control sample contained a residue of 0.08 mg/kg.

[4] Control sample contained a residue of 0.42 mg/kg.

[5] Control sample contained a residue of 0.43 mg/kg.

Sugar Beet

Six supervised trials with azoxystrobin on sugar beet were conducted in the United States in 2015/2016. Trials were conducted using a flowable suspension concentrate formulation containing 239.4 g azoxystrobin/L and 239.4 g fludioxonil/L which was applied once to mature sugar beet roots post-harvest at 4.7 g ai/tonne.

Samples of sugar beets (at least 12 per sample) were collected at maturity and the tops removed. The application was made as a direct spray to the roots, which were tumbled during application to ensure uniform application and to simulate commercial practice. Following the application, the roots were allowed to dry for approximately 2-3 hours (with the exception of trial 04) prior to storage. In trial 04, additional samples of sugar beet roots were taken for processing.

All samples of roots were and maintained in frozen storage (-20 °C) for periods of up to 265 days prior to analysis.

Residues of azoxystrobin and R230310 in sugar beet were determined using the analytical method RAM 305/03 with an LOQ of 0.01 mg/kg per analyte.

The results of the trials are summarised in Table 5. Residues in all control samples and residues of R230310 in treated samples were <LOQ.

Table 5 Residues of azoxystrobin on sugar beet in the United States[1]

Trial Information	Crop (Variety)	Country (Region)	Application Rate (g ai/t)	Water Volume (L/t)	BBCH Growth Stage at Application	Crop Part	Azoxystrobin residue, mg/kg (mean)
cGAP	Sugar beet	United States	4.7	1.9	Mature (post-harvest)		
TK0044248-01 2015/2016	Sugar beet (BTS 60RR27 MP)	United States (Verona, WI)	4.3	1.9	BBCH 49	Root	0.92, 1.7 (1.3)
TK0044248-02 2015/2016	Sugar beet (3574X0853)	United States (Ephrata, WA)	4.8	2.1	BBCH 49	Root	2.6, 2.2 (2.4)
TK0044248-03 2015/2016	Sugar beet (9425RR4M)	United States (Geneva, MN)	4.8	2.1	BBCH 49	Root	0.73, 0.72 (0.73)
TK0044248-04 2015/2016	Sugar beet (SX1521N)	United States (St. Cloud, MN)	4.8	2.1	BBCH 49	Root	1.9, 2.1 (2.0)

Trial Information	Crop (Variety)	Country (Region)	Application Rate (g ai/t)	Water Volume (L/t)	BBCH Growth Stage at Application	Crop Part	Azoxystrobin residue, mg/kg (mean)
cGAP	Sugar beet	United States	4.7	1.9	Mature (post-harvest)		
TK0044248-05 2015/2016	Sugar beet (Select Harvest SUGB14151J)	United States (Wyoming, IL)	4.8	2.1	BBCH 49	Root	1.1, 1.0 (1.1)
TK0044248-06 2015/2016	Sugar beet (Green Valley Lot# 160210)	United States (Richland, IA)	4.5	2.1	BBCH 49	Root	1.4, 1.3 (1.4)

Notes:

[1] Reference: TK0044248.

FATE OF RESIDUES DURING PROCESSING

One processing trial on sugar beet was conducted in Minnesota, United States in 2015/2016 as part of the magnitude of residues study on sugar beet roots (Trial TK0044248-04). The field trial contained one untreated plot and two treated plots. In the treated plots, one post-harvest application of a SC formulation containing a nominal concentration of 239.4 g/L azoxystrobin and 235.9 g/L fludioxonil was made at a rate equivalent to 4.8 g ai/t.

Sugar beet roots were collected immediately after treatment and stored frozen prior to shipment to the processing facility. The roots were cleaned and sliced, and the following processed fractions were produced: raw juice, wet pulp, pressed pulp, press water, dried pulp, thick juice, raw sugar, refined sugar, and molasses.

Raw juice and wet pulp fractions were prepared by placing sliced sugar beet roots (cossettes) in water baths set at 88–92 °C for 30 to 45 seconds, followed by 68–74 °C for at least 9 minutes. Following diffusion, the raw juice was sieved, and the raw juice and wet pulp fractions were collected and placed into frozen storage.

Pressed pulp fractions were prepared from the diffused cossettes using a hydraulic press. Dried beet pulp was produced by drying the pressed pulp at 54–71 °C until the final moisture content was ≤ 15 percent. Pressed and dried pulp samples, in addition to the press water, were collected and placed in freezer storage.

Raw juice was mixed at high temperature (80–85 °C) at pH 10.5 until precipitation. Centrifugation was used to separate the mud and juice. The juice was then mixed again at high temperature (80–85 °C) at pH 9.1–9.3. The juice was centrifuged and filtered to obtain a clear juice (i.e., thin juice). This was again mixed at 80–85 °C, and the pH was reduced to 8.8–9.0 with sodium bisulfite. Evaporation under vacuum at 29 °C was performed until a thick juice (50–60 percent solids) was obtained. Following filtration, thick juice fractions were collected and placed into frozen storage.

Further evaporation of this juice was performed until 70–80 percent solids juice (syrup) was achieved. Following seeding with white sugar, the solution was allowed to cool, and the raw sugar fractions were collected and placed into frozen storage. Sugar and molasses were separated by centrifugation and placed into frozen storage.

In order to obtain ensiled beet pulp, pressed pulp was vacuum sealed in bags and exposed to elevated temperatures (*ca.* 40–46 °C) for 2 days. Following this, after 12 days storage at ambient temperature, the ensiled pulp was collected and placed into frozen storage until arrival at the analytical laboratory.

All samples of sugar beet processed commodities were and maintained in frozen storage (-20 °C) for periods of up to 265 days prior to analysis.

Processed fractions were analysed for residues of azoxystrobin by method RAM 305/03, validated for determination of azoxystrobin in sugar beet roots and processed fractions to an LOQ of 0.01 mg/kg.

Residues of azoxystrobin in sugar beet roots (RAC) were 2.0 mg/kg. Residues were diluted in all the processed fractions of treated sugar beet, with processing factors ranging from 0.017 (refined sugar) to 0.47 (dried pulp).

Table 6 Azoxystrobin residues in sugar beet and processed commodities[1]

Trial Number	Crop (Variety)	Country (Region)	Application Rate[2,3] (g ai/t)	Water volume[3] (L/t)	Crop Part Azoxystrobin residue, mg/kg	(mean)[2,3]	Processing Factors[4]
TK0044248-04 2015/2016	Sugar beet (SX1521N)	United States (St. Cloud, MN)	4.8	2.1	Root	1.9, 2.1 (2.0)	-
					Raw juice	0.56	0.29
					Thick juice	0.21	0.11
					Raw sugar	0.32	0.16
					Refined sugar	0.033	0.017
					Molasses	0.38	0.20
					Wet pulp	0.034	0.018
					Ensiled pulp	0.24	0.12
					Dried pulp	0.93	0.47
					Pressed pulp	0.33	0.17
					Press water	0.54	0.27

Notes:

[1] Reference: TK0044248

[2] Mean of duplicate samples (RAC)

[3] Mean of triplicate samples (processed commodities)

[4] Processing factor = residue in processed commodity ÷ residue in unprocessed commodity (RAC)

APPRAISAL

Azoxystrobin was first evaluated for toxicology and residues by the JMPR in 2008. It was evaluated for residues by the JMPR in 2011, 2012, 2013, 2017 and 2019. An ADI of 0–0.02 mg/kg bw was established and an ARfD was considered unnecessary.

The residue definition for compliance with the MRL and for dietary risk assessment for plant and animal commodities is parent azoxystrobin. The residue is fat soluble.

Azoxystrobin was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional MRLs in 2021 and rescheduled to the 2022 JMPR. The current Meeting received additional analytical methods, storage stability data, GAP information and residue trial data from uses on mango, papaya, sugar beet, and sugar beet processed commodities.

Methods of analysis

Residue analytical method RAM 305/03 was used for the analysis of azoxystrobin in the supervised residue trials on mango, papaya, sugar beet, and the processing study on sugar beet. Method RAM 305 was previously validated as version RAM 305/01 in a wide range of crops and crop types by the 2008 JMPR. Method RAM 305/03 is procedurally the same as the version RAM 01 and validation data evaluated for RAM 305/01 are applicable to RAM 305/03.

Recoveries and percentRSDs for RAM 305/03 were within the acceptable range (70–120 percent). The LOQ is 0.01 mg/kg for all versions of the method and all plant commodities tested.

The Meeting concluded that RAM 305/03 was sufficiently validated and is suitable to measure azoxystrobin in mango peel and flesh, papaya peel and flesh, and sugar beet and its processed commodities.

Stability of pesticides residues in stored analytical samples

In 2008, the JMPR evaluated data on the stability of azoxystrobin in plant and animal commodities stored frozen at ≤-18 °C. The 2008 Meeting concluded that residues of azoxystrobin were stable for at least 24 months in high water and high starch commodities.

The Meeting concluded that the storage stability data are adequate to support the storage durations in studies provided to the current Meeting.

Results of supervised residue trials on crops

Mango

The cGAP for azoxystrobin on mango from Brazil is four foliar-directed applications at 120 g ai/ha with a 7-day PHI and 14-day re-treatment interval (RTI), followed by a post-harvest dip or spray application at 120 g ai/hL.

In independent trials matching the cGAP for foliar + post-harvest dip applications, residues of azoxystrobin in whole fruit were (n=4): 1.67, 1.93, 2.11, and 2.67 mg/kg.

In independent trials matching the cGAP for foliar + post-harvest spray applications, residues of azoxystrobin in whole fruit were (n=4): 1.66, 2.16, 2.31, and 2.54 mg/kg.

The Meeting considered the foliar + post-harvest dip and foliar + post-harvest spray applications independent and agreed to combine the data sets to estimate a maximum residue level.

Residues of azoxystrobin in mangos (whole fruit) from trials approximating the cGAP were (n=8): 1.66, 1.67, 1.93, 2.11, 2.16, 2.31, 2.54, and 2.67 mg/kg.

The Meeting estimated a maximum residue level (based on the mean + 4 SD) of 4 mg/kg (Po) for azoxystrobin in mangoes and withdrew its previous recommendation.

Residues in mango flesh from trials approximating cGAP were (n=8): 0.02 (4), 0.05, 0.06 (2), and 0.07 mg/kg.

The Meeting estimated an STMR of 0.035 mg/kg for mango flesh.

Papaya

The cGAP for azoxystrobin on papaya from Brazil is four foliar-directed applications at 100 g ai/ha with a 3 day PHI and 14 day re-treatment interval (RTI), followed by a post-harvest dip or spray application at 120 g ai/hL.

In independent trials matching the cGAP for foliar + post-harvest dip applications, residues of azoxystrobin were (n=4): 1.12, 1.13, 1.22, and 2.40 mg/kg.

In independent trials matching the cGAP for foliar + post-harvest spray applications, residues of azoxystrobin were (n=4): 0.93, 1.31, 1.33, and 1.50 mg/kg.

The Meeting considered the foliar + post-harvest dip and foliar + post-harvest spray applications independent and agreed to combine the data sets to estimate a maximum residue level.

Residues of azoxystrobin in mangos (whole fruit) from trials approximating the cGAP were (n=8): 0.93, 1.12, 1.13, 1.22, 1.31, 1.33, 1.50, and 2.40 mg/kg.

The Meeting estimated a maximum residue level (based on mean + 4 SD) of 4 mg/kg (Po) for azoxystrobin in papaya to replace its previous recommendation.

Residues in papaya flesh from trials approximating the cGAP were (n=8): 0.01, 0.05 (2), 0.10 (2), 0.12, 0.13, and 0.17 mg/kg.

The Meeting estimated an STMR of 0.10 mg/kg for papaya flesh.

Sugar beet

The critical GAP in the United States consists of one post-harvest application at a rate of 4.7 g ai/tonne roots.

In independent trials matching the cGAP, residues of azoxystrobin in sugar beet roots were (n=6): 0.73, 1.1, 1.3, 1.4, 2.0, 2.4 mg/kg.

The Meeting estimated a maximum residue level (based on mean + 4 SD) of 4 mg/kg (Po) and an STMR of 1.35 mg/kg for azoxystrobin in sugar beets. Furthermore, the Meeting withdrew its previous recommendation of 1 mg/kg for azoxystrobin in root and tuber vegetables except potato and recommended a new maximum residue level of 1 mg/kg for azoxystrobin in root and tuber vegetables except potato and sugar beet.

Fate of residues during processing

Processing factors and residue estimates for sugar beet processed commodities are summarised below.

Table 7 Processing factors and residue estimates for azoxystrobin

Raw commodity	STMR	Processed commodity	Processing Factors	STMR-P/ Median-P
Sugar beet root	1.35	Molasses	0.20	0.27
		Refined sugar	0.017	0.023
		Dried pulp	0.47	0.635
		Ensiled pulp	0.12	0.162

The Meeting estimated the STMR-Ps/median-Ps listed in Table 1 for use in dietary risk assessment and estimation of animal dietary burdens.

Residues in animal commodities

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the JMPR. The dietary burdens, estimated using the 2018 OECD Feed diets listed in Appendix XIV Electronic attachments to the 2016 Edition of the FAO manual, are presented in Annex 6.

Of the commodities evaluated by the current Meeting, only sugar beet molasses, dried pulp, and ensiled pulp need consideration with respect to livestock diets.

Table 8 shows the dietary burdens calculated by the 2013 JMPR and the current Meeting.

Table 8 Estimated maximum and mean dietary burdens of farm animals

	Livestock dietary burden, azoxystrobin, ppm of dry matter diet (2013/2022)							
	Japan		**United States-Canada**		**European Union**		**Australia**	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.96/6.9	0.96/1.8	17/25	12/16	61/63	25/26	72/83❶	51/51❸
Dairy cattle	16/19	3.0/3.5	30/45	12/14	74/81❷	29/31❹	46/51	20/22
Poultry – broiler	1.4/1.4	1.4/1.4	1.7/1.7	1.7/1.7	2.2/5.7	1.9/5.0	1.7/1.7	1.7/1.7
Poultry – layer	1.4/1.4	1.4/1.4	1.7/1.7	1.7/1.7	21/25❺	9.5/12❻	1.7/1.7	1.7/1.7

Notes:

❶ Highest maximum beef or dairy cattle burden suitable for MRL estimates for mammalian tissues.

❶ Highest maximum dairy cattle burden suitable for MRL estimates for milk

❸ Highest mean beef or dairy cattle burden suitable for STMR estimates for mammalian tissues

❹ Highest mean dairy cattle burden suitable for STMR estimates for milk.

❺ Highest maximum poultry broiler or layer burden suitable for MRL estimates for poultry eggs and tissues.

❻ Highest mean poultry broiler or layer burden suitable for STMR estimates for poultry eggs and tissues.

The Meeting noted that the new estimations did not result in a signification change of the dietary burdens of farm animals. Based on the minor change in livestock dietary burden, the Meeting did not recalculate residues in animal commodities or revise its recommendations for maximum residue levels.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed in Annex 1 are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with the MRL and for dietary risk assessment for plant and animal commodities: *azoxystrobin*.

The residue is fat-soluble.

Table 9 Recommendations for residues of azoxystrobin from the 2022 JMPR

CCN	Crop/Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
FI 0345	Mango	4 (Po)	0.7	0.035	--
FI 0350	Papaya	4 (Po)	0.3	0.1	--
VR 0596	Sugar beet	4 (Po)	--	1.35	--
VR 0075	Group of root and tuber vegetables except potato	W	1	0.23	--
VR 0075	Group of root and tuber vegetables except potato and sugar beet	1	--	0.23	--
For dietary risk assessment and/or animal dietary burden calculation					
DM 0596	Sugar beet molasses	--	--	0.27	--
DM 3523	Sugar beet refined sugar	--	--	0.023	--
AM 3599	Sugar beet dried pulp	--	--	Median: 0.635	--
	Sugar beet ensiled pulp	--	--	Median: 0.162	--

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for azoxystrobin is 0–0.2 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for azoxystrobin were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the previous and present JMPR. The results are shown in Annex 3 of the 2022 JMPR Report. The IEDIs ranged 3–20 percent of the maximum ADI.

The Meeting concluded that the long-term intake of residues of azoxystrobin from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2008 Meeting determined that the establishment of an acute reference dose is unnecessary for azoxystrobin. The Meeting concluded that the acute dietary exposure to residues of azoxystrobin, from uses considered by the present Meeting, is unlikely to present a public health concern.

REFERENCES

Study Report	Author	Year	Citation
TK0044248	Emily Shepard	2017	Magnitude of the Residues on Sugarbeets and in Sugarbeet Processed Commodities Azoxystrobin + Fludioxonil Residues on Sugarbeets Following Post-Harvest Treatment in 2015
LBS19053	Fernanda Campos Mastrotti Pereira	2020	Magnitude of the Residues of Fludioxonil, Azoxystrobin and its Isomer R230310 in Mango Fruits
LBS19052	Fernanda Campos Mastrotti Pereira	2020	Magnitude of Residues of Fludioxonil, Azoxystrobin and its Isomer R230310 in Papaya Fruits
VR-016/20	Marta B. Bento Magagnato	2020	Validation Study of Analytical Methodology for Residue Analysis of Active Ingredient Azoxystrobin (IC15504) and its Isomer R230310 in Mango Fruits (Pulp and Peel) – RAM 305/03
VR-015-20	Marta B. Bento Magagnato	2020	Validation Study of Analytical Methodology for Residue Analysis of Active Ingredient Azoxystrobin (IC15504) and its Isomer R230310 in Papaya Fruits (Pulp and Peel) – RAM 305-03

BENZOVINDIFLUPYR (261)

First draft prepared by T. van der Velde-Koerts, Centre for Nutrition, Prevention and Health Services (VPZ), National Institute for Public Health and the Environment (RIVM), The Netherlands

EXPLANATION

Benzovindiflupyr is a broad-spectrum fungicide first evaluated by JMPR in 2013 for Toxicology and in 2014 for Residues. The compound was re-evaluated in 2016, 2018 and 2019 for additional uses.

An ADI of 0–0.05 mg/kg bw and an ARfD of 0.1 mg/kg bw were established by the 2013 JMPR. The 2014 JMPR Meeting recommended the residue definition for plant and animal commodities (for compliance with MRLs and for estimation of dietary intake) as: benzovindiflupyr. The residue is fat soluble.

Benzovindiflupyr was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2021 JMPR and rescheduled for evaluation by the 2022 JMPR. The Meeting received additional information from the manufacturer on the toxicity of metabolites, method of residue analysis, use patterns, supervised residue trials (blueberries, dried ginseng, sugar beets and maize) and processing studies on sugar beets.

RESIDUE ANALYSIS

Analytical methods used in supervised residue trials

HPLC-MS/MS Method GRM042.03A

The Meeting received additional reduced validation data for blueberries, dried ginseng roots, sugar beet commodities and maize commodities for HPLC-MS/MS Method GRM042.03A (dated 4 August 2011). This method was summarized and evaluated by the JMPR 2014 and was considered valid for the determination of parent and metabolite SYN 546039 (free and conjugated) in the range 0.01–0.1 mg/kg for apples, grapes, wheat (forage, grain, hay, straw, flour), spinach, lettuce, peanuts, coffee beans, carrot (roots and leaves), turnip (roots and leaves), radish (roots and leaves), orange juice and sugarcane. The valid range was extended to 0.5 mg/kg for wheat forage and wheat hay.

Blueberries

Blueberries were analysed for benzovindiflupyr using a modification of method GRM042.03A. The modification was described in [Byeongseok, 2019, VV-619742 and Clark, 2016, 2017] and was dated 8 December 2016 (equivalent to 23 January 2017). The modifications involved direct determination of the parent compound without clean-up and a change in HPLC-MS/MS conditions. The metabolite SYN 546039 was not determined.

Blueberries (10 g) were extracted by homogenization in 80/20 (v/v) acetonitrile/water followed by centrifugation. The supernatants were diluted with acetonitrile/water (50:50, v:v) and analysed for benzovindiflupyr by UPLC-MS/MS using negative ion – turbo ion spray ionization – Q-trap triple quadrupole mass spectrometry with multiple reaction monitoring (MRM). Benzovindiflupyr was monitored at m/z 395.9 (Q1) and 90.9 (Q3) for quantification and at m/z 395.9 (Q1) and 368 (Q3) or 324 (Q3) for confirmation.

Dried ginseng roots

Dried ginseng roots were analysed for benzovindiflupyr and the metabolite SYN 546039 including its conjugates using a modification of method GRM042.03A. The modification (WO 9.222 v1) was described in [Lennon, 2019, VV-547840] and was dated 18 October 2017. The modification included a change in acid hydrolysis concentration to release the SYN 546039 aglycon, change in clean-up procedures, the use of matrix matched standards for SYN 546039 and a change in HPLC-MS/MS instrumentation and conditions.

Homogenised dried ginseng roots (5 g) were pre-soaked with water for 30 min. The samples were extracted by homogenization with 80/20 (v/v) acetonitrile/water, followed by centrifugation. An aliquot of the supernatant was removed, the acetonitrile was evaporated off and 1 M HCl added. The acidified aliquot was partitioned four times with iso-hexane to separate parent off.

The combined iso-hexane fractions containing the parent compound were evaporated to dryness, dissolved in acetonitrile/water (50:50, v/v) and analysed for benzovindiflupyr by UPLC-MS/MS using negative ion – electrospray ionization (ESI-) – Q-trap triple quadrupole mass spectrometric detection with multiple reaction monitoring (MRM). Benzovindiflupyr was monitored at m/z 396 (Q1) and 91 (Q3) for quantification and at m/z 396 (Q1) and 368 (Q3) for confirmation.

The acidic aqueous fraction containing the metabolite SYN 546039 and its conjugates was heated in a water bath at 100°C and agitated for 6 hours to hydrolyse conjugates of SYN 546039. The samples were cooled and cleaned up on an Agilent Bond Elut Phenyl Modified (PH) SPE column, followed by clean-up with aminopropyl functionated silica gel (PSA). The eluant was diluted with acetonitrile/water (50:50, v/v) and analysed for SYN 546039 by UPLC-MS/MS using negative ion – electrospray ionization (ESI-) – Q-trap triple quadrupole mass spectrometric detection with multiple reaction monitoring (MRM). SYN 546039 was monitored at m/z 412 (Q1) and 91 (Q3) for quantification and at m/z 412 (Q1) and 340 (Q3) for confirmation using matrix matched standards.

Sugar beet tops, sugar beet roots, dried pulp

Sugar beet commodities were analysed for benzovindiflupyr and the metabolite SYN 546039 including its conjugates using a modification of method GRM042.03A. The modification was described in [Dorsey, 2019, VV-547512; Shepard, 2019, VV-547573 and Rodgers, 2015a] and was dated 23 October 2015 (sugar cane). Modifications included a change in acid hydrolysis concentration to release the SYN 546039 aglycon, change in clean-up and HPLC-MS/MS instrumentation and conditions.

Sugar beet tops, roots, and dried pulp samples were extracted with acetonitrile:water (80:20, v:v) using a homogenizer followed by centrifugation. Aliquots of the extract were diluted with acetonitrile:water (50:50, v:v) and analysed for benzovindiflupyr by HPLC-MS/MS using positive ion – turbo ion spray (TIS) – triple quadrupole mass spectrometric detection with multiple reaction monitoring (MRM). Benzovindiflupyr was monitored at m/z 398 (Q1) and 342 (Q3) for quantification and at m/z 398 (Q1) and 322 (Q3) or 286 (Q3) for confirmation.

For the analysis of the metabolite SYN 546039 (including its conjugates), additional aliquots of the acetonitrile:water extracts were evaporated in a heated water bath to remove the acetonitrile. Aqueous 1 M HCl was added, followed by three partitions with iso-hexane. The acidic, aqueous fraction was collected and heated at 100 °C for six hours in a heating block with regular, manual agitation to hydrolyse conjugates of SYN 546039. The samples were cooled and taken through a Waters Oasis HLB SPE cartridge. SYN 546039 was eluted from the cartridge with acetonitrile:water (50:50, v/v). Samples were diluted with acetonitrile:water (50:50, v/v) and analysed for SYN 546039 by HPLC-MS/MS using positive ion–turbo ion spray (TIS)–triple quadrupole mass spectrometric detection with multiple reaction

monitoring (MRM). SYN 546039 was monitored at m/z 414 (Q1) and 159 (Q3) for quantification and at m/z 414 (Q1) and 238 (Q3) for confirmation.

Sugar beet refined sugar and sugar beet molasses

Sugar beet commodities were analysed for benzovindiflupyr and the metabolite SYN 546039 including its conjugates using modifications of method GRM042.03A [Dorsey, 2019, VV-547512; Shepard, 2019, VV-547573 and Rodgers, 2015b, Rodgers, 2015c] dated 30 November 2015 (molasses) and 17 December 2015 (refined sugar). The modification included a change in extraction solvent, change in acid hydrolysis concentration to release the SYN 546039 aglycon, change in clean-up and HPLC-MS/MS instrumentation and conditions.

Refined sugar samples were dissolved in water and molasses samples were dissolved in acetonitrile:water (50:50, v:v). Aliquots of the molasses samples were evaporated in a heated water bath to remove the acetonitrile. Aliquots of the sugar and molasses samples were acidified with aqueous 1 M HCl. The acidified aliquot was partitioned with iso-hexane to separate parent compound off. The iso-hexane fraction was diluted with acetonitrile/water (50:50, v/v) and analysed for benzovindiflupyr by HPLC-MS/MS using positive ion – turbo ion spray (TIS) – triple quadrupole mass spectrometric detection with multiple reaction monitoring (MRM). Benzovindiflupyr was monitored at m/z 398 (Q1) and 342 (Q3) for quantification and at m/z 398 (Q1) and 322 (Q3) or 286 (Q3) for confirmation

The acidic, aqueous fraction containing SYN 546039 and its conjugates was heated at 100 °C for six hours in a heating block to hydrolyse conjugates of SYN 546039. The samples were cooled and taken through a Waters Oasis HLB SPE cartridge. SYN 546039 was eluted from the cartridge with acetonitrile:water (50:50, v:v). Samples were diluted with acetonitrile:water (50:50, v:v) then analysed for SYN 546039 by HPLC-MS/MS using positive ion – turbo ion spray – triple quadrupole mass spectrometric detection with multiple reaction monitoring (MRM). SYN 546039 was monitored at m/z 414 (Q1) and 159 (Q3) for quantification and at m/z 414 (Q1) and 238 (Q3) for confirmation.

Maize forage, fodder and grains

Maize commodities were analysed for benzovindiflupyr and the metabolite SYN 546039 including its conjugates using a modification of method GRM042.03A. The modification is described in [Banman, 2020, VV-872218]. The modification involved direct determination of the parent compound without clean-up, a change in acid hydrolysis concentration to release the SYN 546039 aglycon and a change in HPLC-MS/MS conditions.

Maize forage (10 g) was extracted by homogenization in 80/20 (v/v) acetonitrile/ water, followed by centrifugation. Maize stover and maize grains, 5 g sub samples were pre-soaked with water prior to homogenization with 80/20 (v/v) acetonitrile/ water. An aliquot of the supernatant was diluted with acetonitrile/water (50:50, v:v) and analysed for benzovindiflupyr by HPLC-MS/MS with negative ion – electrospray ionization (ESI) – tandem mass spectrometry with multiple reaction monitoring (MRM). Benzovindiflupyr was monitored at m/z 396.1 (Q1) and 91 (Q3) for quantification and at m/z 398 (Q1) and 368 (Q3) for confirmation.

For the analysis of the metabolite SYN 546039 (including its conjugates), additional aliquots of the acetonitrile:water extracts were evaporated in a heated water bath to remove the acetonitrile. Aqueous 1 M HCl was added, followed by three partitions with iso-hexane. The acidic, aqueous fraction was collected and heated at 100 °C for six hours in a heating block with regular, manual agitation to hydrolyse conjugates of SYN 546039. The samples were cooled and taken through a Waters Oasis HLB SPE cartridge. SYN 546039 was eluted from the cartridge with acetonitrile:water (50:50, v:v). Samples

were diluted with acetonitrile:water (50:50, v/v) and analysed for SYN 546039 by HPLC-MS/MS with negative-ion – electrospray ionization (ESI) – triple quadrupole mass spectrometry with multiple reaction monitoring (MRM). SYN 546039 was monitored at m/z 395.5 (Q1) and 90.9 (Q3) for quantification and at m/z 395.5 (Q1) and 368 (Q3) for confirmation.

The analytical method was validated by spiking macerated samples with benzovindiflupyr just before extraction and by spiking the hydrolysates with SYN 546039 (just after hydrolysis at 100 °C and subsequent cooling). Validation results for blueberries, dried ginseng roots, sugar beet commodities and maize commodities are shown in Table 1 (benzovindiflupyr) and Table 2 (metabolite SYN 546039).

Remark by the reviewer:

- Samples were fortified with the free form of SYN536039 just after the acid hydrolysis procedure and thus validation only covers the clean-up part. The JMPR 2014 report indicates that acceptable reduced validation data were available, where SYN 546039 was added before extraction. In addition, the JMPR 2014 report indicates that acceptable validation data are available for the hydrolysis of SYN546039 conjugates using 0.5 M HCl in a radio-validation study. The acid concentration has changed from 0.5 M HCl in the original method to 1 M HCl in the current method. As the hydrolysis efficiency is acceptable at the 0.5 M HCl treatment, it can be assumed that the hydrolysis efficiency is also acceptable at 1 M HCl.
- HPLC-MS/MS method GRM042.03A modifications are considered reduced validated for the determination of benzovindiflupyr in the range 0.01–3.0 mg/kg in maize stover, 0.01–2.0 mg/kg in blueberries and 0.01–1.0 mg/kg in dried ginseng roots, sugar beet commodities (roots, tops, dried pulp, molasses, refined sugar) and 0.01–0.1 mg/kg in other maize commodities (grain, forage).
- HPLC-MS/MS method GRM042.03A modifications are considered reduced validated for the determination of SYN546039 (including its conjugates) in the range 0.01–1.0 mg/kg in sugar beet commodities (roots, tops, dried pulp, molasses, refined sugar) and 0.01–0.1 mg/kg in dried ginseng roots and maize commodities (grain, forage, stover).

Table 1 Validation results for benzovindiflupyr using HPLC-MS/MS method GRM042.03A or its modified versions

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r percent	control samples mg/kg (n)	calibration	reference, method
blueberries	0.01	0.01 0.02 0.1 2	6 3 3 3	104 99 103 97	100-110 97-100 101-105 95-101	3 2 2 3	<0.01 (10)	5 replicate points 0.02-0.5 ng/mL in solvent linear r>0.999	[Byeongseok. 2019, VV-619742] method validation + concurrent recovery
dried ginseng roots	0.01	0.01 0.1 1.0	6 4 3	71 71 76	63-78 62-86 69-82	5 11 7	<0.01 (4)	4 replicate points 0.08-8 ng/mL in solvent linear R^2>0.98	[Lennon, 2019, VV-547840] method validation + concurrent recovery
sugar beet tops	0.01	0.01 1.0	3 3	99 98	95-105 96-101	5.1 2.5	<0.01 (22)	6 replicate points 0.02-1.0 ng/ml in solvent 1/× weighing r>0.999	[Dorsey, 2019, VV-547512] method validation
sugar beet roots	0.01	0.01 1.0	3 3	90 99	86-97 95-101	6.9 3.3	<0.01 (22)	6 replicate points 0.02-1.0 ng/ml	[Dorsey, 2019, VV-547512]

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r percent	control samples mg/kg (n)	calibration	reference, method
								in solvent 1/× weighing r>0.999	method validation
sugar beet dried pulp	0.01	0.01 1.0 2.0	3 3 1	98 102 107	94-101 99-108	3.6 4.6 -	<0.01 (2)	6 replicate points 0.02-1.0 ng/ml in solvent 1/× weighing r>0.999	[Dorsey, 2019, VV-547512] method validation
sugar beet refined sugar	0.01	0.01 1.0	3 3	96 91	84-109 88-94	13 3.4	<0.01 (2)	6 replicate points 0.02-1.0 ng/ml in solvent 1/× weighing r>0.999	[Dorsey, 2019, VV-547512] method validation
sugar beet molasses	0.01	0.01 1.0	3 4	91 88	88-93 82-92	2.8 5.1	<0.01 (2)	6 replicate points 0.02-1.0 ng/ml in solvent 1/× weighing r>0.999	[Dorsey, 2019, VV-547512] method validation + concurrent recoveries
maize forage	0.01	0.01 0.1 3.0	3 3 1	92 99 96	86-100 98-99 -	8.0 0.4 -	<0.01 (10)	7 replicate points 2-500 ng/mL in solvent 1/× weighing R^2>0.98	[Banman, 2020, VV-872218] method validation
maize grains	0.01	0.01 0.1	3 3	99 104	94-104 98-108	5.0 5.0	<0.01 (14)	8 replicate points 2-1000 ng/mL in solvent 1/× weighing R^2>0.99	[Banman, 2020, VV-872218] method validation
maize stover	0.01	0.01 0.1 3.0	3 3 3	106 103 103	103-108 101-105 100-107	2.2 1.9 3.5	<0.01 (12)	7 replicate points 2-500 ng/mL in solvent 1/× weighing R^2>0.98	[Banman, 2020, VV-872218] method validation

Table 2 Validation results for metabolite SYN 546039 using HPLC-MS/MS method GRM042.03A or its modified versions

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r percent	control samples mg/kg (n)	calibration	reference, method
dried ginseng roots	0.01	0.01 0.1 [a]	7 3	69 112	65-74 110-115	3 3	<0.01 (4)	4 replicate points 0.08-8 ng/mL matrix matched linear R2>0.98	[Lennon, 2019, VV-547840] method validation
sugar beet tops	0.01	0.01 1.0 [a]	3 3	96 99	93-98 96-101	2.4 2.4	<0.01 (22)	6 replicate points 0.02-1.0 ng/ml in solvent 1/× weighing r>0.9999	[Dorsey, 2019, VV-547512] method validation
sugar beet roots	0.01	0.01 1.0	3 3	96 97	92-99 96-99	3.8 1.4	<0.01 (22)	6 replicate points 0.02-1.0 ng/ml in solvent	[Dorsey, 2019, VV-547512] method validation

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean range	RSD_r percent	control samples mg/kg (n)	calibration	reference, method
		[a]					1/× weighing r>0.9999	
sugar beet dried pulp	0.01	0.01 1.0 [a]	3 3	94 91-96 96 95-98	2.5 1.4	<0.01 (2)	6 replicate points 0.02-1.0 ng/ml in solvent 1/× weighing r>0.9999	[Dorsey, 2019, VV-547512] method validation
sugar beet refined sugar	0.01	0.01 1.0 [a]	3 3	80 75-84 100 98-101	5.6 1.7	<0.01 (2)	6 replicate points 0.02-1.0 ng/ml in solvent 1/x weighing r>0.9999	[Dorsey, 2019, VV-547512] method validation
sugar beet molasses	0.01	0.01 1.0 [a]	3 3	88 80-95 102 100-103	8.8 1.4	<0.01 (2)	6 replicate points 0.02-1.0 ng/ml in solvent 1/x weighing r>0.9999	[Dorsey, 2019, VV-547512] method validation
maize forage	0.01	0.01 0.1 [a]	3 3	90 82-99 86 82-93	9.5 7.0	<0.01 (10)	8 replicate points 2-500 ng/mL in solvent 1/x weighing R2>0.98	[Banman, 2020, VV-872218] method validation
maize grains	0.01	0.01 0.1 [a]	3 3	98 95-102 89 87-90	4.2 1.5	<0.01 (10)	8 replicate points 2-500 ng/mL in solvent 1/x weighing R2>0.99	[Banman, 2020, VV-872218] method validation
maize stover	0.01	0.01 0.1 [a]	3 3	75 69-81 76 75-77	7.8 1.0	<0.01 (10)	8 replicate points 2-500 ng/mL in solvent 1/x weighing R2>0.99	[Banman, 2020, VV-872218] method validation

Notes:

[a] SYN 546039 was spiked after hydrolysis.

USE PATTERN

The Meeting received new authorised labels from the United States for lowbush blueberries, ginseng, sugar beet and maize. These are summarised in Table 3.

The label indicates a plant back interval of 6 months (180 days), except for crops mentioned in the United States label (i.e. lowbush blueberries, bulb vegetables, rape seed (canola), wheat, barley, triticale, rye, oat, maize (field corn, popcorn), sweet corn, cotton, cucurbit vegetables, fruiting vegetables, ginseng, grasses grown for seed (bluegrass, bromegrass, fescue, orchard grass, ryegrass), pulses (dry legumes), peanuts, potatoes, soya beans, sugar beets, sugar cane, tomatoes, tuberous and corm vegetables).

Table 3 Registered outdoor pre-harvest uses of benzovindiflupyr

Crop	Country	Form (g ai/L)	Application				PHI, days
			Method	Rate kg ai/ha	Water L/ha	Number (RTI in days)	
Blueberries lowbush	United States	EC 100	foliar spray [d]	0.076 max seasonal rate 0.15	[c]	2 (10-14)	1
Ginseng	United States	EC 100	foliar spray [d]	0.076 max seasonal rate 0.31	[c]	4 (14)	15
Sugar beet	United States	EC 100 [a]	in furrow or banded soil application followed by a foliar spray [d]	0.075 [b] maximum seasonal rate 0.15	[c]	1 soil + 1 foliar	soil at 2-8 leaf stage; foliar up to BBCH 31
	United States	EC 100 [a]	foliar spray [d]	0.075 [b] max seasonal rate 0.15	[c]	2 (5-14)	up to BBCH 31
Maize (field corn; popcorn)	United States	EC 100 [a]	foliar spray [d]	0.029-0.051 max seasonal rate 0.10	[c]	2 (14)	7
Maize [e]	Canada	EC 100	foliar spray (aerial or field sprayers)	0.030-0.075 max seasonal rate 0.15	[f]	2 (7)	7 [g h]

Notes:

[a] For foliar sprays, the addition of a spreading/penetrating type adjuvant such as organo-silicon blends with either non-ionic surfactants (NIS) or vegetable based crop oils (COC); or vegetable based COC (not mineral); or NIS with at least 90 percent concentration is recommended. Sugar beet soil applications: tank mixtures with crop oil concentrates (COC) or methylated spray oil (MSO) may result in sugar beet crop injury.

[b] Dose rates for in-furrow or banded soil applications are based on planted row spacing and row width.

[c] For foliar sprays, thorough coverage is necessary to provide good disease control. Ground equipment: apply in a minimum of 93 L/ha (10 GPA) for sugar beet (soil and foliar applications) and maize, 187 L/ha (20 GPA) for lowbush blueberries, 467 L/ha (50 GPA) for ginseng. Aerial equipment: apply in a minimum of 19 L/ha (2 GPA) for lowbush blueberries, ginseng, maize and sugar beet foliar applications. Do not apply through any ultra-low volume sprays.

[d] Foliar sprays may be applied by all types of spray equipment commonly used for making ground and aerial applications. Ginseng, sugar beets and maize may also be treated by chemigation (i.e. irrigation equipment).

[e] Maize includes field corn, sweet corn, popcorn and specialty including all cultivars and/or hybrids of these, including seed production.

[f] For foliar sprays, thorough coverage is necessary to provide good disease control. Ground equipment: apply in a minimum of 200 L/ha for maize. Aerial equipment: apply in a minimum of 45 L/ha. Do not apply through any ultra-low volume sprays.

[g] PHI of 7 days for forage, sweet corn and maize grain

[h] Rotational crop restriction of 180 days. No rotational crop restrictions for tuberous and corm vegetables (including potatoes), pulses (including soya beans); fruiting vegetables (including cucurbits), cereals, maize and rapeseed.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received supervised trials to support additional MRLs for benzovindiflupyr- (Table 4).

Table 4 Summary of supervised trials

Group	Commodity	Table
Berries and other small fruits	Blueberries	Table 5

Group	Commodity	Table
Subgroup of bush berries		
Root and tuber vegetables Subgroup of root vegetables	Dried ginseng	Table 6
	Sugar beet roots	Table 7
Cereal grains Subgroup of maize cereals	Maize grains	Table 8
Cereal grains and grasses feed products Subgroup of products with high water content	Maize forage	-
Cereal grains and grasses feed products Subgroup of products with low water content	Maize stover	Table 9
Miscellaneous feed products Subgroup of products with high water content	Sugar beet tops	Table 10

In many trials duplicate field samples were taken and analysed separately; in the residue trials summary tables, the individual residue results for duplicate field samples were presented together with their mean (in parenthesis). When residues were not quantifiable they were shown as below the LOQ (e.g. < 0.01 mg/kg). Residues, application rates and spray concentrations were rounded to two significant figures; unrounded figures were used to calculate mean residues. Results were not corrected for concurrent method recoveries. Data used in estimation of MRL, HR and STMR values were underlined in the residue trials summary tables. Where higher residue values occurred at longer PHIs than the cGAP, these higher values were underlined and used in the estimation.

Subgroup of Bush berries (004B)

Blueberries

The Meeting received nine supervised residue trial data on highbush and lowbush blueberries conducted outdoors in Canada in 2016 [Byeongseok, 2019, VV-619742]. Highbush and lowbush blueberries were sprayed two times with an EC formulation in the range between 0.074–0.079 kg ai/ha for each application, using 246–287 L/ha water. Two trials were conducted at exaggerated rates. Non-ionic surfactants were added to the tank mix. The application was by tractor mounted CO_2 sprayer or hand-held CO_2 sprayer. No rain occurred within 24 hours after the first or second application, except for trial 154.

Sampling: For lowbush blueberries, mature fruits were harvested with a hand rake in a diagonal swath across the plot avoiding at least 0.5 m from plot ends. Individual plots were at least 60 m^2. Samples were collected from 12–14 areas within the plot and from high and low areas of the plant, which contained sheltered and exposed fruits. Collected berries were placed on a clean screen and a fan was used to remove leaves and stems. Two replicate samples were collected from each plot. Each sample weighed between 0.5–0.9 kg.

For highbush blueberries (and lowbush trial 288), mature fruits were handpicked from two sides of the row, avoiding the bush at the row ends. Individual plots consisted of at least 6 plants. Samples were collected from at least 12 areas in the plot, from inside and outside, top and bottom, exposed and sheltered areas of bushes. Stems and leaves were removed while harvesting. Two replicate samples were collected from each plot. Each sample weighed between 0.5–0.9 kg.

Storage: Blueberries were frozen within 7 hrs after harvest and were stored for 155–227 days (7.5 months) at -10 °C or lower. Actual storage temperatures reached -5.9 °C for trial 160.

Analysis: Benzovindiflupyr was determined in the homogenised berries using a modification of HPLC-MS/MS method GRM042.03A with an LOQ of 0.01 mg/kg. Individual concurrent recoveries ranged from 98–108 percent at 0.01–2.0 mg/kg. Residues in the control samples were <LOQ (n=10). Table 5

The results for replicate field samples and their average are shown in Table 5. Average benzovindiflupyr concentrations ranged between 0.22–0.87 mg/kg. Highest benzovindiflupyr concentration in an individual sample was 1.1 mg/kg benzovindiflupyr (trial 159).

Remarks by the reviewer:

- The United States label refers to lowbush blueberries only.
- Sample sizes of blueberries in study VV-619742 (0.5–0.9 kg) are lower than recommended in the FAO manual (at least 1 kg) for representative samples.
- The demonstrated storage stability of 24 months at -18 °C in high acid commodity groups (oranges) by JMPR 2014 covers the storage period of 227 days for blueberries in study VV-619742. However, as the samples in study VV-19742 were stored frozen at a higher temperature, i.e., -10 °C (one trial at -5.9 °C), it is not clear whether degradation of residues occurred.
- The modification of HPLC-MS/MS method GRM042.03A is reduced validated for the determination of benzovindiflupyr in blueberries in the range 0.01–2.0 mg/kg. The benzovindiflupyr concentrations reported in study VV-619742 are covered by the validation of this analytical method.
- SYN 546039 was not analysed for in the blueberry samples.

Table 5 Residues in blueberries from field trials in the United States

BLUEBERRIES Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Conc in kg ai/hL	Method; Last Treatment date & GS	DAT	Parent (mg/kg)	[Reference], trial
Low bush blueberries								
Upper Rawdon, NS, Canada, 2016, (Lowbush: Wild Clones)	EC [a]	2 (10)	0.075 0.076	0.030 0.030	foliar broadcast; 04 Aug; fruiting 95 percent blue	1	0.64, 0.67; (0.65)	[Byeongseok, 2019, VV-619742], trial AAFC16-039R-153]
Dean, NS, Canada, 2016, (Lowbush: Wild Clones)	EC [a]	2 (10)	0.074 0.074	0.030 0.030	foliar broadcast; 18 Aug; fruiting; 95 percent blue	1	0.68, 0.70; (0.69) [b]	[Byeongseok, 2019, VV-619742] , trial AAFC16-039R 154]
Milford Field, NS, Canada, 2016, (Lowbush: Wild Clones)	EC [a]	2 (10)	0.075 0.075	0.030 0.030	foliar broadcast; 25 Jul; fruiting; 85 percent blue	1	0.44, 0.51; (0.48)	[Byeongseok, 2019, VV-619742], trial AAFC16-039R-155]
Caledonia, NS, Canada, 2016, (Lowbush: Wild Clones)	EC [a]	2 (10)	0.075 0.074	0.030 0.030	foliar broadcast; 25 Jul; fruiting; 90 percent blue	1	0.51, 0.51; (0.51)	[Byeongseok, 2019, VV-619742], trial AAFC16-039R-156]
Mt. Thom,	EC	2	0.076	0.028	foliar broadcast;	1	0.75,	[Byeongseok, 2019,

BLUEBERRIES Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Conc in kg ai/hL	Method; Last Treatment date & GS	DAT	Parent (mg/kg)	[Reference], trial
NS, Canada, 2016, (Lowbush: Wild clones)	[a]	(10)	0.077	0.028	15 Aug; BBCH 82-89 mixed		0.98; (0.87)	VV619742], trial AAFC16-039R-288]
High bush blueberries								
Jordan Station, ON, Canada, 2016, (Highbush: Bluecrop)	EC [a]	2 (10)	0.077 0.079	0.030 0.030	foliar directed; 28 Jul; coloured fruit (mature)	1	0.67, 1.1; (0.86)	[Byeongseok, 2019, VV-619742], trial AAFC16-039R-159]
Langley, BC, Canada, 2016, (Highbush: Liberty)	EC [a]	2 (9)	0.078 0.078	0.027 0.027	foliar directed; 04 Aug; 90 percent mature fruit	1	0.52, 0.56; (0.54)	[Byeongseok, 2019, VV-619742], trial AAFC16-039R-160]
L'Acadie, QC, Canada, 2016, (Highbush: Patriot)	EC [a]	2 (10)	0.23 0.23	0.030 0.030	foliar directed; 21 Jul; mature fruit	1 5 7 11 14	0.34, 0.38; (0.36) 0.28, 0.33; (0.30) 0.26, 0.28; (0.27) 0.19, 0.25; (0.22) 0.23, 0.29; (0.26)	[Byeongseok, 2019, VV-619742], trial AAFC16-039R-157]
L'Acadie, QC, Canada, 2016, (Highbush: Norland)	EC [a]	2 (10)	0.23 0.23	0.030 0.030	foliar directed; 21 Jul; mature fruit	1	0.48, 0.55; (0.51)	[Byeongseok, 2019, VV-619742], trial AAFC16-039R-158]

Notes:

[a] 0.2 percent v/v Agral 90 non-ionic surfactant (NIS) was added to the tank mix.

[b] First rain started within 3 hrs after the second application (0.2 mm rain) in trial 154.

Subgroup of Root Vegetables (016A)

Ginseng – dried ginseng

The Meeting received four supervised residue trial data on ginseng conducted outdoors in the United States in 2016 [Lennon, 2019, VV-547840]. Three to five year old ginseng plants under a shade structure were sprayed four times with an EC or SC formulation in the range between 0.073–0.080 kg ai/ha for each application, using 626–682 L/ha water. Non-ionic surfactants were added to the tank mix. The applications were by CO_2 backpack sprayer. No rain occurred within 24 hours after each of the applications, except for trial MI182.

Ginseng plants were grown in different soil types to allow production of different shapes of ginseng roots: long thin roots are expected from sandy soils and shorter roots with more branching are expected from heavier soils.

Sampling: Fresh ginseng roots were collected from 12 impartially selected areas of each plot and avoiding 15 cm inches from bed sides and 1.8 m from bed ends. Roots were collected by using potato forks to loosen the soil around the roots after which they were handpicked. Two replicate samples were collected from each plot. Each sample weighed 4.1–4.5 kg (9–10 lbs).

Roots were hand washed by softly agitating the roots until the majority of the dirt was removed. Roots were spread onto drying racks in a commercial dryer. Fresh ginseng roots were dried for 6–8 days to an estimated 70 percent to 90 percent dry matter content using a commercial protocol. Dried ginseng root samples weighed at least 0.91 kg (2 lbs), except for trials MI214 and MI215, where samples weighed 0.77–0.87 kg (1.71–1.93 lbs).

The commercial protocol consisted of forced air-drying in a commercial tray dryer at 38 °C (32–38 °C) at a relative humidity of 45 percent (30–45 percent) and an air-flow rate of 0.7 ft/s [Answers to Questions, July 2021] Typical drying times under these conditions are 8–14 days depending on the root diameter and tray loading rate. Actual dry matter content of the individual ginseng root samples was not measured nor recorded [Answers to Questions, July 2021].

Storage: Dried ginseng roots were frozen within 1 hr after the drying was complete and were stored for 399–460 days (15 months) at -18 °C or lower.

Analysis: Benzovindiflupyr and metabolite SYN 546039 (including its conjugates) were determined in the dried ginseng roots using a modification of HPLC-MS/MS method GRM042.03A with an LOQ of 0.01 mg/kg for each analyte. Mean concurrent recoveries in dried ginseng roots ranged from 71–71 percent at 0.01–1.0 mg/kg for benzovindiflupyr and 69–112 percent at 0.01–0.1 mg/kg for SYN 546039. Residues in the control samples were < LOQ (n=4) for each analyte.

The results for replicate field samples and their average are shown in Table 6. Average benzovindiflupyr concentrations in dried ginseng roots ranged between 0.030–0.14 mg/kg. Highest residue in an individual sample was 0.16 mg/kg benzovindiflupyr (trial 16-MI215). SYN 546039 (including conjugates) residues were < 0.01 mg/kg in each individual sample.

Remarks by the reviewer:

- Sample sizes of fresh ginseng roots in study VV-547840 (12 roots; 4.1–4.5 kg) are compliant with the recommendations in the FAO manual (12 roots; at least 2 kg) for representative samples.
- The demonstrated storage stability of 24 months at -18 °C in high starch commodity groups (wheat grain, potato) and dried commodities (wheat straw, dried fruits) by JMPR 2014 covers the storage period of 399–460 days (15 months) for dried ginseng roots in study VV-547840.
- The modification of HPLC-MS/MS method GRM042.03A is reduced validated for the determination of benzovindiflupyr in dried ginseng roots in the range 0.01–1.0 mg/kg and SYN546039 including conjugates in the range 0.01–0.1 mg/kg. The benzovindiflupyr and SYN546039 (including conjugates) concentrations in study VV-547840 are covered by the validation of this analytical method.
- According to the Codex Classification, ginseng should comply with Codex Standard 295R-2009. This regional standard has been replaced by Codex Standard 321-2015. Codex Standard 321-2015 stipulates that dried ginseng roots should contain no more moisture than 14.0 percent (i.e. have a dry matter content of at least 86.0 percent). The actual dry matter content of the

individual dried ginseng root samples from the supervised field trials were not reported, but were estimated at 70–90 percent in the study report.

Table 6 Residues in dried ginseng roots from field trials in the United States

DRIED GINSENG ROOTS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date & GS	Soil type	DAT + DT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial
Hatley, WI, United States, 2016, (American)	EC [a]	4 (15, 13, 13)	0.076 0.077 0.077 0.077	foliar broadcast; 30 Aug; fruiting	loam	15+6	0.095, 0.094; (0.094) [b]	<0.01 <0.01 (<0.01)	[Lennon, 2019, VV-547840], trial 11760.16-MI182]
Athens, WI, United States, 2016, (American)	EC [a]	4 (15, 14, 13)	0.080 0.077 0.078 0.076	foliar broadcast; 19 Jul; fruiting	silt loam	15+7	0.034, 0.033; (0.034)	<0.01 <0.01 (<0.01)	[Lennon, 2019, VV-547840], trial 11760.16-MI214
Wausau, WI, United States, 2016, (American)	EC [a]	4 (15, 13, 13)	0.076 0.080 0.073 0.076	foliar broadcast; 19 Jul; fruiting	loam	15+7	0.13, 0.16; (0.14)	<0.01 <0.01 (<0.01)	[Lennon, 2019, VV-547840],, trial 11760.16-MI215
Mosinee, WI, United States, 2016, (American)	EC [a]	4 (15, 13, 13)	0.074 0.075 0.075 0.076	foliar broadcast; 19 Jul; fruiting	sand loam	0+8 2+6 8+7 15+7 21+8.	0.049, 0.053; (0.051) 0.046, 0.048; (0.047) 0.033, 0.027; (0.030) 0.056, 0.056; (0.056) 0.068, 0.068; (0.068)	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	[Lennon, 2019, VV-547840], trial 11760.16-MI216]

Notes:

DAT + DT = harvest at indicated days after treatment followed by drying time (in days) after which dried ginseng roots were stored frozen.

[a] Scanner non-ionic surfactants (NIS) was added to the tank mix at an unstated concentration.

[b] First rain started 1 hr after the second application (0.13 inches of rain) for trial MI182.

Sugar beet roots

The Meeting received 24 supervised residue trial data on sugar beets conducted outdoors in the United States in 2017 and 2018 and in Canada in 2017 [Dorsey, 2019, VV-547512; Shepard, 2019, VV-547573]. Sugar beet plants were treated with two applications of a WG or EC formulation in the range between 0.072–0.083 kg ai/ha for each application, using 93–410 L/ha water. The first application was performed as an in-furrow or banded soil application at sowing/planting (BBCH 00). The second application was

performed at BBCH 30–32 as a banded foliar application, except United States trials TK0296310 -03 and -05 where a broadcast foliar application was performed and Canadian trials TK03034334-03 and -04 where the second application was at BBCH 19–32 and BBCH 12–32, respectively. The foliar application was made in conjunction with either a non-ionic surfactant (NIS) or crop oil concentrate (COC) tank mix adjuvant. Two United States trials (TK0296310 -01 and -02) included an additional plot treated at an exaggerated rate to obtain samples for processing. The applications were made by handheld, backpack, tractor- or ATV-mounted sprayers (ATV = all-terrain vehicle). Information on rain within 24 hrs after each application was not stated in the report.

Sampling: From the United States trials at least 12 sugar beet plants were collected at normal commercial harvest (BBCH 47–50, i.e. when beets have reached 70–100 percent of their normal size). Sugar beet plants in trial TK0296310-07 were harvested at BBCH 39 (when leaves cover 90 percent of the soil, and roots are small). Sugar beet plants were separated into tops and roots. Two replicate samples were collected from each plot. Sample weights were not stated in the report.

The sample weights for the 12 roots from the immature sugar beet plants from trial TK0296310-07 were 5.6 kg (13.0 lbs) for each of the replicate samples [Answers to questions, July 2021].

Sugar beet plants from the Canadian trials were collected at maturity (BBCH 49); sugar beet plants in trial TK03043334-03 (at DAT 69) were harvested at BBCH 39–49, which is a broad range of beets having reached 0 to 100 percent of their normal size). Two replicate samples were collected from each plot. The sample weights and number of sampled sugar beet plants were not stated in the report.

In the Canadian trials 12 sugar beet plants were harvested for each replicate sample. The small roots from the immature sugar beet plants from trial TK03043334-03 weighted 5.6–9.4 kg for each of the replicate samples at each pre-harvest interval [Answers to questions, July 2021].

Storage: Samples were frozen and stored for 2.0–14.2 months at -10 °C or lower.

Analysis: Benzovindiflupyr and metabolite SYN 546039 (including its conjugates) were determined in the sugar beet roots using a modification of HPLC-MS/MS method GRM042.03A with an LOQ of 0.01 mg/kg for each analyte. Individual concurrent recoveries in sugar beets roots ranged from 79–109 percent at 0.01–1.0 mg/kg for benzovindiflupyr and 76–116 percent at 0.01–0.1 mg/kg for SYN 546039. Residues in the control samples were < LOQ (n=22) for each analyte.

The results for replicate field samples and their average are shown in Table 7. Average benzovindiflupyr concentrations in sugar beet roots ranged between <0.01–0.073 mg/kg. Highest residue in an individual sample was 0.076 mg/kg benzovindiflupyr (trial TK0296310–05). SYN 546039 (including conjugates) residues were < 0.01 mg/kg in each individual sugar beet root sample.

Remarks by the reviewer:

- Two of the Canadian trials, TK-0304334-04 and -06 took place at the same location. As the planting and application differed by 30 days, these trials are considered to be independent.
- Sample sizes in the United States study VV-547512 and the Canadian study VV-547573 are compliant with the recommended sample size in the FAO manual (at least 12 plants).
- In trial TK0296310-07, the sugar beet plants were harvested at BBCH 39 and in trial TK03043334-03, the sugar beet plants were harvested at BBCH 39–49. Although BBCH 39 reflects immature plants, sample weights for these 12 roots were at least 5.6 kg (460 g/root) and can thus be considered representative for MRL derivation.

- The demonstrated storage stability of 24 months at -18 °C in high starch commodity groups (wheat grain, potato) by JMPR 2014 covers the storage period of 2.0–14.2 months for sugar beet roots in study VV-547512.
- The modification of HPLC-MS/MS method GRM042.03A is reduced validated for the determination of benzovindiflupyr and SYN 546039 (including its conjugates) in sugar beet roots in the range 0.01–1.0 mg/kg. The benzovindiflupyr and SYN 546039 (including conjugates) concentrations in studies VV-547512 and VV-547573 are covered by the validation of this analytical method.

Table 7 Residues in sugar beet roots from field trials in Canada and the United States after combined soil + foliar treatment

SUGAR BEET ROOTS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date & GS	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial Soil type
Gardner, ND, United States, 2017 (SVRR 336)	WG [a]	2 (40)	0.077 0.080	in furrow soil + banded foliar; 3 Jul; BBCH 30-31	81	0.011, <0.01; (0.010)	<0.01 <0.01 (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–01 Soil: clay
	WG [a]	2 (40)	0.38 0.39	idem	81	0.014, 0.018, 0.033; (0.022)	<0.01 <0.01 <0.01 (<0.01)	[b]
Northwood, ND, United States, 2017 (Hilleshog 4022RR)	WG [a]	2 (32)	0.080 0.078	in furrow soil + banded foliar; 20 Jun; BBCH 31-32	93	<0.01, <0.01; (<0.01)	<0.01 <0.01 (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–02 Soil: loam
	WG [a]	2 (32)	0.40 0.39	idem	93	0.015; 0.020, <0.01; (0.015)	<0.01, <0.01;] <0.01 (<0.01)	[b]
Richland, IA, United States, **2017** (Hilleshog 4302RR)	WG [a]	2 (32)	0.078 0.079	in furrow soil + broadcast foliar; 5 Jul; BBCH 31	90 95 100 105 110	0.016, 0.011 (0.014) 0.011, <0.01; (0.010) 0.010, 0.012; (0.011) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–03 Soil: silt clay loam
Fitchburg, WI, United States, 2017 (SVRR 336)	WG [a]	2 (29)	0.080 0.077	banded soil + banded foliar; 29 Jun; BBCH 30-31	90 96	<0.01, <0.01; (<0.01) <0.01, <0.01;	<0.01, <0.01; (<0.01) <0.01, <0.01;	[Dorsey, 2019, VV-547512], trial TK0296310–04 Soil: silt loam

SUGAR BEET ROOTS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date & GS	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial Soil type
					99 106 109	(<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	(<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	
Aurora, SD, United States, **2017** (Anaconda RR)	WG [a]	2 (42)	0.081 0.080	in furrow soil + broadcast foliar; 17 Aug; BBCH 31	48	0.076, 0.070; (0.073)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–05 Soil: loam
Carrington, ND, United States, 2017 (Hilleshog 4022RR)	WG [a]	2 (36)	0.079 0.079	in furrow soil + banded foliar; 5 Jul; BBCH 31	79	0.012, 0.013; (0.012)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–06 Soil: sand loam
Larned, KS, United States, 2017 (BTS 8512 PRO 200)	WG [a]	2 (34)	0.079 0.079	in furrow soil + banded foliar; 5 Jul; BBCH 31	68	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–07 Soil: loam
Lewistown, UT, United States, 2017 (BTS 60RR27 Large)	WG [a]	2 (33)	0.078 0.083	in furrow soil + banded foliar; 6 Jul; BBCH 31-32	83	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–08 Soil: sand loam
Paso Robles, CA, United States, 2017 (70RR99)	WG [a]	2 (39)	0.078 0.078	in furrow soil + banded foliar; 23 Jul; BBCH 31	74	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–09 Soil: sand loam
Fresno, CA, United States, 2017 (Newbie)	WG [a]	2 (25)	0.076 0.078	banded soil + banded foliar; 16 Jun; BBCH 31	122	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–10 Soil: loam sand
Ephrata, WA, United States, 2017 (150370-222-HMR-1)	WG [a]	2 (31)	0.076 0.079	in furrow soil + banded foliar; 19 Jun; BBCH 31	92	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–11 Soil: sand loam
Rupert,	WG	2	0.082	in furrow soil	119	<0.01,	<0.01,	[Dorsey, 2019, VV-

SUGAR BEET ROOTS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date & GS	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial Soil type
ID, United States, 2017 (BTS 27RR20)	[a]	(31)	0.079	+ banded foliar; 19 Jun; BBCH 31		<0.01; (<0.01)	<0.01; (<0.01)	547512], trial TK0296310–12 Soil: loam sand
Richland, IA, United States, **2018** (Hilleshog 4302RR)	WG [a]	2 (30)	0.078 0.079	in furrow soil + banded foliar; 13 Jul; BBCH 31	75	<0.01, 0.013; (0.012)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–13 Soil: silt clay loam
Aurora, SD, United States, **2018** (Anaconda RR)	WG [a]	2 (38)	0.078 0.081	in furrow soil + banded foliar; 23 Jul; BBCH 31-32	65	0.059, 0.048; (0.054)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–14 Soil: loam
St. Marc-sur-Richelieu, QC, Canada, 2017 (9221RR)	EC [a]	2 (40)	0.076 0.079	banded soil + banded foliar; 14 Jul; BBCH 31	42 47 53 59 63	0.016, 0.017; (0.016) 0.010, 0.012; (0.011) 0.020, 0.033; (0.026) 0.016, 0.015; (0.016) 0.012, 0.012; (0.012)	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-01 Soil: loam
Elm Creek, MB, Canada, 2017 (SV36152RR)	EC [a]	2 (35)	0.076 0.077	banded soil + banded foliar; 7 July; BBCH 31	82	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-02 Soil: sand
Taber, AB, Canada, 2017 (SV36152RR)	EC [a]	2 (40)	0.078 0.076	in furrow soil + banded foliar; 6 Jul; BBCH 19-32	69 74 78 84 91	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-03 Soil: sand loam

SUGAR BEET ROOTS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date & GS	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial Soil type
Taber, AB, Canada, 2017 (9221RR)	EC [a]	2 (37)	0.077 0.073	banded soil + banded foliar; 2 Aug; BBCH 12-32	72	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-04 Soil: sand loam
Bow Island, AB, Canada, 2017 (9221RR)	EC [a]	2 (37)	0.078 0.076	in furrow soil + banded foliar; 2 Aug; BBCH 31	77	0.014, 0.015; (0.014)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-05 Soil: loam
Bow Island, AB, Canada, 2017 (SV36152 RR)	EC [a]	2 (41)	0.077 0.077	in furrow soil + directed foliar; 7 Jul; BBCH 31	68	<0.01; <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573] trial TK0304334-06 Soil: loam
Broderick, SK, Canada, 2017 (9221RR)	EC [a]	2 (41)	0.074 0.074	in furrow soil + banded foliar; 18 Jul; BBCH 31	55	<0.01; <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-07 Soil: silt loam
Kipp, AB, Canada, 2017 (SV36152RR)	EC [a]	2 (46)	0.078 0.072	banded soil + directed foliar; 12 Jul; BBCH 31	62	<0.01; <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-08 Soil: sand clay loam

Notes:

[a] For the foliar applications crop oil concentrates (COC) or non-ionic surfactants (NIS) were added to the tank mix. **COC**: Superb HC 0.5 percent-1.0 percent v/v (United States trial 01, 06, 08); Prime oil 0.5-1.0 percent v/v (United States trial 03, 05, 13, 14), Chem Spray 1 percent v/v (United States trial 04), Herbimax 0.6 percent v/v (United States trial 09), **NIS**: Preference 0.25 percent v/v (United States, trial 02, 12), Induce 0.12 percent v/v (United States trial 07, Kinetic 0.3 percent v/v (United States trial 10), R-11 0.13 percent v/v (United States trial 11), Agral 90 0.2 percent v/v (Canada trial 01, 02, 03, 04, 05, 06, 07, 08).

[b] Samples used in the sugar beet root processing study.

Subgroup of Maize Cereals (020E)

Maize

Data from supervised residue trials on maize in the United States were summarized and evaluated by the JMPR 2016. The Meeting received an additional eight supervised residue trial data on maize conducted outdoors in the United States in 2019 [Banman, 2020, VV-872218]. Plots with maize plants were treated with two broadcast foliar sprays with an EC formulation in the range between 0.050–0.053 kg ai/ha for each application using 121–224 L/ha water. Non-ionic surfactants or crop oil concentrates were added to the tank mix. The application was by backpack or tractor-mounted sprayer. Information on rain within 24 hrs after each application was not stated in the report.

Sampling: Whole maize plants were harvested at BBCH 89 and separated into maize grains and remaining plants. Maize grains were collected from at least 12 areas of the plot. Two replicate samples were collected from each plot. Each sample weighed at least 1 kg.

Storage: Samples were frozen and stored for 212–344 days (9.4–11.3 months) at -10 °C or lower.

Analysis: Benzovindiflupyr and metabolite SYN 546039 (including its conjugates) were determined in the maize grains using HPLC-MS/MS method GRM042.03A with an LOQ of 0.01 mg/kg for each analyte. Individual concurrent recoveries in maize grains ranged from 89–120 percent at 0.01–0.1 mg/kg for benzovindiflupyr and 82–98 percent at 0.01–0.1 mg/kg for SYN 546039. Residues in the control samples were < LOQ (n=10) for each analyte.

The results for replicate field samples and their average are shown in Table 8. The average benzovindiflupyr concentrations in maize grains ranged between <0.01–0.016 mg/kg. Highest residue in an individual sample was 0.019 mg/kg benzovindiflupyr (TK0462705-03 – P3). SYN 546039 (including conjugates) residues were < 0.01 mg/kg in each individual maize grain sample.

Remarks by the reviewer:

- Sample sizes of maize grains in study VV-872218 (1 kg) are compliant with the recommendation in the FAO manual (at least 1 kg) for representative samples.
- The demonstrated storage stability of 24 months at -18 °C in high starch commodity groups (wheat grain, potato) by JMPR 2014 covers the storage period of 212–344 days (9.4–11.3 months) for maize grains in study VV-872218.
- The modification of HPLC-MS/MS method GRM042.03A is reduced validated for the determination of benzovindiflupyr and SYN546039 (including conjugates) in maize grains in the range 0.01–0.1 mg/kg. The benzovindiflupyr and SYN 546039 (including conjugates) concentrations in study VV-872218 are covered by the validation of this analytical method.

Table 8 Residues in maize grains from field trials in the United States

MAIZE GRAINS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date; GS at last treatment;	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial
Stewardson, IL, United States, 2019, (Pioneer 1197AM1)	EC [a]	2 (14)	0.051 0.053	Broadcast foliar; 4 Oct; BBCH 87-89	6 10 13 17 20	<0.01, <0.01; (<u>0.01</u>) <0.01 <0.01 <0.01 <0.01	<0.01, <0.01; (<0.01) <0.01 <0.01 <0.01 <0.01	[Banman, 2020, VV-872218]; trial TK0462705-01 – P3
Atlantic, IA, United States, 2019, (DKC 64-35 RIB)	EC [a]	2 (14)	0.053 0.052	Broadcast foliar; 8 Oct; BBCH 89	7 10 15 19 22	<0.01, <0.01; (<u>0.01</u>) <0.01 <0.01 <0.01 <0.01	<0.01, <0.01; (<0.01) <0.01 <0.01 <0.01 <0.01	[Banman, 2020, VV-872218]; trial TK0462705-02 –P3
Northwood, ND, United States, 2019,	EC [a]	2 (13)	0.052 0.052	Broadcast foliar; 16 Oct; BBCH 87-	7 11	0.014, 0.019; (<u>0.016</u>) 0.010	<0.01, <0.01; (<0.01) <0.01	[Banman, 2020, VV-872218]; trial TK0462705-03

MAIZE GRAINS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date; GS at last treatment;	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial
(DKC 34-82 RIB)				89;	15 18 21	0.010 0.010 <0.01	<0.01 <0.01 <0.01	– P3
Fitchburg, WI, United States, 2019, (Pioneer P9188R)	EC [a]	2 (15)	0.052 0.051	Broadcast foliar; 23 Oct BBCH 87	7 9 13 16 21	<0.01, <0.01; <u>(<0.01)</u> <0.01 <0.01 <0.01 <0.01	<0.01, <0.01; (<0.01) <0.01 <0.01 <0.01 <0.01	[Banman, 2020, VV-872218]; trial TK0462705-04R -P3
Richland, IA, United States, 2019, (210-79DGVT2P RIB)	EC [a]	2 (13)	0.052 0.052	Broadcast foliar; 30 Sept BBCH 87	7 9 14 17 21	<0.01, <0.01; <u>(<0.01)</u> <0.01 <0.01 <0.01 <0.01	<0.01, <0.01; (<0.01) <0.01 <0.01 <0.01 <0.01	[Banman, 2020, VV-872218]; trial TK0462705-05 – P3
Manilla, IN, United States, 2019, (Pioneer 1197)	EC [a]	2 (14)	0.052 0.052	Broadcast foliar; 5 Nov BBCH 89	7	<0.01, <0.01; <u>(<0.01)</u>	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; trial TK0462705-06R -P3
York, NE, United States, 2019, (P1138AM)	EC [a]	2 (13)	0.052 0.051	Broadcast foliar; 24 Sept BBCH 89	9	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; trial TK0462705-07 - P3
Stafford, KS, United States, 2019, (P0805 AM)	EC [a]	2 (14)	0.052 0.050	Broadcast foliar; 18 Sept BBCH 89	6	<0.01, <0.01; <u>(<0.01)</u>	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; trial TK0462705-08 - P3

Notes:

[a] Non-ionic surfactants (NIS) or crop oil concentrates (COC) were added to the tank mix**: NIS:** Preference 0.25 percent v/v [Trial 01, 03, 05), Chem Surf 80 NIS 0.25 percent v/v (Trial 02), NIS 0.25 percent v/v (trial 04), Cornbelt Premier 90 0.032 percent v/v (trial 07), Induce 0.25 percent v/v (trial 08). Or **COC**: SuperB 0.5 percent v/v (trial 06),

Cereal grains and grasses feed products – subgroup of products with high water content (050B)

Maize forage

Data from supervised residue trials on sweet corn forage and maize forage from the United States were summarized and evaluated by the JMPR 2016. The Meeting received an additional eight supervised residue trial data on maize forage conducted outdoors in the United States in 2019 [Banman, 2020, VV-872218]. As the whole plants were harvested at BBCH 85–89, they are considered not representative for green forage plants, as the plants already lost moisture and thus may have concentrated their residues. The trials were listed together with maize stover.

Cereal grains and grasses feed products – subgroup of products with low water content (051B)

Maize stover

Data from supervised residue trials on sweet corn stover and maize stover in the United States were summarized and evaluated by the JMPR 2016. The Meeting received an additional eight supervised residue trial data on maize whole plants and maize stover conducted outdoors in the United States in 2019 [Banman, 2020, VV-872218]. Plots with maize plants were treated with two broadcast foliar sprays with an EC formulation in the range between 0.050–0.053 kg ai/ha for each application using 121–224 L/ha water. Non-ionic surfactants or crop oil concentrates were added to the tank mix. The application was by backpack or tractor-mounted sprayer. Information on rain within 24 hours after each application was not stated in the report.

Sampling: Plot P2 at each location (trials submitted as maize forage whole plants but considered stover) was treated at growth stage BBCH 75–85 and whole maize plants were harvested at BBCH 85–87. Plot P3 at each location (trials submitted as maize stover) was treated at growth stage BBCH 87–89 and whole maize plants were harvested at BBCH 89 and separated into maize grains and remaining plants (stover). Maize whole plants and maize stover were collected from at least 12 plants and from 12 areas of the plot. Two replicate samples were collected from each plot. Each sample weighed at least 1 kg.

Storage: Samples were frozen and stored for 212–344 days (9.4–11.3 months) at -10 °C or lower.

Analysis: Benzovindiflupyr and metabolite SYN 546039 (including its conjugates) were determined in the maize whole plants and maize stover using HPLC-MS/MS method GRM042.03A with an LOQ of 0.01 mg/kg for each analyte. Mean concurrent recoveries in maize whole plants and maize stover ranged from 94–103 percent at 0.01–3.0 mg/kg for benzovindiflupyr and 74–82 percent at 0.01–0.1 mg/kg for SYN 546039. Residues in the control samples were < LOQ (n=10) for each analyte.

The results for replicate field samples and their average are shown in Table 9. The average benzovindiflupyr concentrations in maize whole plants and maize stover ranged between 0.10–2.9 mg/kg. Highest residue in an individual sample was 2.9 mg/kg benzovindiflupyr. SYN 546039 (including conjugates) residues were < 0.01 mg/kg in each individual maize whole plant and maize stover sample.

Remarks by the reviewer:

- Trials submitted as maize forage (Plot P2 at each location) were treated at growth stage BBCH 75–85 and whole maize plants were harvested at BBCH 85–87. As the whole plants were harvested at BBCH 85–89, they are considered not representative for green forage plants, as the plants already lost moisture and thus may have concentrated their residues. The trials were listed together with maize stover.
- Sample sizes of whole maize plants and maize stover in study VV-872218 (12 plants) are compliant with the recommendation in the FAO manual (at least 12 plants) for representative samples.
- The demonstrated storage stability of 24 months -18 °C in dried commodity groups (wheat straw) by JMPR 2014 covers the storage period of 212–344 days (9.4–11.3 months) for maize whole plants and maize stover in study VV-872218.
- The modification of HPLC-MS/MS method GRM042.03A is reduced validated for the determination of benzovindiflupyr in maize whole plants and maize stover in the range 0.01–3.0 mg/kg and SYN 546039 (including conjugates) in the range 0.01–0.1 mg/kg. The benzovindiflupyr and SYN 546039 (including conjugates) concentrations in study VV-872218 are covered by the validation of this analytical method.

Table 9 Residues in maize stover (whole plant and remaining plant) from field trials in the United States

MAIZE STOVER Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date; GS at last treatment;	GS at harvest	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial
Stewardson, IL, United States, 2019, (Pioneer 1197AM1)	EC [a]	2 (14)	0.051 0.052	Broadcast foliar; 29 Aug; BBCH 83-85	85-87	7	0.14, 0.19; (0.17)	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; TK0462705-01 – P2 (whole plant)
	EC [a]	2 (14)	0.051 0.053	Broadcast foliar; 4 Oct; BBCH 87-89	89	6	1.1, 1.3; (1.2)	<0.01, <0.01; (<0.01)	TK0462705-01 – P3 (remaining plant)
Atlantic, IA, United States, 2019, (DKC 64-35 RIB)	EC [a]	2 (15)	0.052 0.051	Broadcast foliar; 23 Aug BBCH 81-83	85-87	7	0.26, 0.31; (0.28)	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; trial TK0462705-02 – P2 (whole plant)
	EC [a]	2 (14)	0.053 0.052	Broadcast foliar; 8 Oct; BBCH 89	89	7	0.68, 1.0; (0.86)	<0.01, <0.01; (<0.01)	[TK0462705-02 –P3 (remaining plant)
Northwood, ND, United States, 2019, (DKC 34-82 RIB)	EC [a]	2 (13)	0.051 0.051	Broadcast foliar; 10 Sept BBCH 83-85	85	8	0.28 0.25; (0.26)	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; trial TK0462705-03 – P2 (whole plant)
	EC [a]	2 (13)	0.052 0.052	Broadcast foliar; 16 Oct; BBCH 87-89;	89	7	1.4, 1.3; (1.3)	<0.01, <0.01; (<0.01)	TK0462705-03 – P3 (remaining plant)
Fitchburg, WI, United States, 2019, (Pioneer P9188R)	EC [a]	2 (13)	0.052 0.052	Broadcast foliar; 5 Sept; BBCH 83-85	85-87	8	0.20, 0.19; (0.20)	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218; TK0462705-04R – P2 (whole plant)
	EC [a]	2 (15)	0.052 0.051	Broadcast foliar; 23 Oct BBCH 87	89	7	2.8, 2.9; (2.9)	<0.01, <0.01; (<0.01)	TK0462705-04R -P3 (remaining plant)
Richland, IA, United States, 2019, (210-79DGVT2P RIB)	EC [a]	2 (14)	0.052 0.051	Broadcast foliar; 9 Aug BBCH 83	85	7	0.16, 0.17; (0.16)	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; TK0462705-05 – P2 (whole plant)
	EC [a]	2 (13)	0.052 0.052	Broadcast foliar; 30 Sept BBCH 87	89	7	1.3, 1.9; (1.6)	<0.01, <0.01; (<0.01)	TK0462705-05 – P3 (remaining plant)
Manilla,	EC	2	0.052	Broadcast	85	8	1.1,	<0.01,	[Banman, 2020,

MAIZE STOVER Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date; GS at last treatment;	GS at harvest	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial
IN, United States, 2019, (Pioneer 1197)	[a]	(13)	0.052	foliar; 10 Sept BBCH 75			1.1; (1.1)	<0.01; (<0.01)	VV-872218]; trial TK0462705-06R – P2 (whole plant)
	EC [a]	2 (14)	0.052 0.052	Broadcast foliar; 5 Nov BBCH 89	89	7	1.7, 1.7; (1.7)	<0.01, <0.01; (<0.01)	TK0462705-06R -P3 (remaining plant)
York, NE, United States, 2019, (P1138AM))	EC [a]	2 (15)	0.052 0.052	Broadcast foliar, 16 Aug BBCH 85	87	6	0.18, 0.18; (0.18)	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; TK0462705-07 – P2 (whole plant)
	EC [a]	2 (13)	0.052 0.051	Broadcast foliar; 24 Sept BBCH 89	89	9	0.81, 1.1; (0.95)	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; trial TK0462705-07 -P3 (remaining plant)
Stafford, KS, United States, 2019, (P0805 AM))	EC [a]	2 (14)	0.054 0.050	Broadcast foliar, 1 Aug BBCH 75-79	85-87	8	0.11, 0.10; (0.10)	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; trial TK0462705-08 – P2 (whole plant)
	EC [a]	2 (14)	0.052 0.050	Broadcast foliar; 18 Sept BBCH 89	89	6	2.1, 2.4; (2.3)	<0.01, <0.01; (<0.01)	[Banman, 2020, VV-872218]; trial TK0462705-08 -P3 (remaining plant)

Notes:

[a] Non-ionic surfactants (NIS) or crop oil concentrates (COC) were added to the tank mix**: NIS:** Preference 0.25 percent v/v [Trial 01, 03, 05), Chem Surf 80 NIS 0.25 percent v/v (Trial 02), NIS 0.25 percent v/v (trial 04), Cornbelt Premier 90 0.032 percent v/v (trial 07), Induce 0.25 percent v/v (trial 08). Or **COC**: SuperB 0.5 percent v/v (trial 06),

Miscellaneous feed products – subgroup of products with high water content (052A)

Sugar beet tops

The Meeting received 22 supervised residue trial data on sugar beets conducted outdoors in the United States in 2017 and 2018 and Canada in 2017 and 2018 [Dorsey, 2019, VV-547512; Shepard, 2019, VV-547573]. Sugar beets were treated with two applications of a WG or EC formulation in the range between 0.072–0.083 kg ai/ha for each application, using 93–410 L/ha water. The first application was performed as an in-furrow or banded soil application at sowing/planting (BBCH 00). The second application was

performed at BBCH 30–32 as a banded foliar application, except United States trials TK0296310 -03 and -05 where a broadcast foliar application was performed and Canadian trials TK03034334-03 and -04 where the second application was at BBCH 19–32 and BBCH 12–32, respectively. The foliar application was made in conjunction with either a non-ionic surfactant (NIS) or crop oil concentrate (COC) tank mix adjuvant. The applications were made by handheld, backpack, tractor- or ATV-mounted sprayers (ATV = all-terrain vehicle). Information on rain within 24 hours after each application was not stated in the report.

Sampling: From the United States trials at least 12 sugar beet plants were collected at normal commercial harvest (BBCH 47–50). Sugar beet plants in trial TK0296310-07 were harvested at BBCH 39. Sugar beet plants were separated into tops and roots. Two replicate samples were collected from each plot. Sample weights were not stated in the report.

The sample weights for the 12 tops from the immature sugar beet plants from trial TK0296310-07 were 2.0–2.5 kg (4.5–5.5 lbs) for each of the replicate samples [Answers to questions, July 2021].

Sugar beet plants from the Canadian trials were collected at maturity (BBCH 49); sugar beet plants in trial TK03043334-03 (at DAT 69) were harvested at BBCH 39–49. Sugar beet plants were separated into tops and roots. Two replicate samples were collected from each plot. The sample weights and number of sampled sugar beet plants were not stated in the report.

In the Canadian trials 12 sugar beet plants were harvested for each replicate sample. The small roots from the immature sugar beet plants from trial TK03043334-03 weighted 5.6–9.4 kg for each of the replicate samples at each pre-harvest interval. The tops from the immature sugar beet plants from trial TK03043334-03 weighted 2.5–4.4 kg for each of the replicate samples at each pre-harvest interval [Answers to questions, July 2021].

Storage: Samples were frozen and stored for 2.0–14.2 months at -10 °C or lower.

Analysis: Benzovindiflupyr and metabolite SYN 546039 (including its conjugates) were determined in the sugar beet tops using a modification of HPLC-MS/MS method GRM042.03A with an LOQ of 0.01 mg/kg for each analyte. Individual concurrent recoveries in sugar beets tops ranged from 88–107 percent at 0.01-0.1 mg/kg for benzovindiflupyr and 81–112 percent at 0.01–0.1 mg/kg for SYN 546039. Residues in the control samples were < LOQ (n=22) for each analyte.

The results for replicate field samples and their average are shown in Table 10. The average benzovindiflupyr concentrations in sugar beet tops range between <0.01-0.055 mg/kg. Highest residue in an individual sample was 0.079 mg/kg benzovindiflupyr (TK0296310–14). SYN 546039 (including conjugates) residues were < 0.01 mg/kg in each individual sugar beet top sample, except in:

- trial TK0304334-01 (St. Marc sur Richelieu, QC, Canada) where SYN 546039 was found at a mean level of 0.015 mg/kg at DAT 42 (individual 0.013/0.017 mg/kg), 0.014 mg/kg at DAT 47 (individual 0.012/0.016 mg/kg), <0.01 mg/kg at DAT 53, 0.012 mg/kg at DAT 59 (individual 0.011/0.014 mg/kg) and 0.01 mg/kg at DAT 63 (individual <0.01/0.010 mg/kg);
- Trial TK0304334-03 (Taber, AB, Canada) where SYN 546039 was found at a mean level of 0.013 mg/kg at DAT 69 (individual 0.012/0.014 mg/kg) and < 0.01 mg/kg at DAT 74, 78, 84, 91.

Remarks by the reviewer:

- Two of the Canadian trials, TK-0304334-04 and -06 took place at the same location. As the planting and application differed by 30 days, these trials are considered to be independent;
- Sample sizes in the United States study VV-547512 and the Canadian study VV-547573 are compliant with the recommended sample size in the FAO manual (at least 12 plants);

- In trial TK0296310-07, the sugar beet plants were harvested at BBCH 39 and in trial TK03043334-03, the sugar beet plants were harvested at BBCH 39–49. Although BBCH 39 reflects immature plants, sample weights were at least 5.6 kg for these 12 roots (460 g/root) and at least 2.5 kg for these 12 tops and can thus be considered representative for MRL derivation;
- The demonstrated storage stability of 24 months -18 °C in high water commodity groups (spinach) by JMPR 2014 covers the storage period of 2.0–14.2 months for sugar beet tops in study VV-547512;
- The modification of HPLC-MS/MS method GRM042.03A is reduced validated for the determination of benzovindiflupyr and SYN 546039 (including conjugates) in sugar beet tops in the range 0.01-1.0 mg/kg. The benzovindiflupyr and SYN 546039 (including conjugate) concentrations in studies VV-547512 and VV-547573 are covered by the validation of this analytical method.

Table 10 Residues in sugar beet tops from field trials in Canada and the United States after combined soil + foliar treatment

SUGAR BEET TOPS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date & GS	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial Soil type
Gardner, ND, United States, 2017 (SVRR 336)	WG [a]	2 (40)	0.077 0.080	in furrow soil + banded foliar; 3 Jul; BBCH 30-31	81	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–01 Soil: clay
Northwood, ND, United States, 2017 (Hilleshog 4022RR)	WG [a]	2 (32)	0.080 0.078	in furrow soil + banded foliar; 20 Jun; BBCH 31-32	93	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–02 Soil: Loam
Richland, IA, United States, **2017** (Hilleshog 4302RR)	WG [a]	2 (32)	0.078 0.079	in furrow soil + broadcast foliar; 5 Jul; BBCH 31	90 95 100 105 110	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, 0.011; 0.010 <0.01, <0.01 (<0.01) <0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–03 Soil: silt clay loam
Fitchburg, WI, United States, 2017 (SVRR 336)	WG [a]	2 (29)	0.080 0.077	banded soil + banded foliar; 29 Jun; BBCH 30-31	90 96	<0.01, <0.01; (<0.01) <0.01, <0.01;	<0.01, <0.01; (<0.01) <0.01, <0.01;	[Dorsey, 2019, VV-547512], trial TK0296310–04 Soil: silt loam

SUGAR BEET TOPS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date & GS	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial Soil type
					99 106 109	(<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	(<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	
Aurora, SD, United States, **2017** (Anaconda RR)	WG [a]	2 (42)	0.081 0.080	in furrow soil + broadcast foliar; 17 Aug; BBCH 31	48	0.012, 0.015; (0.014)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–05 Soil: loam
Carrington, ND, United States, 2017 (Hilleshog 4022RR)	WG [a]	2 (36)	0.079 0.079	in furrow soil + banded foliar; 5 Jul; BBCH 31	79	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–06 Soil: sand loam
Larned, KS, United States, 2017 (BTS 8512 PRO 200)	WG [a]	2 (34)	0.079 0.079	in furrow soil + banded foliar; 5 Jul; BBCH 31	68	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–07 Soil: loam
Lewistown, UT, United States, 2017 (BTS 60RR27 Large)	WG [a]	2 (33)	0.078 0.083	in furrow soil + banded foliar; 6 Jul; BBCH 31-32	83	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–08 Soil: sand loam
Paso Robles, CA, United States, 2017 (70RR99)	WG [a]	2 (39)	0.078 0.078	in furrow soil + banded foliar; 23 Jul; BBCH 31	74	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–09 Soil: sand loam
Fresno, CA, United States, 2017 (Newbie)	WG [a]	2 (25)	0.076 0.078	banded soil + banded foliar; 16 Jun; BBCH 31	122	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–10 Soil: loam sand
Ephrata, WA, United States, 2017 (150370-222-HMR-1)	WG [a]	2 (31)	0.076 0.079	in furrow soil + banded foliar; 19 Jun; BBCH 31	92	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–11 Soil: sand loam
Rupert,	WG	2	0.082	in furrow soil	119	<0.01,	<0.01,	[Dorsey, 2019, VV-

SUGAR BEET TOPS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date & GS	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial Soil type
ID, United States, 2017 (BTS 27RR20)	[a]	(31)	0.079	+ banded foliar; 19 Jun; BBCH 31		<0.01; (<0.01)	<0.01; (<0.01)	547512], trial TK0296310–12 Soil: loam sand
Richland, IA, United States, **2018** (Hilleshog 4302RR)	WG [a]	2 (30)	0.078 0.079	in furrow soil + banded foliar; 13 Jul; BBCH 31	75	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–13 Soil: silt clay loam
Aurora, SD, United States, **2018** (Anaconda RR)	WG [a]	2 (38)	0.078 0.081	in furrow soil + banded foliar; 23 Jul; BBCH 31-32	65	0.079, 0.031; (0.055)	<0.01, <0.01; (<0.01)	[Dorsey, 2019, VV-547512], trial TK0296310–14 Soil: loam
St. Marc-sur-Richelieu, QC, Canada, 2017 (9221RR)	EC [a]	2 (40)	0.076 0.079	banded soil + banded foliar; 14 Jul; BBCH 31	42 47 53 59 63	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	0.013, 0.017 (0.015) 0.016, 0.012; (0.014) <0.01, <0.01; (<0.01); 0.014, 0.011 (0.012) <0.01, 0.010; (0.01)	[Shepard, 2019, VV-547573], trial TK0304334-01 Soil: loam
Elm Creek, MB, Canada, 2017 (SV36152RR)	EC [a]	2 (35)	0.076 0.077	banded soil + banded foliar; 7 July; BBCH 31	82	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019, VV-547573], trial TK0304334-02 Soil: sand
Taber, AB, Canada, 2017 (SV36152RR)	EC [a]	2 (40)	0.078 0.076	in furrow soil + banded foliar; 6 Jul; BBCH 19-32	69 74 78 84 91	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	0.014, 0.012; (0.013) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-03 Soil sand loam

SUGAR BEET TOPS Location, Country, Year (Variety)	Form	No (RTI, days)	Rate in kg ai/ha	Method; Last Treatment date & GS	DAT	Parent (mg/kg)	SYN 546039 + conjugates (mg/kg)	[Reference], trial Soil type
Taber, AB, Canada, 2017 (9221RR)	EC [a]	2 (37)	0.077 0.073	banded soil + banded foliar; 2 Aug; BBCH 12-32	72	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-04 Soil: sand loam
Bow Island, AB, Canada, 2017 (9221RR)	EC [a]	2 (37)	0.078 0.076	in furrow soil + banded foliar; 2 Aug; BBCH 31	77	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-05 Soil: loam
Bow Island, AB, Canada, 2017 (SV36152 RR)	EC [a]	2 (41)	0.077 0.077	in furrow soil + directed foliar; 7 Jul; BBCH 31	68	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573] trial TK0304334-06 Soil: loam
Broderick, SK, Canada, 2017 (9221RR)	EC [a]	2 (41)	0.074 0.074	in furrow soil + banded foliar; 18 Jul; BBCH 31	55	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-07 Soil silt loam
Kipp, AB, Canada, 2017 (SV36152RR)	EC [a]	2 (46)	0.078 0.072	banded soil + directed foliar; 12 Jul; BBCH 31	62	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	[Shepard, 2019 VV-547573], trial TK0304334-08 Soil: sand clay loam

Notes:

[a] For the foliar applications crop oil concentrates (COC) or non-ionic surfactants (NIS) were added to the tank mix. **COC**: Superb HC 0.5 percent-1.0 percent v/v (United States trial 01, 06, 08); Prime oil 0.5-1.0 percent v/v (United States trial 03, 05, 13, 14), Chem Spray 1 percent v/v (United States trial 04), Herbimax 0.6 percent v/v (United States trial 09), **NIS**: Preference 0.25 percent v/v (United States, trial 02, 12), Induce 0.12 percent v/v (United States trial 07, Kinetic 0.3 percent v/v (United States trial 10), R-11 0.13 percent v/v (United States trial 11), Agral 90 0.2 percent v/v (Canada trial 01, 02, 03, 04, 05, 06, 07, 08).

[b] Samples used in the sugar beet root processing study.

Additional metabolite information

The Meeting received new toxicological information on metabolites and the relevance of these metabolites for the residue definition was therefore re-assessed. Field samples from all trials were analysed for SYN 546039 (including its conjugates) and field trials from pulses and oilseeds were additionally analysed for SYN545720, using validated analytical methods, summarized and assessed by the 2014 and 2016 JMPR. As the 2016 JMPR did not summarize the residue information on the metabolites, the JMPR 2016 supervised trials data were re-summarized. Also the JMPR 2014 and 2019 trials were reproduced below. The results of the JMPR 2022 trials were included in the conclusion below.

Presence of SYN 546039 (hydroxy-benzovindiflupyr) including its conjugates:

- Pome fruit (apple and pear, Table 11, Table 12). SYN 546039 was not detected (< 0.01 mg/kg) in any of the apple and pear trials at 30 and 60 days after the last application (DALA). In two apple trials conducted at 5× exaggerated rates. SYN 546039 ranged from <0.01–0.012 mg/kg at 30 DALA;
- Berries and other small fruit (blueberries, grapes, Table 5, Table 13). SYN 546039 was found in most grape trials. Levels ranging from <0.01–0.057 mg/kg were found at 21 DALA and < 0.01–0.22 mg/kg at 45 DALA. In two grape trials conducted at 5× exaggerated rates SYN 546039 ranged from 0.071–0.12 mg/kg at 21 DALA. SYN 546039 was not analysed in the blueberry trials;
- Bulb vegetables (bulb onions, green onions, Table 14, Table 15). SYN 546039 was not detected (<0.01 mg/kg) in any of the bulb onion trials. It was detected in most of the green onion trials at levels ranging from < 0.01–0.049 mg/kg;
- Cucurbits (cucumbers, summer squashes and melons, Table 16, Table 17, Table 18). SYN 546039 was not detected (< 0.01 mg/kg) in most cucurbit trials at PHI 0 days, except for one summer squash trial and one melon trial. In these two trials SYN 546039 ranged from < 0.01–0.020 mg/kg in summer squashes and <0.01–0.018 mg/kg in melons. In another decline trial on melons, SYN 546039 ranged from 0.013–0.014 mg/kg at longer PHIs of 7–14 days;
- Fruiting vegetables other than cucurbits (sweet peppers, chili peppers, tomatoes, Table 19, Table 20, Table 21). SYN 546039 was not detected (<0.01 mg/kg) in any of the pepper trials at PHI 0–21 days. SYN 546039 was found in one tomato trial at PHI 0 days at 0.014–0.021 mg/kg and it was found in two tomato trials at a longer PHI of 14 days where it ranged from <0.01–0.016 mg/kg;
- Pulses (beans, soya beans, peas, Table 22, Table 23, Table 24). SYN 546039 was not detected (<0.01 mg/kg) in any of the dry bean trials at PHI 0–21 days or dry soya bean trials at PHI 0–35 days. SYN 546039 was found in one dry pea trial at 14 days at <0.01–0.037 mg/kg; in all other dry pea trials SYN 546039 was not detected (<0.01 mg/kg) at PHI 0–21 days;
- Root and tuber vegetables (ginseng, sugar beet roots, potato, Table 6, Table 7, Table 25). SYN 546039 was not detected (<0.01 mg/kg) in any of the ginseng trials at PHI 0–21 days, in any of the sugar beet trials at PHI 42–122 days and in any of the potato trials at PHI 0–29 days;
- Cereal grains (barley, wheat, maize, sweet corn, Table 26, Table 27, Table 8, Table 28, Table 29). SYN 546039 was not detected (< 0.01 mg/kg) in most grain trials at PHI 16–52 days for barley, PHI 10–61 days for wheat, PHI 6–22 days for maize and PHI 6–7 days for sweet corn, except for four barley trials and one wheat trial. In these trials SYN 546039 ranged from <0.01–0.040 mg/kg in barley grain at PHI 23–52 days and 0.011 mg/kg at PHI 41 days for wheat grain. In one barley or two wheat trials conducted at 5× exaggerated rate, SYN 546039 ranged from 0.088–0.091 mg/kg at PHI 26 days for barley grain and <0.01–0.017 mg/kg at PHI 34–41 days for wheat grain;
- Sugar cane canes (Table 30): SYN 546039 was not detected (<0.01 mg/kg) in any of the trials at PHI 20–40 days;
- Oilseeds (rapeseed, cottonseed, peanuts, Table 31, Table 32, Table 33): SYN 546039 was not detected (<0.01 mg/kg) in any of the rapeseed trials at normal rate at PHI 25–40 days or any of the peanut trials at PHI 7–44 days. It was however found in several of the cotton seed trials in the range <0.01–0.034 mg/kg at PHI 40–48 days. In addition it was found in the trials conducted at 5x exaggerated rate in the range <0.01–0.12 mg/kg (n=2) at PHI 44–45 days for cottonseed and in the range 0.011–0.046 mg/kg (n=2) at PHI 31 days for rapeseeds;

- Coffee beans (Table 34): SYN 546039 was not detected (<0.01 mg/kg) in most of the green coffee bean trials at PHI 21–35 days. In one trial SYN 546039 was found at 0.02 mg/kg at PHI 21 days;
- Legume forage (pea vines, soya forage, Table 35, Table 36): SYN5456039 was found in all pea vine trials in the range 0.039–0.77 mg/kg at PHI 0–21 days and in all soya forage trials in the range 0.028–0.53 mg/kg at PHI 0–14 days. Trials on peanut forage were not conducted;
- Legume fodder (pea hay, soya fodder, peanut hay, Table 37, Table 38, Table 39): SYN5456039 was detected in all pea hay trials in the range 0.074–4.4 mg/kg at PHI 0–21 days, all soya hay trials in the range 0.091–4.6 mg/kg at PHI 0–14 days and in almost all peanut hay trials in the range <0.01–1.8 mg/kg at PHI 30 days;
- Cereal forage (wheat forage, sweet corn/maize forage, Table 40, Table 41, Table 42): SYN 546039 was found in almost all wheat forage trials in the range <0.01–0.38 mg/kg at PHI 0–14 days and many of the sweet corn and maize forage trials at <0.01–0.055 mg/kg at PHI 1–17 days. Trials on barley forage were not conducted;
- Cereal fodder (barley hay/straw, wheat hay/straw, sweetcorn/maize stover, Table 43, Table 44, Table 45, Table 46, Table 47, Table 48): SYN 546039 was found in all barley hay and straw trials in the range 0.01–1.0 mg/kg at PHI 0–52 days and in almost all wheat hay and straw trials in the range <0.01–1.3 mg/kg at PHI 0–61 days. SYN 546039 was found in many of the sweet corn and maize stover trials in the range <0.01–0.16 mg/kg at PHI 2–17 days;
- Oilseed forage/fodder (Cotton seed gin trash, Table 49): SYN 546039 was found in all gin trash trials in the range 0.11–1.1 mg/kg at PHI 45–49 days. Trials on rapeseed peanut forage were not conducted. Peanut hay is listed under legume hay;
- Sugar beet tops (Table 10). SYN 546039 was not detected (< 0.01 mg/kg) in most sugar beet tops at PHI 48–122 days, except in two trials where SYN 546039 was found at <0.01–0.017 mg/kg at PHI 42–69 days;

  Presence of SYN545720 (cleavage product) in seeds of pulses, oilseeds and coffee beans:

- Pulses (beans, soya beans, peas, Table 22, Table 23, Table 24). SYN545720 was not detected (<0.01 mg/kg) in any of the dry bean seed trials at PHI 0–21 days and dry soya bean seed trials at PHI 0–28 days. SYN545720 was found in one dry pea trial at PHI 14 days at 0.017–0.026 mg/kg; in all other dry pea trials SYN545720 was not detected (<0.01 mg/kg) at PHI 0–22 days;
- Oilseeds (rapeseed, cotton seed, peanuts, Table 31, Table 32, Table 33). SYN545720 was not detected (<0.01 mg/kg) in any of the cottonseed trials at PHI 35–56 days, peanut trials at PHI 7–44 days and rapeseed trials at PHI 25–40 days;
- Coffee beans (Table 34). SYN545720 was not detected (<0.01 mg/kg) in any of the green coffee bean trials at PHI 21–35 days.

Pome fruit

Residue information apples and pears was reproduced from the JMPR 2016 evaluation and extended with the additional metabolite information.

Apple

Table 11 Residues in apple from field trials in Canada and the United States (JMPR 2016)

APPLE Location, Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent mg/kg	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
United States, North Rose, NY 2010, (Cortland)	EC	4 **(7,7,7)**	0.050 0.050 **0.051** **0.051**	81 [29 Jul]	30	0.024, 0.036 (0.030)	<0.01, <0.01; (<0.01)	TK0025156 E03-0481 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	**0.051** **0.051** **0.051** 0.050	**75** **[29 Jun]**	60	0.011, <0.01 (0.01)	<0.01, <0.01; (<0.01)	
United States, North Rose, NY 2010, (Ida Red)	EC	4 **(6,7,7)**	**0.051** **0.051** **0.051** **0.051**	85 [26 Aug]	**32**	0.040, 0.037 (0.039)	<0.01, <0.01; (<0.01)	TK0025156 E03-0482 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	0.051 0.051 0.051 0.051	**75** **[29 Jul]**	60	0.028, 0.015 (0.021)	<0.01, <0.01; (<0.01)	
United States, Hereford, PA 2010, (Starkrimson)	EC	4 (8,7,6)	0.050 0.049 **0.050** 0.050	78	**29**	0.062, 0.086 (0.074)	<0.01, <0.01; (<0.01)	TK0025156 E04-0483 (Storage: 1.9 months)
	EC	4 (7,7,7)	**0.051** **0.050** **0.051** **0.051**	**75**	60	0.057, 0.057 (0.057)	<0.01, <0.01; (<0.01)	
United States, Alto, GA 2010, (Arkansas, Black)	EC	4 **(7,7,7)**	0.050 0.050 0.050 0.050	78	30	0.093, 0.041 (0.067)	<0.01, <0.01; (<0.01)	TK0025156 E12-0484 (Storage: 1.9 months)
	EC	4 **(6,7,7)**	0.050 0.051 0.051 0.050	**75**	60	0.080, 0.055 (0.067)	<0.01, <0.01; (<0.01)	
United States, Conklin, MI 2010, (Red Delicious)	EC	4 **(7,7,7)**	0.050 0.050 0.050 0.050	79	**30** **31**	0.019, 0.018 (0.019)	<0.01, <0.01; (<0.01)	TK0025156 C03-0485 (Storage: 1.9 months)
	EC	4 **(7,6,8)**	0.050 0.050 0.050 0.050	**76**	60	<0.01, 0.01 (0.01)	<0.01, <0.01; (<0.01)	
United States, Conklin, MI 2010, (Golden Delicious)	EC	4 (7,7,7)	0.050 **0.051** **0.051** 0.050	79	**31**	0.087, 0.051 (0.069)	<0.01, <0.01; (<0.01)	TK0025156 C03-0486 (Storage: 1.9 months)
	EC	4 **(7,6,8)**	0.050 0.050 0.050 0.050	**76**	60	0.034, 0.043 (0.038)	<0.01, <0.01; (<0.01)	
	EC	**4 (6,7,7)**	**0.252** **0.252**	**79** **[20 Aug]**	**31**	**0.46, 0.45**	**0.010, 0.012**	

APPLE Location, Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent mg/kg	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
			0.251 **0.250**			**(0.46)**	**(0.011)**	
United States, Los Molinos, CA 2010, (Summer field)	EC	4 **(7,7,7)**	**0.051** **0.051** **0.050** **0.051**	79	30	0.034, <0.01 (**<u>0.022</u>**)	<0.01, <0.01; (<0.01)	TK0025156 W23-0488 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	**0.051** **0.051** **0.051** **0.051**	**75**	60	0.012, 0.020 (0.016)	<0.01, <0.01; (<0.01)	
United States, Ephrata, WA 2010, (Red Delicious)	EC	4 **(7,7,9)**	**0.051** **0.051** **0.051** **0.051**	87 **[8 Sept]**	**28**	0.074, 0.081 (0.078)	<0.01, <0.01; (<0.01)	TK0025156 W18-0489 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	0**.051** 0.050 0.050 0.050	**76** **[6 Aug]**	**61**	0.053, 0.061 (0.057)	<0.01, <0.01; (<0.01)	
	EC	**4** **(7,7,9)**	**0.255** **0.255** **0.254** **0.252**	**87** **[8 Sept]**	**28**	**0.66,** **0.50** **(0.58)**	**<0.01,** **<0.01** **(<0.01)**	
United States, Ephrata, WA 2010, (Breabum)	EC	4 **(7,7,7)**	**0.051** **0.051** **0.051** 0.050	87	20, 25 30 35, 40	0.12, 0.075 0.069, 0.078 (0.074) 0.086, 0.067	<0.01, <0.01; <0.01, <0.01; (<0.01) <0.01, <0.01;	TK0025156 W18-0490 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	0.050 **0.051** **0.051** 0.050	**80**	**59**	0.094, 0.097 (<u>0.096</u>)	<0.01, <0.01; (<0.01)	
United States, Hood River, OR 2010, (Jonagold)	EC	4 **(7,7,7)**	0.050 0.050 0.050 0.050	**77**	20 25 **32** 35 **39**	0.071 0.050 0.040 0.048 **(0.044)** 0.049 0.046	<0.01, <0.01; <0.01, <0.01; (<0.01) <0.01 <0.01	TK0025156 W20-0491 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	**0.050** **0.051** **0.050** **0.051**	**73**	60	0.033 0.041 **(0.037)**	<0.01, <0.01; (<0.01)	
United States, Hood River, OR 2010, (Honey Crisp)	EC	4 **(7,7,7)**	0.050 0.050 **0.051** **0.051**	77	**29**	0.065, 0.056 (**<u>0.060</u>**)	<0.01, <0.01; (<0.01)	TK0025156 W20-0492
	EC	4 **(7,6,8)**	**0.052** **0.051** **0.051** **0.051**	**75**	60	0.019, 0.023 **(0.021)**	<0.01, <0.01; (<0.01)	
United States,	EC	4	**0.051**	85	30	0.17,	<0.01,	TK0025156

APPLE Location, Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent mg/kg	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
Madera, CA 2010, (Fuji)		**(7,7,7)**	0.052 0.050 0.052			0.15 (0.16)	<0.01; (<0.01)	E19-0487 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	**0.051** 0.052 0.050 0.052	**79**	60	0.033, 0.027 (0.030)	<0.01, <0.01; (<0.01)	
United States, Madera. CA **2011,** (Fuji)	EC	4 **(7,7,7)**	**0.049** **0.050** **0.050** **0.050**	85	**29**	0.058, 0.023 **(0.040)**	<0.01, <0.01; (<0.01)	TK0025156 TK0025156-19 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	**0.050** **0.050** **0.049** **0.050**	**77**	60	0.013, <0.01 (0.012)	<0.01, <0.01; (<0.01)	
Canada, Berwick, NS 2011, (McIntosh)	EC	4 **(7,7,7)**	0.050 0.051 0.052 0.050	77-81	30	0.038, 0.057 (0.048)	<0.01, <0.01; (<0.01)	CER05906/11 T960 (Storage: 1.9 months)
	EC	4 **(7,7,8)**	**0.050** **0.051** **0.050** **0.051**	**73**	59	0.029, 0.015 (0.022)	<0.01, <0.01; (<0.01)	
Canada, St. George, ON 2011, (Empire)	EC	4 **(7,7,7)**	0.051 0.049 0.051 0.050	76-78	**20** **25** 31 . . **35** **40**	**0.036** **0.032** 0.036, 0.039 (0.038) **0.029** **0.033**	<0.01 <0.01 <0.01, <0.01; (<0.01) <0.01 <0.01	CER05906/11 T961 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	**0.049** **0.047** **0.047** **0.048**	**74-75**	61	0.028, 0.025 **(0.026)**	<0.01, <0.01; (<0.01)	
Canada, Branchton, ON 2011, (McIntosh)	EC	4 **(7,7,7)**	0.049 0.051 0.049 0.048	77-79 [25 Aug]	29	0.038, 0.038 (0.038)	<0.01, <0.01; (<0.01)	CER05906/11 T962 (Storage: 1.9 months)
	EC	4 **(7,6,7)**	**0.048** **0.049** **0.051** **0.050**	**74-75** **[25 Jul]**	60	0.025, 0.020 **(0.022)**	<0.01, <0.01; (<0.01)	
Canada, Branchton, ON 2011, (Imperial Gala)	EC	4 **(7,7,7)**	0.050 0.050 0.051 0.050	76-78 [25 Aug]	29	0.033, 0.051 (0.042)	<0.01, <0.01; (<0.01)	CER05906/11 T963 (Storage: 1.9 months)
	EC	4 **(7,6,7)**	**0.048** **0.051** **0.051** **0.050**	**75** **[25 Jul]**	60	0.026, 0.035 **(0.030)**	<0.01, <0.01; (<0.01)	
Canada, Okanagan Falls, BC	EC	4 **(8,7,7)**	0.053 0.052 0.051	78-81	30	0.036, 0.026 (0.031)	<0.01, <0.01; (<0.01)	CER05906/11 T964 (Storage: 1.9

APPLE Location, Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent mg/kg	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
2011, (McIntosh)			0.053					months)
	EC	**5 (8,6,7,7)**	**0.049 0.052 0.051 0.051 0.051**	**75-77**	52	0.013, 0.017 (0.015)	<0.01, <0.01; (<0.01)	

Pear

Table 12: Residues in pear from field trials in Canada and the United States (JMPR 2016)

PEAR Location Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Orefield, PA 2010, (Bartlett)	EC	4 **(5,6,8)**	0.050 **0.051 0.051** 0.052	77	**29**	0.018, 0.023 **(0.020)**	<0.01, <0.01; (<0.01)	TK0025156 E04-0493 (Storage: 1.9 months)
	EC	4 **(5,8,6)**	**0.050 0.051 0.050 0.051**	**75**	**57**	<0.01, <0.01 **(<0.01)**	<0.01, <0.01; (<0.01)	
United States, Lindsay, CA 2010, (Olympic)	EC	4 **(6,7,7)**	**0.051 0.051 0.051 0.050**	79 **[18 Aug]**	30	0.069, 0.055 (0.062)	<0.01, <0.01; (<0.01)	TK0025156 W32-0494 (Storage: 1.9 months)
	EC	4 **(6,7,7)**	**0.050 0.051 0.051 0.051**	79 **[21 Jul]**	**58**	0.032, 0.038 (0.035)	<0.01, <0.01; (<0.01)	
United States, Madera. CA 2010, **(Hosui Asian)**)	EC	4 **(7,7,7)**	**0.051 0.050 0.051 0.051**	85	30	0.077, 0.056 **(0.066)**	<0.01, <0.01; (<0.01)	TK0025156 E19-0495 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	0.050 0.051 0.051 0.050	**79**	60	0.011, 0.016 (0.014)	<0.01, <0.01; (<0.01)	
United States, Hood River, OR 2010, (Red d'Anjou)	EC	4 **(7,7,7)**	0.050 0.050 0.050 0.050	**75**	**29**	0.084, 0.089 **(0.086)**	<0.01, <0.01; (<0.01)	TK0025156 W20-0498 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	**0.052 0.050 0.050 0.051**	**74**	**61**	0.026, 0.019 **(0.022)**	<0.01, <0.01; (<0.01)	
United States, Ephrata, WA 2010, (Concorde)	EC	4 **(7,7,7)**	0.050 **0.051** 0.050 0.050	84 [20 Aug]	**31**	0.11, 0.090 (0.10)	<0.01, <0.01; (<0.01)	TK0025156 W18-0496 (Storage: 1.9 months)
	EC	4	**0.051**	**75**	60	0.025,	<0.01,	

PEAR Location Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
		(7,8,6)	**0.051 0.051 0.050**	**[22 Jul]**		0.030 (0.028)	<0.01; (<0.01)	
United States, Ephrata, WA 2010, (Bartlett)	EC	4 **(7,7,7)**	0.050 **0.051 0.051 0.051**	80 [6 Aug]	**31**	0.082, 0.10 (0.091)	<0.01, <0.01; (<0.01	TK0025156 W18-0497 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	**0.051 0.050 0.051 0.051**	**75 [8 Jul]**	60	0.015, 0.015 (0.015)	<0.01, <0.01; (<0.01)	
United States, Ephrata, WA **2011, (Hosui Asian**)	EC	4 **(7,7,7)**	**0.049 0.050 0.050 0.050**	85 **[29 Aug]**	**29**	0.019, 0.033 (0.026)	<0.01, <0.01; (<0.01)	TK0025156 TK0025156-20 (Storage: 1.9 months)
	EC	4 **(7,7,7)**	**0.050 0.050 0.052 0.050**	77 **[29 Jul]**	60	0.017, 0.014 (0.016)	<0.01, <0.01; (<0.01)	
Canada, Simcoe, ON 2011, (Bartlett)	EC	4 **(7,6,8)**	0.051 0.051 0.050 0.052	74-75	30	0.064, 0.050 (0.057)	<0.01, <0.01; (<0.01)	CER05907/11 T966 (Storage: 1.9 months)
	EC	4 **(6,7,7**)	**0.048 0.053 0.051 0.055**	**73-74**	60	0.030, 0.019 **(0.024)**	<0.01, <0.01; (<0.01)	
Canada, Beamsville, ON 2011, (Bosc)	EC	4 **(7,7,7)**	0.054 0.054 0.054 0.053	76-78 [17 Aug]	30	0.042, 0.038 (0.040)	<0.01, <0.01; (<0.01)	CER05907/11 T965 (Storage: 1.9 months)
	EC	4 **(7,7,8)**	**0.051 0.052 0.052 0.053**	**74-75 [19 Jul]**	59	0.022, 0.015 **(0.018)**	<0.01, <0.01; (<0.01)	
Canada, Beamsville, ON 2011, (D'Anjou	EC	4 **(7,7,7)**	0.049 0.048 0.050 0.049	76-77 [17 Aug]	30	0.041, 0.030 (0.036)	<0.01, <0.01; (<0.01)	CER05907/11 T967 (Storage: 1.9 months)
	EC	4 **(7,7,8)**	**0.049 0.051 0.049 0.050**	**74-75 [19 Jul]**	59	0.014, 0.011 **(0.012)**	<0.01, <0.01; (<0.01)	
Canada, St. Catharines, ON 2011, (Bartlett)	EC	4 **(7,6,8**)	0.048 0.049 0.048 0.050	74-76	**20 26** 30 . . **36 40**	**0.060 0.044** 0.062, 0.055 **(0.058) 0.048 0.026**	<0.01 <0.01 <0.01, <0.01; (<0.01) <0.01 <0.01	CER05907/11 T968 (Storage: 1.9 months)
	EC	4 **(6,6,7)**	**0.049 0.049 0.048**	**74**	60	0.011, 0.011 ((0.011)	<0.01, <0.01; (<0.01)	

PEAR Location Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
			0.051					
Canada, Okanagan Falls, BC 2011, (Bartlett)	EC	4 **(7,8,6)**	0.051 0.052 0.049 0.050	76-78	31	0.036, 0.052 (0.044)	<0.01, <0.01; (<0.01)	CER05907/11 T969 (Storage: 1.9 months)
	EC	4 **(6,8,6)**	**0.051 0.053 0.051 0.051**	**74-77**	59	0.019, 0.026 **(0.022)**	<0.01, <0.01; (<0.01)	

Grapes

Residue information on grapes was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information.

Table 13 Residues in grapes from field trials in the United States (JMPR 2016)

GRAPES Location, Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
United States, Dundee, NY 2010, (Concord)	EC	4 (7,7,7)	**0.077 0.077 0.077 0.076**	85 [31 Aug]	21	0.21, 0.27 **(0.24)**	<0.01, 0.013 (0.012)	TK0025158 E03-0501 (Storage: 486 days)
	EC	4 (7,7,7)	**0.077 0.075 0.076 0.076**	**81 [7 Aug]**	45	0.15, 0.17 (0.16)	0.053, 0.059 (0.056)	
United States, Dundee, NY 2010, (Vidal)	EC	4 (7,7,7)	**0.076 0.077 0.077 0.076**	85 [24 Sept]	21	0.23, 0.48 **(0.36)**	<0.01, <0.01; (<0.01)	TK0025158 E03-0502 (Storage: 462 days)
	EC	4 (7,7,7)	**0.077 0.076 0.078 0.077**	**83 [31 Aug]**	45	0.36, 0.26 (0.31)	0.060, 0.045 (0.052)	
United States, Artios, CA 2010, (Rubi Red)	EC	4 (7,7,7)	0.075 0.074 0.075 0.075	83 **[13 Aug]**	21	0.59, 0.73 (0.66)	0.028, 0.013; (0.020)	TK0025158 W23-0503 (Storage: 504 days)
	EC	4 (7,7,7)	**0.074 0.075 0.075 0.074**	**77 [20 Jul]**	45	0.81, 0.72 **(0.76)**	0.16; 0.22; (0.19)	
United States, Kerman, CA 2010, (Thompson Seedless)	EC	4 (6,7,7)	**0.077 0.077 0.077 0.077**	81 **[26 Jul]**	21	0.11, 0.11 (0.11)	0.010, 0.024 (0.017)	TK0025158 W28-0504 (Storage: 522 days)
	EC	4 **(7,7,7)**	**0.077 0.077 0.077**	**75 [2 Jul]**	45	0.25, 0.21 (0.23)	0.082, 0.080 (0.081)	

GRAPES Location, Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
			0.077					
United States, Hickman, CA 2010, (Chardonnay)	EC	4 (7,7,7)	**0.075 0.076 0.076 0.076**	85 **[20 Aug]**	11 16 21 26 31	0.49 0.52 0.36, 0.48 **(0.42)** 0.44 0.47	0.023; 0.034; 0.034, 0.036; (0.035) 0.036 0.048	TK0025158 W26-0505 (Storage: 507 days)
	EC	4 **(8,6,7)**	**0.077 0.076 0.076 0.076**	**79 [28 Jul]**	45	0.45, 0.34 **(0.40)**	0.11, 0.091 (0.10)	
United States, Delano, CA 2010, (Thompson Seedless)	EC	4 (7,6,6)	**0.076 0.077 0.076 0.077**	47 **[17 Aug]**	21	0.090, 0.11 (0.10)	0.015, 0.015 (0.015)	TK0025158 W33-0506 (Storage: 500 days)
	EC	4 **(7,8,13)**	**0.077 0.077 0.078 0.076**	**47 [29 Jul]**	45	0.061, 0.042 (0.052)	0.023, 0.018 (0.020)	
	EC	**4 (7,6,6)**	**0.384 0.384 0.382 0.383**	**47 [17 Aug]**	**21**	**0.99, 1.4 (1.2)**	**0.089, 0.071 (0.080)**	
United States, Madera, CA 2010, (Thompson Seedless)	EC	4 (7,7,7)	**0.078 0.078 0.077 0.078**	85 [9 Aug]	21	0.14, 0.14 (0.14)	0.031, 0.032 (0.032)	TK0025158 W29-0507 (Storage: 508 days)
	EC	4 (7,7,7)	**0.076 0.076 0.076 0.078**	**77 [16 Jul]**	45	0.12, 0.10 (0.11)	0.039, 0.026 (0.032)	
	EC	**4 (7,7,7)**	**0.391 0.375 0.376 0.391**	**85 [9 Aug]**	**21**	**1.1, 0.69 (0.92)**	**0.11, 0.12 (0.12)**	
United States, Madera, CA 2010, (Thompson Seedless)	EC	4 (7,7,7)	**0.077 0.078 0.078 0.079**	85 [13 Aug]	21	0.10, 0.23 **(0.16)**	0.012, 0.016; (0.014)	TK0025158 E19-0510 (Storage: 504 days)
	EC	4 (7,7,7)	**0.077 0.078 0.077 0.078**	**79 [20 Jul]**	45	0.12, 0.14 (0.13)	0.023, 0.018 (0.020)	
United States, Lindsay, CA 2010, (Crimson)	EC	4 (7,7,7)	0.075 0.076 0.076 0.077	88 **[27 Aug]**	21	0.43, 0.38 **(0.40)**	0.027, 0.028 (0.028)	TK0025158 W32-0508 (Storage: 497 days)
	EC	4 **(6,7,7)**	0.076 0.078 0.076 0.076	**81 [27 Jul]**	45	0.39, 0.30 **(0.34)**	0.045, 0.037 (0.041)	

GRAPES Location, Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
United States, Fresno, CA 2010, (Thompson Seedless)	EC	4 (7,7,7)	**0.077 0.078 0.078 0.079**	83 [13 Aug]	11 16 21 26 31	0.13 0.042 0.094, 0.083 **(0.089)** 0.041 0.056	0.016; <0.01; 0.018, 0.012; (0.015) <0.01, <0.01	TK0025158 E19-0509 (Storage: 514 days)
	EC	4 (7,7,7)	**0.077 0.078 0.078 0.078**	**79 [20 Jul]**	45	0.094, 0.074 (0.084)	0.028, 0.017 (0.022)	
United States, Ephrata, WA 2010, (White Riesling)	EC	4 (7,7,7)	**0.076 0.076 0.077 0.076**	85 [16 Sept]	21	0.55, 0.55 (0.55)	0.026, 0.025; (0.026)	TK0025158 W18-0511 (Storage: 470 days)
	EC	4 (7,7,7)	**0.076 0.076 0.076 0.076**	**82 [23 Aug]**	45	0.40, 0.39 (0.40)	0.069, 0.086 (0.078)	
United States, Ephrata, WA 2010, (Chardon-nay)	EC	4 (7,7,7)	0.076 0.076 0.076 0.076	87 [2 Sept]	21	0.49, 0.35 (0.42)	0.057, 0.041; (0.049)	TK0025158 W18-0512 (Storage: 484 days)
	EC	4 (7,7,7)	**0.076 0.076 0.076 0.077**	**80 [9 Aug]**	45	0.27, 0.25 (0.26)	0.072, 0.066; (0.069)	
United States, Fresno, CA **2011,** (Ruby Red)	EC	4 (7,7,7)	0.075 0.075 0.076 0.075	83 [25 Aug]	**22**	0.038, 0.040 (0.039)	<0.01, <0.01 (<0.01)	TK0025158 TK0025158- 13 (Storage: 126 days)
	EC	4 **(7,9,5)**	**0.075 0.076 0.075 0.074**	**77 [1 Aug]**	**46**	0.043, 0.037 (0.040)	<0.01, <0.01 (<0.01)	
United States, Fresno, CA **2011,** (Thompson Seedless)	EC	4 (7,7,7)	**0.075 0.075 0.075 0.076**	83 [25 Aug]	21	0.15, 0.16 (0.16)	0.018, 0.023 (0.020)	TK0025158 TK0025158- 14 (Storage: 126 days)
	EC	4 (7,7,7)	0.075 0.075 0.075 0.075	**77 [1 Aug]**	45	0.10, 0.086 (0.093)	0.070, 0.041 (0.056)	
United States, Dundee, NY 2011, (Concord)	EC	4 (7,7,7)	0.077 0.077 0.076 0.077	83 [31 Aug]	21	0.41, 0.44, 0.31 (0.39)	0.017, 0.021, 0.015 (0.018)	TK0044874 TK0044874-01 (Storage: 161 days)
	WG	4 (7,7,7)	0.076 0.076 0.076 0.076		21	0.38, 0.38, 0.34 (0.37)	0.016, 0.014, 0.011 (0.014)	
United States,	EC	4	0.077	81	21	0.18,	0.042,	TK0044874

GRAPES Location, Year (variety)	Form.	no	kg ai/ha	BBCH [last application date]	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
Madera, CA 2011, (Thompson Seedless)		(7,7,7)	0.077 0.077 0.078	[1 Aug]		0.17, 0.11 (0.15)	0.045, 0.030 (0.039)	TK0044874-02 (Storage: 191 days)
	WG	4 (7,7,7)	**0.077** **0.077** **0.076** **0.077**		21	0.14, 0.14, 0.15 (0.14)	0.023, 0.022, 0.024 (0.023)	
United States, Templeton, CA 2011, (Marsanne)	EC	4 (6,7,8)	0.080 0.079 0.074 0.078	81 **[26 Aug]**	21	0.089, 0.087, 0.062 (0.079)	0.018, 0.026, 0.021 (0.022)	TK0044874 TK0044874-03 (Storage: 166 days)
	WG	4 (6,7,8)	0.078 0.079 0.077 0.076		21	0.13, 0.10, 0.10 (0.11)	0.012, <0.01, <0.01 (0.011)	

Bulb vegetables

Residue information on bulb onions and green onions was reproduced from the JMPR 2019 evaluation.

Bulb onions

Table 14: Residues in bulb onions bulbs from field trials in the United States and Canada (JMPR 2019)

BULB ONIONS Location, Year (variety)	Form	RTI	Rate (kg ai/ha)	Growth stage at final application	DALT	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report Trials No Reference Storage period
United States Parlier, CA 2013 (Candy)		7	0.077 0.076 0.076 0.077	Vegetative	0 2 7 14	0.014 0.014 (0.014) 0.011 <0.01 (0.010) 0.014 <0.01 (0.012) <0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01) <0.01 <0.01 (<0.01) <0.01 <0.01 (<0.01) <0.01 <0.01 (<0.01)	IR-4 PR No. 11130 CA73 Lennon, 2016, BENZOVINDI_001 storage: 16 months
United States Las Cruces, NM 2013 (Texas Yellow Grano)		6-7	0.073 0.077 0.075 0.077	Mature bulb	6	0.012 0.011 (0.011)	<0.01 <0.01 (<0.01	IR-4 PR No. 11130 NM13 Lennon, 2016, storage: 17 months
United States Willard, OH 2013 (Candy)		6-8	0.075 0.078 0.075 0.077	Vegetative	8	0.015 0.014 (0.015)	<0.01 <0.01 (<0.01)	IR-4 PR No. 11130 OH*10 Lennon, 2016,

BULB ONIONS Location, Year (variety)	Form	RTI	Rate (kg ai/ha)	Growth stage at final application	DALT	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report Trials No Reference Storage period
								storage: 15 months
United States Moxee, WA 2013 (Candy)	WG	6-7	0.077 0.076 0.074 0.075	Vegetative	7	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	IR-4 PR No. 11130 WA*19 Lennon, 2016, storage: 14 months
	EC	6-7	0.077 0.077 0.076 0.076	Vegetative	7	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	
United States Holtville, CA 2014 (Koda)	WG	6-7	0.074 0.075 0.073 0.075	Mature bulbs (2-4" diameter)	6	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	IR-4 PR No. 11130 CA72 Lennon, 2016, storage: 6.6 months
	EC	6-7	0.076 0.076 0.073 0.076	Mature bulbs (2-4" diameter)	6	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	
United States Weslaco, TX 2014 (Sierra Blanca)	WG	7	0.075 0.074 0.074 0.074	Vegetative	7	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	IR-4 PR No. 11130 TX11 Lennon, 2016, storage: 8.0 months
	EC	7	0.077 0.075 0.076 0.076	Vegetative	7	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	
Canada[b] Harrow, ON 2013 (Lasalle)		6-7	0.074 0.076 0.076 0.074	7 leaves	8	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	IR-4 PR No. 11130 ON05 Lennon, 2016, storage: 15 months
Canada[b] Harrow, ON 2013 (Pulsar)		6-7	0.074 0.078 0.085 0.076	~8 leaves	8	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	IR-4 PR No. 11130 ON06 Lennon, 2016, storage: 15 months
Canada[b] Ste. Clotilde, QC 2013 (Trailblazer)		6-8	0.072 0.075 0.073 0.068	8+ leaves; bulb almost at size	8	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	IR-4 PR No. 11130 QC08 Lennon, 2016, storage: 15 months
Canada[b] Ste. Clotilde, QC 2013 (Frontier)		6-8	0.074 0.074 0.083 0.076	8+ leaves; bulb almost at size	8	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	IR-4 PR y No. 11130 QC09 Lennon, 2016, storage: 15 months

Notes:

[a] Mean of replicate field samples [individual values]

[b] It was noted that trial ON5 and ON6, as well as QC8 and QC9 were performed at the same location and year and therefore could not be considered as independent. Hence, the highest residue value from each of these locations was selected.

Green onions

Table 15 Residues in green onions whole plant from field trials in the United States and Canada (JMPR 2019)

GREEN ONIONS Location, Year (variety)	Form	RTI	Rate (kg ai/ha)	Growth Stage at final appl	DALT	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; trial no Reference Storage period
United States Willard, OH 2013 (Evergreen white bunching)		6-8	0.096 0.074 0.078 0.076	Vegetative	7	0.060 0.053 (0.056)	0.012 0.010 (0.011)	IR-4 PR No. 11130 OH*17 Lennon, 2016, BENZOVINDI_001 Max. frozen storage: 16 months
United States Willard, OH 2013 (Ishikura Improved bunching)	WG	6-8	0.081 0.075 0.081 0.076	Vegetative	6	0.042 0.060 (0.051)	<0.01 <0.01 (<0.01)	IR-4 PR No. 11130 OH*18 Lennon, 2016, BENZOVINDI_001 Max. frozen storage: 16 months
	EC	6-8	0.078 0.075 0.075 0.083	Vegetative	6	0.14 0.17 (0.16)	0.038 0.043 (0.041)	
United States Aurora, OR 2013 (Green Banner)		6-8	0.077 0.077 0.077 0.078	Vegetative	7	0.11 0.11 (0.11)	0.011 0.010 (0.011)	IR-4 PR No. 11130 OR16 Lennon, 2016, BENZOVINDI_001 Max. frozen storage: 17 months
Canada Ste. Clotilde, QC 2013 (Tokyo long white)		7-8	0.074 0.075 0.074 0.076	5 leaves	7	0.20 0.20 (0.20)	0.047 0.049 (0.048)	IR-4 PR No. 11130 QC20 Lennon, 2016, BENZOVINDI_001 Max. frozen storage: 16 months

Notes:

[a] Mean of replicate field samples [individual values]

[b] It was noted that trial OH17 and OH18 were performed at the same location and year and therefore could not be considered as independent. Hence, the highest residue value from this location was selected.

Cucurbits

Residue information on cucumbers, summer squash and melons was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information.

Cucumber

Table 16 Residues in cucumber from field trials in the United States (JMPR 2016)

CUCUMBER Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Chula, GA	EC	4 (7)	0.077 0.077	89	0	0.018, <0.010,	<0.01, <0.01	TK0058639 TK0058639-07

CUCUMBER Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
2011, (Thunder)			0.077 0.075			<0.010 (0.013)	<0.01; (<0.01)	(Storage: 10.2 months)
	WG	4 (7)	0.076 0.077 0.076 0.076		0	<0.010, 0.012, 0.018 (0.013)	<0.01 <0.01, <0.01; (<0.01)	
United States, Suffolk, NC 2011, (Marketer)	EC	4 (7)	0.076 0.076 0.077 0.078	65	0	<0.01, 0.010 (0.010)	<0.01, <0.01; (<0.01)	TK0058639 TK0058639-08 (Storage: 10.2 months)
United States, Hobe Sound, FL 2011, (Impact)	EC	4 (7)	0.076 0.076 0.077 0.077	71	0	0.025, 0.047, 0.084 (0.052)	<0.01 <0.01, <0.01; (<0.01)	TK0058639 TK0058639-09 (Storage: 10.2 months)
	WG	4 (7)	0.077 0.076 0.077 0.077		0	0.052, 0.039, 0.057 (0.049)	<0.01 <0.01, <0.01; (<0.01)	
United States, Northwood, ND 2011, (Marketmore 76)	EC	4 (7)	0.077 0.077 0.075 0.077	85	0	<0.01, <0.01, <0.01 (<0.01)	<0.01 <0.01, <0.01; (<0.01)	TK0058639 TK0058639-10 (Storage: 10.2 months)
	WG	4 (7)	0.076 0.074 0.076 0.077		0	<0.01, <0.01, <0.01 (<0.01)	<0.01 <0.01, <0.01; (<0.01)	
United States, Campbell, MN 2011, (Speedway)	EC	4 (7)	0.079 0.077 0.077 0.076	73	0	0.014, 0.022 (0.018)	<0.01, <0.01; (<0.01)	TK0058639 TK0058639-11 (Storage: 10.2 months)
United States, Hinton, OK 2011, (Calypso)	EC	4 (7)	0.077 0.076 0.073 0.075	84	0	0.038, 0.028 (0.033)	<0.01, <0.01; (<0.01)	TK0058639 TK0058639-12 (Storage: 10.2 months)

Summer squash

Table 17 Residues in summer squash from field trials in the United States (JMPR 2016)

SUMMER SQUASH Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Alton, NY 2011, (Superpik F1)	EC	4 (7)	0.079 0.078 0.051 0.078	79	0	0.024, 0.019 (0.022)	<0.01, <0.01; (<0.01)	TK0058639 TK0058639-13 (Storage: 9.4 months)

SUMMER SQUASH Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Chula, GA 2011, (Dixie)	EC	4 (7)	0.077 0.076 0.075 0.078	89	0	0.013, 0.022, 0.031 (<u>0.022</u>)	<0.01, 0.012, 0.020 (0.014)	TK0058639 TK0058639-14 (Storage: 9.4 months)
	WG	4 (7)	0.077 0.076 0.076 0.077		0	0.019, 0.021, 0.023 (0.021)	<0.01 <0.01, <0.01; (<0.01)	
United States, Hobe Sound, FL 2011, (Fortune)	EC	4 (7)	0.076 0.077 0.077 0.076	75	0	0.042, 0.072, 0.032 (0.049)	<0.01 <0.01, <0.01; (<0.01)	TK0058639 TK0058639-15 (Storage: 9.4 months)
	WG	4 (7)	0.076 0.076 0.077 0.077		0	0.028, 0.035, 0.086 (<u>0.050</u>)	<0.01, <0.01, <0.01; (<0.01)	
United States, York, NE 2011, (Black Beauty Zucchini)	EC	4 (7)	0.076 0.075 0.075 0.078	76	0	0.014, 0.018, 0.019 (<u>0.017</u>)	<0.01 <0.01, <0.01; (<0.01)	TK0058639 TK0058639-16 (Storage: 9.4 months)
	WG	4 (7)	0.076 0.076 0.076 0.076		0	<0.01, 0.017, 0.018 (0.015)	<0.01 <0.01, <0.01; (<0.01)	
United States, Porterville, CA 2011, (Black Beauty)	EC	4 (7)	0.076 0.076 0.077 0.079	89	0 1 3 7 14	0.012, 0.026 (0.019) <u>0.023</u> 0.018 0.010 <0.01	<0.01, <0.01; (<0.01) <0.01 <0.01 <0.01 <0.01	TK0058639 TK0058639-17 (Storage: 9.4 months)

Melons

Table 18 Residues in melons from field trials in the United States (JMPR 2016)

MELONS Location; Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Chula, GA 2011, (Cantaloupe: Athena)	EC	4 (7)	0.075 0.076 0.076 0.076	89	0	0.046, 0.046, 0.051 (0.048)	<0.01 <0.01, <0.01; (<0.01)	TK0058639 TK0058639-01 (Storage: 9.7 months)
	WG	4 (7)	0.077 0.076 0.076 0.077		0	0.066, 0.045, 0.049 (<u>0.053</u>)	<0.01 <0.01, <0.01; (<0.01)	

MELONS Location; Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Rice, MN 2011, (Cantaloupe: Honey Rock)	EC	4 (7)	0.076 0.078 0.077 0.077	76	0	0.13, 0.12, 0.16 (0.14)	0.018, 0.018, 0.017 (0.018)	TK0058639 TK0058639-02 (Storage: 9.7 months)
	WG	4 (7)	0.077 0.076 0.076 0.075		0	0.14, 0.090, 0.062 (0.097)	0.011, <0.01, <0.01 (0.010)	
United States, Wall, TX 2011, (Cantaloupe: Jumbo Hales Best)	EC	4 (7)	0.076 0.077 0.078 0.076	89	0	0.029, 0.069 (0.049)	<0.01, <0.01; (<0.01)	TK0058639 TK0058639-03 (Storage: 9.7 months)
United States, Porterville, CA 2011, (Cantaloupe: Hales Best Jumbo)	EC	4 (7)	0.076 0.077 0.077 0.077	89	0	0.026, 0.033, 0.018 (0.026)	<0.01 <0.01, <0.01; (<0.01)	TK0058639 TK0058639-4 (Storage: 9.7 months)
	WG	4 (7)	0.077 0.077 0.076 0.079		0	0.016, 0.031, 0.011 (0.019)	<0.01 <0.01, <0.01; (<0.01)	
United States, Paso Robles, CA 2011, (Cantaloupe: Top Mark)	EC	4 (7)	0.078 0.076 0.075 0.077	82	0	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	TK0058639 TK0058639-5 (Storage: 9.7 months)
United States, Sanger, CA 2011, (Oro Rico)	EC	4 (7)	0.078 0.078 0.078 0.078	89	0 1 3 7 14	0.14, 0.096 (0.12) 0.11 0.12 0.11 0.068	<0.01, <0.01 (<0.01) <0.01 <0.01 0.014 0.013	TK0058639 TK0058639-6 (Storage: 9.7 months)

Fruiting vegetables other than cucurbits

Residue information on sweet peppers, chili peppers and tomatoes was reproduced from the JMPR 2016 evaluation and extended with the additional metabolite information.

Sweet peppers

Table 19 Residues in sweet peppers from field trials in United States (JMPR 2016)

SWEET PEPPERS Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Athens, GA	EC	4 (7)	0.077 0.076	79	0 .	0.081, 0.10,	<0.01 <0.01,	TK0058641 TK0058641-13

SWEET PEPPERS Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
2011, (bell: Yolo)			0.077 0.078		 **14**	0.10 (0.093) **0.029,** **0.026** **(0.028)**	<0.01; (<0.01) <0.01, <0.01; (<0.01)	(Storage: 9.5 months)
	WG	4 (7)	0.077 0.075 0.077 0.076	79	0	0.085, 0.11, 0.071 (0.089)	<0.01 <0.01, <0.01; (<0.01)	
United States, Winter Garden, FL 2011, (bell: Patriot)	EC	4 (7)	0.077 0.076 0.076 0.074	87	0 . **14**	0.51, 0.72, 0.62 (0.62) **0.29,** **0.34** **(0.32)**	<0.01 <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641 TK0058641-14 (Storage: 5.2-6.2 months) Two different brands of crop oil concentrate adjuvants were used.
	WG	4 (7)	0.076 0.077 0.076 0.073	87	0	0.62, 0.51, 0.48 (0.54)	<0.01 <0.01, <0.01; (<0.01)	
United States, Stafford, KS 2011, (bell: Better Belle)	EC	4 (7)	0.075 0.076 0.076 0.074	73 [15 Aug]	0 . **14**	0.047, 0.033 (0.040) **0.024,** **0.013** **(0.018)**	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641 TK0058641-15 (Storage: 10 months)
United States, Wall, TX 2011, (bell: Camelot)	EC	4 (7)	0.075 0.076 0.076 0.074	89	0 . **14**	0.057, 0.062 **(0.060)** **0.032,** **0.040** **(0.036)**	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641 TK0058641-16 (Storage: 9.4 months)
United States, Arroyo Grande, CA 2011, (bell: Crusader)	EC	4 (7)	0.078 0.086 0.085 0.087	89	0 . **14**	0.11, 0.085, 0.12 (0.10) **0.041,** **0.041** **(0.041)**	<0.01 <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641 TK0058641-17 (Storage: 6.8 months)
	WG	4 (7)	0.076 0.084 0.084 0.085	89	0	0.11, 0.095, 0.082 (0.096)	<0.01 <0.01, <0.01; (<0.01)	
United States, Corning, CA 2011, (non-bell: Lamuyo #943)	EC	4 (7)	0.078 0.078 0.077 0.077	89	0 . **14**	0.38, 0.32 (0.35) **0.34,** **0.13** **(0.24)**	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641 TK0058641-21 (Storage: 10.2-11.2 months)

Chili peppers

Table 20 Residues in chili peppers from field trials in the United States (JMPR 2016)

CHILI PEPPERS Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Sanger, CA 2011, (bell: Grande Rio)	EC	4 (7)	0.078 0.079 0.078 0.079	89	0 1 **3** **7** **14** **21**	**0.37,** **0.27** **(0.32)** 0.36 **0.26** **0.22** **0.13,** **0.17** **(0.15)** **0.33**	<0.01, <0.01; (<0.01) <0.01, <0.01; <0.01, <0.01, <0.01; (<0.01) <0.01	TK0058641 TK0058641-18 (Storage: 8.6 months)
United States, Larned, KS 2011, (non-bell: Tam Hot Jalapeno)	EC	4 (7)	0.077 0.076 0.076 0.076	74	0 . **14**	0.065, 0.043 (0.054) **0.014,** **0.017** **(0.016)**	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641 TK0058641-19 (Storage: 8.9 months)
United States, Wall, TX 2011, (non-bell: Grande Jumbo Hybrid, Jalapeno)	EC	4 (7)	0.078 0.077 0.077 0.077	89 [8 Nov]	0 14	0.029, 0.047 **(0.038)** 0.066, 0.055 **(0.060)**	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641 TK0058641-20 (Storage: 9.6 months)

Tomatoes

Table 21 Residues in tomatoes from field trials in the United States (JMPR 2016)

TOMATO Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Alton, NY 2011, (POLBIG F1)	EC	4 (7)	0.076 0.076 0.076 0.076	86	0 . **14**	0.038, 0.041 (0.040) **0.029,** **0.027** **(0.028)**	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641-01 (Storage: 8.1 months)
United States, Jeffersonville, GA 2011, (Red Bounty)	EC	4 (7)	0.078 0.078 0.078 0.078	85	0 . **14**	0.051, 0.045, 0.064 (0.053) **0.022,** **0.027** **(0.024)**	<0.01 <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641-02 (Storage: 6.9 months)
	WG	4 (7)	0.075 0.075 0.075 0.074	85	0	0.050, 0.045, 0.039 **(0.045)**	<0.01 <0.01, <0.01; (<0.01)	

TOMATO Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Winter Garden, 2011, (Large Cherry)	EC	4 (7)	0.077 0.076 0.076 0.077	84	0 . **14**	0.40, 0.46 (0.43) **0.26,** **0.20** **(0.23)**	<0.01, <0.01 (<0.01) 0.014, <0.01 (0.012)	TK0058641-03 (Storage: 5.6 months)
United States, Hobe Sound, FL 2011, (FL47)	EC	4 (7)	0.077 0.075 0.076 0.077	77	0 . **14**	<0.01, <0.01 (<0.01) **<0.01,** **<0.01** **(<0.01)**	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641-04 (Storage: 4.8 months)
United States, Rice, MN 2011, (Arkansas Traveler)	EC	4 (7)	0.077 0.077 0.077 0.077	73	0 . **14**	0.046, 0.024, 0.070 (0.047) **0.042,** **0.033** **(0.038)**	<0.01 <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641-05 (Storage: 9.0 months)
	WG	4 (7)	**0.077** **0.077** **0.076** **0.076**	73	0	0.074, 0.043, 0.065 (0.061)	<0.01 <0.01, <0.01; (<0.01)	
United States, Porterville, CA 2011, (Roma VF)	EC	4 (7)	0.077 0.076 0.078 0.080	89	0 . **14**	0.073, 0.078, 0.27 (0.14) **0.098,** **0.059** **(0.078)**	<0.01 <0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641-06 (Storage: 8.4 months)
	WG	4 (7)	0.079 0.077 0.078 0.076	89	0	0.20, 0.086, 0.076 (0.12)	<0.01 <0.01, <0.01; (<0.01)	
United States, Paso Robles, CA 2011, (Galilea, Roma)	EC	4 (7)	0.076 0.077 0.076 0.077	86	0 . **14**	0.099, 0.071 (0.085) **0.040,** **0.049** **(0.044)**	<0.01, <0.01; (<0.01) <0.01, <0.01; (<0.01)	TK0058641-07 (Storage: 7.7 months)
United States, Kerman, CA 2011, (Roma)	EC	4 (7)	0.077 0.077 0.077 0.077	85	0 14	0.18, 0.18 (0.18) 0.15, 0.25 (0.20)	<0.01, <0.01 (<0.01) 0.016, 0.016 (0.016)	TK0058641-08 (Storage : 9.1 months)
United States, Visalia, CA 2011, (Romas)	EC	4 (7)	0.077 0.074 0.076 0.078	88	0 14	0.083, 0.094 **(0.088)** 0.11, 0.11	<0.01, <0.01; (<0.01) <0.01, <0.01;	TK0058641-09 (Storage: 8.5 months)

TOMATO Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
						(0.11)	(<0.01)	
United States, Sanger, CA 2011, (Quality 27)	EC	4 (7)	0.077 0.076 0.076 0.076	85 [17 Aug]	**0** **1** 3 **7** **14** **21**	**0.034,** **0.073** **(0.054)** **0.033** 0.066 **0.057** **0.043,** **0.066** **(0.054)** **0.028**	<0.01, <0.01; (<0.01) <0.01, <0.01; <0.01, <0.01, <0.01; (<0.01) <0.01	TK0058641-10
United States, Sanger, CA 2011, (Shasta)	EC	4 (7)	0.078 0.081 0.077 0.080	89 [1 Nov]	0	0.39, 0.36 (0.38)	<0.01, <0.01; (<0.01)	TK0058641-11 (Storage: 6.8-7.8 months)
	EC	4 (7)	0.38 0.38 0.38 0.38	89	0	2.1, 1.6 **(1.8)**	0.021, 0.014 **(0.018)**	
United States, Kettleman City CA 2011, (8004)	EC	4 (7)	0.076 0.077 0.077 0.077	89	0	0.052, 0.036 (0.044)	<0.01, <0.01; (<0.01)	TK0058641-12 (Storage: 9.6 months)
	EC	4 (7)	0.37 0.38 0.38 0.38	89	0	0.41, 0.24 **(0.32)**	<0.01, <0.01; (<0.01)	

Pulses

Residue information on dry bean seeds, dry pea seeds and dry soya bean seeds were reproduced from the JMPR 2014 and 2016 evaluations and extended with additional metabolite information.

Dry beans

Table 22 Residues in dry bean seeds from field trials in the United States and Canada (JMPR 2016)

DRY BEANS Location, Year (variety)	Form; no	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
United States Clarence, MO, 2011 (navy bean: HMS Medalist))	EC 2 (7)	0.075 0.075	R8/95	14	<0.01, <0.01, <0.01 (<0.01)	<0.01 <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01; (<0.01)	TK0058625 TK0058625-06 storage interval: 3.9 months
	WG 2 (7)	0.079 0.076	R8/95	14	<0.01, <0.01, 0.013 (0.011)	<0.01 <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01; (<0.01)	

DRY BEANS Location, Year (variety)	Form; no	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
United States Perley, ND 2011, (bean: Navigator)	EC 2 (7)	0.078 0.078	91	14	<0.01, <0.01, <0.01 (<0.01)	<0.01 <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01; (<0.01)	TK0058625 TK0058625-07 storage interval: 4.4 months
	WG 2 (7)	0.078 0.078	91	14	<0.01, <0.01, <0.01 (<0.01)	<0.01 <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01; (<0.01)	
United States York, NE, 2011, (bean: Marquis- GT)	EC 2 (7)	0.076 0.076	81	14	0.040, 0.048 (0.044)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0058625 TK0058625-08 storage interval: 4.8 months
United States Campbell, MN, 2011, (navy bean: HMS Medalist)	EC 2 (7)	0.076 0.076	79	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0058625 TK0058625-09 storage interval: 4.7 months
United States Gardner, ND, 2011, (bean: Maverick)	EC 2 (7)	0.078 0.077	95	0 7 14 21	0.019 <0.01 <0.01, <0.01 (<0.01) <0.01	<0.01 <0.01, <0.01, <0.01; (<0.01) <0.01,	<0.01 <0.01, <0.01, <0.01; (<0.01) <0.01	TK0058625 TK0058625-10 storage interval: 4.3 months（ 14DALA）
United States Hinton, OK, 2011, (Dwarf Horticulture Taylor Bean)	EC 2 (7)	0.075 0.076	79	14	<0.01, <0.01, 0.011 (0.010)	<0.01 <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01; (<0.01)	TK0058625 TK0058625-11 storage interval: 1.8 months
	WG 2 (7)	0.076 0.077	79	14	<0.01, <0.01, <0.01 (<0.01)	<0.01 <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01; (<0.01)	
United States Wall, TX, 2011, (bean: Pinto III)	EC 2 (7)	0.076 0.075	85	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0058625 TK0058625-12 storage interval: 2.7 months
United States Madera, CA, 2011, (bean: UC-8537)	EC 2 (7)	0.078 0.078	79	14	0.033, 0.056 **(0.044)**	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0058625 TK0058625-13 storage interval: 5.6 months
United States American Falls, ID, 2011, (bean: Pinto)	EC 2 (7)	0.076 0.074	77	14	0.018, 0.021 (0.020)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0058625 TK0058625-14 storage interval: 4.4 months
Canada St-Marc-Sur- Richelieu, QC, 2011, (bean: Hooter)	EC 2 (7)	0.075 0.074	78	3 7 14	<0.01 <0.01 0.011 <0.01 **(0.010)**	<0.01, <0.01; <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01, <0.01; (<0.01)	CER05904/11 T950 storage interval: 5.5-6.1 months

DRY BEANS Location, Year (variety)	Form; no	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
				21	0.010	<0.01,	<0.01	
Canada St-Marc-Sur-Richelieu, QC, 2011, (bean: Etna)	EC 2 (7)	0.076 0.074	78	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	CER05904/11 T951 storage interval: 5.8 months
	WG 2 (7)	0.074 0.078	78	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	CER05904/11 T951 storage interval: 5.8 months
Canada Vanscoy, SK, 2011, (bean: AC Pintoba)	EC 2 (7)	0.075 0.075	81	15	0.066, 0.089 (0.078)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	CER05904/11 T952 storage interval: 4.9-5.2 months
	WG 2 (7)	0.23 0.23	81	15	**0.23, 0.24 (0.23)**	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	
Canada Taber, AB, 2011, (bean: Great Northern)	EC 2 (7)	0.076 0.076	88	15	0.010, <0.01 (0.010)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	CER05904/11 T953 storage interval: 5.1-5.3 months
	WG 2 (7)	0.23 0.23	88	15	0.02, 0.012 (0.016)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	
Canada Minto, MB, 2011, (bean: Pintabo)	EC 2 (7)	0.075 0.075	81	16	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	CER05904/11 T954 storage interval: 5.6 months

Dry soya beans

Table 23 Residues in dry soya bean seeds from field trials in the United States and Brazil (JMPR 2014, JMPR 2016)

DRY SOYA BEANS Location, year, (Variety) Soil type	Form No (Interval days)	kg ai/ha	GS, date (last appl)	DALT	parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
United States Elko, SC, 2010, (Asgrow 7502)	EC 2 (7)	0.076 0.076	95	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 E11-0421 storage interval: 7.1 months
United States Seven Springs, NC, 2010 (AG5605)	EC 2 (7)	0.076 0.076	89	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 E10-0422 storage interval: 7.9 months
United States Cheneyville, LA,	EC 2 (7)	0.075 0.076	95 [1 Sept]	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 E17-0423 storage interval:

DRY SOYA BEANS Location, year, (Variety) Soil type	Form No (Interval days)	kg ai/ha	GS, date (last appl)	DALT	parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
2010, (Pioneer 94M80)								9.4 months
United States Cheneyville, LA, 2010, (Asgrow 5335)	EC 2 (7)	0.078 0.075	97 [4 Oct]	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 E17-0424 storage interval: 8.3 months
United States Pollard, AR, 2010, (Pioneer 94M80)	EC 2 (7)	0.076 0.076	88	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C23-0425 storage interval: 8.2 months
United States Northwood, ND, 2010, (90Y41)	EC 2 (7)	0.077 0.077	85	0 7 14 21 28	<0.01 <0.01 <0.01 , <0.01 (<0.01) <0.01 <0.01	<0.01 <0.01, <0.01, <0.01; (<0.01) <0.01, <0.01;	<0.01 <0.01, <0.01, <0.01; (<0.01) <0.01, <0.01	TK0002561 C13-0426 storage interval: 8.5 months
United States Sharon, ND, 2010, (90Y41)	EC 2 (7)	0.076 0.076	85	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C13-0427 storage interval: 8.6 months
United States Gardner, ND, 2010, (0509239)	EC 2 (7)	0.093 0.094	95	14	<0.01, 0.026 (0.018)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C03-0428 storage interval: 8.4 months
	EC 2 (7)	0.38 0.39	95	14	0.059, 0.079 (0.069)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	
United States Dudley, MO, 2010, (Asgrow 5403)	EC 2 (7)	0.077 0.077	86	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C23-0429 storage interval: 8.2 months
United States Fisk, MO, 2010, (Jake)	EC 2 (7)	0.076 0.077	87	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C23-0430 storage interval: 8.0 months
United States Fitchburg, WI, 2010, (S21-N6)	EC 2 (7)	0.076 0.076	95	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C08-0431 storage interval: 8.7 months
United States Bagley, IA, 2010, (P3Y13-N203)	EC 2 (7)	0.077 0.075	93	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C30-0432 storage interval: 8.5 months
	EC 2 (7)	0.38 0.39	93	14	0.012, <0.01 **(0.011)**	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	
United States Oregon, MO,	EC 2	0.077 0.081	R6-R7	14	<0.01, <0.01	<0.01, <0.01;	<0.01, <0.01;	TK0002561 C19-0433

DRY SOYA BEANS Location, year, (Variety) Soil type	Form No (Interval days)	kg ai/ha	GS, date (last appl)	DALT	parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
2010, (Pioneer 93Y70)	(7)				(<0.01)	(<0.01)	(<0.01)	storage interval: 8.5 months
United States York, NE, 2010, (93Y12)	EC 2 (7)	0.076 0.076	97	14	<0.01, 0.017, <0.01, 0.012, <0.01, <0.01 (0.012)	<0.01, <0.01; <0.01, <0.01; <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01; <0.01, <0.01, <0.01; (<0.01)	TK0002561 C33-0434 storage interval: 8.6-8.8 months
United States Lesterville, SD, 2010, (Latham, L2560R, LS-0991236)	EC 2 (7)	0.076 0.075	81	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C16-0435 storage interval: 8.8 months
United States Marysville, OH, 2010, (SG-329-RR)	EC 2 (7)	0.077 0.077	87	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C01-0436 storage interval: 8.4 months
United States Clarence, MO, 2010, (Asgrow 3803 RR)	EC 2 (7)	0.076 0.077	R7	0 7 14 21 28	0.12 0.032 0.011, 0.011 (0.011) <0.01 <0.01	<0.01 <0.01, <0.01; (<0.01) <0.01, <0.01	<0.01 <0.01, <0.01; (<0.01) <0.01, <0.01;	TK0002561 C20-0437 storage interval: 8.6 months (14DALA)
United States Richland, IA, 2010, (Pioneer 92Y80)	EC 2 (7)	0.076 0.075	81	14	0.077, 0.051 (0.064)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C18-0438 storage interval: 8.5 months
United States Campbell, MN, 2010, (AG 0808)	EC 2 (7)	0.076 0.076	93	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C11-0439 storage interval: 9.0 months
United States Geneva, MN, 2010, (Pioneer 91Y70)	EC 2 (7)	0.077 0.076	85	14	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	TK0002561 C09-0440 storage interval: 9.2 months
Holambra, SP, Brazil, 2010-2011, (CD 214 RR) Soil: Sa/Si/Cl= 52.0/12.5/35.5	EC 3 (19, 14)	0.030 0.030 0.030	BBCH 76- 78, 30-Mar	21 28 35	<0.01 0.020 0.020	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11063; M11063-DMO1
	EC 3 (19, 14)	0.045 0.045 0.045	BBCH 76- 78; 30-Mar	21 28 35	<0.01 <0.01 0.020	<0.01 <0.01 <0.01	<0.01 <0.01 0.010	M11063; M11063-DMO1 (c)
Holambra,	EC	0.030	BBCH 76-	21	<0.01	<0.01	<0.01	M11075;

DRY SOYA BEANS Location, year, (Variety) Soil type	Form No (Interval days)	kg ai/ha	GS, date (last appl)	DALT	parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
SP, Brazil, 2010-2011, (CD214 RR) Soil: Sa/Si/Cl= 52.0/12.5/35.5	3 (19, 14)	0.030 0.030	78, 30-Mar	28 35	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	M11075-DMO3 (c)
	EC 3 (19, 14)	0.050 0.050 0.050	BBCH 76- 78, 30-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-DMO3 (c)
Holambra, SP, Brazil, 2010-2011, (CD214 RR) Soil: Sa/Si/Cl= 52.0/12.5/35.5	WG 3 (19, 14)	0.030 0.030 0.030	BBCH 76- 78, 30-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-DMO1 (c)
	WG 3 (19, 14)	0.045 0.045 0.045	BBCH 76- 78, 30-Mar	21 28 35	0.030 <0.01 0.010	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-DMO1 (c)
Bandeirantes, PR, Brazil, 2010-2011, (BMX Potencia RR) Soil: Sa/Si/Cl= 2.0/18.0/80.0	EC 3 (22, 14)	0.030 0.030 0.030	BBCH 79, 1-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11063; M11063-DMO2
	EC 3 (22, 14)	0.045 0.045 0.045	BBCH 79, 1-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11063; M11063-DMO2
Bandeirantes, PR, Brazil, 2010-2011, (BMX Potencia RR) Soil: Sa/Si/Cl= 2.0/18.0/80.0	EC 3 (22, 14)	0.030 0.030 0.030	BBCH 79, 1-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-DMO2
	EC 3 (22, 14)	0.050 0.050 0.050	BBCH 79, 1-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-DMO2
Bandeirantes, PR, Brazil, 2010-2011, (BMX Potencia RR) Soil: Sa/Si/Cl= 2.0/18.0/80.0	WG 3 (22, 14)	0.030 0.030 0.030	BBCH 79, 1-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-DMO2
	WG 3 (22, 14)	0.045 0.045 0.045	BBCH 79, 1-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-DMO2
Uberlândia, MG, Brazil, 2010-2011, (SYN 9070 RR) Soil: Sa/Si/Cl= 15.1/15.5/69.3	EC 3 (22, 14)	0.030 0.030 0.030	BBCH 80, 9-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11063; M11063-JJB1
	EC 3 (22, 14)	0.045 0.045 0.045	BBCH 80, 9-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11063; M11063-JJB1
Uberlândia, MG, Brazil, 2010-2011, (SYN 9070 RR)	EC 3 (20, 14)	0.030 0.030 0.030	BBCH 80, 9-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-JJB1

DRY SOYA BEANS Location, year, (Variety) Soil type	Form No (Interval days)	kg ai/ha	GS, date (last appl)	DALT	parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
Soil: Sa/Si/Cl= 15.1/15.5/69.3	EC 3 (20, 14)	0.050 0.050 0.050	BBCH 80, 9-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-JJB1
Uberlândia, MG, Brazil, 2010-2011, (SYN 9070 RR) Soil: Sa/Si/Cl= 15.1/15.5/69.3	WG 3 (22, 14)	0.030 0.030 0.030	BBCH 80, 9-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-JJB1
	WG 3 (22, 14)	0.045 0.045 0.045	BBCH 80, 9-Mar	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-JJB1
Engenheiro Coelho, SP, Brazil, 2010-2011, (Vallosa) Soil: Sa/Si/Cl= 58.0/8.5/33.5	EC 3 (59, 14)	0.030 0.030 0.030	BBCH 73- 74, 25-Apr	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11063; M11063-AMA
	EC 3 (59, 14)	0.045 0.045 0.045	BBCH 73- 74, 25-Apr	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11063; M11063-AMA
Engenheiro Coelho, SP, Brazil, 2010-2011, (Valiosa) Soil: Sa/Si/Cl= 44.0/13.8/42.2	EC 3 (59, 14)	0.030 0.030 0.030	BBCH 73- 74, 25-Apr	14 21 28	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-AMA
	EC 3 (59, 14)	0.050 0.050 0.050	BBCH 73- 74, 25-Apr	14 21 28	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-AMA
Engenheiro Coelho, SP, Brazil, 2010-2011 (Valiosa) Soil: Sa/Si/Cl= 44.0/13.8/42.2	WG 3 (59, 14)	0.030 0.030 0.030	BBCH 73- 74, 25-Apr	14 21 28	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-AMA
	WG 3 (59, 14)	0.045 0.045 0.045	BBCH 73- 74, 25-Apr	14 21 28	<0.01 <0.01 0.010	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-AMA
Rio Verde, GO, Brazil, 2010-2011, (Valiosa) Soil: Sa/Si/Cl= 42.5/8.8/48.7	EC 3 (23, 14)	0.030 0.030 0.030	BBCH 79, 24-Febr	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-JJB2 (c)
	EC 3 (23, 14)	0.050 0.050 0.050	BBCH 79, 24-Febr	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-JJB2 (c)
Rio Verde, GO, Brazil, 2010-2011, (Valiosa) Soil: Sa/Si/Cl= 42.5/8.8/48.7	WG 3 (23, 14)	0.030 0.030 0.030	BBCH 79, 24-Febr	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-JJB2 (c)
	WG 3 (23,	0.045 0.045 0.045	BBCH 79, 24-Febr	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-JJB2

DRY SOYA BEANS Location, year, (Variety) Soil type	Form No (Interval days)	kg ai/ha	GS, date (last appl)	DALT	parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
	14)							(c)
Rondonópolis, MT, Brazil, 2010-2011, (TMG 132) Soil: Sa/Si/Cl= 61.0/0.1/39.0	EC 3 (23, 14)	0.030 0.030 0.030	BBCH 79, 24-Febr	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-JJB3
	EC 3 (23, 14)	0.050 0.050 0.050	BBCH 79, 24-Febr	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11075; M11075-JJB3
Rondonópolis MT, Brazil, 2010-2011 (TMG 132) Soil: Sa/Si/Cl= 61.0/0.1/39.0	WG 3 (23, 14)	0.030 0.030 0.030	BBCH 79, 24-Febr	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-JJB3
	WG 3 (23, 14)	0.045 0.045 0.045	BBCH 79, 24-Febr	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11086; M11086-JJB3

Dry peas

Table 24 Residues in dry pea seeds from field trials in the United States and Canada (JMPR 2016)

DRY PEAS Location, Year (variety)	Form; no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 mg/kg	Report; Trial; (remarks)
United States Hinton, OK, 2011, (pea: Alaska)	EC 2 (7)	0.076 0.076	73	14	0.12, 0.10, 0.094 (0.11)	0.037, 0.027, 0.012 (0.025)	0.017, 0.021, 0.022 (0.020)	TK0058625 TK0058625-01 storage interval: 1.8-2.2 months
	WG 2 (7)	0.075 0.076	73	14	0.038, 0.023, 0.021 (0.027)	<0.01 <0.01 <0.01 (<0.01)	0.025, 0.022, 0.026 (0.024)	
United States American Falls, ID, 2011, (pea: Little Marvel)	EC 2 (7)	0.072 0.076	77	14	0.022, <0.01, 0.010 (**0.014**)	<0.01, <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01; (<0.01)	TK0058625 TK0058625-03 storage interval: 4.8 months
	WG 2 (7)	0.077 0.080	67	14	<0.01, <0.01, 0.023 (**0.014**)	<0.01 <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01; (<0.01)	
United States Jerome, ID, 2011, (SNO 112 0490N14) United States,	EC 2 (7)	0.078 0.076	78	0 7 14 21	<0.01 <0.01 <0.01 <0.01 (<0.01) <0.01	<0.01 <0.01 <0.01 <0.01 (<0.01) <0.01	<0.01 <0.01 <0.01 <0.01 (<0.01) <0.01	TK0058625 TK0058625-04 storage interval: 5.8-6.5 months

DRY PEAS Location, Year (variety)	Form; no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 mg/kg	Report; Trial; (remarks)
(field pea)								
United States Hillsboro, OR, 2011, (pea: Blue Bird)	EC 2 (7)	0.076 0.076	75	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0058625 TTK0058625-05 storage interval: 6.3 months
United States Madera, CA, 2011, (Dundale)	EC 2 (7)	0.076 0.076	89	14	<0.01, <0.01 <0.01 (<0.01)	<0.01 <0.01 <0.01 (<0.01)	<0.01 <0.01 <0.01 (<0.01)	TK0058625 TK0058625-15 storage interval: 1.5 months
	WG 2 (7)	0.078 0.079	45	14	0.028, 0.017, 0.027 **(0.024)**	<0.01, <0.01, <0.01; (<0.01)	<0.01 <0.01, <0.01; (<0.01)	
Canada Vanscoy, SK, 2011, (pea: CDC Bronco)	EC 2 (7)	0.075 0.074	82	15	<0.01, 0.097 **(0.054)**	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	CER05905/11 T955 storage interval: 5.6 months
	WG 2 (7)	0.23 0.23	82	15	0.039, 0.028 (0.034)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	
Canada Perdue, SK. 2011, (pea: CDC Bronco)	EC 2 (7)	0.075 0.074	79	15	0.028, 0.038 (0.033)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	CER05905/11 T956 storage interval: 5.3 months
Canada Minto, MB. 2011, (pea: CDC Golden)	EC 2 (7)	0.075 0.075	83	2 6 16 22	0.12 0.023 **<0.01** <0.01; **(<0.01)** <0.01	<0.01 <0.01 <0.01, <0.01; (<0.01) <0.01	<0.01 <0.01 <0.01, <0.01; (<0.01) <0.01,	CER05905/11 T957 storage interval: 5.3-6.2 months
Canada Boissevain, MB, 2011, (pea: CDC Meadow)	EC 2 (7)	0.076 0.074	81	16	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	CER05905/11 T958 storage interval: 5.5-5.6 months
	WG 2 (7)	0.23 0.23	81	16	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	
Canada Rosthern, SK 2011 (pea: Meadow）	EC 2 (7)	0.073 0.075	79	16	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	Report: CER05905/11 T959 storage interval: 5.6 months
	WG 2 (7)	0.23 0.23	79	16	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	<0.01, <0.01; (<0.01)	

Potatoes

Residue information in potatoes was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information.

Table 25 Residues in potato from field trials in the United States (JMPR 2016)

POTATO Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Alton, NY 2011, (Reba)	EC	IF 4F (7)	IF 0.10 F 0.077 F 0.076 F 0.076 F 0.076	47	14	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-01 (Storage: 7.1 months)
	WG	IF 4F (7)	IF 0.10 F 0.076 F 0.076 F 0.076 F 0.076	47	14	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	
United States, North Rose, NY 2011, (Genesee)	EC	IF 4F (7)	IF 0.10 F 0.078 F 0.078 F 0.078 F 0.078	79	14	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-02 (Storage: 8.5 months)
United States, Jeffersonville, GA **2012,** (Red Pontiac)	EC	IF 4F (7)	IF 0.10 F 0.078 F 0.078 F 0.077 F 0.077	93	14	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-03 (Storage: 1.7 months)
	EC	4F (7)	F 0.079 F 0.077 F 0.076 F 0.078	93	14	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	
United States, Oviedo, FL **2011-2012,** (Red LaSoda)	EC	IF 4F (7)	IF 0.098 F 0.078 F 0.077 F 0.077 F 0.077	49	14	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-04 (Storage: 3.7 months)
United States, York, NE 2011, (Russet Norkotah)	EC	IF 4F (7)	IF 0.099 F 0.077 F 0.077 F 0.077 F 0.075	75	14	<0.01, 0.010, <0.01 (0.010)	<0.01, <0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-06 (Storage: 7.6 months)
	WG	IF 4F (7)	IF 0.10 F 0.077 F 0.077 F 0.077 F 0.076	75	14	<0.01, <0.01, 0.010 (0.010)	<0.01, <0.01, <0.01 (<0.01)	
United States, Bagley, IA 2011, (Kennebec)	EC	IF 4F (7)	IF 0.098 F 0.077 F 0.076 F 0.071 F 0.076	75	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-07 (Storage: 8.6 months)
	EC	4F (7)	F 0.074 F 0.078 F 0.074 F 0.078	75	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
United States, Oregon, MO 2011, (Red	EC	IF 4F (7)	IF 0.11 F 0.079 F 0.080 F 0.080	49 [18 Aug]	14	0.011 0.017 (0.014)	<0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-05 (Storage: 8.3 months)

POTATO Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
Pontiac)			F 0.079				[c]	
United States, Oregon, MO 2011, (Yukon Gold)	EC	IF 4F (7)	IF 0.11 F 0.079 F 0.080 F 0.080 F 0.079	93 [4 Aug]	14	0.013, 0.012 **(0.013)**	<0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-08 (Storage: 8.8 months)
United States, Jerome, ID 2011, (Ranger Russet)	EC	IF 4F (7)	IF 0.10 F 0.077 F 0.076 F 0.077 F 0.077	47	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-09 (Storage: 8.5 months)
United States, Sanger, CA 2011, (Red La Soda)	EC	IF 4F (7)	IF 0.10 F 0.076 F 0.077 F 0.075 F 0.077	46	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-10 (Storage: 8.8 months)
United States, American Falls, ID 2011, (Russet Burbank)	EC	IF 4F (7)	IF 0.098 F 0.077 F 0.076 F 0.076 F 0.077	47	14	0.011, <0.01, <0.01 **(0.010)**	<0.01, <0.01 <0.01 (<0.01)	TK0058640 ; TK0058640-11 (Storage: 7.5 months)
	WG	IF 4F (7)	IF 0.099 F 0.076 F 0.082 F 0.077 F 0.078	47 [13 Sept]	14	**0.010**, 0.012, 0.018 **(0.013)**	<0.01, <0.01 <0.01 (<0.01)	
United States, American Falls, ID 2011, (Dark Red Norland)	EC	IF 4F (7)	IF 0.097 F 0.083 F 0.077 F 0.078 F 0.072	49 [30 Aug]	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-12 (Storage: 8.0 months)
United States, American Falls, ID 2011, (Norkotah)	EC	IF 4F (7)	IF 0.097 F 0.072 F 0.077 F 0.077 F 0.075	49	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-13 (Storage: 7.7 months)
	EC	4F (7)	F 0.080 F 0.076 F 0.082 F 0.076	49 [6 Sept]	14	0.011, <0.01 **(0.010)**	<0.01, <0.01 (<0.01)	
United States, Ephrata, WA 2011, (Umatilla)	EC	IF 4F (7)	IF 0.099 F 0.076 F 0.076 F 0.076 F 0.076	47	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0058640 ; TK0058640-14 (Storage: 8.1 months)
United States, Rupert, ID 2011, (Russet Burbank)	EC	IF 4F (7)	IF 0.10 F 0.076 F 0.074 F 0.076 F 0.076	96 [31 Aug]	**0** **7** **14** **21** **29**	**<0.01,** **0.017** **0.012,** **0.012** **0.015**	<0.01, <0.01 <0.01 <0.01, <0.01	TK0058640 ; TK0058640-15 (Storage: 7.7 months)
United States, Rupert, ID	EC	IF 4F	IF 0.10 F 0.078	77	**13**	<0.01, <0.01	<0.01, <0.01	TK0058640 ;

POTATO Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
2011, (Russett Burbank)		(7)	F 0.081 F 0.077 F 0.077	[31 Aug]		(<0.01)	(<0.01)	TK0058640-16 (Storage: 7.9-8.5 months)
	EC	**IF 4F (7)**	**IF 0.510 F 0.376 F 0.376 F 0.376 F 0.376**	**77 [31 Aug]**	**13**	**0.039, 0.046 0.043, <0.01 0.015, <0.01 (0.027)**	**<0.01, <0.01 <0.01, <0.01 <0.01, <0.01 (<0.01)**	

Notes:

IF = Single in-furrow application; 4F = four foliar applications

Cereals

Residue information on barley grain, wheat grain, maize grain and sweet corn on the cob was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information.

Barley

Table 26 Residues in barley grain from field trials in the United States and Canada (JMPR 2016)

BARLEY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Germansville, PA 2010, (Nomini)	EC	2 (14	0.077 0.076	BBCH 71	**52**	0.30, 0.58, 0.65, 0.50, 0.70, 0.51 (0.54)	0.026, 0.027, 0.022, 0.026, 0.019, 0.030 (0.025)	TK0002559; E04-0381 (Storage: 9.0-12.7 months)
United States, Northwood, ND 2010, (Pinnacle)	EC	2 **(15)**	0.076 0.076	Feekes 10.5.2	**47**	0.013, 0.015 (0.014)	<0.01, <0.01 (<0.01)	TK0002559; C13-0382 (Storage: 6.8-7.1 months)
	EC	**2 (15)**	**0.379 0.382**	**Feekes 10.5.2**	**47**	**0.10, 0.10 (0.10)**	**<0.01, <0.01 (<0.01)**	
United States, Richland, IA **2011,** (winter barley Para-mount 66)	EC	2 **(12)**	0.076 0.076 **= ? 0.377 0.381**	Feekes 11.1	**26**	2.3, 3.1 (2.7)	0.091, 0.088 (0.090)	TK0002559; C18-0383 data not used due to mis-application
	EC	**2 (12)**	**0.377 0.381 = ? 0.076 0.076**	**Feekes 11.1**	**26**	**0.70, 0.70 (0.70)**	**0.014, 0.018 (0.016)**	

BARLEY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Clarence, MS 2010, (Lacey)	EC	2 **(11)**	0.074 0.077	BBCH 71	**23**	0.45, 0.54, 0.58, 0.62, 0.65, 0.70 (0.59)	0.029, 0.030, 0.039, 0.034, 0.040, 0.040 (0.035)	TK0002559; C20-0384 (Storage: 8.4-12.1 months)
United States, Jamestown, ND 2010, (Tradition)	EC	2 (14)	**0.078** 0.077	BBCH 71	**19** 26 **33** **40**	**0.46** 0.35, 0.30 (0.32) **0.31** **0.30**	<0.01 <0.01, <0.01 (<0.01) <0.01 <0.01	TK0002559; C12-0385 (Storage: 7.2 months)
United States, Grand Island, NE 2010, (Baronesse)	EC	2 **(13)**	0.077 0.076	BBCH 71	**16**	0.26, 0.27, 0.25, 0.27, 0.24 (0.26)	<0.01, <0.01 <0.01, <0.01 <0.01, (<0.01))	TK0002559; C33-0386 (Storage: 8.2-10.3 months)
United States, Carrington, ND 2010, **(spring barley: Pinnacle)**	EC	2 **(15)**	0.077 0.076	Feekes 10.5.4	**41**	0.028, 0.030 (0.029)	<0.01, <0.01 (<0.01)	TK0002559; C13-0387 (Storage: 7.2 months)
United States, Lake Andes, SD 2010, (Tradition)	EC	2 **(15)**	0.076 0.077	**BBCH 71-73**	**34**	0.29, 0.31 (0.30)	<0.01, <0.01 (<0.01)	TK0002559; C16-0388 (Storage: 7.3 months)
United States, Berthoud, CO 2010, (Coors 69)	EC	2 (14)	0.077 0.077	Feekes 10.5.4	**28**	0.069, 0.088 **(0.078)**	<0.01, <0.01 (<0.01)	TK0002559; W12-0389 (Storage: 8.1 months)
United States, Madera, CA 2010, (Recleaned Whole Barley)	EC	2 (14)	0.076 0.077	BBCH 71	**23**	0.089, 0.12 (0.10) ***	<0.01, <0.01 (<0.01)	TK0002559; W29-0390 (Storage: 8.9 months) *** Residue of 0.047 mg/kg parent in control sample; data not used
United States, Hermiston, OR 2010, (Radiant)	EC	2 **(7)**	0.076 0.077	BBCH 84	**29**	0.35, 0.36 (0.36)	<0.01, <0.01 (<0.01)	TK0002559; W21-0391 (Storage: 7.4 months)
United States, Jerome, ID 2010, (Foster)	EC	2 **(13)**	0.077 0.077	BBCH 71	**41**	0.059, 0.063 (0.061)	<0.01, <0.01 (<0.01)	TK0002559; W16-0392 (Storage: 7.5 months)
Canada Taber, AB 2011, (CDC Earl)	EC	2 (14)	0.079 0.079	**71-73**	22	0.88, 0.95 (0.92)	<0.01, <0.01 (<0.01)	CER05902/11; T929 (Storage: 2.8 months)
Canada	EC	2	0.074	**69-71**	41	0.16,	<0.01,	CER05902/11;

BARLEY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
Elgin, MB 2011, (AC Metcalfe)		(14)	0.075	[04-Aug]		0.13 (0.15)	<0.01 (<0.01)	T930 (Storage: 2.3 months)
Canada Elgin, MB 2011, (Tradition)	EC	2 **(13)**	0.075 0.074	**71-75** [04-Aug]	36	0.25, 0.33 (0.29)	<0.01, 0.01 (0.01)	CER05902/11; T932 (Storage: 2.4 months)
Canada Rosthern, SK 2011, (Metcalfe))	EC	2 (14)	0.079 0.076	**69-73**	35	0.12, 0.12 (0.12)	<0.01, <0.01 (<0.01)	CER05902/11; T933 (Storage: 2.8 months)
Canada Duck Lake, SK 2011, (Metcalfe)	EC	2 **(20)**	0.080 **0.078**	**69-72**	44	0.098, 0.093 (0.096)	<0.01, <0.01 (<0.01)	CER05902/11; T934 (Storage: 2.0 months)
Canada Fort Sask. AB 2011, (Coalition)	EC	2 **(8)**	0.075 0.075	**69-71**	37	0.38, 0.46 (0.42)	<0.01, <0.01 (<0.01)	CER05902/11; T935 (Storage: 2.0 months)
Canada Wellwood, MB 2011, (Conlon)	EC	2 (14)	0.072 0.075	71	34	0.23, 0.19 (0.21)	<0.01, <0.01 (<0.01)	CER05902/11; T936 (Storage: 3.0 months)
Canada Minto, MB 2011, (Copeland)	EC	2 (14)	0.075 0.074	**71-73** [25-Jul]	25	0.12, 0.11 (0.12)	<0.01, <0.01 (<0.01)	CER05902/11; T931 (Storage: 3.1 months)
Canada Minto, MB 2011, (Legacy)	EC	2 **(15)**	0.075 0.075	**71-73** [21-Jul]	**21** **29** **35** 43	**0.12** **0.14,** **0.14** **(0.14)** **0.16** 0.17	<0.01, <0.01, <0.01 (<0.01) <0.01, <0.01	CER05902/11; T937 (Storage: 3.0 months)

Wheat

Table 27 Residues in wheat grain from field trials in the United States and Canada (JMPR 2016)

WHEAT Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
United States, Seven Springs, NC 2010, (Pioneer 26R15)	EC	2 **(15)**	**0.077** 0.078	71	**21**	0.037, 0.044 **(0.040)**	<0.01, <0.01 (<0.01)	TK0002558 E10-0351 (Storage: 9.6 months)
United States, Shelbyville, MO 2010, **(soft red winter wheat: Erine)**	EC	2 **(16)**	**0.079** 0.077	Feekes 10.5	**38**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002558 C20-0354 (Storage: 9.0 months)

WHEAT Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
United States, Richland, IA 2010, (**soft red winter wheat:** Wilcross748)	EC	2 (14)	0.076 0.076	Feekes 10.5.4	**10**	0.025, 0.044 (**0.034**)	<0.01, <0.01 (<0.01)	TK0002558 C18-0355 (Storage: 8.9 months)
United States, Milford Center, OH 2010, (Croplan Genetics 8614)	EC	2 **(13)**	0.077 0.077	**Feekes 10.5.4**	**14**	0.041, 0.050, (0.046)	<0.01, <0.01 (<0.01)	TK0002558 C01-0356 (Storage: 8.8 months)
	EC	2 (14)	0.077 0.077	**Feekes 5 + 14 days**	**35**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
United States, Macon, MO 2010, (**soft red winter wheat**: V9710)	EC	2 **(16)**	0.077 0.077	Feekes 10.5.1	**41**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002558 C20-0357 (Storage: 9.1 months)
United States, Carrington, ND 2010, (**soft white s**pring **wheat:** AP-604-CL)	EC	2 **(11)**	0.076 0.075	Feekes 10.5.4	**41**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002558 C13-0359 (Storage: 7.4 months)
	EC	2 **(13)**	**0.077** **0.077**	**BBCH 45**	**52**	<0.01, 0.015 (**0.012**)	<0.01, <0.01 (<0.01)	
United States, Carrington, ND 2010, (**hard red spring wheat**: Faller)	EC	2 **(11)**	0.077 0.075	Feekes 10.5.4	**41**	<0.01, <0.01, (<0.01)	<0.01, <0.01 (<0.01)	TK0002558 C13-0361 (Storage: 6.9-7.4 months)
	EC	2 **(13)**	0.077 **0.077**	**BBCH 45**	**52**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
United States, Jamestown, ND 2010, (**wheat: durum**: variety not known))	EC	2 (14)	0.077 0.076	71	**28** **36** **42** **49**	0.016 0.014, 0.017 (0.016) 0.010 0.021	<0.01 <0.01 <0.01 (<0.01) <0.01 <0.01	TK0002558 C12-0360 (Storage: 5.8-6.4 months)
	EC	2 (14)	0.077 0.076	**33**	**59** **67** **73** **80**	<0.01 <0.01 <0.01 (<0.01), <0.01 <0.01	<0.01 <0.01 <0.01 (<0.01) <0.01 <0.01	
United States, Lake Andes, SD 2010, (**hard white spring wheat**: Argent)	EC	2 **(15)**	0.076 0.076	**71-73**	**34**	0.026, 0.012, (0.019)	<0.01, <0.01 (<0.01)	TK0002558 C16-0362 (Storage: 7.5 months)
	EC	2 **(13)**	0.076 **0.075**	**59-63**	**44**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
United States, Grand Island, NE 2010,	EC	2 **(13)**	0.077 0.077	71	**31**	0.014, 0.015, (**0.014**)	<0.01, <0.01 (<0.01)	TK0002558 C33-0363 (Storage: 8.6 months)

WHEAT Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
(hard red winter wheat: NE 01643)								
	EC	2 (14)	0.077 0.077	**30**	**57**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
United States, Johnstown, CO 2010, (**winter wheat**: Yuma)	EC	2 **(15)**	0.078 0.077	Feekes 10.5.4	**26**	0.046 0.087 **(0.066)**	<0.01, <0.01 (<0.01)	TK0002558 W12-0364 (Storage: 8.4-8.9 months)
United States, Eaton, CO 2010, (**winter wheat: Jagalene**)	EC	2 (14)	0.076 0.078	Feekes 10.5.4	**23** **30** **37** **41**	0.037 0.017, 0.023 (0.020) 0.041 0.040	<0.01 <0.01, <0.01 (<0.01) <0.01, <0.01	TK0002558 W12-0365 (Storage: 8.2-8.9 months)
United States, Milliken, CO 2010, (**hard red winter wheat:** Bill Brown)	EC	2 **(15)**	0.078 0.076	Feekes 10.5.4	**26**	0.086, 0.060 **(0.073)**	<0.01, <0.01 (<0.01)	TK0002558 W12-0369 (Storage: 8.9 months)
United States, Rupert, ID 2010, (**hard white spring wheat:** Klassic)	EC	2 (14)	0.076 **0.078**	71	**39**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002558 W15-0370 (Storage: 7.9 months)
United States, Fisk, MO **2011,** (**soft red winter wheat**: Beretta)	EC	2 **(16)**	0.076 0.076	73	**18**	0.069, 0.013 (0.041)	<0.01, <0.01 (<0.01)	TK0002558 C23-0352 (Storage: 5.4-8.5 months)
United States, Raymondville, TX **2011,** (**hard red winter wheat**: Caudillo)	EC	2 (14)	**0.079** **0.079**	Feekes 10.5.4	**44**	0.022, 0.018 (0.020)	<0.01, <0.01 (<0.01)	TK0002558 W08-0358 (Storage: 6.1 months)
United States, Uvalde, TX **2011,** (**hard red winter wheat**: Tam 203)	EC	2 **(12)**	0.075 0.075	71	**34**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002558 W07-0366 (Storage: 5.5 months)
	EC	**2** **(12)**	**0.378** **0.378**	**71**	**34**	**0.41,** **0.42** **(0.42)**	0.017, 0.016 (0.016)	
United States, Wall, TX **2011,** (**hard red winter wheat:** Coronado)	EC	2 **(12)**	0.077 **0.078**	**BBCH 71** **Feekes** **10.5.4**	**37**	0.012, 0.012 (0.012)	<0.01, <0.01 (<0.01)	TK0002558 W40-0367 (Storage: 5.2 months)
United States, Levelland, TX **2011,** (**hard red winter wheat**: Weather	EC	2 (14)	0.075 **0.076**	Feekes 10.5.4	**35**	0.042, 0.076 (0.059)	<0.01, <0.01 (<0.01)	TK0002558 W39-0368 (Storage: 5.3 months)

WHEAT Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
master)								
United States, Valley City, ND 2011, (**winter wheat:** Falcon)	EC	2 (14)	0.079 0.076	73	**23**	0.026, 0.017, 0.019 **(0.022)**	<0.01 <0.01, <0.01 (<0.01)	TK0048907 TK048907-01 (Storage: 5.8-6.4 months)
	WG	2 (14)	0.077 0.077	73	**23**	0.024, 0.025, 0.020 (0.023)	<0.01 <0.01, <0.01 (<0.01)	
United States, Jamestown, ND 2011, (**winter wheat**: Overland)	EC	2 (14)	0.077 0.077	73	**22**	0.024, 0.021, 0.035 (0.027)	<0.01 <0.01, <0.01 (<0.01)	TK0048907 TK048907-02 (Storage: 4.9 months)
	WG	2 (14)	0.077 0.077	73	**22**	0.027, 0.010, 0.011 (0.016)	<0.01 <0.01, <0.01 (<0.01)	
United States, Northwood, ND **2011,** **(hard red winter wheat**: Jerry)	EC	2 (14)	0.077 **0.079**	Feekes 10.5.4 **[24 Jun]**	**41**	<0.01, <0.01, (<0.01)	<0.01, <0.01 (<0.01)	TK0002558 C13-0353 (Storage: 3.7 months)
	EC	2 **(16)**	**0.080** 0.078	**33** **[4 Jun]**	**61**	<0.01, <0.01 **(<0.01)**	<0.01, <0.01 (<0.01)	
	EC	**2** **(14)**	**0.381** **0.381**	**Feekes 10.5.4** **[24 Jun]**	**41**	**0.026,** **0.030** **(0.028)**	<0.01, <0.01 (<0.01)	
United States, Northwood, ND 2011, (**hard red spring wheat:** Faller)	EC	2 (14)	0.076 0.077	71 **[20 Jul]**	**33**	<0.01, <0.01, <0.01 (<0.01)	<0.01 <0.01, <0.01 (<0.01)	TK0048907 TK048907-03 (Storage: 4.2 months)
	WG	2 (14)	0.077 0.076	71 **[20 Jul]**	**33**	<0.01, <0.01, <0.01 (<0.01)	<0.01 <0.01, <0.01 (<0.01)	
Canada Vanscoy, SK 2011, (Infinity))	EC	2 (**13**) **0.074**	0.074	**69-71**	36	0.023, 0.038 (**0.030**)	<0.01, <0.01 (<0.01)	CER05901/11; T916 (Storage: **71 days)**
Canada Kinley, SK 2011, (Infinity)	EC	2 (**24**)	0.079 0.075	**69-71**	41	0.048, 0.036 (0.042)	<0.01, <0.01 (<0.01)	CER05901/11; T917 (Storage: **47 days)**
Canada Taber, AB 2011, (Superb)	EC	2 **(15)**	0.079 0.078	**67-71**	32	0.027, 0.027 (0.027)	<0.01, <0.01 (<0.01)	CER05901/11; T918 (Storage **37-67 days)**
Canada Boissevain, MB 2011, (**Spring wheat**: Harvest)	EC	2 **(13)**	0.076 0.075	**69-71** **[4 Aug]**	40	0.029, 0.022 (0.026)	<0.01, <0.01 (<0.01)	CER05901/11; T919 (Storage **54 days)**
Canada	EC	2	0.075	**69-71**	43	<0.01,	<0.01,	CER05901/11;

WHEAT Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
Boissevain, MB 2011, (**Spring wheat**: Kane)		**(13**	0.075	**[4 Aug]**		0.014 (0.012)	<0.01 (<0.01)	T920 (Storage **51 days)**
Canada Rosthern, SK 2011, (Infinity)	EC	2 (14)	0.071 0.072	**69-71**	52	<0.01, 0.012 (0.011)	<0.01, <0.01 (<0.01)	CER05901/11; T921 (Storage: **51 days)**
Canada Blaine Lake, SK 2011, (Infinity)	EC	2 **(15)**	0.073 0.076	**69-71**	54	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05901/11; T922 (Storage: **52 days)**
Canada Duck Lake, SK 2011, (Infinity)	EC	2 **(21)**	0.080 0.076	**69-71**	41	0.026, 0.037 (0.032)	0.011, 0.011 (0.011)	CER05901/11; T923 (Storage: **39-60 days)**
Canada Kipp, AB 2011, (Superb)	EC	2 **(15)**	0.077 0.077	**69-71**	42	0.021, 0.013 (0.017)	<0.01, <0.01 (<0.01)	CER05901/11; T924 (Storage: **39 days)**
Canada Alvena, SK 2011, (Goodeve – Ac Intrepid)	EC	2 (14)	0.078 0.071	**70-71**	28	0.025, 0.025 (0.025)	<0.01, <0.01 (<0.01)	CER05901/11; T925 (Storage: **68 days)**
Canada Fort Sask. AB, 2011, (Harvest)	EC	2 **(8)**	0.076 0.074	**69-71**	44	0.027, 0.023 (0.025)	<0.01, <0.01 (<0.01)	CER05901/11; T926 (Storage: **38 days)**
Canada Minto, MB 2011, (Superb)	EC	2 (14)	0.079 0.075	**71-73** **[2 Aug]**	**27** 35 . **42** **48**	**0.021** 0.014, 0.023 **(0.018)** **0.024** **0.022**	<0.01 <0.01, <0.01 (<0.01) <0.01, <0.01	CER05901/11; T927 (Storage: **48-68 days)**
Canada Minto, MB 2011, (**spring wheat**: AC Barrie)	EC	2 (14)	0.077 0.076	**69-71** **[2 Aug]**	**27** 35 . **42** **48**	**0.022** 0.041, 0.012 (0.026) **0.026** **0.013**	<0.01 <0.01, <0.01 (<0.01) <0.01, <0.01	CER05901/11; T928 (Storage: **48-69 days)**

Maize and popcorn

Table 28 Residues in maize and popcorn (grain) from field trials in the United States (JMPR 2016)

MAIZE GRAINS Location, Year (variety)	Form	No	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
United States, Germansville, PA 2010, (TA 290-11)	EC	4 (14)	0.080 0.078 0.086 0.077	R6 BBCH 89	7	<0.01, <0.01; (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; E04-0451 (Storage: 5.6 months)
United States, Athens, GA 2010, (32B10)	EC	4 (14)	0.076 0.076 0.075 0.075	86-88	7	<0.01, <0.01; (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; E12-0452 (Storage: 5.4-5.9 months)
United States, Gardner, ND 2010, (Int65D85R)	EC	4 (14)	0.076 0.077 0.077 0.076	96	**-3B** **2** 7 **12** **17**	**0.015** **0.019** 0.014, 0.015; (0.014) **0.012** **0.013**	<0.01 <0.01 <0.01 <0.01 (<0.01) <0.01 <0.01	TK0002562; C12-0453 (Storage: 4.8 months)
United States, Northwood, ND 2010, (DKC35-19/ A1002669)	EC	4 (14)	0.076 0.076 0.076 0.076	89	**-3B** **2** 7 **12** **17**	**0.028** **0.021** 0.016, 0.018 (0.017) **0.015** **0.016**	<0.01 <0.01 <0.01 <0.01 (<0.01) <0.01 <0.01	TK0002562; C13-0454 (Storage: 4.3 months)
United States, Fisk, MO 2010, (RL8950HB)	EC	4 (14)	0.076 0.076 0.076 0.077	BBCH 95 50 percent leaves changed colour	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; C23-0455 (Storage: 6.3 months)
United States, Oregon , MO 2010, (Pioneer 32T16)	EC	4 (14)	0.078 0.076 0.078 0.075	**late R5, just turning R6**	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; C19-0456 (Storage: 5.6 months)
United States, Fitchburg, WI 2010, (37Y12)	EC	4 (14)	0.076 0.077 0.076 0.075	96	7	<0.01, 0.019 (0.010)	<0.01, <0.01 (<0.01)	TK0002562; C08-0457 (Storage: 5.0 months)
United States, Bagley, IA 2010, (33D47)	EC	4 (14)	0.074 0.077 0.080 0.077	97	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; C30-0458 (Storage: 4.8 months)
United States Bolckow, MO 2010, (Mycogen2 K718)	EC	4 (14)	0.077 0.077 0.077 0.076	**late R5**	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; C19-0459 (Storage: 5.7 months)

MAIZE GRAINS Location, Year (variety)	Form	No	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
United States, Sharon, ND 2010, (DKC35-19/ A1002669)	EC	4 (14)	0.077 0.077 0.076 0.076	89	7	0.014, 0.016 (0.015)	<0.01, <0.01, (<0.01)	TK0002562; C13-0460 (Storage: 4.3 months)
United States, Lesterville, SD 2010, (Golden Harvest H-8254 3000 GT, var. 162X579 14WP917)	EC	4 (14)	0.076 0.077 0.077 0.077	92	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; C16-0461 (Storage: 5.2 months)
United States, Richwood, OH 2010, (DKC57-66 VT3/RR2)	EC	4 (14)	0.077 0.077 0.077 0.077	**85/87**	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562; C01-0462 (Storage: 4.7 months)
United States, Clarence, MO 2010, (Pioneer 33D49)	EC	4 (14)	0.076 0.077 0.082 0.078	**R6**	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; C20-0463 (Storage: 5.6 months)
United States, Osceola, NE 2010, (4947RB)	EC	4 (14)	0.077 0.076 0.076 0.077	89	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; C33-0464 (Storage: 5.1 months)
United States, Campbell, MN 2010, (DKC 38-89)	EC	4 (14)	0.076 0.076 0.076 0.076	85	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; C11-0465 (Storage: 5.4 months)
United States, Geneva, MN 2010, (Pioneer 38M60)	EC	4 (14)	0.078 0.076 0.075 0.076	R6 BBCH 89	7	<0.01, 0.019 (0.014)	<0.01 <0.01 (<0.01)	TK0002562; C09-0466 (Storage: 5.1 months)
United States, Perry, IA 2010, (P1162XR)	EC	4 (14)	0.075 0.078 0.076 0.080	97	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; C30-0467 (Storage: 4.8 months)
United States, York, NE 2010, (X723 14WP.0)	EC	4 (14)	0.076 0.076 0.076 0.077	89	7	0.017 <0.01 (0.014)	<0.01 <0.01 (<0.01)	TK0002562; C33-0468 (Storage: 5.2 months)

MAIZE GRAINS Location, Year (variety)	Form	No	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial (remarks)
	EC	**4 (14)**	**1.36 1.36 1.36 1.36**	**89**	**7**	**0.025 0.027 (0.026)**	<0.01 <0.01 (<0.01)	idem for processing
United States, Anabel, MO 2010, (33T57)	EC	4 (14)	0.074 0.075 0.074 0.080	**R6**	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; C20-0469 (Storage: 5.6 months)
United States, Raymondville, TX 2010, (HG284162)	EC	4 (14)	0.080 0.078 0.078 0.078	87 physio logical maturity	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; W08-0470 (Storage: 7.1 months)
United States, Levelland, TX 2010, (AP2504, Popcorn grain)	EC	4 (14)	0.076 0.075 0.075 0.077	**ripe grain**	7	<0.01, <0.01 (<0.01)	<0.01 <0.01 (<0.01)	TK0002562; W39-0471 (Storage: 5.9 months)
United States, Wall, TX 2011, (Hybrid 111RM GT/CB/LL/RW)	EC	**4 (8,6,6)**	**0.077 0.075 0.077 0.077**	**89 (29 Aug)**	**6**	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	**TK0058623; 01 (Storage: 191 days)**
	WG	**4 (8,6,6)**	**0.076 0.076 0.077 0.077**	**89 (29 Aug)**	**6**	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	
United States, Bagley, IA 2011, (111RM)	EC	**4 (7,7,7)**	**0.074 0.075 0.078 0.078**	**95 (5 Oct)**	**7**	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	**TK0058623; 02 (Storage: 153 days)**
	WG	**4 (7,7,7)**	**0.077 0.076 0.077 0.074**	**95 (5 Oct)**	**7**	<0.01, <0.01 (<0.01)	<0.01, <0.01; (<0.01)	
United States, Rice, MN 2011, (DKC 35-43)	EC	**4 (7,7,7)**	**0.076 0.076 0.077 0.077**	**96 (28 Sept)**	**7**	<0.01 <0.01 <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	**TK0058623; 03 (Storage: 160 days)**
	WG	**4 (7,7,7)**	**0.077 0.076 0.077 0.076**	**96 (28 Sept)**	**7**	**0.011, 0.011; 0.018 (0.013))**	<0.01, <0.01, <0.01; (<0.01)	

Notes:

Bold = data modified or not available in JMPR 2016

Sweet corn

Table 29 Residues in sweet corn ears from field trials in the United States (JMPR 2016)

SWEET CORN Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Germansville, PA 2010, (Extra Tender 274A)	EC	4 (7)	0.077 0.076 0.077 0.077	73	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562 E04-0472 (Storage: Ears 7.2 months)
United States, North Rose, NY 2010, (Serendipity)	EC	4 (7)	0.078 0.078 0.080 0.078	75	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562 E04-0473 (Storage: Ears 6.0 months)
United States, Athens, GA 2010, (Silver King)	EC	4 (7)	0.076 0.076 0.077 0.076	73	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562 E04-0474 (Storage: Ears 7.1 months)
United States, Oviedo, FL 2010, (Silver Queen)	EC	4 (7)	0.075 0.077 0.076 0.078	71	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562 E04-0475 (Storage: Ears 8.7 months)
United States, Gardner, ND 2010, (Zea Mays GH4927)	EC	4 (7)	0.077 0.076 0.077 0.076	75	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562 C12-0476 (Storage: Ears 6.4 months)
United States, Bagley, IA 2010, (Not listed)	EC	4 (7)	0.074 0.071 0.077 0.074	73	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562 C30-0477 (Storage: Ears 6.7 months)
United States, Oregon, MO 2010, (Bodacious)	EC	4 (7)	0.078 0.082 0.080 0.082	73	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562 C19-0478 (Storage: Ears 7.3 months)
United States, Centerville, SD 2011, (Kandy Korn)	EC	4 (7)	0.075 0.075 0.078 0.076	73	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562 C16-0479 (Storage: Ears 6.5 months)
United States, Clarence, MO 2010, (Incredible)	EC	4 (7)	0.077 0.077 0.076 0.076	85	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562 C20-0480 (Storage: Ears 7.2 months)
United States, Porterville, CA 2010, (Bodacious)	EC	4 (**7,5,6**)	0.076 0.076 0.076 0.076	99	**3DB 2** 7 **12 17**	**<0.01, <0.01** <0.01, <0.01 (<0.01) **<0.01, <0.01**	<0.01, <0.01 <0.01, <0.01 (<0.01) <0.01, <0.01	TK0002562 W32-0481 (Storage: Ears 7.5-8.2 months)
United States, Rupert, ID 2010, (Sugarbuns)	EC	4 (7)	0.072 0.076 0.076 0.080	77	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002562 W15-0482 (Storage: Ears 6.9 months)
United States,	EC	4	0.076	79	7	<0.01,	<0.01,	TK0002562

SWEET CORN Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
Hillsboro, OR 2010, (Honey and Pearls)		(7)	0.075 0.077 0.077			<0.01 (<0.01)	<0.01 (<0.01)	W21-0483 (Storage: Ears 6.0 months)
United States, Wall, TX 2011, (Hybrid 111RM GT/CB/LL/RW)	**EC**	**4 (7,8,8)**	**0.078 0.077 0.077 0.079**	**71 (23 Jul)**	7	<0.01, <0.01, <0.01, (<0.01)	<0.01, <0.01, <0.01 (<0.01)	**TK0058623; 01 (Storage: 5.9 months)**
United States, Bagley, IA 2011, (111RM)	EC	**4 (7,8,6)**	**0.075 0.073 0.076 0.080**	**73 (11 Aug)**	7	<0.01, <0.01, <0.01, (<0.01)	<0.01 <0.01, <0.01 (<0.01)	**TK0058623; 02 (Storage: 5.3 months)**
	WG	**4 (7,8,6)**	**0.077 0.077 0.076 0.078**	**73 (11 Aug)**	**7**	**<0.01, <0.01 (<0.01)**	<0.01, <0.01 (<0.01)	
United States, Rice, MN 2011, (DKC 35-43)	EC	**4 (7,7,7)**	**0.076 0.076 0.076 0.076**	**73 (24 Aug)**	7	<0.01, <0.01, <0.01, (<0.01)	<0.01 <0.01, <0.01 (<0.01)	**TK0058623; 03 (Storage: 4.9 months)**
	WG	**4 (7,7,7)**	**0.076 0.076 0.077 0.076**	**73 (24 Aug)**	**7**	**<0.01, <0.01, <0.01, (<0.01)**	<0.01 <0.01, <0.01 (<0.01)	

Bold = modified compared to JMPR 2016 evaluation

3DB = 3 days before the last application

Sugar cane

Residue information on sugar cane stalks was reproduced from the JMPR 2016 and JMPR 2019 evaluation and extended with additional metabolite information.

Table 30 Residues in sugar cane canes in field trials in the United States and Brazil (JMPR 2016 and 2019)

SUGAR CANE Location Year (variety)	Form	no (RTI)	Rate kg ai/ha	Growth stage at final appl	DALT	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report Trial No Reference Storage period
United States Oviedo, FL 2014 (1446)	EC	3 (14)	0.078 0.072 0.085	BBCH 3	29	0.10 0.10 (0.10)	<0.01 <0.01 (<0.01)	TK0161217-01 Hampton, 2016, Max. frozen storage: 11 months
	WG	2 (14)	0.077 0.078 0.081	BBCH 3	29	0.25 0.17 (0.21)	<0.01 <0.01 (<0.01)	
United States Okeechobee, FL		3 (14)	0.077 0.080	BBCH 4	20 25	0.078 0.056	<0.01 <0.01	TK0161217-02 Hampton, 2016,

SUGAR CANE Location Year (variety)	Form	no (RTI)	Rate kg ai/ha	Growth stage at final appl	DALT	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report Trial No Reference Storage period
2014 (CP731547K)			0.078		29 35 40	0.11 0.11 (0.11) 0.14 0.12	<0.01 <0.01 (<0.01) <0.01 <0.01	Max. frozen storage: 12 months
United States Hobe Sound, FL 2014 (CP881762K)		3 (14)	0.077 0.078 0.076	BBCH 4 (reaching crop maturity)	31	0.084 0.052 (0.068)	<0.01 <0.01 (<0.01)	TK0161217-03 Hampton, 2016, Max. frozen storage: 12 months
United States Washington, LA 2014 (LCP-85-384)	EC	3 (14)	0.076 0.080 0.077	130 to 140 inches	30	0.081 0.046 (0.064)	<0.01 <0.01 (<0.01)	TK0161217-04 Hampton, 2016, Max. frozen storage: 13 months
	WG	3 (14)	0.077 0.080 0.078	130 to 140 inches	30	0.16 0.089 **(0.12)**	<0.01 <0.01 (<0.01)	
United States Cheneyville, LA 2014 (HoCP540)		3 (14)	0.076 0.081 0.080	BBCH 8 (~16 node)	20 28 35 38	0.061 0.065 0.059 (0.062) 0.040 0.043	<0.01 <0.01 <0.01 (<0.01) <0.01 <0.01	TK0161217-05 Hampton, 2016, Max. frozen storage: 12 months
United States Morrow, LA 2014 (L01-299)		3 (14)	0.082 0.087 0.076	15 to 16 nodes	30	0.033 0.028 **(0.030)**	<0.01 <0.01 (<0.01)	TK0161217-06 Hampton, 2016, Max. frozen storage: 13 months
United States Raymondville, TX 2015 (1210)	EC	3 (12-16)	0.081 0.078 0.078	BBCH 39 (max stem length)	30	0.068 0.072 (0.070)	<0.01 <0.01 (<0.01)	TK0161217-07 Hampton, 2016, Max. frozen storage: 6.8 months
	WG	3 (12-16)	0.080 0.077 0.078	BBCH 39 (max stem length)	30	0.045 0.029 (0.037)	<0.01 <0.01 (<0.01)	
United States Puunene, HI 2014 (78-4135)		3 (14)	0.076 0.076 0.076	BBCH 4 Mature Stalks	31	<0.01 0.016 (0.013)	<0.01 <0.01 (<0.01)	TK0161217-08 Hampton, 2016, Max. frozen storage: 14 months
Brazil, Mirassol, **2010-2011,** (SP 84 2025)	EC	5 (30)	0.030 0.030 0.030 0.030 0.030	42 **[6 Apr]**	20 30 40	<0.01 <0.01 0.02	<0.01 <0.01 <0.01	M11013 M11013-AMA1 (Storage: 5 months)
Brazil, Mirassol, **2010-2011,** (SP 84 2025)	WG	5 (30)	0.030 0.030 0.030 0.030 0.030	42 **[6 Apr]**	20 30 40	<0.01 <0.01 0.02	<0.01 <0.01 <0.01	M11019 M11019-AMA1 (Storage: 5 months)
Brazil, Jaboticabal, **2010-2011,** (RB 5453)	EC	5 (30)	0.030 0.030 0.030 0.030 0.030	39 **[29 Mar]**	20 30 40	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11013 M11013-AMA2 (Storage: 5 months)
Brazil, Jaboticabal,	WG	5 (30)	0.030 0.030	39 **[29 Mar]**	20 30	<0.01 <0.01	<0.01 <0.01	M11019 M11019-AMA2

SUGAR CANE Location Year (variety)	Form	no (RTI)	Rate kg ai/ha	Growth stage at final appl	DALT	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report Trial No Reference Storage period
2010-2011, (RB 5453)			0.030 0.030 0.030		40	<0.01	<0.01	(Storage: 5 months)
Brazil, Jaboticabal, **2010-2011,** (SP 89-1115)	**EC**	5 (30)	0.090 0.090 0.090 0.090 0.090	**38-39** **[14 Apr]**	30	**0.02**	<0.01	M11007 M11007-AMA2 (Storage: 5 months)
	EC	5 (30)	0.150 0.150 0.150 0.150 0.150	**38-39** **[14 Apr]**	30	**0.07**	<0.01	
Brazil Bandeirantes, **2010-2011,** (RB 72454)	EC	5 (30)	0.030 0.030 0.030 0.030 0.030	39 **[4 May]**	20 30 40	0.02 0.02 0.02	<0.01 <0.01 <0.01	M11013 M11013-DMO (Storage: 5 months)
Brazil Bandeirantes, **2010-2011,** (RB 72454)	WG	5 (30)	0.030 0.030 0.030 0.030 0.030	39 **[4 May]**	20 30 40	0.02 0.02 0.02	<0.01 <0.01 <0.01	M11019 M11019-DMO (Storage: 5 months)
Brazil Tupaciguara, **2010-2011,** (SP 86155)	EC	5 (30)	0.030 0.030 0.030 0.030 0.030	38 **[1 Jun]**	20 30 40	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11013 M11013-JJB (Storage: 5 months)
Brazil Tupaciguara, **2010-2011,** (SP 86155)	WG	5 (30)	0.030 0.030 0.030 0.030 0.030	38 **[1 Jun]**	20 30 40	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11019 M11019-JJB (Storage: 5 months)
Brazil Rio das Pedras, **2010-2011,** (RB 85 7515)	EC	5 (30)	0.030 0.030 0.030 0.030 0.030	**47-48** **[19 Febr]**	20 30	0.02 <0.01	<0.01 <0.01	M11013 M11013- RWC1 (Storage: 5 months)
Brazil Rio das Pedras, **2010-2011,** (RB 85 7515)	WG	5 (30)	0.030 0.030 0.030 0.030 0.030	**47-48** **[19 Febr]**	20 30	0.02 0.02	<0.01 <0.01	M11019 M11019- RWC1 (Storage: 5 months)
Brazil Rio das Pedras, **2010-2011,** (RB 85 7515)	**EC**	5 (30)	0.090 0.090 0.090 0.090 0.090	**37-39** **[27 Apr]**	30	**0.03**	<0.01	M11007 M11007- RWC2 (Storage: 5 months)
	EC	5 (30)	0.15 0.15 0.15 0.15 0.15	**37-39** **[27 Apr]**	30	**0.05**	<0.01	

SUGAR CANE Location Year (variety)	Form	no (RTI)	Rate kg ai/ha	Growth stage at final appl	DALT	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report Trial No Reference Storage period
Brazil **Holambra,** **2010-2011,** **(RB 85 7515)**	EC	5 (30)	0.030 0.030 0.030 0.030 0.030	**47-48** **[19 Febr]**	20 30 40	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11013 M11013- RWC2 (Storage: 5 months)
Brazil **Holambra,** **2010-2011,** **(RB 85 7515)**	WG	5 (30)	0.030 0.030 0.030 0.030 0.030	**47-48** **[19 Febr]**	20 30 40	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11019 M11019- RWC2 (Storage: 5 months)
Brazil Holambra, **2010-2011,** (RB 85 7515)	**EC**	5 (30)	0.090 0.090 0.090 0.090 0.090	**38-39** **[26 Apr]**	30	**0.04**	<0.01	M11007 M11007- RWC1 (Storage: 5 months)
	EC	5 (30)	0.15 0.15 0.15 0.15 0.15	**38-39** **[26 Apr]**	30	**0.10**	<0.01	
Brazil Santa Lucia, **2010-2011,** (SP 81-3250)	**EC**	5 (30)	0.090 0.090 0.090 0.090 0.090	**38-39** **[15 Apr]**	30	**0.03**	<0.01	M11007 M11007- AMA1 (Storage: 5 months)
	EC	5 (30)	0.15 0.15 0.15 0.15 0.15	**38-39** **[15 Apr]**	30	**0.04**	<0.01	

Oilseeds

Residue information on rapeseed, cotton seeds and peanut nutmeat was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information.

Rapeseed

Table 31 Residues on rapeseed seed from field trials in Canada (JMPR 2016)

RAPE SEED Location Year (variety)	Form; no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
Canada Elm Creek, MB, 2011, (1841 RR)	EC 1	0.081	**69-73**	29	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T938 storage interval: 86 days
Canada Morden, MB,	EC 1	0.082	**67-69**	30	0.054, 0.070	<0.01, <0.01	<0.01, <0.01	CER05903/11 T938C

RAPE SEED Location Year (variety)	Form; no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
2011, (1841 RR)					(0.062)	(<0.01)	(<0.01)	storage interval: 90 days
Canada Kinley, SK, 2011, (1841 RR)	EC 1	0.076	**67-71 [2 Aug]**	30	0.021, 0.024 (0.023)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T939 storage interval: 80 days
Canada Kinley, SK, 2011, (72-55RR)	EC 1	0.076	**69-73 [4 Aug]**	29	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T940 storage interval: 67 days
Canada Elgin, MB, 2011, (72-55RR)	EC 1	0.076	68 **[16 Jul]**	30	0.11, 0.094 (0.10)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T941 storage interval: 93 days
Canada Elgin, MB, 2011, (1841 RR)	EC 1	0.074	68 **[17 Jul]**	31	0.027, 0.028, (0.028)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T948 storage interval: 96 days
	EC 1	**0.222**	**68 [17 Jul]**	**31**	**0.12**	0.011	<0.01	
Canada Blaine Lake, SK, 2011, (72-55RR)	EC 1	0.074	**78-79**	30	0.011, <0.01 (0.011)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T942 storage interval: 67 days
Canada Rosthern, SK, 2011, (1841 RR)	EC 1	0.076	**73-76 [5 Aug]**	31	0.013, <0.01 (0.012)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T943 storage interval: 75 days
Canada Rosthern, SK, 2011, (72-55RR)	EC 1	0.075	73-76 **[5 Aug]**	31	0.023, 0.014 (0.019)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T949 storage interval: 78 days
	EC 1	**0.230**	**73-76 [5 Aug]**	**31**	**0.13**	0.046	<0.01	
Canada Alvena, SK, 2011, (1841 RR)	EC 1	0.070	**65-66**	31	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T945 storage interval: 75 days
Canada Fort Sask. AB, 2011, (72-55RR)	EC 1	0.077	**67-71**	32	0.051, 0.039 (0.045)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T946 storage interval: 61 days
Canada Minto, MB, 2011, (72-55RR)	EC 1	**0.074**	67 **[18 Jul]**	35	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	CER05903/11 T944 storage interval: 88 days
Canada Minto, MB, 2011, (1841 RR)	EC 1	0.074	67 **[18 Jul]**	25 30 . . 35 40	0.046 0.033, 0.029 (0.031) 0.023 0.021	<0.01 <0.01, <0.01 (<0.01) <0.01 <0.01	<0.01 <0.01, <0.01 (<0.01) <0.01 <0.01	CER05903/11 T947 storage interval: 88-92 days

Cotton seed

No trials were selected for cotton seed.

Table 32 Residues in cotton seed from field trials in United States (JMPR 2016)

COTTON SEED Location Year (variety)	Form no (RTI)	kg ai/ha	BBCH	DA LA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
United States, Elko, SC 2010, (Delta Pine-Land 161)	EC 3 (14)	0.075 0.076 0.076	83	**47**	0.025, 0.035 (0.030)	<0.01; 0.013 (0.012)	<0.01; <0.01 (<0.01)	TK0025157 E11-0521 (Storage: 7.8 months)
United States, Cheneyville, LA, 2010, (Phytogen 375 WRF)	EC 3 (14)	0.077 0.077 0.078	77	**35** **40** 45 . . **50** **55**	**<0.01** **<0.01** <0.01, <0.01 (<0.01) **<0.01** **<0.01**	<0.01 0.012 <0.01, <0.01 (<0.01) <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 (<0.01) <0.01 <0.01	TK0025157 E17-0522 (Storage: **8.9** months)
United States, Fisk MS 2010, (DP 164 B2RF)	EC 3 (14)	0.077 0.076 0.077	77	**41**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0025157 C23-0523 (Storage: **8.5** months)
United States, Proctor, AR2010, (DyanGro 2400RF)	EC 3 (14)	0.076 0.076 0.076	85	**35** **40** **44** **.** **.** **50** **56**	**0.023** **<0.01** 0.018, 0.028 (0.023) <0.01 <0.01	0.021 <0.01 0.015 0.021 (0.018) <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 (<0.01) <0.01 <0.01	TK0025157 C24-0524 (Storage: **8.6** months)
United States, Batesville, TX, 2010, (DPL0949)	EC 3 (14)	0.075 0.076 0.078	76	**44**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0025157 W07-0525 (Storage: 9.6 months)
United States, Uvalde, TX 2010, (Stoneville 5458B2RF)	EC 3 (14)	0.076 0.076 0.074	78	**42**	0.019, 0.024 (0.022)	0.023, 0.033 (0.028)	<0.01, <0.01 (<0.01)	TK0025157 W07-0526 (Storage: 9.6 months)
	EC 3 (14)	**0.381 0.381 0.381**	**79**	**42**	**0.055 0.046 (0.050)**	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	
United States, Wall, TX 2010, (Fibermax 1740 B2F)	EC 3 (14)	0.076 0.076 0.075	84	**49**	0.028, 0.032 (0.030)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0025157 W40-0527 (Storage: Seed 13.1 months)
United States, Wolfforth, TX, 2010, (FM9058)	EC 3 (14)	0.078 0.077 0.075	**81**	**43**	0.070, 0.086 (0.078)	<0.01, 0.011 (0.010)	<0.01 <0.01 (<0.01)	TK0025157 W39-0528 (Storage: Seed 7.8 months)
United States, Levelland, TX, 2010, (FM9180B2F)	EC 3 (14)	0.078 0.076 0.076	89	**44**	0.081, 0.069 (0.075)	0.034 0.018 (0.026)	<0.01 <0.01 (<0.01)	TK0025157 W39-0529 (Storage: Seed **7.9** months)
	EC 3 (14)	**0.381 0.392 0.381**	**89**	**44**	**0.69**	0.14	<0.01	
United States,	EC	0.076	67	45	<0.01,	<0.01,	<0.01,	TK0025157

COTTON SEED Location Year (variety)	Form no (RTI)	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
Fresno, CA 2010, (PHY775 WRF ACALA)	3 (14)	0.077 0.076			<0.01 (<0.01)	<0.01 (<0.01)	<0.01 (<0.01)	W19-0530 (Storage: 8.6 months)
United States, Madera, CA 2010, (Acala RiataRR)	EC 3 (14)	0.078 0.077 0.077	72	**44**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0025157 W29-0531 (Storage: 8.3 months)
United States, Stratford, CA 2010, (DP 949B2RF)	EC 3 (14)	0.075 0.075 0.076	81	**44**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0025157 W33-0532 (Storage:8.5 months)
United States, Fisk, MO 2011, (DP 0912 B2RF)	EC 3 (14)	0.077 0.076 0.077	80	**46**	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	TK0058642 TK0058642-01 (Storage: 4.7 months)
	WG 3 (14)	0.077 0.076 0.076	80	**46**	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	
United States, Greenville, MS 2011, (ST 5458 B2RF)	EC 3 (14)	0.077 0.078 **0.076**	77	45	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	TK0058642 TK0058642-02 (Storage: 5.1 months)
	WG 3 (14)	0.078 0.077 0.077	77	45	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	
United States, Uvalde, TX 2011, (DPL 0935)	EC3 (14)	0.075 0.076 0.075	77	**43**	<0.01, <0.01, <0.01 (<0.01)	<0.01, 0.011 <0.01 (0.010)	<0.01, <0.01, <0.01 (<0.01)	TK0058642 TK0058642-03 (Storage: Seed 6.6 months)
	WG 3 (14)	0.076 0.077 0.076	77	**43**	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	
United States, Levelland, TX 2011, (FM9180 B2F)	EC 3 (14)	0.075 0.077 0.077	81	**48**	0.044, 0.047, 0.033 (0.041)	0.013 0.013 <0.01 (0.012)	<0.01, <0.01, <0.01 (<0.01)	TK0058642 TK0058642-04 (Storage: Seed 4.0 months)
	WG 3 (14)	0.076 0.077 0.076	81	**48**	0.030, 0.031, 0.029 (0.030)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	

Peanuts

Table 33 Residues in peanut nutmeat from field trials in Brazil and the United States (JMPR 2016)

PEANUT Location, Year (variety)	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
Brazil, Jaboticabal SP 2010-2011, (Runner IAC 886)	EC 4 (14)	0.050 0.050 0.050 0.050	76	7 **14** **21**	0.020 **<0.01** **<0.01**	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	A17961A M11082-AMA1
	WG 4 (14)	0.050 0.050 0.050 0.050	76	7 **14** **21**	<0.01 **<0.01** **<0.01**	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	**A181826B** **M11093-AMA1**
Brazil, Matão–SP 2010-2011, (Runner IAC 886)	WG 4 (14)	0.050 0.050 0.050 0.050	76	7 14 21	<0.01 0.020 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	A181826B M11093-AMA2
Brazil, Bálsamo- SP 2010-2011, (Runner IAC 886)	EC 4 (14)	0.050 0.050 0.050 0.050	77	7 **14** **21**	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	A17961A M11082-AMA3
	WG 4 (14)	0.050 0.050 0.050 0.050	77	7 **14** **21**	<0.01 **<0.01** **<0.01**	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	**A181826B** **M11093-AMA3**
Brazil, Engenheiro Coelho–SP 2010-2011, (Tatu Vermelho)	EC 4 (14)	0.050 0.050 0.050 0.050	71	7 **14** **21**	<0.01 **<0.01** **<0.01**	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	A17961A M11082-AMA4
	WG 4 (14)	0.050 0.050 0.050 0.050	71	7 14 21	<0.01 **<0.01** **<0.01**	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	**A181826B** **M11093-AMA4**
Brazil, Bandeirantes–PR 2010-2011, (Tatu)	EC 4 (14)	0.050 0.050 0.050 0.050	85	7 **14** **21**	<0.01 **<0.01** **<0.01**	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	A17961A M11082-DMO
	WG 4 (14)	0.050 0.050 0.050 0.050	85	7 **14** **21**	<0.01 **<0.01** **<0.01**	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	**A181826B** **M11093-DMO**
Brazil, Uberlândia–MG 2010-2011, (Tatu)	EC 4 (14)	0.050 0.050 0.050 0.050	88	7 **14** **21**	<0.01 **<0.01** **<0.01**	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	**A17961A** **M11082-JJB** **(Portuguese version only)**
	WG 4 (14)	0.050 0.050 0.050 0.050	88	7 **14** **21**	<0.01 **<0.01** **<0.01**	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	A181826B M11093-JJB
United States, Elko, SC 2010, (Gregory)	EC 3 (14)	0.10 0.10 0.10	79	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002560 E11-0401 (Storage: 8.2 months)
United States, Seven Springs,	EC 3	0.10 0.10	69	30	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	TK0002560 E10-0402

PEANUT Location, Year (variety)	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
NC 2010, (Perry)	(14)	0.10			(<0.01)	(<0.01)	(<0.01)	(Storage: 8.4 months)
United States, Hawkinsville, GA 2010, (Georgia 06-G)	EC 3 (14)	0.10 0.10 0.10	79	**23** 30 - - **37** **44**	**<0.01** <0.01, <0.01 (<0.01) **<0.01** **<0.01**	**<0.01** <0.01, <0.01 (<0.01) **<0.01** **<0.01**	**<0.01** <0.01, <0.01 (<0.01) **<0.01** **<0.01**	TK0002560 E12-0403 (Storage: 9.6 months)
United States, Suffolk, VA 2010, (Champs)	EC 3 (14)	0.10 0.10 0.10	75 **[22 Sept]**	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002560 E07-0404 (Storage: 7.3 months)
United States, Suffolk, VA 2010, (Champs)	EC 3 (14)	0.10 0.10 0.10	75 **[22 Sept]**	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002560 E07-0405 (Storage: 7.4 months)
United States, Suffolk, VA, 2010, (Georgia 6G)	EC 3 (14)	0.10 0.10 0.10	79 **[15 Sept]**	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002560 E11-0407 (Storage: 8.2 months)
United States, Pikeville, NC 2010, (Gregory)	EC 3 (14)	**0.095** 0.10 0.10	79	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002560 E10-0406 (Storage: 7.1-8.0 months)
United States, Unadilla, GA 2010, (Georgia 6G)	EC 3 (14)	**0.099** 0.10 **0.099**	79	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002560 E12-0408 (Storage: 9.6 months)
United States, Malone, FL 2010, (Georgia Greene)	EC 3 (14)	0.10 0.10 0.10	Pod Fill	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002560 E14-0409 (Storage: 8.9 months)
United States, Charlotte, TX 2010, (Florida Runner 07)	EC 3 (14)	0.10 0.10 0.10	86	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002560 W07-0410 (Storage: 7.1 months)
United States, Dilley, TX 2010, (TamRum OL-1)	EC 3 (14)	0.10 0.10 0.10	86	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002560 W07-0411 (Storage: 7.1 months)
United States, Levelland, TX 2010, (Tamspan 90)	EC3 (14)	0.10 0.099 0.10	Ma turing nuts	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002560 W39-0412 (Storage: 8.7 months)
United States, Pineview, GA 2011, (Georgia 06-G)	EC 3 (14)	0.098 0.098 0.099	**77-79**	30	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	TK0047558 TK0047558-01 (Storage: 3.0 months)
	WG 3 (14)	0.10 0.099 0.10	**77-79**	30	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	

PEANUT Location, Year (variety)	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
United States, Charlotte, TX 2011, (Georgia 09)	EC 3 (14)	0.099 0.10 0.10	84	30	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	TK0047558 TK0047558-02 (Storage: 6.7 months)
	WG 3 (14)	0.10 0.10 **0.099**	84	30	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	
United States, Hinton, OK 2011, (Tamnut OL06)	EC 3 (14)	0.10 0.10 0.098	79	30	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	TK0047558 TK0047558-03 (Storage: 2.1 months)
	WG 3 (14)	0.10 0.10 0.10	79	30	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	

Coffee beans

Residue information on coffee beans was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information.

Table 34 Residues in green coffee beans$ from field trials in Brazil (JMPR 2016)

GREEN COFFEE BEANS Location Year (variety)	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
Brazil Indianopolis, MG 2010, (Mundo Novo)	**WG** 3 (60)	0.060 0.060 0.060	83	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11085 M11085-JJB1 (Storage: 1.6-2.1 months)
Brazil Indianopolis, MG 2010, (Mundo Novo)	**EC** 3 (60)	0.050 0.050 0.050	83	21 28 35	0.020 <0.01 <0.01	0.020 <0.01 <0.01	<0.01 <0.01 <0.01	**M11074** **M11074-JJB1** **(Storage 1.4-2.4 months)**
Brazil Araguari, MG 2010, (Mundo Novo)	**WG** 3 (60)	0.060 0.060 0.060	83	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11085 M11085-JJB2 (Storage: 1.6-2.1 months)
Brazil Araguari, MG 2010, (Mundo Novo)	**EC** 3 (60)	0.050 0.050 0.050	83	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	**M11074** **M11074-JJB2** **(Storage 1.4-2.4 months)**
Brazil Sao Goncalo do Sapucai, MG 2010, (Mundo Novo)	**WG** 3 (60)	0.060 0.060 0.060	81	21 28 35	0.020 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11085 M11085-RWC1 (Storage: 2.0-2.5 months)
Brazil Sao Goncalo do Sapucai, MG 2010,	**EC** 3 (60)	0.050 0.050 0.050	81	21 28 35	0.020 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	**M11074** **M11074-RWC1** **(Storage 1.5-2.5 months)**

GREEN COFFEE BEANS Location Year (variety)	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	SYN 545720 (mg/kg)	Report; Trial; (remarks)
(Mundo Novo)								
Brazil Campinas, SP 2010, (Catuai Vermelho IAC 144)	**WG** 3 (60)	0.060 0.060 0.060	79	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11085 M11085-RWC2 (Storage: 1.9-2.4 months)
Brazil Campinas, SP 2010, (Catuai Vermelho IAC 144)	**EC** 3 (60)	0.050 0.050 0.050	79	21 28 35	<u><0.01</u> <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	**M11074** **M11074-RWC2** **(Storage 1.4-2.4 months)**
Brazil Linhares, ES 2010, (Conilon)	**WG** 3 (60)	0.060 0.060 0.060	78	21 28 35	0.020 0.020 0.020	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11085 M11085-RWC3 (Storage: 1.9-2.3 months)
Brazil Linhares, ES 2010, (Conilon)	**EC** 3 (60)	0.050 0.050 0.050	78	21 28 35	<u>0.070</u> 0.050 0.050	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	**M11074** **M11074-RWC3** **(Storage 1.4-2.3 months)**
Brazil Taiuva, SP 2010, (Catuai Amarelo)	**WG** 3 (60)	0.060 0.060 0.060	81	21 28 35	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	M11085 M11085-AMA (Storage: 2.3-2.7 months)
Brazil Taiuva, SP 2010, (Catuai Amarelo)	**EC** 3 (60)	0.050 0.050 0.050	81	21 28 35	<u><0.01</u> <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	**M11074** **M11074-AMA** **(Storage: 1.8-2.7 months)**

Notes:

$ Coffee berries were dried (21 days in a greenhouse at room temperature (AMA), 3 days in a dryer at 38°C (JJB1, JJB2), by the sun (RWC1, RWC2, RWC3)) Dried coffee berries were peeled to get the green coffee beans.

Legume vines/forage

Residue information on pea vines and soya bean forage was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information. SYN545720 was not analysed in pea vines or soya bean forage. Information on soya bean forage from trials in Brazil is not available (JMPR 2014). Trials on peanut forage were not conducted.

Pea vines

Table 35 Residues in pea vines from field trials in Canada and the United States (JMPR 2016)

PEA VINES Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States Hinton, OK, 2011, (Alaska)	EC	2 (7)	**0.075** **0.076**	73	14	0.35, 0.64, 0.30 **(<u>0.43</u>)**	0.42, 0.36, 0.25 (0.34)	TK0058625 TK0058625-01 storage interval: 3.7 months
	WG	2	**0.077**	73	14	0.37,	0.048,	TK0058625

PEA VINES Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
		(7)	**0.077**			0.43, 0.48 (0.43)	0.039, 0.048 (0.045)	TK0058625-01 storage interval: 3.7 months
United States American Falls, ID, 2011, (Little Marvel)	EC	2 (7)	**0.077** **0.080**	**67**	14	0.34, 0.28, 0.26 (0.29)	0.23, 0.23, 0.17 (0.21)	TK0058625 TK0058625-03 storage interval: 5.6 months
	WG	2 (7)	**0.075** **0.072**	**67**	14	0.27, 0.22, 0.19 (0.23)	0.15, 0.14, 0.12 (0.14)	TK0058625 TK0058625-03 storage interval: 5.6 months
United States Jerome, ID, 2011, (SNO 112 0490N14)	EC	2 (7)	**0.075** **0.078**	**33**	0 7 14 . 21	3.9 1.2 0.27, 0.28 (0.28) 0.17	0.11 0.16 0.13, 0.15 (0.14) 0.15	TK0058625 TK0058625-04 storage interval: 7.3 months
United States Hillsboro, OR, 2011, (Blue Bird)	EC	2 (7)	**0.075** **0.078**	75	14	0.58, 0.64 (0.61)	0.65, 0.77 (0.71)	TK0058625 TK0058625-05 storage interval: 6.3 months
United States Madera, CA, 2011, (Dundale)	EC	2 (7)	**0.078** **0.079**	**45**	14	0.44, 0.61, 0.40 **(0.48)**	0.57, 0.62, 0.54 (0.58)	TK0058625 TK0058625-15 storage interval: 2.5 months
	WG	2 (7)	**0.076** **0.076**	45	14	0.97, 0.88, 0.94 **(0.93)**	0.24, 0.25, 0.22 (0.24)	TK0058625 TK0058625-15 storage interval: 2.5 months

Soya forage

No trials were selected for soya forage.

Table 36 Residues in soya forage from field trials in the United States (JMPR 2016)

SOYA FORAGE Location; Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States Elko, SC, 2010, (Asgrow 7502)	EC	2 (7)	0.076 0.076	69	0	4.0, 2.4 (3.2)	0.082, 0.063 (0.072)	TK0002561 E11-0421 storage interval: 9.7 months
United States Seven Springs, NC, 2010, (AG5605)	EC	2 (7)	0.077 0.077	68	0	6.1, 5.2 **(5.6)**	0.22, 0.16 (0.19)	TK0002561 E10-0422 storage interval: 10.0 months

SOYA FORAGE Location; Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States Cheneyville, LA, 2010, (Pioneer 94M80)	EC	2 (7)	0.075 0.077	**74-75 [8 Jul]**	0	2.9, 2.8 **(2.8)**	0.063, 0.048 (0.056)	TK0002561 E17-0423 storage interval: 11.3 months
United States Cheneyville, LA, 2010, (Asgrow 5335)	EC	2 (7)	0.078 **0.080**	75 **[20 Aug]**	0	3.1, 2.1 (2.6)	0.23, 0.15 (0.19)	TK0002561 E17-0424 storage interval: 10.0 months
United States Pollard, AR, 2010, (Pioneer 94M80)	EC	2 (7)	0.077 0.076	69	0	5.8, 5.1 **(5.4)**	0.28, 0.24 (0.26)	TK0002561 C23-0425 storage interval: 10.3 months
United States Northwood, ND, 2010, (90Y41)	EC	2 (7)	0.076 0.076	40 percent PD	0 . 3 7 14	4.2, 3.8 (4.0) 2.1 1.7 0.88	0.21, 0.14 (0.18) 0.22 0.26 0.19	TK0002561 C13-0426 storage interval: 10.2 months
United States Sharon, ND, 2010, (90Y41)	EC	2 (7)	0.077 0.077	30 percent PD	0	4.4, 4.2 (4.3)	0.12, 0.12 (0.12)	TK0002561 C13-0427 storage interval: 10.7-10.8 months
United States Gardner, ND, 2010, (0509239)	EC	2 (7)	0.075 0.077	72	0	4.6, 4.6 (4.6)	0.22, 0.23 (0.22)	TK0002561 C03-0428 storage interval: 10.0 months
United States Dudley, MO, 2010, (Asgrow 5403)	EC	2 (7)	0.076 0.076	69	0	3.1, 3.4 **(3.2)**	0.12, 0.20 (0.16)	TK0002561 C23-0429 storage interval: 10.7 months
United States Fisk, MO, 2010, (Jake)	EC	2 (7)	0.076 0.077	69	0	3.4, 3.9 **(3.6)**	0.028, 0.036 (0.032)	TK0002561 C23-0430 storage interval: 10.3 months
United States Fitchburg, WI, 2010, (S21-N6)	EC	2 (7)	0.075 0.075	75	0	2.1, 1.8 (2.0)	0.039, 0.032 (0.036)	TK0002561 C08-0431 storage interval: 10.5-11.9 months
United States Bagley, IA, 2010, (P3Y13-N203)	EC	**2 (8)**	0.077 0.077	75	0	2.9, 2.7 (2.8)	0.091, 0.093 (0.092)	TK0002561 C30-0432 storage interval: 9.9 months
United States Oregon, MO, 2010, (Pioneer 93Y70)	EC	2 (7)	0.075 0.076	R3-R4	0	2.8, 2.3 (2.6)	0.27, 0.38 (0.32)	TK0002561 C19-0433 storage interval: 10.2 months
United States York, NE, 2010, (93Y12)	EC	2 (7)	0.076 0.076	73	0	3.4, 3.7 (3.6)	0.53, 0.51 (0.52)	TK0002561 C33-0434 storage interval: 11.0 months
United States Lesterville, SD,	EC	2 (7)	0.075 0.076	71	0	3.2, 2.9	0.089, 0.085	TK0002561 C16-0435

SOYA FORAGE Location; Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
2010, (Latham, L2560R, LS-0991236)						**(3.0)**	(0.087)	storage interval: 10.5 months
United States Marysville, OH, 2010, (SG-329-RR)	EC	2 (7)	0.026 0.077	75	0	2.6, 2.9 (2.8)	0.070, 0.089 (0.080)	TK0002561 C01-0436 TK0002561 C01-0436 storage interval: 9.9 months
United States Clarence, MO, 2010, (Asgrow 3803 RR)	EC	2 (7)	0.075 0.076	R2	0 . . 3 7 14	4.9, 5.1 (5.0) 1.0 0.78 0.21	0.090, 0.063 (0.076) 0.19 0.27 0.13	TK0002561 C20-0437 storage interval: 10.8 months
United States Richland, IA, 2010, (Pioneer 92Y80)	EC	2 (7)	0.077 0.077	70	0	2.2, 3.5 (2.8)	0.041, 0.065 (0.053)	TK0002561 C18-0438 storage interval: 10.3 months
United States Campbell, MN, 2010, (AG 0808)	EC	2 (7)	0.076 0.076	69	0	4.4, 3.9 (4.2)	0.52, 0.26 (0.39)	TK0002561 C11-0439 storage interval: 10.8 months
United States Geneva, MN, 2010, (Pioneer 91Y70)	EC	2 (7)	0.077 0.075	**72**	0	5.4, 5.1 **(5.2)**	0.69, 0.62 (0.66)	TK0002561 C09-0440 storage interval: 10.7 months

Legume hay

Residue information on pea hay, soya bean hay and peanut hay was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information. SYN545720 was not analysed in pea hay, soya bean hay or peanut hay. Residue information on soya bean hay from trials in Brazil (JMPR 2014) is not available.

Pea hay

Table 37 Residues in pea hay from field trials in Canada and the United States (JMPR 2016)

PEA HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trials; (remarks)
United States Hinton, OK, 2011, (Alaska)	EC	2 (7)	**0.075 0.076**	73	14	1.2, 1.8, 1.9 (1.6)	0.81, 1.0, 1.4 (1.1)	TK0058625 TK0058625-01 storage interval: 3.4 months
	WG	2 (7)	**0.077 0.077**	73	14	2.1, 1.8, 2.3 (2.2)	0.074, 0.14, 0.13 (0.11)	

PEA HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trials; (remarks)
United States American Falls, ID, 2011, (Little Marvel)	EC	2 (7)	**0.077** **0.080**	**67**	14	1.5, 0.81, 1.0 **(1.1)**	1.2, 0.91, 1.1 (1.1)	TK0058625 TK0058625-03 storage interval: 5.7 months
	WG	2 (7)	**0.075** **0.072**	**67**	14	0.92, 0.49, 0.88 **(0.76)**	0.61, 0.28, 0.60 (0.50)	
United States Jerome, ID, 2011, (SNO 112 0490N14)	EC	2 (7)	**0.075** **0.078**	**33**	0 7 14 . 21	23 7.5 1.7, 1.9 (1.8) 0.25	2.9 2.7 0.95, 0.89 (0.92) 0.18	TK0058625 TK0058625-04 storage interval: 7.4 months
United States Hillsboro, OR, 2011, (Blue Bird)	EC	2 (7)	**0.075** **0.078**	75	14	2.3, 3.9 (3.1)	2.6, 4.4 (3.5)	TK0058625 TK0058625-05 storage interval: 6.4 months
United States Madera, CA, 2011, (Dundale)	EC	2 (7)	**0.078** **0.079**	**45**	14	2.8, 2.9, 2.6 **(2.8)**	3.0, 3.1, 2.8 (3.0)	TK0058625 TK0058625-15 storage interval: 2.6 months
	WG	2 (7)	**0.076** **0.076**	**45**	14	3.9, 3.2, 3.8 **(3.6)**	1.4, 1.1, 1.3 (1.3)	

Soya hay

No trials were selected for soya hay.

Table 38 Residues in soya hay from field trials in the United States (JMPR 2016)

SOYA HAY Location; Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States Elko, SC, 2010, (Asgrow 7502)	EC	2 (7)	0.076 0.076	69	0	15, 17 (16)	0.16, 0.12 (0.14)	TK0002561 E11-0421 storage interval: 9.5 months
United States Seven Springs, NC, 2010, (AG5605)	EC	2 (7)	0.077 0.077	68	0	16, 17 **(16)**	1.8, 1.1 (1.4)	TK0002561 E10-0422 storage interval: 9.8 months
United States Cheneyville, LA, 2010, (Pioneer 94M80)	EC	2 (7)	0.075 0.077	**74-75** **[8 Jul]**	0	7.1, 8.7 (7.9)	0.51, 0.69 (0.60)	TK0002561 E17-0423 storage interval: 11.1 months

SOYA HAY Location; Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Cheneyville, LA, 2010, (Asgrow 5335)	EC	2 (7)	0.078 **0.080**	75 **[20 Aug]**	0	12, 12 (12)	0.68, 0.58 (0.63)	TK0002561 E17-0424 storage interval: 9.9 months
United States Pollard, AR, 2010, (Pioneer 94M80)	EC	2 (7)	0.077 0.076	69	0	36, 36 (36)	2.4, 3.8 (3.1)	TK0002561 C23-0425 storage interval: 10.1-10.2 months
United States Northwood, ND, 2010, (90Y41)	EC	2 (7)	0.076 0.076	40 percent PD	0 . 3 7 14	13, 13 (13) 13 5.0 3.6	0.56, 0.59 (0.58) 1.5 0.56 1.4	TK0002561 C13-0426 storage interval: 10.2 months
United States Sharon, ND, 2010, (90Y41)	EC	2 (7)	0.077 0.077	30 percent PD	0	20, 21 **(20)**	0.58, 0.48 (0.53)	TK0002561 C13-0427 storage interval: 10.7 months
United States Gardner, ND, 2010, (0509239)	EC	2 (7)	0.075 0.077	72	0	11, 12 (12)	1.1, 1.3 (1.2)	TK0002561 C03-0428 storage interval: 8.5-8.6 months
United States Dudley, MO, 2010, (Asgrow 5403)	EC	2 (7)	0.076 0.076	69	0	12, 12 (12)	0.59, 0.48 (0.54)	TK0002561 C23-0429 storage interval: 10.3 months
United States Fisk, MO, 2010, (Jake)	EC	2 (7)	0.076 0.077	69	0	16, 17 **(16)**	0.091, 0.10 (0.096)	TK0002561 C23-0430 storage interval: 10.2 months
United States Fitchburg, WI, 2010, (S21-N6)	EC	2 (7)	0.075 0.075	75	0	9.1, 7.9, 9.5 (8.8)	1.1, 0.85, 0.35 (0.77)	TK0002561 C08-0431 storage interval: 10.4-12.9 months
United States Bagley, IA, 2010, (P3Y13-N203)	EC	**2** **(8)**	0.077 0.077	75	0	11, 10 **(10)**	0.39, 0.37 (0.38)	TK0002561 C30-0432 storage interval: 9.7 months
United States Oregon, MO, 2010, (Pioneer 93Y70)	EC	2 (7)	0.075 0.076	R3-R4	0	7.0, 9.0 (8.0)	0.49, 0.67 (0.58)	TK0002561 C19-0433 storage interval: 10.0 months
United States York, NE, 2010, (93Y12)	EC	2 (7)	0.076 0.076	73	0	19, 18 **(18)**	4.6, 3.7 (4.2)	TK0002561 C33-0434 storage interval: 10.8 months
United States Lesterville, SD, 2010, (Latham, L2560R, LS-0991236)	EC	2 (7)	0.075 0.076	71	0	13, 16 **(14)**	2.9, 2.8 (2.8)	TK0002561 C16-0435 storage interval: 10.0-10.3 months
United States Marysville, OH,	EC	2 (7)	0.026 0.077	75	0	10, 9.5	3.2, 2.6	TK0002561 C01-0436

SOYA HAY Location; Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
2010, (SG-329-RR)						**(9.8))**	(2.9)	TK0002561 C01-0436 storage interval: 9.7 months
United States Clarence, MO, 2010, (Asgrow 3803 RR)	EC	2 (7)	0.075 0.076	R2	0 3 7 14	17, 11 (14) 3.7 1.4 0.36	0.39, 0.39 (0.39) 0.63 0.55 0.45	TK0002561 C20-0437 storage interval: 10.6 months
United States Richland, IA, 2010, (Pioneer 92Y80)	EC	2 (7)	0.077 0.077	**70**	0	10, 6.4 (8.2)	0.31, 0.16 (0.24)	TK0002561 C18-0438 storage interval: 10.1 months
United States Campbell, MN, 2010, (AG 0808)	EC	2 (7)	0.076 0.076	69	0	15, 13 (14)	1.2, 1.5 (1.4)	TK0002561 C11-0439 storage interval: 10.6 months
United States Geneva, MN, 2010, (Pioneer 91Y70)	EC	2 (7)	0.077 0.075	72	0	17, 16 **(16)**	1.3, 2.0 (1.6)	TK0002561 C09-0440 storage interval: 10.5 months

Peanut hay

Table 39 Residues in peanut hay from field trials in the United States (scaling factor 0.75)

PEANUT HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
United States, Elko, SC 2010, (Gregory)	EC	3 (14)	0.10 0.10 0.10	79	30	3.1, 2.6 **(2.8)** Scaled: 2.1	0.29, 0.24 (0.26) Scaled: 0.20	TK0002560 E11-0401 (Storage: 7.7 months)
United States, Seven Springs, NC 2010, (Perry)	EC	3 (14)	0.10 0.10 0.10	69	30	2.7, 3.2 (3.0) Scaled: 2.2	0.25, 0.28 (0.26) Scaled: 0.20	TK0002560 E10-0402 (Storage: 8.0 months)
United States, Hawkinsville, GA 2010, (Georgia 06-G)	EC	3 (14)	0.10 0.10 0.10	79	**23** 30 . . **37** **44** .	**6.7,** 8.2, 7.1 **(7.6)** **4.9** **6.0** Scaled: 5.7	0.26, 0.33 0.29 (0.31) 0.29 0.24 Scaled: 0.23	TK0002560 E12-0403 (Storage: 9.2 months)
United States, Suffolk, VA	EC	3 (14)	0.10 0.10	75 **[22 Sept]**	30	2.4, 2.5	0.34, 0.41	TK0002560 E07-0404

PEANUT HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
2010, (Champs)			0.10			(2.4) Scaled: 1.8	(0.38) Scaled: 0.28	(Storage: 6.9 months)
United States, Suffolk, VA 2010, (Champs)	EC	3 (14)	0.10 0.10 0.10	75 **[22 Sept]**	30	3.1, 3.1 (3.1) Scaled: 2.3	0.44, 0.47 (0.46) Scaled: 0.34	TK0002560 E07-0405 (Storage: 6.9 months)
United States, Suffolk, VA, 2010, (Georgia 6G)	EC	3 (14)	0.10 0.10 0.10	79 **[15 Sept]**	30	4.1, 3.3 (3.7) Scaled: 2.8	0.31, 0.30 (0.30) Scaled: 0.23	TK0002560 E11-0407 (Storage: 7.7 months)
United States, Pikeville, NC 2010, (Gregory)	EC	3 (14)	**0.095** 0.10 0.10	79	30	1.7, 1.8 (1.8) Scaled: 1.3	0.19, 0.23 (0.21) Scaled: 0.16	TK0002560 E10-0406 (Storage: 7.1-7.5 months)
United States, Unadilla, GA 2010, (Georgia 6G)	EC	3 (14)	**0.099** 0.10 **0.099**	79	30	6.2, 6.3 **(6.2)** Scaled: 4.7	0.34, 0.33 (0.34) Scaled: 0.25	TK0002560 E12-0408 (Storage: 9.2 months)
United States, Malone, FL 2010, (Georgia Greene)	EC	3 (14)	0.10 0.10 0.10	Pod Fill	30	10, 7.8 (9.0) Scaled: 6.8	0.68, 0.64 (0.66) Scaled: 0.50	TK0002560 E14-0409 (Storage: 8.5 months)
United States, Charlotte, TX 2010, (Florida Runner 07)	EC	3 (14)	0.10 0.10 0.10	86	30	2.8, 2.9 **(2.8)** Scaled: 2.1	0.13, 0.12 (0.12) Scaled: 0.094	TK0002560 W07-0410 (Storage: 6.6 months)
United States, Dilley, TX 2010, (TamRum OL-1)	EC	3 (14)	0.1008 0.0997 0.1008	86	30	**9.1,** **7.4** **8.2** **5.1** **6.7** **6.4** **(7.2)** Scaled: 5.3	1.8, 1.5 1.3 0.66 1.4 1.4 (1.3) Scaled: 1.0	TK0002560 W07-0411 (Storage: 6.6-20.3 months)
United States, Levelland, TX 2010, (Tamspan 90)	EC	3 (14)	0.10 0.099 0.10	Maturing nuts	30	2.8, 2.7 (2.8) Scaled: 2.1	0.15, 0.14 (0.14) Scaled: 0.11	TK0002560 W39-0412 (Storage: 8.2 months)
United States, Pineview, GA 2011, (Georgia 06-G)	EC	3 (14)	0.098 0.098 0.099	**77-79**	30	6.7, 7.4, 7.0 (7.0) Scaled: 5.3	0.75, 0.74 0.64 (0.71) Scaled:	TK0047558 TK0047558-01 (Storage: 5.3 months)

PEANUT HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
							0.53	
	WG	3 (14)	0.10 0.099 0.10	**77-79**	30	5.4, 7.2, 4.4 (5.7)	0.25, 0.25, 0.17 (0.22)	
United States, Charlotte, TX 2011, (Georgia 09)	EC	3 (14)	0.099 0.10 0.10	84	30	0.51, 0.53, 0.24 (0.43) Scaled: 0.32	0.041, 0.035, 0.018 (0.031) Scaled: 0.024	TK0047558 TK0047558-02 (Storage: 5.5 months)
	WG	3 (14)	0.10 0.10 **0.099**	84	30	0.19, 0.22, 0.16 (0.19)	0.011, <0.01, <0.01 (0.010)	
United States, Hinton, OK 2011, (Tamnut OL06)	EC	3 (14)	0.10 0.10 0.098	79	30	2.3, 2.7, 3.2 (2.7) Scaled: **2.0**	0.27, 0.46 0.59 (0.44) Scaled 0.33	TK0047558 TK0047558-03 (Storage: 4.4 months)
	WG	3 (14)	0.10 0.10 0.10	79	30	1.5, 1.5, 2.0 (1.7)	0.16, 0.19 0.23 (0.19)	

Cereal forage

Residue information on wheat forage, sweet corn forage and maize forage was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information. Trials on barley forage were not conducted.

Wheat forage

Table 40 Residues in wheat forage from field trials in Canada and the United States (JMPR 2016)

WHEAT FORAGE Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Seven Springs, NC 2010, (Pioneer 26R15)	EC	2 **(13)**	0.077 0.078	67	7	0.93, 1.2 **(1.1)**	0.092, 0.11 (0.10)	TK0002558 E10-0351 (Storage: Forage 10.6 months)
United States, Shelbyville, MO 2010, **(soft red winter wheat: Erine)**	EC	2 (14)	0.075 0.078	**Feekes 10**	7	0.47, 0.43 (0.45)	0.15, 0.17 (0.16)	TK0002558 C20-0354 (Storage: Forage 10.4 months)
United States,	EC	2	0.077	Feekes 5	7	0.42,	0.042,	TK0002558

WHEAT FORAGE Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
Richland, IA 2010, (**soft red winter wheat:** Wilcross748)		(14)	0.077			0.34 (0.38)	0.085 (0.064)	C18-0355 (Storage: Forage 10.4 months)
United States, Milford Center, OH 2010, (Croplan Genetics 8614)	EC	2 (14)	0.077 0.077	Feekes 5	7	0.80, 0.83 (0.82)	0.16, 0.14 (0.15)	TK0002558 C01-0356 (Storage: Forage 9.8 months)
United States, Macon, MO 2010, (**soft red winter wheat**: V9710)	EC	2 **(16)**	0.077 **0.078**	Feekes 10.5.1	7	not collected	-	TK0002558 C20-0357
United States, Carrington, ND 2010, (**soft white spring wheat:** AP-604-CL)	EC	2 **(13)**	0.077 **0.077**	45	7	0.67, 0.66 **(0.66)**	0.11, 0.16 (0.14)	TK0002558 C13-0359 (Storage: Forage 8.9 months)
United States, Carrington, ND 2010, (**hard red spring wheat**: Faller)	EC	2 **(13)**	0.077 **0.077**	45	7	0.33, 0.33 (0.33)	0.15, 0.16 (0.16)	TK0002558 C13-0361 (Storage: Forage 8.9 months)
United States, Jamestown, ND 2010, (**wheat: durum**: variety not known))	EC	2 (14)	0.077 0.077	33	**0** **3** 7 **10** **14**	**4.2** **1.9** 1.2, 1.3 **(1.2)** **0.80** **0.38**	0.045 0.067 0.11 0.10 (0.10) 0.097 0.11	TK0002558 C12-0360 (Storage: Forage 7.3-7.8 months)
United States, Lake Andes, SD 2010, (**hard white spring wheat**: Argent)	EC	2 **(13)**	0.076 0.075	**59-63**	7	1.6, 1.3 **(1.4)**	0.20, 0.17 (0.18)	TK0002558 C16-0362 (Storage: Forage 8.8 months)
United States, Grand Island, NE 2010, (**hard red winter wheat: NE 01643)**	EC	2 (14)	0.076 0.077	30	**8**	1.3, 0.49 (0.90)	0.10, 0.090 (0.095)	TK0002558 C33-0363 (Storage: Forage 10.3 months)
United States, Johnstown, CO 2010, (**winter wheat**: Yuma)	EC	2 (14)	0.077 0.077	Feekes 9	7	1.2, 1.4 (1.3)	0.17, 0.15 (0.16)	TK0002558 W12-0364 (Storage: Forage 10.1 months)
United States, Eaton, CO	EC	2 (14)	0.077 0.076	Feekes 9	**0** **3**	**3.9** **1.7**	0.041 0.058	TK0002558 W12-0365

WHEAT FORAGE Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
2010, (**winter wheat: Jagalene**)					7 . **10** **14**	1.5, 1.5 (1.5) **1.8** **0.83**	0.059, 0.068 (0.064)) 0.093 0.071	(Storage: Forage 9.9-10.0 months)
United States, Milliken, CO 2010, (**hard red winter wheat:** Bill Brown)	EC	2 (14)	**0.078** 0.077	**Feekes 9-10**	7	1.2, 1.4 (1.3)	0.071, 0.10 (0.086)	TK0002558 W12-0369 (Storage: Forage 10.1 months)
United States, Rupert, ID 2010, (**hard white spring wheat:** Klassic)	EC	2 (14)	0.078 **0.088**	41	7	0.69, 0.73 (0.71)	0.040, 0.046 (0.043)	TK0002558 W15-0370 (Storage: Forage 9.3 months)
United States, Fisk, MO **2011,** (**soft red winter wheat**: Beretta)	EC	2 **(13)**	0.076 **0.077**	37	**8**	0.61, 0.64 **(0.62)**	0.11, 0.11 (0.11)	TK0002558 C23-0352 (Storage: Forage 3.6 months)
United States, Raymondville, TX **2011,** (**hard red winter wheat**: Caudillo)	EC	2 **(16)**	0.076 0.077	32	**6**	3.0, 3.7 (3.4)	0.27, 0.35 (0.31)	TK0002558 W08-0358 (Storage: Forage 5.1 months)
United States, Uvalde, TX **2011,** (**hard red winter wheat**: Tam 203)	EC	2 (14)	0.076 0.077	45	7	1.2, 1.1 (1.2)	0.083, 0.082 (0.082)	TK0002558 W07-0366 (Storage: Forage 3.5 months)
United States, Wall, TX **2011,** (**hard red winter wheat:** Coronado)	EC	2 **(13)**	0.077 0.078	33	7	2.3, 2.1 (2.2)	0.15, 0.17 (0.16)	TK0002558 W40-0367 (Storage: Forage 3.3 months)
United States, Levelland, TX **2011,** (**hard red winter wheat**: Weather master)	EC	2 (14)	**0.074** **0.074**	32	7	1.4, 2.7 **(2.0)**	0.020, 0.027 (0.024)	TK0002558 W39-0368 (Storage: Forage 3.8 months)
United States, Valley City, ND 2011, (**winter wheat:** Falcon)	EC	2 **(15)**	0.077 **0.077**	49	7	0.24, 0.26, 0.11 (0.20)	0.081, 0.086, 0.095 (0.087)	TK0048907 TK048907-01 (Storage: Forage 5.9 months)
	WG	2 **(15)**	0.076 0.078	49	7	0.37, 0.43, 0.63 (0.48)	0.052, 0.055, 0.067 (0.058)	

WHEAT FORAGE Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Jamestown, ND 2011, (**winter wheat**: Overland)	EC	2 **(15)**	0.077 0.078	40	7	0.58, 0.67, 0.94 (0.73)	0.080, 0.091, 0.13 (0.10)	TK0048907 TK048907-02 (Storage: Forage 6.2 months)
	WG	2 **(15)**	0.077 0.078	40	7	0.73, 0.72, 0.68 (0.71)	0.049, 0.054, 0.051 (0.051)	
United States, Northwood, ND **2011,** **(hard red winter wheat**: Jerry)	EC	2 **(16)**	**0.080** 0.078	33	7	1.5, 1.2 (1.4)	0.021, 0.023 (0.022)	TK0002558 C13-0353 (Storage: Forage 2.1 months)
United States, Northwood, ND 2011, (**hard red spring wheat:** Faller)	EC	2 (14)	0.079 0.077	69	7	0.63, 0.47, 0.54 (0.55)	0.38, 0.32, 0.25 (0.32)	TK0048907 TK048907-03 (Storage: Forage 5.3 months)
	WG	2 (14)	0.078 0.078	69	7	0.36, 0.36, 0.51 (0.41)	0.11, 0.13, 0.17 (0.14)	
Canada Vanscoy, SK 2011, (Infinity))	EC	2 (14)	0.074 0.073	**57-59**	7	0.59, 1.5 **(1.0)**	0.047, 0.069 (0.058)	CER05901/11; T916 (Storage: 3.1 months)
Canada Kinley, SK 2011, (Infinity)	EC	2 (14)	**0.074** **0.073**	**58-61**	7	1.8, 1.8 (1.8)	0.12, 0.096 (0.11)	CER05901/11; T917 (Storage: 2.8 months)
Canada Taber, AB 2011, (Superb)	EC	2 (14)	**0.078** **0.079**	**33-37**	7	1.6, 1.3 **(1.4)**	0.10, 0.054 (0.077)	CER05901/11; T918 (Storage: 3.2 months)
Canada Boissevain, MB 2011, (**Spring wheat**: Harvest)	EC	2 **(13)**	**0.075** **0.078**	**41-45** **[16 Jul]**	6	2.0, 1.7 **(1.8)**	0.14, 0.18 (0.16)	CER05901/11; T919 (Storage: 7.2 months)
Canada Boissevain, MB 2011, (**Spring wheat**: Kane)	EC	2 **(13)**	**0.076** **0.073**	**41-43** **[16 Jul]**	**6**	1.8, 1.3 (1.6)	0.14, 0.13 (0.14)	CER05901/11; T920 (Storage: 5.1 months)
Canada Rosthern, SK 2011, (Infinity)	EC	2 **(13)**	**0.074** **0.076**	**45-59**	**8**	0.35, 0.44 (0.40)	0.051, 0.039 (0.045)	CER05901/11; T921 (Storage: 3.0 months)
Canada Blaine Lake, SK 2011, (Infinity)	EC	2 **(15)**	**0.072** **0.075**	**45-50**	7	<0.01, <0.01 (<0.01) **	<0.01, <0.01 (<0.01)	CER05901/11; T922 (Storage: 6.5 months)
Canada	EC	2	**0.075**	**51-61**	7	0.79,	0.075,	CER05901/11;

WHEAT FORAGE Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
Duck Lake, SK 2011, (Infinity)		(14)	**0.075**			1.1 **(0.94)**	0.10 (0.088)	T923 (Storage: 2.6 months)
Canada Kipp, AB 2011, (Superb)	EC	2 **(15)**	**0.076** **0.079**	**53-58**	6	1.9, 1.9 (1.9)	0.11, 0.10 (0.10)	CER05901/11; T924 (Storage: 2.8 months)
Canada Alvena, SK 2011, (Goodeve – Ac Intrepid)	EC	2 (14)	**0.073** 0.071	**55-59**	7	1.5, 1.0 **(1.2)**	0.078, 0.089 (0.084)	CER05901/11; T925 (Storage: 2.8 months)
Canada Fort Sask. AB, 2011, (Harvest)	EC	2 (14)	**0.075** **0.077**	**41-61**	7	0.44, 0.68 **(0.56)**	0.062, 0.11 (0.086)	CER05901/11; T926 (Storage: 2.8 months)
Canada Minto, MB 2011, (Superb)	EC	2 (14)	**0.076** **0.074**	**37-41** **[7 Jul]**	**4** 7 . **11** **14**	**1.1** 0.90, 0.58 (0.74) **0.26** **0.11**	0.085 0.082, 0.088 (0.085) 0.12 0.14	CER05901/11; T927 (Storage: 3.2 months)
Canada Minto, MB 2011, (**spring wheat**: AC Barrie)	EC	2 (14)	**0.073** 0.076	**37-41** **[7 Jul]**	**4** 7 . **11** **14**	**0.75** 0.73, 0.68 **(0.70)** **0.47** **0.63**	0.098 0.11, 0.10 (0.10) 0.15 0.14	CER05901/11; T928 (Storage: 5.7 months)

Notes:

** The residue levels detected in wheat forage samples from site T922 (samples T922-02 and T922-03) appear to be unexpectedly low (<0.01 ppm); samples were analysed to confirm below LOQ values. These duplicate samples may have been collected from the check plot but it cannot be confirmed.

Sweet corn and maize forage

No trials were selected for sweet corn forage or maize forage from the United States.

Note from the reviewer: Report TK0002562: Sweet corn forage and stover samples were each at the same location and received each the same treatment at the same day. Ears were picked by hand and part of the stalks without ears were harvested as “sweet corn forage” at the same day as the sweet corn ears. The remaining stalks were left in the field and were harvested at a later date as “sweet corn stover”. The growth stage at harvest for sweet corn forage and stover was not stated in the study report.

Table 41 Residues in sweet corn forage (without ears) from field trials in the United States (JMPR 2016)

SWEET CORN FORAGE Location Year (Variety)	Form No (RTI)	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report Trial Remark
United States, Germansville, PA 2010, (Extra Tender 274A)	EC 4 (7)	0.077 0.076 0.077 0.077	73 early milk stage	**6**	2.6, 2.6; (2.6)	0.026, 0.024; (0.025)	TK0002562 E04-0472 (Storage: Forage 7.2 months)
United States, North Rose, NY 2010, (Serendipity)	EC 4 (7)	0.078 0.078 0.080 0.078	75 early mature ears	7	1.3, 1.2; **(1.2)**	0.019, 0.023; (0.021)	TK000256 **E02-0473** (Storage: Forage 6.0 months)
United States, Athens, GA 2010, (Silver King)	EC 4 (7)	0.076 0.076 0.077 0.076	**72-73**	7	1.9, 1.5; (1.7)	0.026, 0.023; (0.024)	TK0002562 **E12-0474** (Storage: Forage 7.1 months)
United States, Oviedo, FL 2010, (Silver Queen)	EC 4 (7)	0.075 0.077 0.076 0.078	71	7	0.34, 0.15; **(0.24)**	<0.01, <0.01; (<0.01)	TK0002562 E04-0475 **E15-0475** (Storage: Forage 8.6-20.3 months)
United States, Gardner, ND 2010, (Zea Mays GH4927)	EC 4 (7)	0.077 0.076 0.077 0.076	75	7	0.79, 1.1; (0.95)	<0.01, 0.015; (0.012)	TK0002562 C12-0476 (Storage: Forage 6.4 months)
United States, Bagley, IA 2010, (Not listed)	EC 4 (7)	0.074 0.071 0.077 0.074	73	**6**	2.2, 1.2; (1.7)	0.042, 0.026; (0.034)	TK0002562 C30-0477 (Storage: Forage 6.7 months)
United States, Oregon, MO 2010, (Bodacious)	EC 4 (7)	0.078 0.082 0.080 0.082	**R3**	7	0.97, 1.1; (1.0)	0.022, 0.025; (0.023)	TK0002562 C19-0478 (Storage: Forage 7.3 months)
United States, Centerville, SD 2011, (Kandy Korn)	EC 4 (7)	0.075 0.075 0.078 0.076	73	7	0.75, 0.58; **(0.66)**	0.018, 0.011; (0.015)	TK0002562 C16-0479 (Storage: Forage 6.6 months)
United States, Clarence, MO 2010, (Incredible)	EC 4 (7)	0.077 0.077 0.076 0.076	**R3-4**	7	0.97, 1.2; (1.1)	0.012, 0.016; (0.014)	TK0002562 C20-0480 (Storage: Forage 7.2 months)
United States, Porterville, CA 2010, (Bodacious)	EC 4 (7)	0.076 0.076 0.076 0.076	**R3**	**-3B 2** 7 **12 17**	**2.4, 2.7,** 2.0, 1.7; **(1.8)** **1.7, 2.0,**	0.026, 0.025, 0.030, 0.028; (0.029) 0.033, 0.030,	TK0002562 W32-0481 (Storage: Forage 7.5-8.2 months)
United States, Rupert, ID 2010, (Sugarbuns)	EC 4 (7)	0.072 0.076 0.076 0.080	77	7	0.23, 0.24; (0.24)	0.021, 0.020; (0.020)	TK0002562 W15-0482 (Storage: Forage 6.9 months)

SWEET CORN FORAGE Location Year (Variety)	Form No (RTI)	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report Trial Remark
United States, Hillsboro, OR 2010, (Honey and Pearls)	EC 4 (7)	0.076 0.075 0.077 0.077	79	7	0.25, 0.67; (0.46)	<0.01, <0.01; (<0.01)	TK0002562 W21-0483 (Storage: Forage 5.9 months)

For maize forage the whole plant was sampled. The growth stage at harvest was not stated in the study reports.

Table 42 Residues in maize (field corn) forage from field trials in the United States (JMPR 2016)

MAIZE FORAGE Location Year (variety)	Form no (RTI)	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
United States, Germansville, PA 2010, (TA 290-11)	EC 4 (7)	0.078 0.078 0.075 0.077	75 milk stage	7	0.68, 0.74; (0.71)	0.014, 0.012; (0.013)	TK0002562; E04-0451 (Storage: 7.0 months)
United States, Athens, GA 2010, (32B10)	EC 4 (7)	0.076 0.076 0.076 0.076	**36-39**	7	0.43, 0.23; (0.33)	<0.01, <0.01; (<0.01)	TK0002562; E12-0452 (Storage: 7.7 months)
United States, Gardner, ND 2010, (Int65D85R)	EC 4 (7)	0.077 0.077 0.077 0.077	65	**1** **4** 7 **10** **13**	**1.5,** **2.2,** 0.83, 0.63; **(0.73)** **0.82,** **0.47**	<0.01, 0.011, <0.01, <0.01; (<0.01) <0.01, <0.01	TK0002562; C12-0453 (Storage: 6.0-6.1 months)
United States, Northwood, ND, 2010, (DKC35-19/ A1002669)	EC 4 (7)	0.078 0.075 0.076 0.076	83	**1** **4** 7 **10** **13**	**0.98,** **1.2,** 0.57, 0.66; **(0.62)** **0.39,** **0.44**	0.013, 0.014, 0.014, 0.014; (0.014) <0.01, 0.015,	TK0002562; C13-0454 (Storage: 6.1 months)
United States, Fisk, MO 2010, (RL8950HB)	EC 4 (7)	0.076 0.077 0.076 0.076	85 dough stage	7	0.78, 1.2; **(0.97)**	0.020, 0.020; (0.020)	TK0002562; C23-0455 (Storage: 6.6-6.8 months)
United States, Oregon , MO 2010, (Pioneer 32T16)	EC 4 (7)	0.078 0.078 0.080 0.080	**R4 to early R5**	7	0.39, 0.45; (0.42)	0.055, 0.071; (0.063)	TK0002562; C19-0456 (Storage: 6.6 months)
United States, Fitchburg, WI 2010, (37Y12)	EC 4 (7)	0.077 0.076 0.076 0.076	85	7	0.44, 0.61; **(0.53)**	<0.01, <0.01; (<0.01)	TK0002562; C08-0457 (Storage: 6.0 months)
United States, Bagley, IA 2010,	EC 4 (7)	0.076 0.077 0.078	71	7	0.30, 0.34; (0.32)	<0.01, <0.01, (<0.01)	TK0002562; C30-0458 (Storage: 7.1 months)

MAIZE FORAGE Location Year (variety)	Form no (RTI)	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
(33D47)		0.073					
United States, Bolckow, MO 2010, (Mycogen2 K718)	EC 4 (7)	0.076 0.077 0.076 0.076	**R4**	7	0.74, **0.66;** (0.70)	0.011, 0.010; (0.011)	TK0002562; C19-0459 (Storage: 6.6 months)
United States, Sharon, ND 2010, (DKC35-19/ A1002669)	EC 4 (7)	0.077 0.076 0.077 0.078	83	7	0.84, 0.53; **(0.69)**	0.013, <0.01; (0.011)	TK0002562; C13-0460 (Storage: 6.1 months)
United States, Lesterville, SD 2010, (Golden Harvest H-8254 3000 GT, var. 162X579 14WP917)	EC 4 (7)	0.074 0.076 0.076 0.076	79	7	0.29, 0.37; (0.33)	<0.01, <0.01; (<0.01)	TK0002562; C16-0461 (Storage: 6.3 months)
United States, Richwood, OH 2010, (DKC57-66 VT3/RR2)	EC 4 (7)	0.077 0.077 0.077 0.077	60	7	0.32, 0.22; (0.27)	<0.01, <0.01; (<0.01)	TK0002562; C01-0462 (Storage: 5.6 months)
United States, Clarence, MO 2010, (Pioneer 33D49)	EC 4 (7)	0.078 0.075 0.075 0.076	**R4**	7	0.53, 1.0; (0.77)	0.026, 0.022; (0.024)	TK0002562; C20-0463 (Storage: 6.7 months)
United States, Osceola, NE 2010, (4947RB)	EC 4 (7)	0.076 0.075 0.076 0.076	85	7	0.70, 0.50; (0.60)	0.012, <0.01; (0.011)	TK0002562; C33-0464 (Storage: 6.2 months)
United States, Campbell, MN 2010, (DKC 38-89)	EC 4 (7)	0.076 0.076 0.076 0.076	83 early dough	7	0.38, 0.18; (0.28)	<0.01, <0.01; (<0.01)	TK0002562; C11-0465 (Storage: 6.1 months)
United States, Geneva, MN 2010, (Pioneer 38M60)	EC 4 (7)	0.076 0.077 0.075 0.076	R3 BBCH 75	7	0.22, 0.16; (0.19)	<0.01, <0.01; (<0.01)	TK0002562; C09-0466 (Storage: 6.6 months)
United States, Perry, IA 2010, (P1162XR)	EC 4 (7)	0.077 0.077 0.075 0.075	71	7	0.24, 0.17; (0.20)	<0.01, <0.01; (<0.01)	TK0002562; C30-0467 (Storage: 7.1 months)
United States, York, NE 2010, (X723 14WP.0)	EC 4 (7)	0.076 0.075 0.076 0.076	85	7	0.51, 0.54; **(0.53)**	<0.01, <0.01; (<0.01)	TK0002562; C33-0468 (Storage: 6.2 months)
United States,	EC	0.077	**late R4**	7	0.91,	0.018,	TK0002562;

MAIZE FORAGE Location Year (variety)	Form no (RTI)	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
Anabel, MO 2010, (33T57)	4 (7)	0.076 0.075 0.075			1.4; (1.2)	<0.01; (0.014)	C20-0469 (Storage: 6.6-12.5 months)
United States, Raymondville, TX 2010, (HG284162)	EC 4 (7)	0.077 0.077 0.078 0.080	85 dough stage	7	1.2, 1.2; (1.2)	0.014, 0.014; (0.014)	TK0002562; W08-0470 (Storage: 8.4 months)
United States, Wall, TX 2011, (Hybrid 111RM GT/CB/LL/RW)	EC 4 (7,8,8)	0.078 0.077 0.077 0.079	71 (23 Jul)	**7**	1.3, 2.1, 1.2; (1.5)	0.021, 0.047, 0.024; (0.030)	TK0058623; 01 (Storage: 227 days)
	WG 4 (7,8,8)	0.075 0.077 0.075 0.077	71 (23 Jul)	**7**	1.4, 2.4, 2.2; (2.0)	<0.01, <0.01, <0.01 (<0.01)	
United States, Bagley, IA 2011, (111RM)	EC 4 (7,8,6)	0.075 0.073 0.076 0.080	73 (11 Aug)	**7**	0.64, 0.64, 0.72; (0.67)	<0.01, <0.01, <0.01; (<0.01)	TK0058623; 02 (Storage:208 days)
	WG 4 (7,8,6)	0.077 0.077 0.076 0.078	73 (11 Aug)	**7**	1.1, 0.84, 0.80; (0.91)	<0.01, <0.01, <0.01; (<0.01)	
United States, Rice, MN 2011, (DKC 35-43)	EC 4 (7,7,7)	0.076 0.076 0.076 0.076	73 (24 Aug)	**7**	0.48, 0.34, 0.53; (0.45)	<0.01, <0.01, <0.01; (<0.01)	TK0058623; 03 (Storage: 195 days)
	WG 4 (7,7,7)	0.076 0.076 0.077 0.076	73 (24 Aug)	**7**	0.55, 0.40, 0.51; (0.49)	<0.01, <0.01, <0.01; (<0.01)	

Cereal hay/straw/stover

Residue information on barley hay/straw, wheat hay/straw and sweetcorn/maize stover was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information.

Barley hay

Table 43 Residues in barley hay from field trials in the United States and Canada (JMPR 2016)

BARLEY HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Germansville, PA 2010, (Nomini)	EC	2 (14)	0.077 0.076	BBCH 71	**6**	2.7, 1.9 (2.3)	0.12, 0.11 (0.12)	TK0002559; E04-0381 (Storage: 9.8 months)
United States, Northwood, ND 2010,	EC	2 **(15)**	0.076 0.076	Feekes 10.5.2	**6**	4.0, 4.0 (4.0)	0.20, 0.22 (0.21)	TK0002559; C13-0382 (Storage:

BARLEY HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
(Pinneacle)								8.1-21.9 months)
United States, Richland, IA **2011,** (Para-mount 66)	EC	2 **(12)**	0.076 0.076	Feekes 11.1	7	8.4, 10 (9.2)	0.42, 0.53 (0.48)	TK0002559; C18-0383 Data not used due to mis-application
United States, Clarence, MS 2010, (Lacey)	EC	2 **(11)**	0.074 0.077	BBCH 71	7	4.6, 3.4 (4.0)	0.24, 0.16 (0.20)	TK0002559; C20-0384 (Storage: 8.8 months)
United States, Jamestown, ND 2010, (Tradition)	EC	2 (14)	**0.078** 0.077	BBCH 71	**0** **3** 6 12 **14**	**11** **6.3** 6.3, 3.2 (4.8) 5.2 **4.3**	0.57 0.40 0.39, 0.29 (0.34) 0.33 0.26	TK0002559; C12-0385 (Storage: 8.7-9.0 months)
United States, Grand Island, NE 2010, (Baronesse)	EC	2 **(13)**	0.077 0.076	BBCH 71	7	2.4, 2.4 (2.4)	0.070, 0.069 (0.070)	TK0002559; C33-0386 (Storage: 8.5 months)
United States, Carrington, ND 2010, (**spring barley: Pinnacle)**	EC	2 **(15)**	0.077 0.076	Feekes 10.5.4	**8**	1.7, 1.6 (1.6)	0.076, 0.094 (0.085)	TK0002559; C13-0387 (Storage: 9.4 months)
United States, Lake Andes, SD 2010, (Tradition)	EC	2 **(15)**	0.076 0.077	**BBCH 71-73**	7	4.8, 5.4 (5.1)	1.0, 0.92 (0.96)	TK0002559; C16-0388 (Storage: 8.2 months)
United States, Berthoud, CO 2010, (Coors 69)	EC	2 (14)	0.077 0.077	Feekes 10.5.4	7	1.5, 1.5 (1.5)	0.084, 0.085 (0.084)	TK0002559; W12-0389 (Storage: 8.7 months)
United States, Madera, CA 2010, (Recleaned Whole Barley)	EC	2 (14)	0.076 0.077	BBCH 71	7	2.8, 2.4 (2.6)	0.17, 0.14 (0.16)	TK0002559; W29-0390 (Storage: 9.4 months)
United States, Hermiston, OR 2010, (Radiant)	EC	2 **(7)**	0.076 0.077	BBCH 84	7	2.4, 2.5 (2.4)	0.041, 0.049 (0.045)	TK0002559; W21-0391 (Storage: 8.0 months)
United States, Jerome, ID 2010, (Foster)	EC	2 **(13)**	0.077 0.077	BBCH 71	7	5.5, 3.9 (4.7)	0.48, 0.37 (0.42)	TK0002559; W16-0392 (Storage: 8.6 months)
Canada Taber, AB 2011, (CDC Earl)	EC	2 (14)	0.079 0.079	**71-73**	7	4.4, 5.5 (5.0)	0.067, 0.086 (0.076)	CER05902/11; T929 (Storage: 6.3 months)
Canada	EC	2	0.074	**69-71**	7	5.4,	0.14,	CER05902/11;

BARLEY HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
Elgin, MB 2011, (AC Metcalfe)		**(13)**	0.075	[04-Aug]		6.8 (6.1)	0.16 (0.15)	T930 (Storage: 6.4 months)
Canada Elgin, MB 2011, (Tradition)	EC	2 **(13)**	0.075 0.074	**71-75** [04-Aug]	7	4.9, 5.2 **(5.0)**	0.25, 0.26 (0.26)	CER05902/11; T932 (Storage: 6.4 months)
Canada Rosthern, SK 2011, (Metcalfe))	EC	2 (14)	0.079 0.076	**69-73**	8	3.4, 4.2 (3.8)	0.12, 0.14 (0.13)	CER05902/11; T933 (Storage: 2.8 months)
Canada Duck Lake, SK 2011, (Metcalfe)	EC	2 **(20)**	0.080 **0.078**	**69-72**	7	1.7, 1.5 (1.6)	0.074, 0.069 (0.072)	CER05902/11; T934 (Storage: 6.3 months)
Canada Fort Sask. AB 2011, (Coalition)	EC	2 **(8)**	0.075 0.075	**69-71**	7	5.2, 5.8 **(5.5)**	0.064, 0.071 (0.068)	CER05902/11; T935 (Storage: 2.0 months)
Canada Wellwood, MB 2011, (Conlon)	EC	2 (14)	0.072 0.075	71	7	9.3, 6.4 **(7.8)**	0.20 0.18 (0.19)	CER05902/11; T936 (Storage: 6.6 months)
Canada Minto, MB 2011, (Copeland)	EC	2 (14)	0.075 0.074	**71-73** [25-Jul]	7	6.3, 6.3 (6.3)	0.17, 0.16 (0.16)	CER05902/11; T931 (Storage: 3.2 months)
Canada Minto, MB 2011, (Legacy)	EC	2 **(15)**	0.075 0.075	**71-73** [21-Jul]	**4** **7** . **11** **14**	**6.6** 5.9, 5.5 (5.7) **5.4** **3.3**	0.28 0.27, 0.28 (0.28) 0.51 0.52	CER05902/11; T937 (Storage: 3.0 months)

Wheat hay

Table 44 Residues in wheat hay from field trials in the United States and Canada (JMPR 2016)

WHEAT HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Seven Springs, NC 2010, (Pioneer 26R15)	EC	2 **(13)**	0.077 0.078	67	7	1.3, 1.6 **(1.4)**	0.16, 0.19 (0.18)	TK0002558 E10-0351 (Storage: Hay 10.7 months)
United States, Shelbyville, MO 2010, **(soft red winter wheat: Erine)**	EC	2 (14)	0.075 0.078	**Feekes 10**	7	2.0, 1.9 (2.0)	0.35, 0.44 (0.40)	TK0002558 C20-0354 (Storage: Hay 10.5 months)

WHEAT HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Richland, IA 2010, (**soft red winter wheat:** Wilcross748)	EC	2 (14)	0.077 0.077	**Feekes 9.5-10**	7	0.49, 1.5 **(1.0)**	0.12, 0.18 (0.15)	TK0002558 C18-0355 (Storage: Hay 10.5 months)
United States, Milford Center, OH 2010, (Croplan Genetics 8614)	EC	2 (14)	0.077 0.077	Feekes 5	7	0.40, 0.67 (0.54)	0.23, 0.33 (0.28)	TK0002558 C01-0356 (Storage: Hay 9.9 months;)
United States, Macon, MO 2010, (**soft red winter wheat**: V9710)	EC	2 **(16)**	0.077 **0.078**	Feekes 10.5.1	7	1.5, 1.8 **(1.6)**	0.20, 0.22 (0.21)	TK0002558 C20-0357 (Storage: Hay 10.4 months)
United States, Carrington, ND 2010, (**soft white spring wheat:** AP-604-CL)	EC	2 **(13)**	0.077 **0.077**	45	7	0.52, 0.92 (0.72)	0.028, 0.27 (0.15)	TK0002558 C13-0359 (Storage: Hay 9.0 months)
United States, Carrington, ND 2010, **(hard red spring wheat**: Faller)	EC	2 **(13)**	0.077 **0.077**	45	7	0.52, 0.61 **(0.56)**	0.34, 0.51 (0.42)	TK0002558 C13-0361 (Storage: Hay 9.0 months)
United States, Jamestown, ND 2010, (**wheat: durum**: variety not known))	EC	2 (14)	0.077 0.077	33	**0** **3** 7 . . **10** **14**	**3.5** **1.6** 1.2, 2.1 **(1.6)** **0.92** **0.49**	0.18 0.23 0.28 0.27 (0.28) 0.32 0.25	TK0002558 C12-0360 (Storage: Hay 7.3-7.9 months)
United States, Lake Andes, SD 2010, (**hard white spring wheat**: Argent)	EC	2 **(13)**	0.076 0.075	**59-63**	7	1.9, 2.5 (2.2)	0.53, 0.53 (0.53)	TK0002558 C16-0362 (Storage: Hay 8.9 months)
United States, Grand Island, NE 2010, (**hard red winter wheat: NE 01643)**	EC	2 (14)	0.076 0.077	30	**8**	2.6, 2.7 **(2.6)**	0.25, 0.23 (0.24)	TK0002558 C33-0363 (Storage: Hay 10.4 months)
United States, Johnstown, CO 2010, (**winter wheat**: Yuma)	EC	2 (14)	**0.078** **0.078**	Feekes 9	7	3.0, 2.7 **(2.8)**	0.52, 0.41 (0.46)	TK0002558 W12-0364 (Storage: Hay 10.2 months)
United States, Eaton, CO 2010, (**winter wheat: Jagalene**)	EC	2 (14)	0.077 0.076	Feekes 9	**0** **3** 7	**6.1** **4.0** 1.6, 1.7	0.087 0.099 0.10, 0.11	TK0002558 W12-0365 (Storage: Hay 10.2 months)

WHEAT HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
					10 **14**	(1.6) **1.2** **1.6**	(0.10) 0.11 0.12	
United States, Milliken, CO 2010, (**hard red winter wheat:** Bill Brown)	EC	2 (14)	**0.078** 0.077	**Feekes 9-10**	7	2.5, 1.2 **(1.8)**	0.16, 0.16 (0.16)	TK0002558 W12-0369 (Storage: Hay 10.3 months)
United States, Rupert, ID 2010, (**hard white spring wheat:** Klassic)	EC	2 (14)	0.078 **0.088**	41	7	4.2, 3.4 (3.8)	0.13, 0.13 (0.13)	TK0002558 W15-0370 (Storage: Hay 9.5 months)
United States, Fisk, MO **2011,** (**soft red winter wheat**: Beretta)	EC	2 **(13)**	0.076 **0.077**	37	**8**	3.1, 2.7 (2.9)	0.28, 0.32 (0.30)	TK0002558 C23-0352 (Storage: Hay 6.2 months)
United States, Raymondville, TX **2011,** (**hard red winter wheat**: Caudillo)	EC	2 **(16)**	0.076 0.077	32	**6**	12, 11 **(12)**	1.3, 1.2 (1.2)	TK0002558 W08-0358 (Storage: Hay 5.2 months)
United States, Uvalde, TX **2011,** (**hard red winter wheat**: Tam 203)	EC	2 (14)	0.076 0.077	45	7	6.8, 5.5 (6.2)	0.33, 0.31 (0.32)	TK0002558 W07-0366 (Storage: Hay 3.5 months)
United States, Wall, TX **2011,** (**hard red winter wheat:** Coronado)	EC	2 **(13)**	0.077 0.078	33	7	9.1, 8.0 (8.6)	0.64, 0.66 (0.65)	TK0002558 W40-0367 (Storage: Hay 3.3 months)q
United States, Levelland, TX **2011,** (**hard red winter wheat**: Weather master)	EC	2 (14)	**0.074** **0.074**	32	7	5.7, 8.6 (7.2)	0.055, 0.067 (0.061)	TK0002558 W39-0368 (Storage: Hay 6.4 months)
United States, Valley City, ND 2011, (**winter wheat:** Falcon)	EC	**2** **(15)**	0.077 **0.077**	49	7	2.4, 2.0, 1.6 (2.0)	0.24, 0.25, 0.26 (0.25)	TK0048907 TK048907-01 (Storage: Hay 6.8 months)
	WG	2 **(15)**	0.076 0.078	49	7	1.4, 2.0, 1.5 (1.6)	0.12, 0.16, 0.090 (0.12)	
United States, Jamestown, ND 2011, (**winter wheat**: Overland)	EC	2 **(15)**	0.077 0.078	40	7	3.6, 3.8, 2.9 (3.4)	0.48, 0.41, 0.33 (0.41)	TK0048907 TK048907-02 (Storage: Hay 7.0 months)

WHEAT HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
	WG	2 **(15)**	0.077 0.078	40	7	2.3, 2.5, 2.7 (2.5)	0.13, 0.14, 0.20 (0.16)	
United States, Northwood, ND **2011,** **(hard red winter wheat**: Jerry)	EC	2 **(16)**	**0.080** 0.078	33 **[4 June]**	7	2.2, 2.1 (2.2)	0.040, 0.040 (0.040)	TK0002558 C13-0353 (Storage: Hay 4.7 months)
United States, Northwood, ND 2011, (**hard red spring wheat:** Faller)	EC	2 (14)	0.079 0.077	69 **[14 Jul]**	7	0.65, 0.79, 0.89 (0.78)	0.22, 0.28, 0.27 (0.26)	TK0048907 TK048907-03 (Storage: Hay 6.1 months)
	WG	2 (14)	0.078 0.078	69 **[14 Jul]**	7	0.68, 0.91, 0.76 (0.78)	0.18, 0.15, 0.15 (0.16)	
Canada Vanscoy, SK 2011, (Infinity))	EC	2 (14)	0.074 0.073	**57-59**	7	4.0, 4.2 (4.1)	0.14, 0.15 (0.14)	CER05901/11; T916 (Storage: 3.1 months)
Canada Kinley, SK 2011, (Infinity)	EC	2 (14)	**0.074** **0.073**	**58-61**	7	8.3, 8.7 (8.5)	0.46, 0.37 (0.42)	CER05901/11; T917 (Storage: 2.8 months)
Canada Taber, AB 2011, (Superb)	EC	2 (14)	**0.078** **0.079**	**33-37**	7	5.7, 6.3 (6.0)	0.13, 0.16 (0.14)	CER05901/11; T918 (Storage: 3.2 months)
Canada Boissevain, MB 2011, (**Spring wheat**: Harvest)	EC	2 **(13)**	**0.075** **0.078**	**41-45** **[16 Jul]**	6	7.4, 6.2, 6.1, 6.0, 7.0, 6.6 (6.6)	0.83, 0.87, 0.42, 0.80, 0.41, 0.75 (0.68)	CER05901/11; T919 (Storage: 7.2 months)
Canada Boissevain, MB 2011, (**Spring wheat**: Kane)	EC	2 **(13)**	**0.076** **0.073**	**41-43** **[16 Jul]**	**6**	4.6, 4.5, 6.3, 5.5, 6.9, 5.9 (5.6)	0.66, 0.52 0.51, 0.57 0.77, 0.91 (0.66)	CER05901/11; T920 (Storage: 5.1 months)
Canada Rosthern, SK 2011, (Infinity)	EC	2 **(13)**	**0.074** **0.076**	**45-59**	**8**	4.1, 3.6 **(3.8)**	0.22, 0.17 (0.20)	CER05901/11; T921 (Storage: 3.0 months)
Canada Blaine Lake, SK 2011,	EC	2 **(15)**	0.073 0.076	**69-71**	6	4.3, 3.5 (3.9)	0.18, 0.18 (0.18)	CER05901/11; T922 (Storage: 6.5 months)

WHEAT HAY Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
(Infinity)								
Canada Duck Lake, SK 2011, (Infinity)	EC	2 (14)	**0.075** **0.075**	**51-61**	7	6.9, 7.3 (7.1)	0.24, 0.26 (0.25)	CER05901/11; T923 (Storage: 2.6 months)
Canada Kipp, AB 2011, (Superb)	EC	2 **(15)**	**0.076** **0.079**	**53-58**	6	7.4, 6.4 (6.9)	0.24, 0.20 (0.22)	CER05901/11; T924 (Storage: 2.8 months)
Canada Alvena, SK 2011, (Goodeve – Ac Intrepid)	EC	2 (14)	**0.073** 0.071	**55-59**	7	6.1, 4.3 (5.2)	0.57, 0.25 (0.41)	CER05901/11; T925 (Storage: 2.8 months)
Canada Fort Sask. AB, 2011, (Harvest)	EC	2 (14)	**0.075** **0.077**	**41-61**	7	2.7, 2.3 (2.5)	0.47, 0.33 (0.40)	CER05901/11; T926 (Storage: 2.8 months)
Canada Minto, MB 2011, (Superb)	EC	2 (14)	**0.076** **0.074**	**37-41** **[7 Jul]**	**4** 7 . **11** **14**	**7.7** 4.8, 4.4 (4.6) **2.7** **1.5**	0.49 0.44, 0.20 (0.32) 0.41 0.39	CER05901/11; T927 (Storage: 3.2 months)
Canada Minto, MB 2011, (AC Barrie)	EC	2 (14)	**0.073** 0.076	**37-41** **[7 Jul]**	**4** 7 . **11** **14**	**6.3** 5.2, 5.6 (5.4) **1.9** **1.1**	0.36 0.38, 0.42 (0.40) 0.39 0.25	CER05901/11; T928 (Storage: 5.7 months)

Barley straw

No trials were selected for barley straw; trials were selected from barley hay and wheat hay.

Table 45 Residues in barley straw from field trials in the United States and Canada (JMPR 2016)

BARLEY STRAW Location Year (variety)	Form.	no	kg ai/ha	BBCH	**DALA**	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
United States, Germansville, PA 2010, (Nomini)	EC	2 (14)	0.077 0.076	BBCH 71	**52**	2.0, 1.8 (1.9)	0.12, 0.30 (0.21)	TK0002559; E04-0381 (Storage: 9.0 months)
United States, Northwood, ND 2010, (Pinneacle)	EC	2 **(15)**	0.076 0.076	Feekes 10.5.2	**47**	0.53, 0.27 (0.40)	0.044, 0.029 (0.036)	TK0002559; C13-0382 (Storage: 7.1 months)
United States, Richland, IA 2010, (Para-	EC	2 **(12)**	0.076 0.076	Feekes 11.1	**26**	**12,** **11** **(11)**	1.0, 0.81 (0.90)	TK0002559; C18-0383 Data not used due to mis-

BARLEY STRAW Location Year (variety)	Form.	no	kg ai/ha	BBCH	**DALA**	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
mount 66)								application
United States, Clarence, MS 2010, (Lacey)	EC	2 **(11)**	0.074 0.077	BBCH 71	**23**	2.1, 1.6 (1.8)	0.11, 0.084 (0.097)	TK0002559; C20-0384 (Storage: 8.4 months)
United States, Jamestown, ND 2010, (Tradition)	EC	2 (14)	**0.078** 0.077	BBCH 71	**19** 26 **33** **40**	**1.7,** 2.0, 2.3 **(2.2)** **3.2** **3.1**	0.14, 0.098, 0.13 (0.11) 0.15 0.16	TK0002559; C12-0385 (Storage: 6.7-7.2 months)
United States, Grand Island, NE 2010, (Baronesse)	EC	2 **(13)**	0.077 0.076	BBCH 71	**16**	3.5, 3.0 (3.2)	0.010, 0.085 (0.048)	TK0002559; C33-0386 (Storage: Straw 8.2 months)
United States, Carrington, ND 2010, (**spring barley: Pinnacle)**	EC	2 **(15)**	0.077 0.076	Feekes 10.5.4	**41**	0.20, 0.22 (0.21)	0.019, 0.029 (0.024)	TK0002559; C13-0387 (Storage: 7.2 months)
United States, Lake Andes, SD 2010, (Tradition)	EC	2 **(15)**	0.076 0.077	**BBCH 71-73**	**34**	5.0, 4.0 (4.5)	0.45, 0.30 (0.38)	TK0002559; C16-0388 (Storage: 7.3 months)
United States, Berthoud, CO 2010, (Coors 69)	EC	2 (14)	0.077 0.077	Feekes 10.5.4	**28**	0.70, 0.96 (0.83)	0.14, 0.17 (0.16)	TK0002559; W12-0389 (Storage: 8.1 months)
United States, Madera, CA 2010, (Recleaned Whole Barley)	EC	2 (14)	0.076 0.077	BBCH 71	**23**	2.1, 2.2 (2.2)	0.15, 0.14 (0.14)	TK0002559; W29-0390 (Storage: 8.9 months)
United States, Hermiston, OR 2010, (Radiant)	EC	2 **(7)**	0.076 0.077	BBCH 84	**29**	2.7, 2.9 (2.8)	0.041, 0.039 (0.040)	TK0002559; W21-0391 (Storage: 7.4 months)
United States, Jerome, ID 2010, (Foster)	EC	2 **(13)**	0.077 0.077	BBCH 71	**41**	1.6, 1.5 (1.6)	0.15, 0.13 (0.14)	TK0002559; W16-0392 (Storage: 7.5-20.9 months)
Canada Taber, AB 2011, (CDC Earl)	EC	2 (14)	0.079 0.079	**71-73**	22	3.3, 5.9 **(4.6)**	0.056, 0.086 (0.071)	CER05902/11; T929 (Storage: 6.3 months)
Canada Elgin, MB 2011, (AC Metcalfe)	EC	2 **(13)**	0.074 0.075	**69-71** [04-Aug]	41	10.0, 5.5 (7.8)	0.16, 0.15 (0.16)	CER05902/11; T930 (Storage: 6.4 months)
Canada Elgin, MB 2011,	EC	2 **(13)**	0.075 0.074	**71-75** [04-Aug]	36	6.0, 5.5 (5.8)	0.19, 0.23 (0.21)	CER05902/11; T932 (Storage: 6.4 months)

BARLEY STRAW Location Year (variety)	Form.	no	kg ai/ha	BBCH	**DALA**	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
(Tradition)								
Canada Rosthern, SK 2011, (Metcalfe))	EC	2 (14)	0.079 0.076	**69-73**	35	1.8, 1.8 (1.8)	0.11, 0.098 (0.10)	CER05902/11; T933 (Storage: 2.8 months)
Canada Duck Lake, SK 2011, (Metcalfe)	EC	2 **(20)**	0.080 **0.078**	**69-72**	44	3.3, 1.5 (2.4)	0.15, 0.080 (0.12)	CER05902/11; T934 (Storage: 6.3 months)
Canada Fort Sask. AB 2011, (Coalition)	EC	2 **(8)**	0.075 0.075	**69-71**	37	3.6, 3.3 **(3.4)**	0.061, 0.058 (0.060)	CER05902/11; T935 (Storage: 2.0 months)
Canada Wellwood, MB 2011, (Conlon)	EC	2 (14)	0.072 0.075	71	34	9.0, 5.1 **(7.0)**	0.51, 0.17 (0.34)	CER05902/11; T936 (Storage: 6.6 months)
Canada Minto, MB 2011, (Copeland)	EC	2 (14)	0.075 0.074	**71-73** [25-Jul]	25	4.2, 3.2 (3.7)	0.13, 0.10 (0.12)	CER05902/11; T931 (Storage: 3.2 months)
Canada Minto, MB 2011, (Legacy)	EC	2 **(15)**	0.075 0.075	**71-73** [21-Jul]	**21** **29** **35** **43**	**1.1** **0.90, 0.82 (0.86)** **0.66** **1.7**	0.19 0.15, 0.14 (0.14) 0.057 0.27	CER05902/11; T937 (Storage: 3.0 months)

Wheat straw

No trials were selected for wheat straw; trials were selected from barley hay and wheat hay.

Table 46 Residues in wheat straw from field trials in the United States and Canada (JMPR 2016)

WHEAT STRAW Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Seven Springs, NC 2010, (Pioneer 26R15)	EC	2 **(15)**	**0.077** 0.078	71	**21**	3.8, 4.3 (4.0)	0.19, 0.22 (0.20)	TK0002558 E10-0351 (Storage: Straw 9.9 months)
United States, Fisk, MO **2011,** (**soft red winter wheat**: Beretta)	EC	2 **(16)**	0.076 0.076	73	**18**	5.5, 5.7 (5.6)	0.39, 0.42 (0.40)	TK0002558 C23-0352 (Storage: Straw 5.4 months)
United States, Shelbyville, MO 2010, (**soft red winter wheat:**	EC	2 **(16)**	0.**079** 0.077	Feekes 10.5	**38**	0.76, 0.67 (0.72)	0.22, 0.17 (0.20)	TK0002558 C20-0354 (Storage: Straw 9.2 months)

WHEAT STRAW Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
Erine)								
United States, Richland, IA 2010, (**soft red winter wheat:** Wilcross748)	EC	2 (14)	0.076 0.076	Feekes 10.5.4	**10**	0.83, 1.2 (1.0)	0.052, 0.071 (0.062)	TK0002558 C18-0355 (Storage: Straw 9.1 months)
United States, Milford Center, OH 2010, (Croplan Genetics 8614)	EC	2 **(13)**	0.077 0.077	**Feekes 10.54**	**14**	0.044, 1.9, **(0.97)**	<0.01, 0.10 (0.055)	TK0002558 C01-0356 (Storage: Straw 9.1 months)
	EC	2 (14)	0.077 0.077	**Feekes 5 + 14 days**	**35**	0.11, 0.10 (0.10)	0.059, 0.057 (0.058)	
United States, Macon, MO 2010, (**soft red winter wheat**: V9710)	EC	2 **(16)**	0.077 0.077	Feekes 10.5.1	**41**	0.38, <0.01 **(0.19)**	0.20, <0.01 (0.10)	TK0002558 C20-0357 (Storage: Straw 9.4 months)
United States, Raymondville, TX **2011,** (**hard red winter wheat**: Caudillo)	EC	2 (14)	**0.079 0.079**	Feekes 10.5.4	**44**	6.7, 7.0 (6.8)	0.64, 0.65 (0.64))	TK0002558 W08-0358 (Storage: Straw 6.1 months)
United States, Carrington, ND 2010, (**soft white s**pring wheat: AP-604-CL)	EC	2 **(11)**	0.076 0.075	Feekes 10.5.4	**41**	0.23, 0.23 (0.23)	0.14, 0.13 (0.14)	TK0002558 C13-0359 (Storage: Straw 7.6 months)
	EC	2 **(13)**	**0.077 0.077**	**BBCH 45**	**52**	0.052, 0.064 **(0.058)**	0.048, 0.046 (0.047)	
United States, Carrington, ND 2010, (**hard red spring wheat**: Faller)	EC	2 **(11)**	0.077 0.075	Feekes 10.5.4	**41**	0.14, 0.13, **(0.14)**	0.093, 0.090 (0.092)	TK0002558 C13-0361 (Storage: Straw 6.9-7.6 months)
	EC	2 **(13)**	0.077 **0.077**	**BBCH 45**	**52**	0.033, 0.035 **(0.034)**	0.030, 0.028 (0.029)	
United States, Lake Andes, SD 2010, (**hard white spring wheat**: Argent)	EC	2 **(15)**	0.076 0.076	**71-73**	**34**	3.6, 3.1 **(3.4)**	0.66, 0.53 (0.60)	TK0002558 C16-0362 (Storage: Straw 7.8 months)
	EC	2 **(13)**	0.076 **0.075**	**59-63**	**44**	1.2, 1.1 **(1.2)**	0.16, 0.19 (0.18)	
United States, Grand Island, NE 2010, (**hard red winter wheat: NE 01643)**	EC	2 **(13)**	0.077 0.077	71	**31**	1.8, 1.7 **(1.8)**	0.38, 0.39 (0.38)	TK0002558 C33-0363 (Storage: Straw 8.9 months)
	EC	2 (14)	0.077 0.077	**30**	**57**	0.14, 0.19 **(0.16)**	0.073, 0.073 (0.073)	
United States, Johnstown, CO	EC	2 **(15)**	0.078 0.077	Feekes 10.5.4	**26**	**3.3**, 4.9	0.21, 0.36	TK0002558 W12-0364

WHEAT STRAW Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
2010, (**winter wheat**: Yuma)						**(4.1)**	**(0.28)**	(Storage: Straw 8.7 months)
United States, Eaton, CO 2010, (**winter wheat: Jagalene**)	EC	2 (14)	0.076 0.078	Feekes 10.5.4	**23** **30** **.** **.** **37** **41**	2.3 2.2, 2.4 **(2.3)** 3.7 3.5	0.15 0.15, 0.12 (0.14) 0.18 0.22	TK0002558 W12-0365 (Storage: Straw 8.0-8.7 months)
United States, Uvalde, TX **2011,** (**hard red winter wheat**: Tam 203)	EC	2 **(12)**	0.075 0.075	71	**34**	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	TK0002558 W07-0366 (Storage: Straw 5.6 months)
United States, Wall, TX **2011,** (**hard red winter wheat:** Coronado)	EC	2 **(12)**	0.077 **0.078**	**BBCH 71** **Feekes** **10.54**	**37**	8.0, 8.7 (8.4)	0.18, 0.18 (0.18)	TK0002558 W40-0367 (Storage: Straw 5.2-9.1 months)
United States, Levelland, TX **2011,** (**hard red winter wheat**: Weather master)	EC	2 (14)	0.075 **0.076**	Feekes 10.5.4	**35**	4.9, 4.5 (4.7)	0.22, 0.20 (0.21)	TK0002558 W39-0368 (Storage: Straw 5.3 months)
United States, Milliken, CO 2010, (**hard red winter wheat:** Bill Brown)	EC	2 **(15)**	0.078 0.076	Feekes 10.5.4	**26**	6.0, 6.4 (6.2)	0.19, 0.16 (0.18)	TK0002558 W12-0369 (Storage: Straw 8.7 months)
United States, Rupert, ID 2010, (**hard white spring wheat:** Klassic)	EC	2 (14)	0.076 **0.078**	71	**39**	0.13, 0.098 (0.11)	0.070, 0.036 (0.053)	TK0002558 W15-0370 (Storage: Straw 7.7 months)
United States, Valley City, ND 2011, (**winter wheat:** Falcon)	EC	2 (14)	0.079 0.076	73	**23**	0.92, 0.98, 2.1, 2.7, 2.4 (1.8)	0.16, 0.15 0.18, 0.18, 0.19 (0.17)	TK0048907 TK048907-01 (Storage: Straw 5.7-6.9 months)
	WG	2 (14)	0.077 0.077	73	**23**	0.79, 0.69, 0.85 (0.78)	0.071, 0.074, 0.072 (0.072)	
United States, Jamestown, ND 2010, (**wheat: durum**: variety not known))	EC	2 (14)	0.077 0.076	71	**28** **36** **42** **49**	1.1 0.84, 0.88 (0.86) 0.74 2.2	0.064 0.069 0.055 (0.062) 0.075 0.083	TK0002558 C12-0360 (Storage: Straw 5.6-6.2 months)
	EC	2 (14)	0.077 0.076	**BBCH 33**	**59** **67**	0.055 0.051, 0.058	0.012 0.011 <0.01	

WHEAT STRAW Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
					73 **80**	**(0.054)** 0.034 0.078	(0.010) <0.01 0.014	
United States, Jamestown, ND 2011, (**winter wheat**: Overland)	EC	2 (14)	0.077 0.077	73	**22**	0.96, 1.2, 0.82 (0.99)	0.15, 0.15, 0.15 (0.15)	TK0048907 TK048907-02 (Storage: Straw 5.7-7.0 months)
	WG	2 (14)	0.077 0.077	73	**22**	1.1, 0.93, 0.61 (0.88)	0.16, 0.15, 0.16 (0.16)	
United States, Northwood, ND **2011,** **(hard red winter wheat**: Jerry)	EC	2 (14)	0.077 **0.079**	Feekes 10.5.4	**41**	0.26, 0.25, **(0.26)**	0.062, 0.072 (0.067)	TK0002558 C13-0353 (Storage: Straw 3.7 months)
	EC	2 **(16)**	**0.080** 0.078	**BBCH 33**	**61**	0.14, **0.17** **(0.16)**	0.040, 0.049 (0.044)	
United States, Northwood, ND 2011, (**hard red spring wheat:** Faller)	EC	2 (14)	0.076 0.077	71	**33**	2.2, 1.8, 1.9 (2.0)	0.60, 0.53, 0.34 (0.49)	TK0048907 TK048907-03 (Storage: Straw 5.1 months)
	WG	2 (14)	0.077 0.076	71	**33**	1.2, 1.2, 1.2 (1.2)	0.31, 0.35, 0.29 (0.32)	
Canada Vanscoy, SK 2011, (Infinity))	EC	2 (**13**)	0.074 **0.074**	**69-71**	36	4.6, 3.2 **(3.9)**	0.27, 0.21 (0.24)	CER05901/11; T916 (Storage: 3.1 months)
Canada Kinley, SK 2011, (Infinity)	EC	2 (**24**)	0.079 0.075	**69-71**	41	3.5, 2.5 (3.0)	0.46, 0.42 (0.44)	CER05901/11; T917 (Storage: 2.8 months)
Canada Taber, AB 2011, (Superb)	EC	2 **(15)**	0.079 0.078	**67-71**	32	4.6, 4.8 (4.7)	0.18, 0.19 (0.18)	CER05901/11; T918 (Storage: 3.2 months)
Canada Boissevain, MB 2011, (**Spring wheat**: Harvest)	EC	2 **(13)**	0.076 0.075	**69-71**	40	0.75, 0.74 **(0.74)**	0.085, 0.080 (0.082)	CER05901/11; T919 (Storage: 7.2 months)
Canada Boissevain, MB 2011, (**Spring wheat**: Kane)	EC	2 **(13**	0.075 0.075	**69-71**	43	1.6, 2.9 (2.2)	0.16, 0.24 (0.20)	CER05901/11; T920 (Storage: 5.1 months)
Canada Rosthern, SK 2011, (Infinity)	EC	2 (14)	0.071 0.072	**69-71**	52	1.6, 0.94 (1.3)	0.31, 0.19 (0.25)	CER05901/11; T921 (Storage: 3.0 months)
Canada Blaine Lake, SK 2011, (Infinity)	EC	2 **(15)**	0.073 0.076	**69-71**	54	0.43, 0.42 **(0.42)**	0.092, 0.086 (0.089)	CER05901/11; T922 (Storage: 6.5 months)

WHEAT STRAW Location, Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
Canada Duck Lake, SK 2011, (Infinity)	EC	2 **(21)**	0.080 0.076	**69-71**	41	1.2, 1.3 **(1.2)**	0.082, 0.083 (0.082)	CER05901/11; T923 (Storage: 2.6 months)
Canada Kipp, AB 2011, (Superb)	EC	2 **(15)**	0.077 0.077	**69-71**	42	2.3, 2.2 **(2.2)**	0.19, 0.21 (0.20)	CER05901/11; T924 (Storage: 2.8 months)
Canada Alvena, SK 2011, (Goodeve – Ac Intrepid)	EC	2 (14)	0.078 0.071	**70-71**	28	2.5, 3.3 (2.9)	0.27, 0.39 (0.33)	CER05901/11; T925 (Storage: 2.8 months)
Canada Fort Sask. AB, 2011, (Harvest)	EC	2 **(8)**	0.076 0.074	**69-71**	44	3.6, 5.1 (4.4)	0.13, 0.12 (0.12)	CER05901/11; T926 (Storage: 2.8 months)
Canada Minto, MB 2011, (Superb)	EC	2 (14)	0.079 0.075	**71-73**	**27** 35 . **42** **48**	**1.6** 4.5, 3.3 (3.9) **3.2** **1.9**	0.11 0.25, 0.19 (0.22) 0.18 0.16	CER05901/11; T927 (Storage: 3.2 months)
Canada Minto, MB 2011, (**spring wheat**: AC Barrie)	EC	2 (14)	0.077 0.076	**69-71**	**27** 35 . **42** **48**	**1.1** 1.1, 1.4 **(1.2)** 1.3 1.6	0.076 0.087, 0.11 (0.098) 0.091 0.11	CER05901/11; T928 (Storage: 5.7 months)

Sweet corn and maize stover

No trials were selected for sweet corn stover or maize stover from the United States.

Note from the reviewer: Report TK0002562: Sweet corn forage and stover samples were each at the same location and received each the same treatment at the same day. Ears were picked by hand and part of the stalks without ears were harvested as "sweet corn forage" at the same day as the sweet corn ears. The remaining stalks were left in the field and were harvested at a later date as "sweet corn stover". Report TK0002562 marks these samples as stover DALA 7. For some of these samples, the actual harvest date was much later than 7 days. For some samples the DALA was indeed 7 days; these samples must be considered replicates of sweet corn forage.

Table 47 Residues in sweet corn stover from field trials in United States (JMPR 2016)

SWEET CORN STOVER Location, Year (variety)	Form.	no	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
United States, Germansville, PA 2010, (Extra Tender	EC	4 (7)	0.077 0.076 0.077 0.077	73 early milk stage	**42**	1.2, 0.91; **(1.0)**	0.028, 0.020; (0.024)	TK0002562 E04-0472 (Storage: Stover 6.9 months)

SWEET CORN STOVER Location, Year (variety)	Form.	no	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)
274A)								
United States, North Rose, NY 2010, (Serendipity)	EC	4 (7)	0.078 0.078 0.080 0.078	75 early mature ears	**14**	1.4, 1.6; (1.5)	0.027, 0.032; (0.029)	TK0002562 **E02-0473** (Storage: Stover 5.7 months)
United States, Athens, GA 2010, (Silver King)	EC	4 (7)	0.076 0.076 0.077 0.076	**72-73**	7 [a]	2.5, 2.5; (2.5)	0.034, 0.043; (0.038	TK0002562 **E12-0474** (Storage: Stover 7.0 months)
United States, Oviedo, FL 2010, (Silver Queen)	EC	4 (7)	0.075 0.077 0.076 0.078	71	**45**	0.13, 0.11 (0.12)	<0.01, <0.01; (<0.01)	TK0002562 **E15-0475** (Storage: Stover 6.8-7.2 months)
United States, Gardner, ND 2010, (Zea Mays GH4927)	EC	4 (7)	0.077 0.076 0.077 0.076	75	**58**	0.31, 0.20; **(0.25)**	0.011, <0.01; (0.010)	TK0002562 C12-0476 (Storage: Stover 4.6 months)
United States, Bagley, IA 2010, (Not listed)	EC	4 (7)	0.074 0.071 0.077 0.074	73	**6** [a]	1.3, 1.4; (1.4)	0.021, 0.026; (0.023)	TK0002562 C30-0477 (Storage: Stover 10.6 months)
United States, Oregon, MO 2010, (Bodacious)	EC	4 (7)	0.078 0.082 0.080 0.082	**R3**	**35**	0.31, 0.39; (0.35)	0.018, 0.024; (0.021)	TK0002562 C19-0478 (Storage: Stover 6.2 months)
United States, Centerville, SD 2011, (Kandy Korn)	EC	4 (7)	0.075 0.075 0.078 0.076	73	**48**	0.21, 0.23; (0.22)	<0.01, 0.011; (0.010)	TK0002562 C16-0479 (Storage: Stover 5.1 months)
United States, Clarence, MO 2010, (Incredible)	EC	4 (7)	0.077 0.077 0.076 0.076	**R3-4**	**17**	3.2, 2.8 (3.0)	0.032 0.031 (0.032)	TK0002562 C20-0480 (Storage: Stover 6.7 months)
United States, Porterville, CA 2010, (Bodacious)	EC	4 (7)	0.076 0.076 0.076 0.076	**R3**	**31** **36** **41** **46** **51**	**3.1** **1.4** 1.8, 1.8 (1.8) **2.0** **1.6**	0.054, 0.025, 0.046, 0.034; (0.040) 0.042, 0.026	TK0002562 W32-0481 (Storage: Stover 6.3-6.9 months)
United States, Rupert, ID 2010, (Sugarbuns)	EC	4 (7)	0.072 0.076 0.076 0.080	77	**56**	0.18, 0.26; **(0.22)**	0.019, 0.016; (0.017)	TK0002562 W15-0482 (Storage: Stover 5.2 months)
United States, Hillsboro, OR 2010, (Honey and Pearls)	EC	4 (7)	0.076 0.075 0.077 0.077	79	**7** [a]	0.48, 0.58 (0.53)	0.012 0.014 (0.013)	TK0002562 W21-0483 (Storage: Stover 5.8 months)

Notes:

[a] replicate sample of sweet corn forage (treatment and harvest at the same day as the sample marked as sweet corn forage)

Table 48 Residues in maize stover (field corn stover) from field trials in the United States (JMPR 2016)

MAIZE STOVER Location Year (variety)	Form.	no	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
United States, Germansville, PA 2010, (TA 290-11)	EC	4 (7)	0.080 0.078 0.086 0.077	R6 BBCH 89	7	5.6, 6.5; **(6.0)**	0.036, 0.035; (0.036)	TK0002562; E04-0451 (Storage: 5.7-9.7 months)
United States, Athens, GA 2010, (32B10)	EC	4 (7)	0.076 0.076 0.075 0.075	86-88	7	2.5, 1.7; (2.1)	0.044, 0.036; (0.040)	TK0002562; E12-0452 (Storage: 5.8-9.9 months)
United States, Gardner, ND 2010, (Int65D85R)	EC	4 (7)	0.076 0.077 0.077 0.076	96	**-3B** **2** 7 **12** **17**	**1.7, 2.8** 2.1, 2.2, **(2.1)** 3.2, 3.1	<0.01, <0.01, <0.01, <0.01; (<0.01) <0.01 <0.01	TK0002562; C12-0453 (Storage: 4.5-5.1 months)
United States, Northwood, ND, 2010, (DKC35-19/ A1002669)	EC	4 (7)	0.076 0.076 0.076 0.076	89	**-3B** **2** 7 **12** **17**	**3.8, 5.6,** 2.9, 3.0**,** **(2.9)** 3.1 **2.1**	<0.01, <0.01, <0.01, <0.01; (<0.01) <0.01 <0.01	TK0002562; C13-0454 (Storage: 4.3 months)
United States, Fisk, MO 2010, (RL8950HB)	EC	4 (7)	0.076 0.076 0.076 0.077	BBCH 95 50 percent leaves changed colour	7	4.3, 8.1 (6.2)	0.064, 0.091 (0.078)	TK0002562; C23-0455 (Storage: 10.3 months)
United States, Oregon , MO 2010, (Pioneer 32T16)	EC	4 (7)	0.078 0.076 0.078 0.075	**late R5, just turning R6**	7	2.6, 2.8 (2.7)	0.064, 0.060 (0.062)	TK0002562; C19-0456 (Storage: 5.6 months)
United States, Fitchburg, WI 2010, (37Y12)	EC	4 (7)	0.076 0.077 0.076 0.075	96	7	2.2, 1.1; **(1.6)**	0.010 <0.01 (0.010)	TK0002562; C08-0457 (Storage: 5.0 months)
United States, Bagley, IA 2010, (33D47)	EC	4 (7)	0.074 0.077 0.080 0.077	97	7	4.8, 5.7; (5.3)	0.012, 0.020; (0.016)	TK0002562; C30-0458 (Storage: 8.8 months)
United States, Bolckow, MO 2010, (Mycogen2 K718)	EC	4 (7)	0.077 0.077 0.077 0.076	**late R5**	7	1.9, 2.0; (2.0)	0.017, 0.034; (0.025)	TK0002562; C19-0459 (Storage: 5.7 months)
United States, Sharon, ND 2010, (DKC35-19/	EC	4 (7)	0.077 0.077 0.076 0.076	89	7	2.3, 3.4 (2.9)	<0.01, <0.01; (<0.01)	TK0002562; C13-0460 (Storage: 4.3 months)

MAIZE STOVER Location Year (variety)	Form.	no	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
A1002669)								
United States, Lesterville, SD 2010, (Golden Harvest H-8254 3000 GT, var. 162X579 14WP917)	EC	4 (7)	0.076 0.077 0.077 0.077	92	7	2.2, 2.6 (2.4)	0.017, 0.019 (0.018)	TK0002562; C16-0461 (Storage: 5.2 months)
United States, Richwood, OH 2010, (DKC57-66 VT3/RR2)	EC	4 (7)	0.077 0.077 0.077 0.077	**85/87**	7	**2.0,** 3.3; (2.7)	0.014, 0.017; (0.016)	TK0002562; C01-0462 (Storage: 4.7 months)
United States, Clarence, MO 2010, (Pioneer 33D49)	EC	4 (7)	0.076 0.077 0.082 0.078	**R6**	7	4.3, 5.4 (4.9)	0.066, 0.071 (0.069)	TK0002562; C20-0463 (Storage: 5.6 months)
United States, Osceola, NE 2010, (4947RB)	EC	4 (7)	0.077 0.076 0.076 0.077	89	7	6.6, 7.1 (6.9)	0.029, 0.038 (0.033)	TK0002562; C33-0464 (Storage: 9.1 months)
United States, Campbell, MN 2010, (DKC 38-89)	EC	4 (7)	0.076 0.076 0.076 0.076	85	7	1.5, 1.4 (1.4)	0.010 <0.01 (0.010)	TK0002562; C11-0465 (Storage: 5.4 months)
United States, Geneva, MN 2010, (Pioneer 38M60)	EC	4 (7)	0.078 0.076 0.075 0.076	R6 BBCH 89	7	**3.2**, 4.0 **(3.6)**	<0.01 0.010 (0.010)	TK0002562; C09-0466 (Storage: 5.2 months)
United States, Perry, IA 2010, (P1162XR)	EC	4 (7)	0.075 0.078 0.076 0.080	97	7	6.3, 2.6; **(4.4)**	0.019, 0.014; (0.016)	TK0002562; C30-0467 (Storage: 4.9 months)
United States, York, NE 2010, (X723 14WP.0)	EC	4 (7)	0.076 0.076 0.076 0.077	89	7	4.6, **2.4; (3.5)**	0.020 0.014 (0.017)	TK0002562; C33-0468 (Storage: 5.2 months)
United States, Anabel, MO 2010, (33T57)	EC	4 (7)	0.074 0.075 0.074 0.080	**R6**	7	3.5, 2.7 (3.1)	0.16 0.13 (0.15)	TK0002562; C20-0469 (Storage: 5.7 months)
United States, Raymondville, TX 2010, (HG284162)	EC	4 (7)	0.080 0.078 0.078 0.078	87 physio logical maturity	7	2.5, 3.7 (3.1)	0.049 0.093 (0.071)	TK0002562; W08-0470 (Storage: 7.1 months)
United States, Levelland, TX	EC	4 (7)	0.076 0.075	**ripe grain**	7	4.0, 3.6	0.016 0.011	TK0002562; W39-0471

MAIZE STOVER Location Year (variety)	Form.	no	kg ai/ha	BBCH at last treatment	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
2010, (AP2504) Popcorn stover			0.075 0.077			(3.8)	(0.013)	(Storage: 5.9 months)
United States, Wall, TX 2011, (Hybrid 111RM GT/CB/LL/RW)	EC	**4 (8,6,6)**	0.077 0.075 0.077 0.077	89 (29 Aug)	**6**	4.3 5.3 4.6 (4.7)	0.032 0.027 0.037 (0.032)	**TK0058623; 01 (Storage: 191 days)**
	WG	**4 (8,6,6)**	0.076 0.076 0.077 0.077	89 (29 Aug)	**6**	4.6, 4.3, 3.7; (4.2)	0.019, 0.019, 0.019; (0.019)	
United States, Bagley, IA 2011, (111RM)	EC	**4 (7,7,7)**	0.074 0.075 0.078 0.078	**95 (5 Oct)**	7	8.9 9.4 8.4 (8.9)	0.013 0.013 0.012 (0.012)	**TK0058623; 02 (Storage: 153 days)**
	WG	**4 (7,7,7)**	0.077 0.076 0.077 0.074	**95 (5 Oct)**	7	13, 8.8, 11; (11)	0.015, 0.018, <0.01; (0.014)	
United States, Rice, MN 2011, (DKC 35-43)	EC	**4 (7,7,7)**	0.076 0.076 0.077 0.077	96 (28 Sept)	7	6.6, 7.0, 11; (8.3)	0.010, 0.010, 0.043; (0.021)	**TK0058623; 03 (Storage: 160 days)**
	WG	**4 (7,7,7)**	0.077 0.076 0.077 0.076	96 (28 Sept)	7	6.3, 7.3, 7.0; (6.9)	<0.01, 0.010, 0.011; (0.010)	

Oilseed forage or fodder

Residue information on cotton seed gin trash was reproduced from the JMPR 2016 evaluation and extended with additional metabolite information. SYN545720 was not analysed in gin trash. Trials on rapeseed or peanut forage were not conducted. Peanut hay is described under legume hay.

No trials were selected for cotton gin trash

Table 49 Residues in cotton gin trash from field trials in the United States (JMPR 2016)

COTTON GIN TRASH Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
United States, Wall, TX 2010, (Fibermax 1740 B2F)	EC	3 (14)	0.076 0.076 0.075	84	**49**	0.23, **0.099 (0.16)**	0.46, 0.15 (0.30)	TK0025157 W40-0527 (Storage: 21.5 months)
United States, Wolfforth, TX 2010,	EC	3 (14)	0.078 0.077 0.075	**81**	**43**	0.61, 0.36 **(0.48)**	0.59, 0.34 (0.46)	TK0025157 W39-0528 (Storage: Gin trash

COTTON GIN TRASH Location Year (variety)	Form.	no	kg ai/ha	BBCH	DALA	Parent (mg/kg)	SYN 546039 + conj (mg/kg)	Report; Trial; (remarks)*
(FM9058)								21.2 months)
United States, Uvalde, TX 2011, (DPL 0935)	EC	3 (14)	0.075 0.076 0.075	77	**43**	0.18, 0.46, 0.19 (0.28)	0.26, 0.82 0.73 (0.60)	TK0058642 TK0058642-03 (Storage: Gin trash 6.8-9.8 months)
	WG	3 (14)	0.076 0.077 0.076	77	**43**	0.46, 0.63, 0.59 (0.56)	0.11, 0.14 0.13 (0.13)	
United States, Levelland, TX 2010, (FM9180B2F)	EC	3 (14)	0.078 0.076 0.076	89	**44**	0.50, 0.76 (0.63)	0.59, 0.74 (0.66)	TK0025157 W39-0529 (Storage: Gin trash 13.1 months)
United States, Levelland, TX 2011, (FM9180 B2F)	EC	3 (14)	0.075 0.077 0.077	81	**48**	1.5, 1.4, 1.6 (1.5)	1.1, 0.79 1.0 (0.96)	TK0058642 TK0058642-04 (Storage: Gin trash 4.1-5.4 months)
	WG	3 (14)	0.076 0.077 0.076	81	**48**	1.1, 1.1, 1.2 (1.1)	0.25, 0.17 0.21 (0.21)	

Fate of residues in storage and processing

In processing

Processing factors (PF) are defined as the residue in the processed commodity divided by the residue in the corresponding raw agricultural commodity (RAC). The residue used to calculate the processing factor is benzovindiflupyr as defined in the residue definition for enforcement and dietary risk assessment. Residues < LOQ were assumed to be at the LOQ for calculation purposes. Processing factors in the tables were rounded to two figures, but were calculated using unrounded figures reported in the study.

Sugar beet processing study

The Meeting received two processing trials on sugar beets [Dorsey, 2019, VV-547512]. Plots located in the United States were treated twice with an exaggerated rate of 0.38–0.40 kg ai/ha using a WG formulation. The first application was performed as an in-furrow soil application at sowing/planting (BBCH 00). The second application was performed as a banded foliar application at BBCH 30–32. Details of the trials used for processing are presented in Table 7 (trials TK0296310 -01 and -02).

Sugar beet plants for processing were collected at normal commercial harvest (BBCH 47–50). Sugar beet plants were separated into tops and roots. Samples were collected and stored frozen prior to shipment to the processing facility. Sugar beet roots were processed into dried pulp, refined sugar and molasses within 1 day after harvest following methods representative of commercial practice.

Processing into dried pulp, molasses and refined sugar consisted of the following steps:

- Cleaning: Sugar beet roots (222.6; 207.3 lbs = 101; 94.0 kg) were cleaned prior to processing. Heavy deposits of soil were removed with a brush and water, and loose leaves and foreign matter were separated from beets to obtain cleaned beets (216.9; 200.7 lbs = 98.4; 91.0 kg).
- Slicing: A portion of the cleaned beets (92.5; 93.2 lbs = 42.0; 42.3 kg) were mechanically sliced into cossettes using a Hobart food chopper.
- Diffusion: Cossettes were first exposed to heated water at 88–92 °C (190–198 °F) for 30–45 seconds and then diffused in 5 kettles containing warm water at 68–74 °C (155–165 °F) for 9 minutes in each kettle. The diffusion water from the 5 kettles was screened (US 100 mesh sieve) to remove any remaining small pieces of beet (cossettes). The diffusion water and the diffused cossettes were collected separately.
- Pressing: Diffused cossettes were dewatered (pressed) in a filter bag and hydraulic press to obtain pressed wet pulp (39.1; 45.0 lbs = 17.7; 20.4 kg). The press water was collected and added to the diffusion water to obtain raw juice.
- Wet pulp drying: The pressed wet pulp was dried in an oven at 54–71 °C (130–160 °F) to achieve a final moisture content of 15 percent or less. The resulting dried pulp (4.3; 4.6 lbs = 1.95; 2.09 kg) was collected.
- Phosphatizing step 1: Raw juice was mixed and the temperature increased to 80–85 °C (176–185 °F). A 20 percent calcium oxide solution was added until the pH was 10.5. If the pH was above 11.2, the pH was lowered to 11.2 with phosphoric acid (3M). A precipitate or mud was formed. Centrifugation separated the mud and phosphatized juice.
- Phosphatizing step 2: The phosphatized juice was mixed and heated to 80–85 °C (176–185 °F). The pH was reduced to 9.1–9.3 with phosphoric acid (3M). After the pH was achieved, the juice was centrifuged and vacuum filtered to separate mud and clear juice. The clear juice was mixed and heated to 80–85 °C (176–185 °F) and the pH was reduced to 8.8–9.0 with sodium bisulphite to obtain thin juice (115.2 lbs).
- Evaporation: The thin juice was evaporated under vacuum until a 50–60 percent solids thick juice was achieved. The temperature was maintained below 85°C (185F). After evaporation, the thick juice was filtered over cotton. The filtered thick juice (6.392 kg) was evaporated under vacuum until a 70–80 percent solids syrup was achieved.
- Crystallisation and centrifugation: A solution of white sugar was added to the syrup and the syrup was allowed to cool. After this 'seeding' step sugar crystals were formed. Crystallised sugar and molasses were separated by centrifugation. Steam was added during centrifugation to remove all residual molasses from the crystallised sugar. Molasses (3.625; 3.764 kg) was collected. Crystallised sugar was dried in an oven at 54–77 °C (130–170 °F) to obtain refined sugar with a final moisture content of 1.0 percent (0.763; 0.625 kg)

Moisture content was determined as 39.92 percent and 43.94 percent in molasses and 3.28 percent and 3.05 percent in dried pulp. The dry matter content (100 percent–moisture content) is therefore 60.08 percent and 56.06 percent in molasses and 96.72 percent and 96.95 percent in dried pulp.

Storage: Samples were frozen and stored for 6.7–7.6 months at -10 °C or lower.

Analysis: Benzovindiflupyr and metabolite SYN 546039 (induding its conjugates) were determined in sugar beet and its processed commodities using a modification of HPLC-MS/MS method GRM042.03A with an LOQ of 0.01 mg/kg for each analyte. Mean concurrent recoveries in sugar beet and its processed commodities ranged from 76–111 percent at 0.01–2.0 mg/kg for benzovindiflupyr and 77–

100 percent at 0.01–1.0 mg/kg for SYN 546039. Residues in the control samples were < LOQ (n=2) for each analyte and matrix.

Processing results for benzovindiflupyr in sugar beet commodities are shown in Table 50. Residues for SYN 546039 (including its oonjugates) were < 0.01 mg/kg in each sample. The results of the two processing trials on sugar beet indicate a concentration of residues of benzovindiflupyr in dried pulp (median PF = 7.1) and a reduction of residues of benzovindiflupyr in refined sugar and molasses (best estimate PF < 0.46).

Remarks by the reviewer:

- No storage stability information is available for processed sugar beet commodities (molasses, dried pulp, sugar). The JMPR 2014 concluded that benzovindiflupyr and SYN546039 (free) are stable for at least 24 months at -18 ºC in crop commodities representative of the high water, high acid, high starch, high protein and high oil commodity groups as well as in wheat straw and for at least 24 months at -10 ºC in various processed commodities such as flour (maize, soya), meal (maize), oil (maize, soya), soya milk, dried fruits (grape, apple) and fruit juice (apple). As storage stability was shown in such a wide range of commodities, storage stability results can be extrapolated to processed sugar beet commodities (molasses, dried pulp, sugar).
- The modification of HPLC-MS/MS method GRM042.03A is reduced validated for the determination of benzovindiflupyr and SYN 546039 (including conjugates) in sugar beet commodities (roots, dried pulp, molasses, refined sugar) in the range 0.01–1.0 mg/kg. The benzovindiflupyr and SYN 546039 (including conjugate) concentrations in study VV-547512 are covered by the validation of this analytical method.
- The pressed wet pulp yields of 19–22 percent and dried pulp yields of 4.6–4.9 percent represent commercial practice. However, the yields for sugar (1.5–1.8 percent) and molasses (8.6–8.9 percent) do not represent commercial practice. Normally, a yield of 14–16 percent refined sugar and up to 3.5 percent molasses (dm 72 percent) is obtained from sugar beets containing 17 percent sucrose. The manufacturer indicates that the study was not set up as a mass balance study and that losses occurring during processing, some of the inputs into the processing procedure, and fractions that were discarded along the process were not weighed/measured [Answers to questions, July 2021]. The processing was conducted according to commercial practices adapted for small-scale equipment in a “batch process". It is not a continuous looping process where, for example, molasses or pulp could be run through the process multiple times to increase overall sugar yield while reducing yield of other components. In addition, the GLP processing lab is working with the beets derived from the field trials, which can vary in water content and sugar content from trial to trial, field to field, and year to year. Commercial production is focused on maximizing sugar output, whereas GLP batch processing is focused on generating representative samples of several commodities (pulp, refined sugar, and molasses) for subsequent residue analysis [Answers to questions, July 2021]. Based on this argumentation, the current reviewer considers the samples representative for the purpose of deriving processing factors.

Table 50 Residues of benzovindiflupyr in sugar beet processed commodities

SUGAR BEET ROOTS Location, Country, Year (Variety)	Application information	DAT	Commodity	Parent (mg/kg)	PF	Yield (percent)	[Reference], trial
Gardner, ND, United States, 2017 (SVRR 336)	In furrow soil + banded foliar BBCH 30-31, Rate: 2x (0.38-0.39) kg ai/ha RTI: 40 days	81	RAC dried pulp refined sugar molasses	0.022 [a] 0.12 <0.01 <0.01	- 5.5 <0.46 <0.46	- 4.6 1.8 8.6	[Dorsey, 2019, VV-547512], TK0296310–01
Northwood, ND, United States, 2017 (Hilleshog 4022RR)	In furrow soil + banded foliar BBCH 31-32 Rate: 2x (0.39-0.40) kg ai/ha RTI: 32 days	93	RAC dried pulp refined sugar molasses	0.015 [b] 0.13 <0.01 <0.01	- 8.7 <0.67 <0.67	- 4.9 1.5 8.9	[Dorsey, 2019, VV-547512], TK0296310–02

Notes:

[a] Mean result of 0.014, 0.018, 0.033 mg/kg, just prior to processing.

[b] Mean result of <0.01, 0.015, 0.020 mg/kg, just prior to processing.

Maize processing study

Maize processing trials have been summarized and evaluated by the JMPR 2016. An overview of the processing factors is given in Table 51.

Overall conclusion on processing factors

An overview of processing factors for benzovindiflupyr in sugar beets and maize are listed in Table 51.

Table 51 Overview of processing factors for sugar beets and maize

	PF – parent only	PF median or best estimate
Sugar beet dried pulp	5.5, 8.7	median 7.1 (n=2)
Sugar beet refined sugar	<0.46, <0.67	best estimate <0.46 (n=2)
Sugar beet molasses	<0.46, <0.67	best estimate <0.46 (n=2)
Maize meal (JMPR 2016)	<0.25	best estimate <0.25 (n=1)
Maize flour (JMPR 2016)	0.25	best estimate 0.25 (n=1)
Maize grits (JMPR 2016)	<0.25	best estimate <0.25 (n=1)
Maize refined oil (dry processing) (JMPR 2016)	<0.25	best estimate <0.25 (n=1)
Maize refined oil (wet processing) (JMPR 2016)	0.50	best estimate 0.50 (n=1)
Maize starch (JMPR 2016)	<0.25	best estimate <0.25 (n=1)
Maize gluten (JMPR 2016)	0.75	best estimate 0.75 (n=1)
Maize bran (JMPR 2016)	0.50	best estimate 0.50 (n=1)
Maize milled by-product (JMPR 2016)	<0.25	best estimate <0.25 (n=1)

APPRAISAL

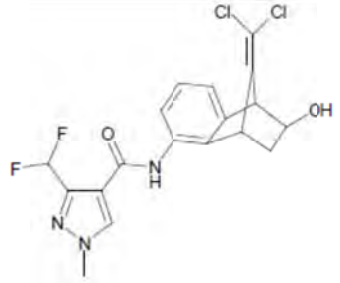
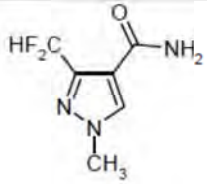
Benzovindiflupyr is a broad-spectrum fungicide first evaluated by JMPR in 2013 for Toxicology and in 2014 for Residues. The compound was re-evaluated in 2016, 2018 and 2019 for additional uses.

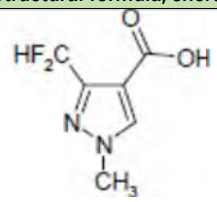
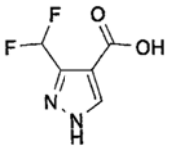
An ADI of 0–0.05 mg/kg bw and an ARfD of 0.1 mg/kg bw were established by the 2013 JMPR. The 2014 JMPR Meeting recommended the residue definition for plant and animal commodities (for compliance with MRLs and for estimation of dietary intake) as: benzovindiflupyr. The residue is fat soluble.

Benzovindiflupyr was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2021 JMPR and rescheduled for evaluation by the 2022 JMPR. The Meeting received additional information from the manufacturer on the toxicity of metabolites, method of residue analysis, use patterns, supervised residue trials (blueberries, dried ginseng, sugar beets and maize) and processing studies on sugar beets.

Chemical Names

Table 52 Metabolites referred to in this appraisal

Code	Structural formula, short name, IUPAC name	JMPR 2014 conclusions
SYN 546039	 hydroxy-benzovindiflupyr CSCD695908 IUPAC: N-[(1RS,2RS,4SR)-9-(dichloromethylene)-2-hydroxy-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide	JMPR 2014: Found in rat goat (milk, liver, kidney, muscle, fat) hen (eggs, liver, muscle, skin with fat) tomato fruit wheat forage/hay/straw wheat grains soya bean forage/hay soya bean seeds rotational crops: lettuce leaves, turnip roots/leaves, wheat forage/hay/straw
SYN 508272	 pyrazole amide CSCC210616 IUPAC: 3- difluoromethyl-1-methyl-1H-pyrazole-4-carboxylic acid amide	JMPR 2014: Found in goat (milk, liver, kidney, muscle, fat) hen (eggs, liver, muscle, skin with fat) wheat hay/straw wheat grains soya bean forage soya bean hay rotational crops: turnip leaves, wheat forage/hay/straw photolysis in water common metabolite with other pyrazole fungicides like bixafen, fluindapyr, fluxapyroxad, inpyrfluxam, isopyrazam and sedaxane

Code	Structural formula, short name, IUPAC name	JMPR 2014 conclusions
NOA 449410	 pyrazole acid CSAA798670 IUPAC: 3-difluoromethyl-1-methyl-1H-pyrazole-4-carboxylic acid	JMPR 2014: Found in: tomato fruit wheat hay/straw wheat grains soya bean forage/hay soya bean seeds rotational crops: lettuce leaves, turnip roots/leaves, wheat forage/hay/straw photolysis in water (not detected in goat and hen commodities) common metabolite with other pyrazole fungicides like bixafen, fluxapyroxad, inpyrfluxam, isopyrazam and sedaxane
SYN 545720	 N-desmethylpyrazole-acid CSCD465008 or R958945, IUPAC: 3-difluoromethyl-1H-pyrazole-4-carboxylic acid CASnr 151734-02-0	JMPR 2014: Found in tomato fruit soya bean forage/hay soya bean seeds rotational crops: lettuce leaves, turnip roots/leaves, wheat forage/hay/straw (not detected in rat, goat and hen commodities) (not found in photolysis in water) common metabolite with other pyrazole fungicides like bixafen, fluxapyroxad, inpyrfluxam, isopyrazam and sedaxane

Methods of analysis

The Meeting received additional validation information for benzovindiflupyr and SYN 546039 in blueberries, dried ginseng roots, sugar beet commodities and maize commodities for analytical methods already considered by the JMPR in 2014. The Meeting concluded that the presented (modified) methods were sufficiently validated and are suitable to quantify benzovindiflupyr and SYN 546039 (including conjugates) in plant commodities.

Stability of pesticide residues in stored analytical samples

The Meeting agreed that the demonstrated storage stability on various representative plant commodities evaluated by the JMPR 2014 covered the residue sample storage intervals used in the field trials and processing studies considered by the current Meeting.

Residue definition

The Meeting received additional toxicological information on hydroxy-benzovindiflupyr (SYN 546039) and the three cleavage products: pyrazole amide (SYN 508272), pyrazole acid (NOA 449410) and N-desmethyl pyrazole acid (SYN 545720). The three cleavage products are common to other pyrazole fungicides like bixafen, fluindapyr, fluxapyroxad, inpyrfluxam, isopyrazam and sedaxane. The 2014 JMPR concluded that

these metabolites were not relevant for the residue definition, because the residue levels were low and/or because the toxicity was much lower than parent.

The current Meeting concluded that the three cleavage products (pyrazole amide, pyrazole acid and N-desmethyl pyrazole acid) are not covered by the health based guidance values of benzovindiflupyr and should be assessed by TTC (Cramer class III).

Hydroxy-benzovindiflupyr (SYN 546039 including conjugates) was found at significant levels in the metabolism studies on legume forage/fodder (9.2–12 percent TRR, 0.34–1.6 mg/kg eq), moderate levels in cereal hay/straw (0.5–3.6 percent TRR, 0.032–0.23 mg/kg eq) and low levels in fruit crops, cereal grains/forage and seeds of pulses/oilseed (0.1–2.1 percent TRR, < 0.01 mg/kg eq).

Actual levels of hydroxy-benzovindiflupyr (including conjugates) in field trials conducted at cGAP were 0.92–3.5 mg/kg in legume hay, 0.15–0.71 mg/kg in legume forage, 0.040–1.2 mg/kg in cereal hay/straw, < 0.01–0.32 mg/kg in cereal forage and 0.018–0.19 mg/kg in grapes. Hydroxy-benzovindiflupyr (including conjugates) levels in other crops were low: < 0.01 mg/kg in pome fruit, bulb onions, dry beans, soya beans, potatoes, sweet corn, sugar cane, rape seed, peanuts, and < 0.01–0.035 mg/kg in fruiting vegetables, dry peas, cereal grains and coffee beans.

Hydroxy-benzovindiflupyr (including conjugates) was found in the livestock metabolism studies evaluated by the 2014 JMPR: mammalian muscle, fat and milk (22 percent–39 percent TRR, < 0.01–0.035 mg/kg eq); mammalian edible offal (22–50 percent TRR, 0.040–0.65 mg/kg eq); and eggs (12–22 percent TRR; 0.014–0.015 mg/kg eq). It was found at low levels in poultry tissues (1.3–5.2 percent TRR, < 0.01 mg/kg eq).

Actual levels of hydroxy-benzovindiflupyr (including conjugates) in feeding studies on lactating cows at the maximum dietary burden were < 0.01 mg/kg in milk and muscle, 0.019 mg/kg in fat and 0.037 mg/kg in mammalian offal. No feeding studies on poultry were available. Based on the metabolism studies, actual levels of hydroxy-benzovindiflupyr (including conjugates) in poultry tissues and eggs are expected to be < 0.01 mg/kg at the maximum dietary burden. The current Meeting concluded that this metabolite is 10 times less toxic than parent.

Because the contribution of hydroxybenzovindiflupyr to the overall dietary risk is low (4.5 percent relative exposure increase), the Meeting confirmed that hydroxy-benzovindiflupyr should not be included in the residue definition for dietary risk assessment for either plant or animal commodities .

The Meeting therefore confirmed its definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities as: benzovindiflupyr.

Results of supervised residue trials on crops

Supervised trials were available for the use of benzovindiflupyr on blueberries, ginseng, sugar beets and maize. Product labels were available from the United States.

Blueberries

Benzovindiflupyr is registered in the United States for a foliar spray on lowbush blueberries with a critical GAP (cGAP) application rate of 2× 0.076 kg ai/ha, a retreatment interval (RTI) of 10 days and a PHI of 1 day.

Because the United States label registered lowbush blueberries only, the Meeting decided that the supervised trials on highbush blueberries could not be matched to this cGAP. Five supervised trials on lowbush blueberries from the United States performed at an application rate of 2× 0.074–0.076 kg ai/ha

with an RTI of 10 days and a PHI of 1 day, approximated this cGAP. Residues of benzovindiflupyr in lowbush blueberries were: 0.48, 0.51, 0.65, 0.69, 0.87 mg/kg (n=5).

The Meeting considered five trials sufficient for the minor crop blueberries and estimated a maximum residue level of 2 mg/kg, a HR of 0.98 mg/kg (individual highest residue) and an STMR of 0.65 mg/kg for benzovindiflupyr in blueberries (FB0020). The recommendation refers to blueberries, as the Codex Classification does not distinguish between lowbush and highbush blueberries (both have code FB0020).

Dried ginseng roots

Benzovindiflupyr is registered in the United States on ginseng with a cGAP rate of 4× 0.076 kg ai/ha, an RTI of 14 days and a PHI of 15 days.

Four supervised trials on ginseng from the United States performed at 4×0.073–0.080 kg ai/ha with an RTI of 13–15 days and a PHI of 15 days approximated this cGAP. Fresh ginseng roots from these trials were dried for 6–8 days until an estimated dry matter content of 70–90 percent was reached. Residues of benzovindiflupyr in dried ginseng roots were: 0.034, 0.068, 0.094, 0.14 mg/kg (n=4), as received.

According to the Codex Classification, ginseng should comply with Codex Standard 295R-2009. This regional standard has been replaced by Codex Standard 321-2015. Codex Standard 321-2015 stipulates that dried ginseng roots should contain no more moisture than 14.0 percent (i.e. have a dry matter content of at least 86.0 percent). The actual dry matter content of the individual dried ginseng root samples from the supervised field trials were not reported, but were estimated at 70–90 percent in the study report.

The Meeting concluded that residues in ginseng roots with an estimated dry matter content of 70–90 percent were not affected by more than 25 percent (100 percent × (86–70) / 86 = 18.6 percent). The Meeting considered four trials sufficient for the minor crop ginseng roots and estimated a maximum residue level of 0.3 mg/kg, a HR of 0.16 mg/kg (highest individual) and an STMR of 0.081 mg/kg for benzovindiflupyr in dried ginseng roots (DV 0604 and DT 0604).

Sugar beet roots

Benzovindiflupyr is registered in the United States on sugar beets with two different GAPs:

A) a single in-furrow or banded soil application at 0.075 kg ai/ha at the 2–8 leaf-stage, followed by a foliar application at 0.075 kg ai/ha up to BBCH 31.

B) two foliar applications at 2× 0.075 kg ai/ha with an RTI of 5 days and the last application applied up to BBCH 31.

The Meeting considered the two foliar applications (GAP B) as cGAP. Because all the supervised trials provided to the Meeting were conducted with the combined soil and foliar treatment (GAP A) with a longer RTI of 25–46 days between the soil and subsequent foliar application, none of the trials matched the cGAP The Meeting decided that the trials were not suitable to derive maximum residue levels.

Maize grains

Benzovindiflupyr is registered in Canada on maize (field corn, popcorn, sweet corn, specialties) with a cGAP of 2× 0.075 kg ai/ha, an RTI of 7 days and a PHI of 7 days. None of the trials provided to the present or previous JMPR Meetings matched with this GAP.

Benzovindiflupyr is registered in the United States on maize (field corn) and popcorn with a cGAP rate of 2× 0.051 kg ai/ha, a retreatment interval (RTI) of 14 days and a PHI of 7 days.

Seven supervised trials on maize from the United States performed at 2× 0.050–0.053 kg ai/ha with an RTI of 13–15 days and a PHI of 6–7 days approximated this cGAP. Residues of benzovindiflupyr in maize grains were: < 0.01 (6), 0.016 mg/kg (n=7).

The Meeting estimated a maximum residue level of 0.02 mg/kg and an STMR of 0.01 mg/kg for benzovindiflupyr in maize (GC 0645). The Meeting decided to extrapolate this MRL to popcorn (GC 0656).

Residues in animal feeds

Maize forage

Benzovindiflupyr is registered in Canada on maize (field corn, popcorn, sweet corn, specialties) with a cGAP of 2× 0.075 kg ai/ha, an RTI of 7 days and a PHI of 7 days. None of the trials provided to the present or previous JMPRs matched with this GAP.

Benzovindiflupyr is registered in the United States on maize (field corn) and popcorn with a cGAP rate of 2× 0.051 kg ai/ha, a retreatment interval (RTI) of 14 days and a PHI of 7 days.

Trials provided as maize forage (whole plant) that could possibly be matched to the United States GAP, were harvested at BBCH 85–89. The Meeting considered these samples not representative for green forage plants, as the plants already lost moisture and thus may have concentrated their residues. The Meeting decided not to derive median and highest residues for maize forage.

Maize stover

Benzovindiflupyr is registered in Canada on maize (field corn, popcorn, sweet corn, specialties) with a cGAP of 2× 0.075 kg ai/ha, a retreatment interval (RTI) of 7 days and a PHI of 7 days. None of the trials provided to the present or previous JMPRs matched with this GAP.

Benzovindiflupyr is registered in the United States on maize (field corn) and popcorn with a cGAP rate of 2× 0.051 kg ai/ha, an RTI of 14 days and a PHI of 7 days.

Seven supervised trials on maize stover (remaining plant) from the United States performed at 2× 0.050–0.053 kg ai/ha with an RTI of 13–15 days and a PHI of 6–7 days approximated this cGAP. Residues of benzovindiflupyr in maize stover were: 0.86, 1.2, 1.3, 1.6, 1.7, 2.3, 2.9 mg/kg (n=7) on an as received basis (assuming a dry matter content of 83 percent derived from the OECD 2018 feed calculator), corresponding to 1.0, 1.5, 1.6, 1.9, 2.0, 2.8, 3.5 mg/kg (n=7) on dry weight basis

The Meeting estimated a maximum residue level of 7 mg/kg, as dry weight and a median residue of 1.6 mg/kg and a highest residue of 2.9 mg/kg as received for maize stover. The Meeting decided to extrapolate these residues to popcorn stover.

Sugar beet leaves and tops

Benzovindiflupyr is registered in the United States on sugar beets with two different GAPs:

A) a single in-furrow or banded soil application at 0.075 kg ai/ha at the 2–8 leaf-stage, followed by a foliar application at 0.075 kg ai/ha up to BBCH 31.

B) two foliar applications at 2× 0.075 kg ai/ha with an RTI of 5 days and the last application applied up to BBCH 31.

The Meeting considered the two foliar applications (GAP B) as cGAP. Because all the supervised trials provided to the Meeting were conducted with the combined soil and foliar treatment (GAP A) with a longer RTI of 25–46 days between the soil and subsequent foliar application, none of the trials matched the cGAP. The Meeting decided that the trials were not suitable to derive maximum residue levels.

Fate of residues during processing

The Meeting received new information on the fate of benzovindiflupyr residues during processing in sugar beet roots. Furthermore, median and highest residues could be derived for maize processed commodities, based on the processing data evaluated by the JMPR 2016.

Table 53 Estimation of processing factors for commodities considered at this and previous Meetings

Raw commodity [STMR/HR]	Processed commodity	Individual processing factors	Mean or best estimate processing factor	STMR-P = STMR-RAC × PF (mg/kg)	HR-P = HR-RAC × PF (mg/kg)
Maize grains [0.01 mg/kg]	Maize meal (JMPR 2016)	< 0.25	< 0.25 (n=1)	0.0025	
	Maize flour (JMPR 2016)	0.25	0.25 (n=1)	0.0025	
	Maize grits (JMPR 2016)	< 0.25	< 0.25 (n=1)	0.0025	
	Maize refined oil (wet processing) (JMPR 2016)	0.50	0.50 (n=1)	0.0050	
	Maize starch (JMPR 2016)	< 0.25	< 0.25 (n=1)	0.0025	
	Maize gluten (JMPR 2016)	0.75	0.75 (n=1)	0.0075	
	Maize bran (JMPR 2016)	0.50	0.50 (n=1)	0.0050	
	Maize milled by-product (JMPR 2016)	< 0.25	< 0.25 (n=1)	0.0025	

Residues in animal commodities

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the current and previous JMPRs and including maize grains and maize stover. The dietary burdens, estimated using the 2018 update of the OECD feed calculator, are presented in Annex 6 of the 2022 JMPR Report and summarised below.

Table 54 Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden: benzovindiflupyr, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	3.035	1.269	6.894	2.559	14.80	4.200	0.661	0.466
Dairy cattle	8.093	3.012	7.519	3.029	14.33	4.065	3.520	1.830
Poultry – broiler	0.196	0.196	0.270	0.270	0.065	0.065	0.047	0.047
Poultry – layer	0.194	0.194	2.154	0.894	0.065	0.065	0.039	0.039

Notes:

Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues.

Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk.

Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues and eggs.

Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

The 2019 JMPR estimated a mean/maximum dietary burden of 5.145/14.80 ppm for beef cattle, 5.145/14.33 ppm for dairy cattle and 0.857/2.075 ppm for layer poultry. The Meeting noted that the contribution of maize grain and maize stover increased the mean dietary burdens for poultry by less than 10 percent. The Meeting therefore confirmed its previous recommendations for maximum residue levels in animal products.

Residue values for exposure calculations

The 2022 JMPR re-evaluated the study reports provided to previous Meetings in order to obtain field trial information for N-desmethyl pyrazole acid (SYN 545720) and Hydroxy-benzovindiflupyr (SYN 546039) (see Annex below). Parent residue values were corrected where necessary (see Annex below). Correct parent residue values were used in the dietary burden calculations, IEDI, IESTI and GECDE estimations conducted by the 2022 JMPR.

Commodity	Compound	Selected residues (mg/kg)
Group of Pome fruit	Parent (corrected values in bold)	**0.020, 0.022**, **0.026**, 0.031, 0.038, 0.039, **0.040**, 0.041, 0.042, 0.044, 0.048, 0.057, **0.058**, **0.060**, 0.062, **0.066**, 0.067, 0.069, 0.074, **0.086**, 0.096, 0.10, 0.16 mg/kg (**n=23**) with HR of 0.17 mg/kg (individual highest residue) and an STMR of 0.057 mg/kg
	SYN 546039including conjugates	< 0.01 (23) mg/kg (n=23) with HR of 0.01 mg/kg and STMR of 0.01 mg/kg
Grapes	Parent (corrected values in bold)	**0.089**, 0.10, 0.11, 0.15, **0.16**, 0.16, 0.23, **0.36,** 0.39, **0.40**, 0.47, 0.55, **0.76** mg/kg (**n=13**) with HR of 0.81 mg/kg (highest individual) and an STMR of 0.23 mg/kg
	SYN 546039including conjugates	0.018, 0.020, 0.022 (2), 0.032, 0.039, 0.041, 0.056, 0.056, 0.10, 0.078, 0.081, 0.19 mg/kg (n=13) with HR of 0.22 mg/kg (highest individual) and an STMR of 0.041 mg/kg– (residues are often higher at higher PHI)
Subgroup of Bulb onions	Parent	JMPR 2019: < 0.01(5), 0.011, 0.012 and 0.015 mg/kg (n=8) with an HR of 0.015 mg/kg and an STMR of 0.01 mg/kg
	SYN 546039including conjugates	< 0.01 (8) mg/kg (n=8) with an HR of 0.01 mg/kg and an STMR of 0.01 mg/kg
Group of Cucurbits	Parent	JMPR 2016: < 0.01 (2), 0.010, 0.013, 0.017, 0.018, 0.022 (2), 0.023, 0.026, 0.033, 0.049, 0.050, 0.052, 0.053, 0.12 and 0.14 mg/kg (n=17) with HR of 0.16 mg/kg (highest individual residue) and an STMR of 0.023 mg/kg based on combined data from cucumbers, summer squash and melons
	SYN 546039including conjugates	< 0.01 (14), 0.014, 0.014, 0.018 (n=17) mg/kg with an HR of 0.018 mg/kg and an STMR of 0.01 mg/kg
Group of Fruiting vegetables other than cucurbits	Parent (corrected values in bold)	< 0.01, 0.040, 0.040, 0.044, 0.053, 0.054, **0.060**, **0.060**, 0.061, 0.085, 0.093, 0.10, 0.11, 0.14, 0.20, 0.35, 0.36, 0.38, 0.43, 0.62 mg/kg (n=20) with an HR of 0.72 mg/kg (highest individual) and an STMR of 0.089 mg/kg
	SYN 546039including conjugates	< 0.01 (19), 0.016 mg/kg with an HR of 0.016 mg/kg and an STMR of 0.01 mg/kg
Subgroup of dry beans (excl soya beans)	Parent (corrected values in bold)	< 0.01 (5), **0.010**, **0.010**, 0.011, 0016, 0.020, 0.044, **0.044**, 0.078 mg/kg (n=13) with an STMR of 0.010 mg/kg
	SYN 546039 including conjugates	< 0.01 (13) mg/kg with an STMR of 0.01 mg/kg
	SYN 545720	< 0.01 (13) mg/kg with an STMR of 0.01 mg/kg
Dry soya beans	Parent (corrected values in bold)	< 0.01 (15), **0.011**, 0.012, 0.018, 0.064 mg/kg (**n=19**) with an STMR of 0.01 mg/kg
	SYN 546039including conjugates	< 0.01 (19) mg/kg with an STMR of 0.01 mg/kg

Commodity	Compound	Selected residues (mg/kg)
	SYN 545720	< 0.01 (19) mg/kg with an STMR of 0.01 mg/kg
Subgroup of dry peas	Parent (corrected values in bold)	< 0.01 (**4**), **< 0.01**, **0.014**, **0.024**, 0.033, **0.054**, 0.11 mg/kg **(n=10)** with an STMR of 0.012 mg/kg
	SYN 546039including conjugates	< 0.01 (9), 0.025 mg/kg (n=10) with an STMR of 0.01 mg/kg
	SYN 545720	< 0.01 (9), 0.020 mg/kg (n=10) with an STMR of 0.01 mg/kg
Potatoes	Parent (corrected values in bold)	< 0.01 (8), 0.010, 0.013, 0.014, 0.015 mg/kg (n=12) with an HR of **0.018 mg/kg** (highest individual) and an STMR of 0.01 mg/kg
	SYN 546039including conjugates	< 0.01 (12) mg/kg (n=12) with an HR of 0.01 mg/kg and an STMR of 0.01 mg/kg
Wheat, rye and triticale	Parent (corrected values in bold)	< 0.01 (8), **0.011, 0.012** (2), **0.014**, 0.017, 0.020, 0.021, 0.025 (2), 0.026 (2), 0.027, **0.030**, 0.032, **0.034**, **0.040**, 0.041, 0.042, 0.046, 0.059, **0.066, 0.073 mg/kg** (n=30) with an STMR of 0.023 mg/kg
	SYN 546039including conjugates	< 0.01 (29), 0.011 mg/kg (n=30) with an STMR of 0.01 mg/kg
Barley and oats	Parent (corrected values in bold)	0.014, 0.029, 0.061, **0.078**, **0.15**, 0.21, 0.26, 0.32, 0.42, 0.54, 0.59 mg/kg (**n=11**) with an STMR of 0.21 mg/kg
	SYN 546039including conjugates	< 0.01 (9), 0.025, 0.035 mg/kg (n=11) with an STMR of 0.01 mg/kg
Sweetcorn on the cob (with husks removed)	Parent	JMPR 2016: < 0.01 (15) mg/kg with an HR of 0.01 mg/kg and an STMR of 0.01 mg/kg based on trials in the United States
	SYN 546039including conjugates	< 0.01 (15) mg/kg (n=15) with an HR of 0.01 mg/kg and an STMR of 0.01 mg/kg
Sugar cane canes (and tops)	Parent (corrected values in bold)	0.013, **0.030**, 0.062, 0.068, 0.070, **0.12**, 0.14, 0.21 mg/kg (n=8) with an HR of 0.25 mg/kg (highest individual) and an STMR of 0.069 mg/kg
	SYN 546039including conjugates	< 0.01 (8) mg/kg (n=8) with an HR of 0.01 mg/kg and an STMR of 0.01 mg/kg
Rapeseed, seed	Parent	JMPR 2016: < 0.01 (2), 0.011, 0.019, 0.023, 0.031, 0.045, 0.062 and 0.10 mg/kg (n=9) with an STMR of 0.023 mg/kg based on trials in Canada
	SYN 546039including conjugates	< 0.01 (9) mg/kg (n=9) with an STMR of 0.01 mg/kg
	SYN 545720	< 0.01 (9) mg/kg (n=9) with an STMR of 0.01 mg/kg
Peanuts, nutmeat	Parent	JMPR 2016 < 0.01 (4), 0.020 (2) mg/kg (n=6) with an STMR of 0.01 mg/kg based on trials in Brazil
	SYN 546039including conjugates	< 0.01 (6) mg/kg (n=6) with an STMR of 0.01 mg/kg
	SYN 545720	< 0.01 (6) mg/kg (n=6) with an STMR of 0.01 mg/kg
Coffee beans, green	Parent	JMPR 2016: < 0.01 (3), 0.020 (2), and 0.070 mg/kg (n=6) with an STMR of 0.015 mg/kg based on trials in Brazil
	SYN 546039including conjugates	< 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.020 mg/kg (n=6) with an STMR of 0.01 mg/kg
	SYN 545720	< 0.01 (6) mg/kg (n=6) with an STMR of 0.01 mg/kg
Legume forage (excl peanuts and soya bean)	Parent (corrected values in bold)	0.28, 0.29, **0.43**, **0.61**, **0.93** mg/kg (n=5) with a highest residue of **0.97** mg/kg (highest individual) and a median residue of 0.43 mg/kg (as received)
	SYN 546039including conjugates	0.15, 0.21, 0.34, 0.58, 0.71 mg/kg (n=5) with a highest residue of 0.77 mg/kg (highest individual) and a median residue of 0.34 mg/kg (as received) Note: different sample choice compared to parent
Legume hay (excl peanuts, soya bean)	Parent (corrected values in bold)	**1.1**, 1.8, 2.2, 3.1, **3.6** mg/kg (n=5) with a highest residue of **3.9** mg/kg (highest individual) and a median residue of 2.2 mg/kg (as received)
	SYN 546039including conjugates	0.92, 1.1, 1.1, 3.0, 3.5 mg/kg (n=5) with a highest residue of 4.4 mg/kg (highest individual) and a median residue of 1.1 mg/kg (as received). Note: different sample choice compared to parent
Peanut hay	Parent (corrected values in bold)	unscaled: 0.43, 1.8, 2.7, 2.8, **2.8**, **2.8**, 3.0, 3.7, **6.2**, 7.0, **7.2**, **7.6**, 9.0 mg/kg (n=13) with a highest residue of 10 mg/kg (highest individual) and a

Commodity	Compound	Selected residues (mg/kg)
		median residue of 3.0 mg/kg (as received) scaled (x0.75): highest individual residue of **7.5** mg/kg and median residue of **2.25** mg/kg (as received)
	SYN 546039including conjugates	unscaled: 0.031, 0.12, 0.14, 0.21, 0.26, 0.26, 0.30, 0.34, 0.31, 0.44, 0.66, 0.71, 1.3 mg/kg (n=13) with a highest residue of 1.8 mg/kg (highest individual) and a median residue of 0.30 mg/kg (as received). scaled (x0.75): highest individual residue of 1.35 mg/kg and median residue of 0.225 mg/kg (as received).
Wheat, barley, oat, rye, triticale forage	Parent (corrected values in bold)	< 0.01, 0.38, 0.40, 0.45, 0.48, 0.55, 0.56, 0.62, 0.66, 0.71, 0.73, 0.74, 0.82, 0.90, 0.94, **1.0**, **1.1**, 1.2, 1.2, 1.2, 1.3, 1.3, 1.4, 1.4, 1.4, 1.8, 1.8, 1.8, 1.9, 2.0, 2.2, 3.4 mg/kg (n=32) with a highest residue of 3.7 mg/kg (highest individual) and a median residue of 1.05 mg/kg (as received)
	SYN 546039including conjugates	< 0.01, 0.022, 0.024, 0.043, 0.045, 0.058, 0.064, 0.077, 0.082, 0.084, 0.086, 0.086, 0.087, 0.088, 0.093, 0.095, 0.10, 0.10, 0.10, 0.11, 0.11, 0.11, 0.15, 0.15, 0.16, 0.16, 0.16, 0.16, 0.16, 0.18, 0.31, 0.32 mg/kg (n=32) with a highest residue of 0.38 mg/kg (highest individual) and median residue 0.0975 mg/kg (as received)
Wheat, barley, oat, rye, triticale hay and straw	Parent (corrected values in bold)	0.54, 0.72, 0.78, **1.0, 1.4**, 1.5, 1.6, 1.6, **1.6, 1.6, 1.8**, **2.0**, **2.0**, 2.2, 2.2, 2.3, **2.4**, 2.5, 2.6, **2.6**, **2.8**, 2.9, 3.4, 3.8, **3.8**, 3.9, 4.0, 4.0, 4.1, 4.7, 5.2, 5.2, 5.4, **5.5**, 6.0, 6.1, 6.2, 6.6, 6.9, 7.1, 7.2, **7.8**, 8.5, 8.6, 12 mg/kg (n=45) with a highest residue of 12 mg/kg and a median residue of 3.4 mg/kg (as received)
	SYN 546039including conjugates	0.040, 0.061, 0.068, 0.070, 0.084, 0.085, 0.10, 0.12, 0.13, 0.14, 0.14, 0.15, 0.15, 0.15, 0.16, 0.16, 0.18, 0.18, 0.19, 0.20, 0.20, 0.21, 0.21, 0.22, 0.24, 0.25, 0.25, 0.26, 0.28, 0.28, 0.30, 0.32, 0.33, 0.40, 0.40, 0.40, 0.41, 0.41, 0.42, 0.42, 0.46, 0.53, 0.65, 0.68, 1.2 mg/kg (n=45) with a highest residue of 1.3 mg/kg (highest individual) and a median residue of 0.21 mg/kg (as received)
Mammals	Parent	STMR: muscle < 0.01, fat 0.010, edible offal 0.012, milk < 0.01 HR: muscle < 0.01, fat 0.019, edible offal 0.064,
	SYN 546039including conjugates	STMR: muscle < 0.01, fat 0.010, edible offal 0.012, milk < 0.01 HR: muscle < 0.01, fat 0.019, edible offal 0.037,
Poultry	Parent	STMR: muscle < 0.01, fat < 0.01, edible offal < 0.01, eggs < 0.01 HR: muscle < 0.01, fat < 0.01, edible offal < 0.01, eggs < 0.01
	SYN 546039including conjugates	STMR: muscle < 0.01, fat < 0.01, edible offal < 0.01, eggs < 0.01 HR: muscle < 0.01, fat < 0.01, edible offal < 0.01, eggs < 0.01

Parent corrected: after re-evaluation of the 2016 JMPR study reports, some parent values were corrected. Bold values in the 2016 JMPR parent data and current parent data indicate which values were corrected.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed in Annex 1 are suitable for establishing maximum residue limits and for IEDI, IESTI and GECDE assessments.

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *benzovindiflupyr*

The residue is fat-soluble.

Table 55 Residue levels suitable for establishing maximum residue limits and for IEDI, IESTI and GECDE assessments

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FB 0020	Blueberries	2		0.65	0.98
DV 0604	Ginseng, dried including red ginseng	0.3		0.081	0.16
DT 0604	Ginseng, dried	0.3		0.081	0.16
GC 0645	Maize	0.02		0.01	
GC 0656	Popcorn	0.02		0.01	
CF 1255	Maize flour			0.0025	
	Maize grits			0.0025	
OR 0645	Maize oil, edible			0.0050	
	Maize starch			0.0025	
CF 3517	Maize gluten			0.0075	
	Maize bran, unprocessed			0.0050	
AS 3358	Maize stover	7 (dw)		1.6 (ar)	2.9 (ar)
AS 0656	Popcorn stover	7 (dw)		1.6 (ar)	2.9 (ar)
CF 0645	Maize meal			0.0025	
	Maize milled by-products			0.0025	

Notes:

(ar) – as received; (dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for benzovindiflupyr is 0–0.05 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for benzovindiflupyr were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the 2022 and previous JMPRs. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 0.27–1.9 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of benzovindiflupry from uses considered by the 2022 and previous JMPRs is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for benzovindiflupyr is 0.1 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for benzovindiflupyr were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2020 JMPR Report.

The IESTIs varied from 0–10 percent of the ARfD for children and 0–9 percent of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of benzovindiflupyr from uses considered by the present Meeting is unlikely to present a public health concern.

Threshold of toxicological concern (TTC) consideration for metabolites

The three cleavage products (pyrazole amide, pyrazole acid and N-desmethyl pyrazole acid) are common to other pyrazole fungicides like bixafen, fluindapyr, fluxapyroxad, inpyrfluxam, isopyrazam and sedaxane. In the absence of overall information on the uses of all active substances and considering the lack of a specific health-based guidance value, the Meeting decided there was insufficient information to perform a combined risk assessment for residues resulting from use with all active substances leading to formation of these three cleavage products. The Meeting concluded that the three cleavage products (pyrazole amide, pyrazole acid and N-desmethyl pyrazole acid) could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day) and that the exposure should be based on the anticipated residues following use of each active substance, separately.

Pyrazole amide (SYN 508272) and pyrazole acid (NOA 449410) were detected at low levels in food crops (<1 percent TRR, < 0.01 mg/kg eq), and animal commodities (< 5 percent TRR, < 0.01 mg/kg eq) in the metabolism studies evaluated by the 2014 JMPR.

N-desmethyl pyrazole acid (SYN 545720, including conjugates) was present at moderate levels in the metabolism studies on seeds of pulses/oilseeds (47 percent TRR, 0.047 mg/kg eq, ratio 47/15 to parent) evaluated by the 2014 JMPR. It was detected at low levels in fruit crops (0.1 percent TRR, < 0.001 mg/kg eq, ratio 0.2/91 to parent). It was not detected in cereals grains or animal commodities.

Actual levels of N-desmethyl pyrazole acid (including conjugates) in field trials conducted at cGAP were < 0.01–0.020 mg/kg in dry pea seeds and < 0.01 mg/kg in dry beans, dry soya bean seeds, rape seeds, peanut nutmeat and green coffee beans.

Based on the benzovindiflupyr uses evaluated by the 2022 and previous JMPRs and the ratios to parent derived from metabolism studies, the Meeting estimated the following dietary exposures:

- Pyrazole amide (SYN 508272): 0.0003–0.0026 µg/kg bw per day (IEDI)
- Pyrazole acid (NOA 449410): 0.0008–0.0045 µg/kg bw per day (IEDI)
- N-desmethyl pyrazole acid (SYN 545720) 0.0075–0.0399 µg/kg bw per day (IEDI). This last estimate could be refined to 0.0052–0.0248 µg/kg bw per day using the field trial information for SYN545720 for pulses, oilseeds and coffee beans (see Annex below).

The Meeting concluded that the estimated dietary exposure to residues of pyrazole amide, pyrazole acid and N-desmethyl pyrazole acid from benzovindiflupyr uses considered by the 2022 and previous JMPRs is below the TTC for Cramer Class III compounds and is unlikely to present a public health concern. Should further benzovindiflupyr uses be considered in the future, these conclusions may need to be re-evaluated.

REFERENCES

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
VV-872218	Banman, C.	2020	Benzovindiflupyr + Propiconazole – Magnitude of the Residue in or on Field Corn – United States 2019 SynTech Research, Stilwell, KS, United States Task and Report No. TK0462705, 31 August 2020 Study No 069SRUS19R0015 Syngenta file No. VV-872218 GLP, Unpublished
VV-	Byeongseok,	2019	Benzovindiflupyr: Magnitude of the Residue on Blueberries, Lowbush and Highbush.

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
619742	A		Pest Management Centre, Agriculture and Agri-Food Canada, Ottawa, ON, Canada Report No. AAFC16-039R, 9 September 2019 Lab ID No AAFC16-039R-279 Syngenta File No. VV-619742, GLP, Unpublished
-	Clark, S	2016	EAG Laboratories Method Modifications to Syngenta "SYN549192 – Analytical Method GRM042.03A for the Determination of SYN545192 and its metabolite SYN 546039 in crops–dated August 4, 2011" ABC Laboratories, CD001 Observations and/or Remarks Study No 84770 – Trial AAFC16-039R-279, dated 8 December 2016 non-GLP, unpublished
-	Clark, S	2017	EAG Laboratories Method Modifications to Syngenta "SYN549192 – Analytical Method GRM042.03A for the Determination of SYN545192 and its metabolite SYN 546039 in crops–dated August 4, 2011" ABC Laboratories, CD001 Observations and/or Remarks Study No 84770 – Trial AAFC16-039R-279, dated 23 January 2017 non-GLP, unpublished
VV-547512	Dorsey, S.	2019	Benzovindiflupyr (A18126B) – Magnitude of the Residues in or on Sugarbeets – United States, 2017 Eurofins EAG Agroscience, LLC, Columbia, MO, United States Task and Report No. TK0296310, 18 June 2019 Study No 85471 Syngenta file No. VV-547512 GLP, Unpublished
VV-547840	Lennon, G.	2019	Benzovindiflupyr + Difenoconazole: Magnitude of the Residue on Ginseng. Rutgers, The State University of New Jersey, Princeton, NJ, United States Report IR-4 PR No. 11760, 21 March 2019 Lab ID number 1176-16-MIR12 Syngenta File No. VV-547840, GLP, unpublished
-	Rodgers, CA	2015a	ABC Laboratories Method Modifications to Syngenta Method GRM042.03A, October 23, 2015 non-GLP, unpublished
-	Rodgers, CA	2015b	ABC Laboratories Method Modifications to Syngenta Method GRM042.03A, November 30, 2015 non-GLP, unpublished
-	Rodgers, CA	2015c	ABC Laboratories Method Modifications to Syngenta Method GRM042.03A, December 17, 2015
VV-547573	Shepard, E.	2019	Benzovindiflupyr (A15457R)– Magnitude of the Residues in or on Sugarbeets – Canada, 2017 Eurofins EAG Agroscience, LLC, Columbia, MO, United States Task No TK0304334 Report No 85475, 18 June 2019 Syngenta file No. VV-547573 GLP, Unpublished
-	Syngenta	2021	Answers to Questions, July 2021

BENZPYRIMOXAN (325)

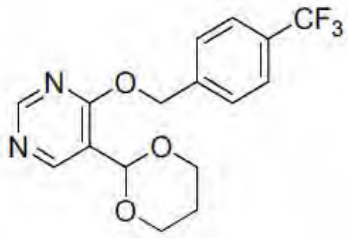
First draft prepared by Makoto Irie and Sachiko Yoda, Food and Agricultural Materials Inspection Center, Japan

EXPLANATION

Benzpyrimoxan is an insect growth regulator having biological activity to rice plant hopper larvae (Hemiptera: Delphacidae), without any adulticidal activity. It is registered for the control of sap sucking insects on rice.

At the Fifty-first Session of the CCPR (2019), benzypyrimoxan was scheduled for the evaluation as a new compound in 2020 and rescheduled to the 2022 JMPR. The Meeting received information on identity, physical and chemical properties, animal and plant metabolism, rotational crop study, environmental fate, analytical methods, GAP information, storage stability, processing, supervised residue trials and farm animal feeding study.

IDENTITY

Common name	Benzpyrimoxan
Chemical name	
IUPAC:	5-(1,3-dioxan-2-yl)-4-[4-(trifluoromethyl)benzyloxy]pyrimidine
CAS:	5-(1,3-dioxan-2-yl)-4-[[4-(trifluoromethyl)phenyl]methoxy]pyrimidine
CAS Registry No:	1449021-97-9
CIPAC No:	Not allocated
Synonyms:	NNI-1501
Structural formula:	
	
Molecular formula:	C16H15F3N2O3
Molecular weight:	340.30

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

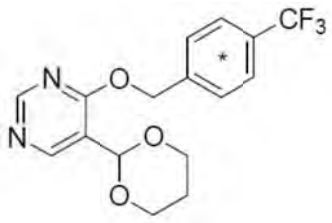
Table 1 Physical and chemical properties of the pure active ingredient

Property	Test material purity	Results	Reference
Appearance (colour, physical state, odour)	99.3%	Pale yellowish white / Powder / Odourless	Ota, 2016 PC-37007, PC-37013, PC-37014
Vapour pressure	99.3%	1.39×10^{-5} Pa at 25 °C	Ota, 2016 PC-37008

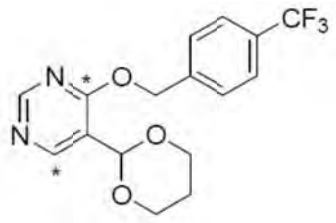
Property	Test material purity	Results	Reference
Henry's Law Constant		Calculation (25 °C) H = 9.39 × 10^{-4} Pa m^3/mol	-
Melting point	99.7%	120.1-121.3°C Capillary tube in a metal block method	Ota, 2016 PC-37016
Boiling point	99.7%	Not confirmed. The test item changed at 225-235 °C Siwoloboff method	Ota, 2016 PC-37017
Thermal stability	99.7%	The test item was stable to about 220 °C. Thermal analysis method	Ota, 2016 PC-37018
Octanol/water partition coefficient	99.3%	log Pow = 3.42 at pH 7.61 Shake Flask method at 24.5 °C	Yonemura, 2017 PC-37033
Solubility in water	99.3%	5.04 mg/L (distilled water) Flask method at 20 ± 0.5 °C pH = 6.79 (24 h), 7.02 (48 h), 6.96 (72 h)	Yaginuma, 2016 PC-37015
Solubility in organic solvents	99.3%	1.95 g/L in heptane 27.9 g/L in methanol 114 g/L in acetone 111 g/L in ethyl acetate 178 g/L in 1,2-dichloroethane 55.8 g/L in *p*-xylene Flask method at 20 ± 0.5 °C	Ota, 2016 PC-37009
Relative density	99.3%	1.440 g/cm^3 at 20 °C	Ota, 2016 PC-37010
Hydrolysis	Radiochemical purity: 99.4% ([phenyl-U-^{14}C]benzpyrimoxan) 98.7% ([pyrimidinyl-4(6)-^{14}C]benzpyrimoxan)	Benzpyrimoxan is hydrolytically stable under neutral and basic conditions (pH 7 and 9, 50 °C), while it is considered to be hydrolytically unstable under acidic conditions (pH 4). Hydrolysis of benzpyrimoxan at pH 4 is highly dependent upon temperature. The degradation half-life of the test substances at pH 4 at 25 °C was 50.4–51.4 days.	Nishimura, 2017 E-37007
Photolysis	Radiochemical purity: 98.2 ([phenyl-U-^{14}C]benzpyrimoxan) 99.3 ([pyrimidinyl-4(6)-^{14}C]benzpyrimoxan)	The DT_{50} of the test substances was 121.6-154.4 days, which were equivalent to 553.2-702.4 days (mean: 627.8 days) under natural sunlight in Tokyo spring.	Murata, 2017 E-37008
Dissociation constant	99.3	pKa = 2.14 Spectrophotometric method	Ota, 2016 PC-37011

METABOLISM AND ENVIRONMENTAL FATE

The metabolism of benzpyrimoxan has been investigated in plants and animals. The fate and behaviour of benzpyrimoxan in plants, animals and the environment were investigated using the [^{14}C] labelled test materials shown in Figure 1



[Phenyl-U-^{14}C]-benzpyrimoxan

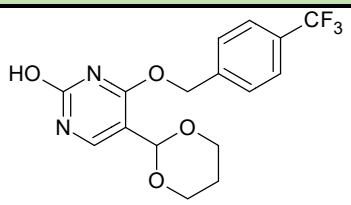
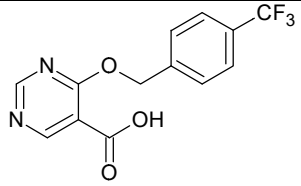
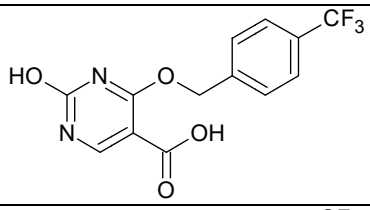
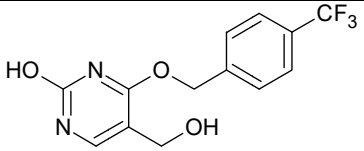
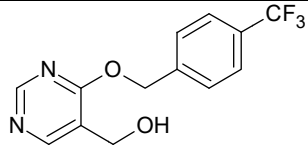


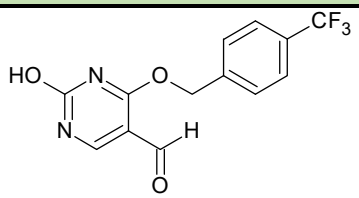
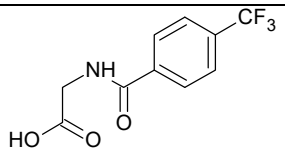
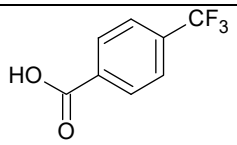
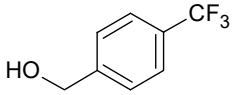
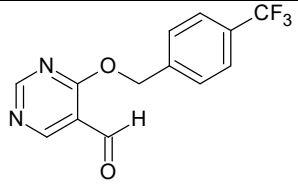
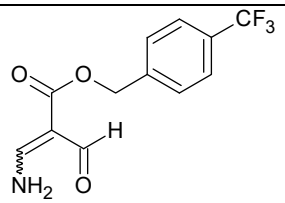
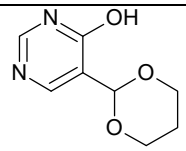
[Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan

Figure 1 [^{14}C]-Labelled test materials used in plant and animal metabolism studies, and the environmental fate studies

The chemical structures of the major metabolite/degradation compounds from the metabolism of benzpyrimoxan are shown in Table 2.

Table 2 Chemical structures of the major degradation compounds from the metabolism of benzpyrimoxan

Compound name		Structure	Found in metabolism studies
Benzpyrimoxan-2-OH DH-04	5-(1,3-dioxan-2-yl)-4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidin-2-ol MW: 356		Rice Livestock Soil
Benzpyrimoxan-acid DH-01	4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidine-5-carboxylic acid MW: 298		Rice Livestock Rat Photolysis
Benzpyrimoxan-acid-2-OH DH-05	2-hydroxy-4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidine-5-carboxylic acid MW: 314		Rice Livestock Rat Soil
Benzpyrimoxan-CH_2OH-2-OH DH-06	5-(hydroxymethyl)-4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidin-2-ol MW: 300		Rice Livestock Rat Soil
Benzpyrimoxan-CH_2OH DH-02	(4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidin-5-yl)methanol MW: 284		Livestock Rat

Compound name		Structure	Found in metabolism studies
Benzpyrimoxan-aldehyde-2-OH DH-07	2-hydroxy-4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidine-5-carbaldehyde MW: 298		Rice Soil
Benzpyrimoxan-benzoly-glycine DH-402	N-[4-(trifluoromethyl)benzoyl]glycine MW: 247		Livestock Rat
4-TFMB DH-101	4-(trifluoromethyl)benzoic acid MW: 190		Rice Rat Soil Photolysis
4-TFMPM DH-102	[4-(trifluoromethyl)phenyl] methanol MW: 176		Rice Rat Soil Hydrolysis Photolysis
Benzpyrimoxan-aldehyde DH-03	4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidine-5-carbaldehyde MW: 282		Soil Hydrolysis
Benzpyrimoxan-enamine-aldehyde DH-08	[4-(trifluoromethyl)phenyl] methyl (2E)-3-amino-2-formylprop-2-enoate MW: 273		Soil (sterilized) Hydrolysis
Benzpyrimoxan-4-OH DH-200	5-(1,3-dioxan-2-yl)pyrimidin-4-ol MW: 182		Hydrolysis Photolysis

Plant metabolism

Plant metabolism studies were performed on rice with [Phenyl-U-^{14}C] and [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan. Metabolites were identified using multiple chromatographic systems and authentic standards.

Rice

The metabolism of benzpyrimoxan was investigated in paddy rice following foliar spray application. Rice was seeded, transplanted and grown in a pot under paddy condition in a greenhouse. [Phenyl-U-^{14}C] or [Pyrimidinyl-4(6)-^{14}C]-labelled benzpyrimoxan was formulated as a suspension concentrate (SC). The SC formulation containing 2 percent of test substance was diluted 200-fold (100 mg ai/L) and 10 mL of the

diluent was sprayed on a whole test plant three times, which was equivalent to 3× 200 g ai/ha (general planting density for paddy rice: 200,000 plants/ha). For applications starting at the milk stage, the plant was sprayed three times at 7 day intervals, at heading stage (BBCH 55) and milk stage (BBCH 61–65 and 73–75). For applications starting at the ripe stage, the plant was sprayed twice at 7 day intervals, at heading stage (BBCH 55) and milk stage (BBCH 61–65), with the third application at BBCH 87–89, 4 weeks after the second application (Yoshizane, 2018: R-37018).

For both stage samples, the samples were taken 7 days after the last application (DALA). Samples of panicle, foliage and root were collected at milk stage. At ripe stage, panicle, straw and root were collected and then panicle was separated into grain and hull after 14 days air drying.

The surface of collected panicle, foliage, straw and hull samples were rinsed with acetonitrile. Panicle and foliage samples collected at milk stage were homogenized and sequentially extracted with acetonitrile/ distilled water (4/1, E1), acetonitrile/ 0.1 mol/L HCl (4/1, E2), acetonitrile/ 1 mol/L HCl (4/1, E3), acetonitrile/ 0.1 mol/L NaOH (4/1, E4) and acetonitrile/ 1 mol/L NaOH (4/1, E5). Grain (husked rice), hull and straw samples collected at ripe stage were crushed into small pieces and then homogenized and extracted as for milk stage samples. The unextracted residues and roots at both stages were collected and combusted after drying. Total radioactive residue (TRR) was determined to be the total of radioactivity in rinses, extracts and unextracted residues. The unextracted residues containing above 0.05 mg eq/kg or 10 percent TRR were treated sequentially with cellulase (for grain, treated with β-amylase), 6 mol/L HCl and 10 mol/L NaOH to characterize the radioactivity.

For quantification of radioactivity, aliquots of rinse and extracts were mixed with a liquid scintillant and subjected to LSC measurement. The unextracted residues and the root samples were combusted by a sample oxidiser. Aliquots of dried residual solid and root were mixed with cellulose powder prior to combustion to improve combustion efficiency. Radioactivity trapped in scintillation vials with scintillant after combustion was subjected to liquid scintillation counting (LSC) measurement.

For identification and quantification of radioactive residue, aliquots of concentrated rinse and extract were applied to two-dimensional TLC chromatography. To confirm identification of radioactive components, radioactivity in rinse and extract fractions was chromatographed on HPLC.

TRRs in panicle and foliage at milk stage were 1.18–1.40 and 1.83–2.44 mg eq/kg, respectively. TRRs in hull and straw at ripe stage were 2.81–4.68 and 3.29–3.69 mg eq/kg, respectively. Relatively small amounts of radioactive residues were detected in grain (0.10–0.25 mg eq/kg) and root (0.05–0.09 mg eq/kg). The major parts of TRR were recovered in surface rinse, neutral extract (acetonitrile/ distilled water (4/1, v/v; E1)) and the acid extract fractions (acetonitrile/ 0.1 mol/L HCl (4/1, v/v; E2)) in all the plant parts at both stages. Rinses and extracts (E1 to E5) were concentrated and subjected to TLC-radioluminography to determine metabolite constituents. The distribution of radioactivity in the extracts are shown in Table 3.

Table 3 Distribution of radioactivity in rice plant treated with ^{14}C-benzpyrimoxan

	Milk stage				Ripe stage					
	Panicle		Foliage		Grain		Hull		Straw	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
[Phenyl-U-^{14}C]-benzpyrimoxan										
Rinse	0.40	28.9	1.20	49.1	N/A	N/A	0.70	14.9	1.15	31.3
ACN/distilled H_2O (E1)	0.91	64.8	1.10	45.2	0.21	85.6	3.37	72.0	1.88	50.9
ACN/0.1 mol/L	0.02	1.4	0.05	2.2	0.01	4.3	0.12	2.5	0.34	9.3

	Milk stage				Ripe stage					
	Panicle		Foliage		Grain		Hull		Straw	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
HCl (E2)										
ACN/1 mol/L HCl (E3)	<0.01	0.5	0.01	0.6	<0.01	1.6	0.04	0.8	0.07	1.8
ACN/0.1 mol/L NaOH (E4)	<0.01	0.3	<0.01	0.3	<0.01	1.1	0.02	0.4	0.02	0.5
ACN/1 mol/L NaOH (E5)	<0.01	0.3	<0.01	0.1	ND	ND	0.01	0.3	0.11	2.9
Rinse and extract (E1-E5)	1.35	96.2	2.38	97.5	0.23	92.6	4.25	90.8	3.57	96.8
Unextracted	0.05	3.8	0.06	2.5	0.02	7.4	0.43	9.2	0.12	3.2
TRR	1.40	100	2.44	100	0.25	100	4.68	100	3.69	100
[Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan										
Rinse	0.29	24.5	0.81	44.2	N/A	N/A	0.43	15.3	0.87	26.6
ACN/distilled H_2O (E1)	0.79	66.7	0.89	48.5	0.07	71.8	1.91	68.0	1.74	53.1
ACN/0.1 mol/L HCl (E2)	0.02	1.7	0.05	3.0	<0.01	5.4	0.11	3.8	0.31	9.4
ACN/1 mol/L HCl (E3)	<0.01	0.3	<0.01	0.4	ND	ND	0.02	0.7	0.06	1.8
ACN/0.1 mol/L NaOH (E4)	<0.01	0.2	<0.01	0.5	ND	ND	<0.01	0.3	0.02	0.5
ACN/1 mol/L NaOH (E5)	ND	ND	<0.01	0.1	ND	ND	<0.01	0.2	0.07	2.3
Rinse and extract (E1-E5)	1.10	93.4	1.77	96.8	0.08	77.2	2.48	88.3	3.08	93.6
Unextracted	0.08	6.6	0.06	3.2	0.02	22.8	0.33	11.7	0.21	6.4
TRR	1.18	100	1.83	100	0.10	100	2.81	100	3.29	100

Notes:

N/A: not applicable; ND: not detected.

The radioactive residues detected in roots were as follows:

Milk stage: 0.08 mg eq/kg ([Phenyl-U-^{14}C]), 0.05 mg eq/kg ([Pyrimidinyl-4(6)-^{14}C]).

Ripe stage: 0.09 mg eq/kg ([Phenyl-U-^{14}C]), 0.09 mg eq/kg ([Pyrimidinyl-4(6)-^{14}C]).

At milk stage, the residues were identified as unchanged benzpyrimoxan and benzpyrimoxan-2-OH (DH-04). Benzpyrimoxan was determined to be 1.00–1.14 mg/kg (81.7–84.7 percent TRR) in panicle and 1.28–1.68 mg/kg (68.8–70.2 percent TRR) in foliage. DH-04 was determined to be 0.05–0.06 mg eq/kg (3.4–4.9 percent TRR) in panicle and 0.20–0.24 mg eq/kg (9.9–10.9 percent TRR) in foliage. Many minor (< 10 percent TRR) metabolites were also detected. After enzyme (-glucosidase and cellulose) hydrolysis of polar metabolites, small amounts (<10 percent TRR) of identified metabolites were detected.

At maturity, the residues were also identified as unchanged benzpyrimoxan and benzpyrimoxan-2-OH (DH-04). Benzpyrimoxan was determined to be 0.05–0.14 mg/kg (48.3–57.2 percent TRR) in grain, 1.68–2.83 mg/kg (59.9–60.6 percent TRR) in hulls and 1.58–1.86 mg/kg (48.2–50.5 percent TRR) in straw. DH-04 was determined to be 0.02–0.04 mg eq/kg (15.4–17.3 percent TRR) in grain, 0.14–

0.24 mg eq/kg (5.0–5.1 percent TRR) in hulls and 0.34–0.39 mg eq/kg (9.3–11.9 percent TRR) in straw. Many minor (<10 percent TRR) metabolites were also detected. After enzyme (-glucosidase and cellulose) hydrolysis of polar metabolites, small amounts (<10 percent TRR) of identified metabolites were detected.

Small amount of radioactivity was liberated from the unextracted residues by decomposing treatment with cellulase (for grain, treated with α-amylase), 6 mol/L HCl and 10 mol/L NaOH, sequentially. Tables 4 and 5 show the metabolites identified in the studies.

Table 4 Summary of radioactive residues in rice following application of [Phenyl-U-^{14}C]-benzpyrimoxan

Components	Milk stage				Ripe stage					
	Panicle		Foliage		Grain[1)]		Hull		Straw	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extract	1.35	96.2	2.38	97.5	0.23	92.6	4.25	90.8	3.57	96.8
Benzpyrimoxan	1.14	81.7	1.68	68.8	0.14	57.2	2.83	60.6	1.86	50.5
BP-acid (DH-01)	ND	ND	0.01	0.5	ND	ND	0.01	0.3	0.05	1.3
BP-2-OH (DH-04)	0.05	3.4	0.24	9.9	0.04	15.4	0.24	5.1	0.34	9.3
BP-acid-2-OH (DH-05)	0.03	2.5	0.11	4.5	<0.01	3.2	0.05	1.1	0.14	3.7
BP-CH_2OH-2-OH (DH-06)	ND	ND	0.05	2.0	ND	ND	ND	ND	0.10	2.6
BP-aldehyde-2-OH (DH-07)	ND	ND	0.03	1.1	ND	ND	ND	ND	<0.01	0.2
DH-101	ND	ND	<0.01	0.3	<0.01	1.4	0.02	0.4	0.08	2.1
DH-102	ND	ND	<0.01	0.4	ND	ND	ND	ND	<0.01	0.2
BP-CH_2OH (DH-02)-conjugate	0.02	1.3	0.02	1.0	<0.01	0.4	0.15	3.1	0.16	4.4
DH-05-conjugate	<0.01	0.2	<0.01	0.2	<0.01	0.3	ND	ND	0.04	1.1
DH-06-conjugate	ND	ND	<0.01	0.2	ND	ND	ND	ND	0.03	0.8
DH-101-conjugate	<0.01	0.3	<0.01	0.2	<0.01	0.7	0.04	0.8	0.04	1.2
DH-102-conjugate	<0.01	0.5	<0.01	0.3	<0.01	0.3	0.07	1.4	ND	ND
Others (sum)[2)]	0.09	6.5	0.20	8.0	0.03[3)]	13.7	0.84[4)]	18.1	0.72[5)]	19.4
Unextracted	0.05	3.8	0.06	2.5	0.02	7.4	0.43	9.2	0.12	3.2
Enxyme treatment - organic phase	<0.01	0.2	<0.01	0.3	0.01	4.1	0.01	0.3	0.05	1.3
Enzyme treatment-aqueous phase	ND	ND	<0.01	0.1	ND	ND	ND	ND	<0.01	0.2
6 mol/L HCl	0.01	1.0	0.01	0.5	ND	ND	0.09	2.0	0.01	0.4
10 mol/L NaOH	0.02	1.3	0.03	1.2	ND	ND	0.11	2.4	0.03	0.7
Unextracted after decomposing	0.02	1.3	0.01	0.4	<0.01	3.3	0.21	4.5	0.02	0.5
TRR	1.40	100	2.44	100	0.25	100	4.68	100	3.69	100

Notes:

ND: not detected.

[1)] Radioactivity was detected up to E4. E5 was not analysed.

[2)] E4 extract data of grain was summed up in here.

[3)] No individual component exceeded < 0.01 mg eq/kg, 3.0%TRR.

[4)] No individual component exceeded 0.21 mg eq/kg, 4.4%TRR.

[5)] No individual component exceeded 0.15 mg eq/kg, 3.9%TRR.

Table 5 Summary of radioactive residues in rice following application of [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan

Components	Milk stage				Ripe stage					
	Panicle		Foliage		Grain[1)]		Hull		Straw	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extract	1.10	93.4	1.77	96.8	0.08	77.2	2.48	88.3	3.08	93.6
Benzpyrimoxan	1.00	84.7	1.28	70.2	0.05	48.3	1.68	59.9	1.58	48.2
BP-acid (DH-01)	ND	ND	0.01	0.8	ND	ND	<0.01	<0.1	0.04	1.3
BP-2-OH (DH-04)	0.06	4.9	0.20	10.9	0.02	17.3	0.14	5.0	0.39	11.9
BP-acid-2-OH (DH-05)	0.02	1.9	0.08	4.2	ND	ND	0.05	1.7	0.12	3.6
BP-CH_2OH-2-OH (DH-06)	ND	ND	0.03	1.5	ND	ND	0.03	1.2	0.09	2.8
BP-aldehyde-2-OH (DH-07)	<0.01	0.5	0.02	0.9	ND	ND	ND	ND	<0.01	0.2
BP-CH_2OH (DH-02)-conjugate	<0.01	0.3	0.02	0.9	ND	ND	0.11	3.8	0.09	2.6
DH-05-conjugate	ND	ND	<0.01	0.2	<0.01	0.7	ND	ND	0.03	0.8
DH-06-conjugate	ND	ND	<0.01	<0.1	ND	ND	ND	ND	0.02	0.7
Others (sum)	0.01	1.2	0.13	7.2	0.01[2)]	10.9	0.47[3)]	16.7	0.70[4)]	21.4
Unextracted	0.08	6.6	0.06	3.2	0.02	22.8	0.33	11.7	0.21	6.4
Enzyme treatment-organic phase	ND	ND	<0.01	0.2	ND	ND	<0.01	0.3	0.03	0.8
Enzyme treatment-aqueous phase	<0.01	0.4	<0.01	0.2	0.01	10.9	<0.01	0.2	0.06	1.7
6 mol/L HCl	0.02	1.7	0.01	0.6	<0.01	8.4	0.07	2.6	0.03	0.9
10 mol/L NaOH	0.03	2.8	0.03	1.5	ND	ND	0.09	3.3	0.06	1.9
Unextracted after decomposing	0.02	1.6	0.01	0.6	<0.01	3.5	0.15	5.3	0.03	1.1
TRR	1.18	100	1.83	100	0.10	100	2.81	100	3.29	100

Notes:

ND: Not detected.

[1)] Radioactivity was detected up to E2. E3, E4 and E5 were not analysed.

[2)] No individual component exceeded < 0.01 mg eq/kg, 3.6%TRR.

[3)] No individual component exceeded 0.08 mg eq/kg, 3.0%TRR.

[4)] No individual component exceeded 0.11 mg eq/kg, 3.5%TRR.

The metabolites accounting for more than 10 percent TRR were benzpyrimoxan and benzpyrimoxan-2-OH (DH-04) in paddy rice.

Figure 2 shows the proposed pathway for benzpyrimoxan in paddy rice.

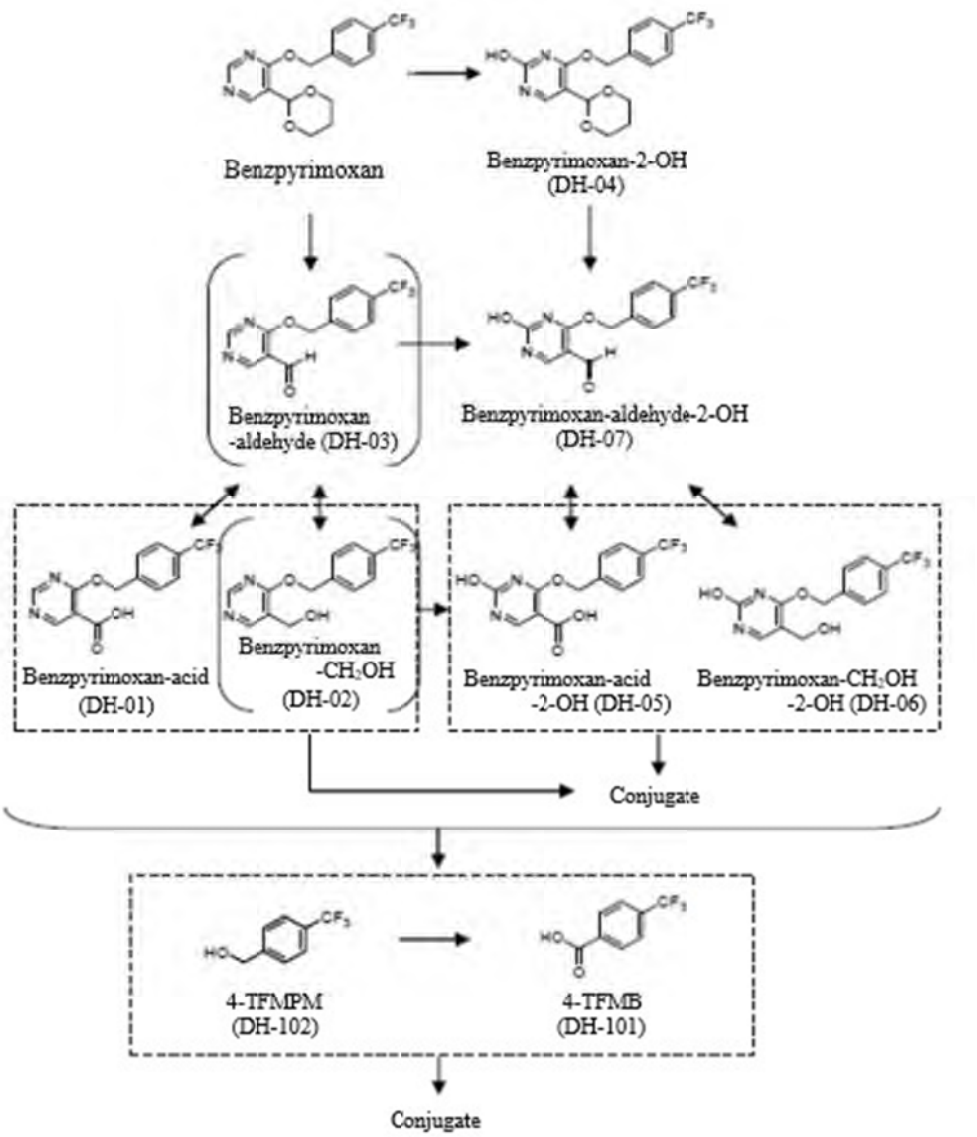


Figure 2 Metabolic Pathway of Benzpyrimoxan in Paddy rice.

Animal metabolism

The Meeting received studies on the metabolism of benzpyrimoxan in rats, lactating goats and laying hens. The metabolism of benzpyrimoxan in laboratory animals (rats) was summarized and evaluated by the WHO Core Assessment Group of the 2022 JMPR.

Lactating goat

The metabolism study in the lactating goat (*Capra hircus*) was conducted with ^{14}C-benzpyrimoxan (Ahn, 2018: R-37010). Lactating goats were dosed with [Phenyl-U-^{14}C]-benzpyrimoxan (1 goat) or [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan (1 goat) orally once daily for 5 consecutive days at a nominal 10 ppm in the diet. Doses were contained in cellulose-filled gelatin capsules. The average actual dose was 13.8 ppm for the phenyl label and 14.7 ppm for the pyrimidinyl label.

Milk, faeces and urine were collected twice a day during the treatment period. The goats were sacrificed approximately 6 hours after the last dose for phenyl label and approximately 8 hours after the last dose for pyrimidinyl label.

The TRR values in the urine, cage wash, bile, whole milk, and skim milk were counted directly by LSC. Portions of each feces sample, homogenized gastrointestinal tracts, and blood samples were analysed for ^{14}C residue by combustion. The radioactive residues in liver, kidney, muscle, fat, and milk fat were measured by solubilization of subsamples.

Samples of liver and kidney were extracted twice with acetonitrile/water (1/1) and then once with acetonitrile at room temperature. The individual extracts were collected separately and analysed by LSC. A portion of the combined acetonitrile/water extracts was analysed by both HPLC and two-dimensional (2-D) TLC. Combined acetonitrile/water extracts of liver and kidney were subjected to β-glucuronidase or sulfatase hydrolysis. Treated samples were analysed by both HPLC and 1 D-/2 D-TLC, co-chromatographed with reference standards.

A sample of whole milk (Day 5) was extracted once with acetonitrile, twice with acetonitrile/water (1/1) and then once with acetonitrile at room temperature. The individual extracts were collected separately and analysed by LSC. A portion of the combined acetonitrile/water extracts was analysed by both HPLC and 2-D TLC.

Samples of skim milk and milk fat (Day 5) near the maximum concentration was extracted twice with acetonitrile/water (1/1) and then once with acetonitrile. The individual extracts were collected separately and analysed by LSC. A portion of the combined acetonitrile/water extracts was analysed by both HPLC and 2-D TLC.

For liver, kidney and milk, initial unextracted residue after the neutral extraction was further extracted twice with acetonitrile/0.1 mol/L HCl (4/1). The individual extracts were collected separately and analysed by LSC. For skim milk and milk fat, unextracted residue after the neutral extraction was dissolved in 0.1 mol/L NaOH and radio assayed by LSC.

Combined fat samples from renal, omental, and subcutaneous fat were extracted with hexane/acetone (4/1) and twice with acetone. The individual extracts were collected separately and analysed by LSC. The acetone/hexane extracts obtained from three individual samples were combined and concentrated to near dryness. Warm hexane was added and the sample was partitioned twice with acetonitrile. The hexane and acetonitrile phases were quantified by LSC. The initial unextracted residue after the acetone extraction was combusted for quantification by LSC.

Unextracted residue samples after the acetonitrile/0.1 mol/L HCl extraction in liver and kidney were characterized by additional extractions with protease, 4 mol/L HCl at 50 °C and 10 mol/L NaOH at 50 °C. The radioactivity recovered from the samples are shown in Table 6.

Table 6 Total recovery of ^{14}C-benzpyrimoxan

	[Phenyl-U-^{14}C]		[Pyrimidinyl-4(6)-^{14}C]	
	mg eq/kg	percent of applied dose	mg eq/kg	percent of applied dose
Urine (total Day 1-5)	-	50.27	-	48.51
Faeces (total Day 1-5)	-	20.15	-	19.71
Milk Day 1 am	ND	ND	ND	ND
pm	0.052	0.007	0.052	0.032
Day 2 am	0.045	0.007	0.048	0.046

	[Phenyl-U-^{14}C]		[Pyrimidinyl-4(6)-^{14}C]	
	mg eq/kg	percent of applied dose	mg eq/kg	percent of applied dose
pm	0.067	0.007	0.060	0.033
Day 3 am	0.053	0.011	0.046	0.047
pm	0.083	0.009	0.095	0.049
Day 4 am	0.052	0.010	0.059	0.051
pm	0.083	0.009	0.093	0.043
Day 5 am	0.052	0.009	0.058	0.052
pm	0.071	0.003	0.088	0.030
Total (Day 1-5)	-	0.07	-	0.38
Liver	0.220	0.27	0.690	0.67
Kidney	0.249	0.04	0.180	0.03
Flank muscle	0.009	<0.01	0.009	<0.01
Loin muscle	0.008	0.01	0.010	0.01
Omental fat	0.009	0.01	0.014	0.02
Subcutaneous fat	0.012	<0.01	0.015	<0.01
Renal fat	0.012	<0.01	0.018	<0.01
Bile	3.738	0.04	3.764	0.14
Blood	0.032	<0.01	0.051	<0.01
GI tract	-	12.23	-	10.89
Cage rinse	-	0.24	-	0.23
Percent of administered dose		83.32		80.57

The overall recovery of radioactivity was 83.3 percent of the applied dose (AD) for phenyl label and 80.6 percent for pyrimidinyl label, respectively. Radioactivity recovered in the urine, feces, and GI tract accounted for 50.3, 20.2 and 12.2 percent AD for phenyl label, and of 48.5, 19.7 and 10.9 percent AD for pyrimidinyl label, respectively Cage wash contained 0.23-0.24 percent AD. Radioactivity in the tissues accounted for <1.0 percent AD. Liver contained 0.27 and 0.67 percent AD for phenyl and pyrimidinyl labels, respectively, kidney contained 0.04 and 0.03 percent AD, and omental fat contained 0.02 percent AD for phenyl label. Other tissues such as muscle and fat contained ≤0.01 percent AD.

TRRs were measured daily in whole milk (separately skim milk and milk fat). For phenyl label, TRRs in skim milk and milk fat reached a plateau at Days 3–5 and were in a range of 0.051–0.083 mg eq/kg in skim milk, 0.057–0.092 mg eq/kg in milk fat, and 0.052–0.083 mg eq/kg in whole milk. For pyrimidinyl label, TRRs also reached a plateau at Days 3-5 and were in a range of 0.055–0.092 mg eq/kg in skim milk, 0.074–0.120 mg eq/kg in milk fat, and 0.058–0.095 mg eq/kg in whole milk. Table 7 shows the TRRs in the samples after extraction.

Table 7 Extractability of radioactivity in tissues and milk of lactating goats

	Extracted				Unextracted		Total
	Neutral solvent[2)]		ACN/0.1 mol/L HCl				
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
[Phenyl-U-^{14}C]							
Liver[1)]	0.180	76.9	0.009	3.8	0.045	19.2	0.234
Kidney	0.217	90.4	0.006	2.5	0.017	7.1	0.240
Muscle (combined)	N/A	N/A	N/A	N/A	N/A	N/A	<0.010

	Extracted				Unextracted		Total
	Neutral solvent[2)]		ACN/0.1 mol/L HCl				
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Fat (combined)	0.007	77.8	N/A	N/A	0.002	22.2	0.009
Milk (Day 5)	0.067	93.1	<0.001	<1.4	0.005	6.9	0.072
[Pyrimidinyl-4(6)-^{14}C]							
Liver[1)]	0.342	56.5	0.010	1.7	0.253	41.8	0.605
Kidney	0.159	85.0	0.004	2.1	0.024	12.8	0.187
Muscle (combined)	N/A	N/A	N/A	N/A	N/A	N/A	<0.010
Fat (combined)	0.007	63.6	N/A	N/A	0.004	36.4	0.011
Milk (Day 5)	0.073	89.0	<0.001	<1.2	0.009	11.0	0.082

Notes:

N/A: Not analysed.

[1)] Neutral solvent extract of liver was treated with β-glucuronidase.

[2)] Neutral solvent extract of milk was partitioned with ethyl acetate, and ethyl acetate was analysed by HPLC and TLC.

In the neutral solvent extract of liver, which was treated with β-glucuronidase, benzpyrimoxan-CH_2OH (DH-02) and its conjugates (in phenyl label, 0.051 mg eq/kg and 21.8 percent TRR; in pyrimidinyl label, 0.019 mg eq/kg and 3.1 percent TRR), benzpyrimoxan-acid-2-OH (DH-05) (in phenyl label, 0.025 mg eq/kg and 10.7 percent TRR; in pyrimidinyl label, 0.039 mg eq/kg and 6.4 percent TRR), and benzpyrimoxan-CH_2OH-2-OH (DH-06) and its conjugate (in phenyl label, 0.036 mg eq/kg and 15.4 percent TRR; in pyrimidinyl label, 0.029 mg eq/kg and 4.8 percent TRR) were identified as major metabolites. Benzpyrimoxan-2-OH (DH-04) was also identified as a minor metabolite. Unknown metabolites in pyrimidinyl label consisted of several metabolites accounting for 0.054 mg eq/kg and 8.9 percent TRR. From the results by LC-MS in ESI positive mode, it was proposed that molecular weight of unknown-1 and unknown-2 was 226.1 ($C_{10}H_{15}O_3N_3$) and 169.1 ($C_{10}H_{16}O_3N_3$), respectively.

In the neutral solvent extract of kidney, benzpyrimoxan-acid (DH-01) accounted for 15.8 percent TRR in phenyl label (0.038 mg eq/kg) and 20.3 percent TRR (0.038 mg eq/kg) in pyrimidinyl label. DH-02 (free and conjugated) accounted for 16.3 percent TRR (0.039 mg eq/kg) in phenyl label and 5.9 percent TRR (0.011 mg eq/kg) in pyrimidinyl label. DH-05 accounted for 54.6 percent TRR (0.131 mg eq/kg) in phenyl label and 39.6 percent TRR (0.074 mg eq/kg) in pyrimidinyl label. DH-06 (free and conjugated) and benzpyrimoxan-benzoly-glycine (DH-402) were identified as minor metabolites.

In the neutral solvent extract of whole milk, skim milk and milk fat, DH-05 was identified as a major metabolite (0.040–0.062 mg eq/kg and 62.5–86.1 percent TRR in phenyl label; 0.036–0.054 mg eq/kg and 49.3–63.5 percent TRR in pyrimidinyl label). DH-06 was a minor residue.

Tables 8 and 9 sumarize the metabolites identified in liver, kidney, fat and milk in both label experiments.

Table 8 Summary of radioactive residues in tissues and milk of lactating goats following application of [Phenyl-U-^{14}C]-benzpyrimoxan in the diet

	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
	Liver		Kidney		Fat (combined)	
Neutral solvent extract	0.180	76.9	0.217	90.4	0.007	77.8
Benzpyrimoxan	ND	ND	ND	ND	ND	ND
BP-acid (DH-01)	ND	ND	0.038	15.8	ND	ND
BP-CH_2OH (DH-02) + DH-02-conjugate	0.051	21.8	0.039	16.3	ND	ND
BP-2-OH (DH-04)	0.006	2.6	ND	ND	ND	ND
BP-acid-2-OH (DH-05)	0.025	10.7	0.131[1)]	54.6[1)]	ND	ND
BP-CH_2OH-2-OH (DH-06) + DH-06-conjugate	0.036	15.4	0.006	2.5	ND	ND
Others (Maximum of other single)	0.062 (0.019)	26.5 (8.1)	0.003 (0.001)	1.3 (0.4)	0.006 (0.006)	66.7 (66.7)
Hexane layer	N/A	N/A	N/A	N/A	0.001	11.1
Aqueous layer	N/A	N/A	N/A	N/A	N/A	N/A
ACN/0.1 mol/L HCl extract	0.009	3.8	0.006	2.5	N/A	N/A
Unextracted[2)]	0.045	19.2	0.017	7.1	0.002	22.2
Protease extract	0.021	9.0	N/A	N/A	N/A	N/A
4 mol/L HCl extract	0.019	8.1	N/A	N/A	N/A	N/A
10 mol/L NaOH extract	0.001	0.4	N/A	N/A	N/A	N/A
Final unextracted	0.004	1.7	N/A	N/A	N/A	N/A
TRR	0.234	100	0.240	100	0.009	100
	Whole milk (Day 5)		Skim milk (Day 5)		Milk fat (Day 5)	
Neutral solvent extract	0.067	93.1	0.073	98.6	0.052	81.3
Benzpyrimoxan	ND	ND	ND	ND	ND	ND
BP-acid (DH-01)	ND	ND	ND	ND	ND	ND
BP-CH_2OH (DH-02) + DH-02-conjugate	ND	ND	ND	ND	ND	ND
BP-2-OH (DH-04)	ND	ND	ND	ND	ND	ND
BP-acid-2-OH (DH-05)	0.062	86.1	0.059	79.7	0.040	62.5
BP-CH_2OH-2-OH (DH-06) + DH-06-conjugate[3)]	0.001	1.4	0.001	1.4	0.002	3.1
Others (Maximum of other single)	0.002 (0.001)	2.8 (1.4)	0.002 (0.001)	2.7 (1.4)	0.007 (0.002)	10.9 (3.1)
Hexane layer	N/A	N/A	N/A	N/A	N/A	N/A
Aqueous layer[4)]	0.002	2.8	0.011	14.9	0.003	4.7
ACN/0.1 mol/L HCl extract	<0.001	<1.4	N/A	N/A	N/A	N/A
Unidentified	0.005	6.9	0.001	1.4	0.012	18.8
TRR	0.072	100	0.074	100	0.064	100

Notes:

N/A: Not applicable, ND: Not detected.

Muscle was not analysed because of low concentration of < 0.01 mg eq/kg.

[1] DH-402 was also included, not exceeded 0.019 mg eq/kg (7.9%TRR) in kidney by TLC analysis.

[2] No individual component exceeded 0.019 mg eq/kg (8.1%TRR) in liver.

[3] DH-06-conjugate was not detected in milk.

[4] No individual component exceeded 0.011 mg eq/kg (2.8%TRR) in whole milk.

Table 9 Summary of radioactive residues in tissues and milk of lactating goats following application of [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan in the diet

	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
	Liver		Kidney		Fat (combined)	
Neutral solvent extract	0.342	56.5	0.159	85.0	0.007	63.6
Benzpyrimoxan	0.014	2.3	ND	ND	ND	ND
BP-acid (DH-01)	ND	ND	0.038	20.3	ND	ND
BP-CH_2OH (DH-02) + DH-02-conjugate	0.019	3.1	0.011	5.9	ND	ND
BP-2-OH (DH-04)	0.019	3.1	ND	ND	ND	ND
BP-acid-2-OH (DH-05)	0.039	6.4	0.074	39.6	ND	ND
BP-CH_2OH-2-OH (DH-06) + DH-06-conjugate	0.029	4.8	0.002	1.1	ND	ND
Others (Maximum of other single)	0.221 (0.054)[2)]	36.5 (8.9)[2)]	0.035 (0.010)	18.7 (5.1)	0.006 (0.006)	54.5 (54.5)
Hexane layer	N/A	N/A	N/A	N/A	0.001	9.1
Aqueous layer	N/A	N/A	N/A	N/A	N/A	N/A
ACN/0.1 mol/L HCl extract	0.010	1.7	0.004	2.1	N/A	N/A
Unextracted[4)]	0.253	41.8	0.024	12.8	0.004	36.4
Protease extract	0.103	17.0	0.013	7.0	N/A	N/A
4 mol/L HCl extract	0.133	22.0	0.008	4.3	N/A	N/A
10 mol/L NaOH extract	0.013	2.1	0.001	0.5	N/A	N/A
Final unextracted	0.003	0.5	0.002	1.1	N/A	N/A
TRR	0.605	100	0.187	100	0.011	100
	Whole milk (Day 5)		Skim milk (Day 5)		Milk fat (Day 5)	
Neutral solvent extract	0.073	89.0	0.076	89.4	0.057	78.1
Benzpyrimoxan	ND	ND	ND	ND	ND	ND
BP-acid (DH-01)	ND	ND	ND	ND	ND	ND
BP-CH_2OH (DH-02) + DH-02-conjugate	ND	ND	ND	ND	ND	ND
BP-2-OH (DH-04)	ND	ND	ND	ND	ND	ND
BP-acid-2-OH (DH-05)	0.048	58.5	0.054	63.5	0.036	49.3
BP-CH_2OH-2-OH (DH-06) + DH-06-conjugate[1)]	0.002	2.4	0.001	1.2	0.002	2.7
Others (Maximum of other single)	0.003 (0.001)	3.7 (1.2)	0.002 (0.002)	2.4 (2.4)	0.010 (0.002)	13.7 (2.7)
Hexane layer	N/A	N/A	N/A	N/A	N/A	N/A
Aqueous layer[3)]	0.020	24.4	0.019	22.4	0.009	12.3
ACN/0.1 mol/L HCl extract	<0.001	<1.2	N/A	N/A	N/A	N/A
Unidentified	0.009	11.0	0.009	10.6	0.016	21.9
TRR	0.082	100	0.085	100	0.073	100

Notes:

N/A: Not applicable, ND: Not detected

Muscle was not analysed because of low concentration of < 0.01 mg eq/kg.

[1] DH-06-conjugate was not detected in milk.

[2] This metabolite was further analysed by LC/MS.

[3] No individual component exceeded 0.007 mg eq/kg (8.5%TRR) in whole milk.

[4] No individual component exceeded 0.043 mg eq/kg (7.1%TRR) and 0.013 mg eq/kg (7.0%TRR) in liver and kidney, respectively

Laying hens

The metabolism study in laying hens (*Gallus gallus domesticus*) was conducted with ^{14}C-benzpyrimoxan (Ahn, 2018: R-37011). Laying hens (2 treatment groups, 10 hens per group) were treated with either [Phenyl-U-^{14}C] or [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan by oral administration. Each hen received a single daily dose for 7 consecutive days at a nominal 10 ppm in the diet. Doses were contained in cellulose-filled gelatin capsules. The average actual dose was 11.1 ppm for the phenyl label and 12.8 ppm for the pyrimidinyl label

Eggs and excreta were collected twice a day during the treatment period. The hens were sacrificed at 6 hours after the last dose, and liver, muscle (breast and leg), gastrointestinal tract and fat samples (abdominal and subcutaneous) were collected.

Cage washes were analysed directly by LSC. Portions of each excreta sample and homogenized gastrointestinal tracts were analysed for ^{14}C residue by combustion via oxidizers. The TRRs in liver, muscle, fat and eggs (whole and separated) were solubilised and measured by LSC after addition of scintillation cocktail.

Eggs and all tissues (except for phenyl labeled musle) contained >0.010 mg eq/kg and were subjected to metabolite analysis. Extracted radiolabeled residues were identified by HPLC using cochromatography with authentic reference standards. The identity of residues was confirmed by TLC comparing the Rf values of standards with the sample they were analysed with.

Samples of liver, muscle and egg were extracted with acetonitrile/water (1/1) and then with acetonitrile. Unextracted residues after the neutral extraction were further extracted with acetonitrile/0.1 mol/L HCl (4/1). The remaining unextracted residue after the weak acidic extraction was combusted for quantification by LSC.

Samples of fat were extracted with hexane/acetone (4/1) and then with acetone. The initial unextracted residue after the acetone extraction was combusted for quantification by LSC. The combined acetone/hexane and acetone extracts from the fat tissues was concentrated to near dryness. Hexane was added and the sample was partitioned with acetonitrile. The hexane and acetonitrile phases were quantified by LSC. Combined acetonitrile layers were analysed by both HPLC and 2D-TLC.

Combined acetonitrile/water extracts of liver (pyrimidinyl-label) were concentrated and subjected to hydrolysis with β-glucuronidase and sulfatase. There was no significant difference between enzyme-treated samples and non-treated samples by HPLC analysis, confirmed by TLC. Unextracted samples after the acetonitrile/0.1 mol/L HCl extraction in liver and egg were characterized by additional extractions with protease, 4 mol/L HCl at 50 °C and 10 mol/L NaOH at 50 °C.

The overall recovery of radioactivity from the group of 10 laying hens was 76.34 percent dose for phenyl label and 87.78 percent dose for pyrimidinyl label, respectively. Radioactivity recovered in the excreta and cage washes accounted for 74.54 percent dose and 0.481 percent dose for phenyl label, and for 85.19 percent dose and 0.382 percent dose for pyrimidinyl label, respectively. Radioactivity in the

tissues accounted for <0.1 percent dose for phenyl label and 0.139 percent dose for pyrimidinyl label (Table 10).

Table 10 Total recovery of ^{14}C-benzpyrimoxan

	[Phenyl-U-^{14}C]		[Pyrimidinyl-4(6)-^{14}C]	
	mg eq/kg	percent of applied dose	mg eq/kg	percent of applied dose
Excreta (total Day 1-7)	-	74.54	-	85.19
Egg white Day 1 am/pm	0.001/0.002	<0.001/<0.001	0.004/0.005	0.001/<0.001
Day 2 am/pm	0.004/0.005	0.001/0.001	0.007/0.008	0.001/0.001
Day 3 am/pm	0.006/0.006	0.001/0.001	0.009/0.012	0.003/0.001
Day 4 am/pm	0.007/0.005	0.001/0.001	0.007/0.008	0.002/0.001
Day 5 am/pm	0.005/0.004	0.001/0.001	0.007/0.009	0.002/0.001
Day 6 am/pm	0.006/0.006	0.001/0.001	0.006/0.007	0.001/0.001
Day 7 am/pm	0.008/0.008	0.002/0.002	0.007/0.007	0.001/0.002
Total (Day 1-7)	0.005	0.015	0.007	0.019
Egg yolk Day 1 am/pm	0.002/0.002	<0.001/<0.001	0.006/0.005	0.001/<0.001
Day 2 am/pm	0.006/0.007	0.001/<0.001	0.004/0.006	<0.001/<0.001
Day 3 am/pm	0.013/0.022	0.001/0.002	0.027/0.040	0.004/0.001
Day 4 am/pm	0.020/0.036	0.002/0.003	0.076/0.098	0.010/0.003
Day 5 am/pm	0.032/0.049	0.003/0.004	0.107/0.160	0.012/0.007
Day 6 am/pm	0.040/0.053	0.004/0.004	0.134/0.185	0.014/0.015
Day 7 am/pm	0.045/0.051	0.004/0.004	0.160/0.197	0.009/0.022
Total (Day 1-7)	0.027	0.033	0.086	0.100
Liver	0.066	0.037	0.188	0.095
Leg muscle	0.006	0.005	0.014	0.009
Breast muscle	0.003	0.003	0.011	0.009
Abdominal fat	0.042	0.011	0.063	0.013
Subcutaneous fat	0.065	0.005	0.176	0.013
GI tract	-	1.209	-	1.952
Cage wash	-	0.481	-	0.382
percent of administered dose		76.34		87.78

TRR were measured daily in eggs (egg white and yolk measured separately). For phenyl label, concentrations of radioactivity both in egg white and yolk reached a plateau at Days 4–7 and were in a range of 0.004–0.008 mg eq/kg in egg white and 0.032–0.053 mg eq/kg in egg yolk. For pyrimidinyl label, concentrations of radioactivity reached a plateau at Days 5–7 and were in a range of 0.006–0.009 mg eq/kg in egg white and 0.134–0.197 mg eq/kg in egg yolk. The extractability of the residues is shown in Table 11.

Table 11 Extractability of radioactivity in tissues and egg of laying hens

	Extracted				Unextracted		Total
	Neutral solvent		ACN/0.1 mol/L HCl				
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
[Phenyl-U-^{14}C]							
Liver	0.036	60.0	0.001	1.7	0.023	38.3	0.060
Muscle (combined)	N/A	N/A	N/A	N/A	N/A	N/A	<0.010

	Extracted				Unextracted		Total
	Neutral solvent		ACN/0.1 mol/L HCl				
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Fat (combined)	0.058	95.1	N/A	N/A	0.003	4.9	0.061
Egg	0.010	66.7	0.001	6.7	0.004	26.7	0.015
[Pyrimidinyl-4(6)-^{14}C]							
Liver	0.097	53.9	0.004	2.2	0.079	43.9	0.180
Muscle (combined)	0.011	78.6	<0.001	<7.1	0.003	21.4	0.014
Fat (combined)	0.121	96.0	N/A	N/A	0.005	4.0	0.126
Egg	0.022	31.4	0.002	2.9	0.046	65.7	0.070

Notes:

N/A: Not applicable

In the neutral solvent extract of liver, benzpyrimoxan-CH_2OH (DH-02) (in phenyl label, 0.013 mg eq/kg and 21.7 percent TRR; in pyrimidinyl label, 0.011 mg eq/kg and 6.1 percent TRR) and benzpyrimoxan-acid-2-OH (DH-05) (in phenyl label, 0.012 mg eq/kg and 20.0 percent TRR; in pyrimidinyl label, 0.050 mg eq/kg and 27.8 percent TRR) were identified as major metabolites. In addition, benzpyrimoxan-CH_2OH-2-OH (DH-06) was also detected as a minor component.

In the initial extract of fat, benzpyrimoxan (in phenyl label, 0.043 mg/kg and 70.5 percent TRR; in pyrimidinyl label, 0.089 mg/kg and 70.6 percent TRR) and DH-02 (in phenyl label, 0.006 mg eq/kg and 9.8 percent TRR; in pyrimidinyl label, 0.018 mg eq/kg and 14.3 percent TRR) were identified as major components. In the neutral solvent extract of muscle and egg, benzpyrimoxan and DH-02 were identified as major components, but were below 0.01 mg eq/kg.

Tables 12 and 13 summarize the compounds indentified in the experiments.

Table 12 Summary of radioactive residues in tissues and egg of laying hens following application of [Phenyl-U-^{14}C]-benzpyrimoxan in the diet

	Liver		Fat (combined)		Egg	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Neutral solvent extract	0.036	60.0	0.058	95.1	0.010	66.7
Benzpyrimoxan	ND	ND	0.043	70.5	0.002	13.3
BP-acid (DH-01)	ND	ND	ND	ND	ND	ND
BP-CH_2OH (DH-02)	0.013	21.7	0.006	9.8	0.004	26.7
BP-acid-2-OH (DH-05)	0.012	20.0	ND	ND	<0.001	<0.1
BP-CH_2OH-2-OH (DH-06)	0.002	3.3	ND	ND	ND	ND
Others (Maximum of others)	0.009 (0.004)[2)]	15.0 (6.7)[2)]	0.009 (0.008)	14.8 (13.1)	0.004 (0.004)	26.7 (26.7)
ACN/0.1 mol/L HCl extract	0.001	1.7	N/A	N/A	0.001	6.7
Unextracted	0.023[1)]	38.3[1)]	0.003	4.9	0.004	26.7
Protease extract	0.004	6.7	N/A	N/A	N/A	N/A
4 mol/L HCl extract	0.015	25.0	N/A	N/A	N/A	N/A
10 mol/L NaOH extract	0.001	1.7	N/A	N/A	N/A	N/A
Final unextracted	0.003	5.0	N/A	N/A	N/A	N/A
TRR	0.060	100	0.061	100	0.015	100

Notes:

N/A: Not applicable, ND: Not detected

Muscle was not analysed because of low concentration of < 0.01 mg eq/kg.

[1] No individual component exceeded 0.007 mg eq/kg (11.7%TRR) by characterization.

Table 13 Summary of radioactive residues in tissues and egg of laying hens following application of [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan in the diet

	Liver		Muscle (combined)		Fat (combined)		Egg	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Neutral solvent extract	0.097	53.9	0.011	78.6	0.121	96.0	0.022	31.4
Benzpyrimoxan	ND	ND	0.006	42.9	0.089	70.6	0.004	5.7
BP-acid (DH-01)	ND	ND	<0.001	<7.1	ND	ND	ND	ND
BP-CH_2OH (DH-02)	0.011	6.1	0.002	14.3	0.018	14.3	0.009	12.9
BP-acid-2-OH (DH-05)	0.050	27.8	<0.001	<7.1	ND	ND	ND	ND
BP-CH_2OH-2-OH (DH-06)	0.006	3.3	ND	ND	ND	ND	ND	ND
Others (Maximum of single)	0.030 (0.005)	16.7 (2.8)	<0.003 (0.002)	<21.4 (14.3)	0.010 (0.008)	7.9 (6.3)	0.009 (0.006)	12.9 (8.6)
ACN/0.1 mol/L HCl extract	0.004	2.2	<0.001	<7.1	N/A	N/A	0.002	2.9
Unextracted	0.079[1)]	43.9[1)]	0.003	21.4	0.005	4.0	0.046[2)]	65.7[2)]
Protease extract	0.022	12.2	N/A	N/A	N/A	N/A	0.026	37.1
4 mol/L HCl extract	0.048	26.7	N/A	N/A	N/A	N/A	0.019	27.1
10 mol/L NaOH extract	0.007	3.9	N/A	N/A	N/A	N/A	0.001	1.4
Final unextracted	0.003	1.7	N/A	N/A	N/A	N/A	<0.000	<0.1
TRR	0.180	100	0.014	100	0.126	100	0.070	100

Notes:

N/A: Not applicable, ND: Not detected

[1] No individual component exceeded 0.037 mg eq/kg (20.6%TRR) by characterization.

Furthermore, these residues consist of some ninhydrin-positive and protein-related residues by TLC.

[2] No individual component exceeded 0.011 mg eq/kg (15.7%TRR) by characterization.

Furthermore, these residues consist of some ninhydrin-positive and protein-related residues by TLC.

Summary of animal metabolism

The metabolic fate of benzpyrimoxan seems to be similar in the goat and the hen and is proposed in Figure 3.

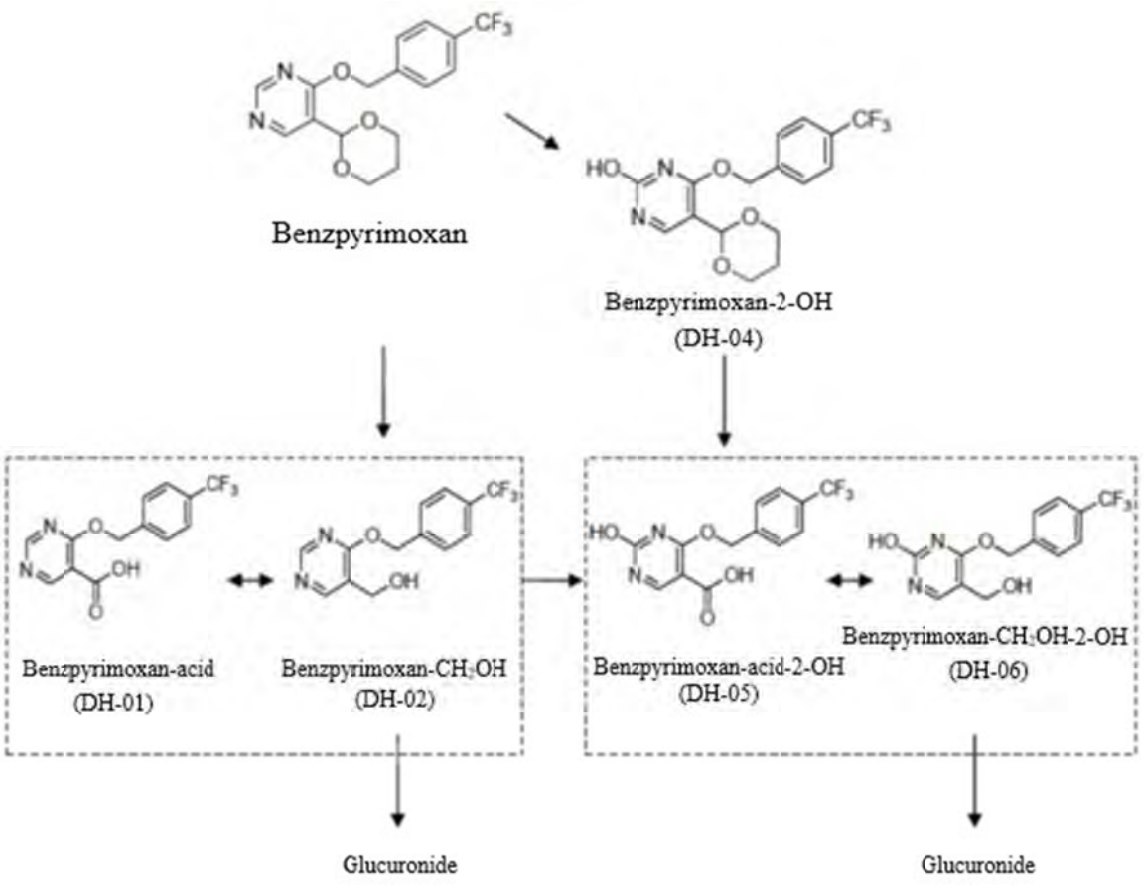


Figure 3 Metabolic pathway of benzpyrimoxan in farm animals

Environmental fate

The Meeting received information on degradation in aerobic soil (paddy and upland), hydrolysis and photodegradation studies.

Degradation in aerobic soil

The soil metabolism study was conducted in aerobic soil under paddy conditions. The test system employed in the study consisted of a 70 g dry weight equivalent of soil flooded with a water layer in a vessel which was connected to a series of traps for organic volatiles and for alkaline volatiles including carbon dioxide using ethylene glycol and 20 percent ethanol amine, respectively. Following a 14-day pre-incubation, [Phenyl-U-^{14}C] and [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan were applied at 0.2 mg ai/kg dry soil, which was equivalent to the proposed maximum field application rate (200 g ai/ha) and incubated in the dark at 25±2 °C under aerobic conditions (Tanaka, 2016: E-37003). Table 14 shows the characteristics of the soil used in the study.

Table 14 Paddy soil characteristics (Tanaka, 2016: E-37003)

Origin location	Kochi, Japan
Soil texture (USDA)	Clay loam
percent Sand	20.4
percent Silt	52.0
percent Clay	27.6

Origin location	Kochi, Japan
pH (KCl)	4.6 [21°C]
pH (water)	5.7 [21°C]
Organic carbon (percent)	1.41
Cation exchange capacity ($cmol_c/kg$)	12.3
Maximum water holding capacity (10 g/kg)	72.0
Bulk density (mg/cm^3)	0.97
Microbial biomass (mg/kg)	
At zero day (pre-incubation)	639
After 120 days	512
After 180 days (post-incubation)	466

Soil samples were analysed at 0, 14, 30, 60, 120 and 180 days after application. Water phase of soil sample was removed after decantation and the soil was extracted with neutral extraction solvent (acetonitrile/distilled water, 4/1), two acidic extraction solvents (acetonitrile/0.1 mol/L HCl, 4/1 and acetonitrile/1.0 mol/L HCl, 4/1). Extracts and trap solutions were subjected to LSC. Unextracted radioactivity obtained after extraction by the solvents was subjected to combustion radioanalysis. Total recovery was determined as the sum of radioactivity in extracts (water and soil phase), trap solution and unextracted, and expressed as percent of applied radioactivity (AR) (Table 15). The extracted fractions were analysed by TLC and HPLC. Additionally, characterization of radioactivity in 20 percent ethanolamine trap media (CO_2 fraction) and unextracted was conducted.

Unextracted radioactivity was fractionated as follows: An aliquot of air-dried soils after solvent extraction derived from 180 day and sterilized soil samples was re-extracted with 1 mol/L NaOH solution at about 50 ºC by shaking for 2 hours followed by centrifugation. The obtained precipitate (humin fraction) was weighed. An aliquot was subjected to combustion radioanalysis. The supernatant was acidified to pH 1–2 by addition of.12 mol/L HCl, and resultant insoluble matter was removed by centrifugation. The supernatant and precipitate obtained by centrifuge were designated as fulvic acid and humic acid fractions, respectively. Fulvic acid fraction was filled up to 25 mL and then an aliquot was subjected to radioanalysis. An aliquot of humic acid fraction was subjected to combustion radioanalysis after reconstituting with 1 mol/L NaOH solution.

Table 15 Mass balance and distribution of benzpyrimoxan applied to paddy soil, in percent applied radioactivity[1)]

	Incubation period (day)						
	0	14	30	60	120	180	180[2)]
[Phenyl-U-^{14}C]							
Water phase	3.1	1.2	0.9	0.9	0.8	0.4	1.5
Soil phase	92.6	95.3	93.5	93.6	94.7	93.8	93.7
Solvent Extracts	92.6	94.4	91.7	91.0	92.0	88.0	88.3
ACN/H_2O	84.7	64.9	53.2	45.2	62.5	58.9	68.7
ACN/0.1 mol/L HCl	7.1	20.5	22.1	21.1	15.5	19.0	15.5
ACN/1.0 mol/L HCl	0.7	9.0	16.4	24.7	14.0	10.2	4.1
Unextracted	ND	1.0	1.8	2.4	2.4	5.1	5.3
Volatiles	N/A	ND	<0.1	0.1	0.3	0.7	N/A
Total Recovery	95.6	96.5	94.5	94.5	95.5	94.3	95.2
[Pyrimidinyl-4(6)-^{14}C]							
Water phase	2.6	1.7	1.3	1.1	0.8	0.5	1.7

	Incubation period (day)						
	0	14	30	60	120	180	180[2)]
[Phenyl-U-^{14}C]							
Soil phase	95.0	93.6	94.4	93.9	93.5	93.1	94.2
Solvent Extracts	95.0	92.4	92.6	91.4	91.1	87.8	86.4
ACN/H_2O	86.8	64.9	57.7	47.3	61.6	59.2	66.7
ACN/0.1 mol/L HCl	7.4	16.8	18.6	19.8	16.5	16.1	15.5
ACN/1.0 mol/L HCl	0.9	10.7	16.2	24.3	13.0	12.4	4.2
Unextracted	<0.1	1.2	1.8	2.5	2.4	5.3	7.8
Volatiles[3)]	N/A	<0.1	0.1	0.2	0.9	1.9	N/A
Total Recovery	97.7	95.3	95.8	95.2	95.2	95.6	95.9

Notes:

N/A: Not applicable, ND: Not detected

[1] Mean of duplicates

[2] Sterilized

[3] Sum of organic volatile and carbon dioxide

Radioactivity in trap solution and unextracted accounted for a maximum of 1.9 and 5.3 percent of AR, respectively. Substantial mineralization ($^{14}CO_2$) was observed with both radiolabels, which indicated that benzpyrimoxan was steadily degraded in paddy soil. Fractionation of unextracted revealed that radioactivity was mainly distributed in humin fraction.

Tables 16 and 17 show the degradantes of benzpyrimoxan in paddy soil. The total recoveries were more than 94.3 percent AR for [Phenyl-U-^{14}C] benzpyrimoxan and more than 95.2 percent AR for [Pyrimidinyl-4(6)-^{14}C] benzpyrimoxan. Regardless of incubation time and sterilized or non-sterilized soil, the radioactivity in the solventextracted fraction accounted for more than about 90 percent of AR. Benzpyrimoxan accounted for more than 70 percent of AR, even after 180 days incubation, which implied that benzpyrimoxan degraded in paddy soil very gradually. The laboratory half-life of benzpyrimoxan in paddy soil was over 1 year.

Several identified and unknown degradates were detected, however, none of which accounted for more than 10 percent of AR throughout the study period for both radiolabels. Benzpyrimoxan-2-OH (DH-04) was the major degradate, which was formed at a maximum of 5.3 percent of AR after 120 days incubation. Benzpyrimoxan-acid (DH-01), benzpyrimoxan-aldehyde (DH-03), benzpyrimoxan-acid-2-OH (DH-05), benzpyrimoxan-CH_2OH-2-OH (DH-06), benzpyrimoxan-aldehyde-2-OH (DH-07), 4-TFMB (DH-101) and 4-TFMPM (DH-102) were detected as minor degradates. Under sterilized condition, benzpyrimoxan-acid (DH-01), benzpyrimoxan-aldehyde (DH-03), benzpyrimoxan-enaminealdehyde (DH-08) and 4-TFMPM (DH-102) were also detected. The results are shown in Tables 16 and 17.

Table 16 Degradation of [Phenyl-U-^{14}C]-benzpyrimoxan in paddy soil under aerobic conditions, in percent of applied radioactivity

	Incubation period (day)						
	0	14	30	60	120	180	180[1)]
Recovery (Water + Soil)	95.6	95.5	92.6	92.0	92.8	88.4	89.8
Benzpyrimoxan	95.3	91.0	83.2	83.2	82.0	72.9	84.3
BP-acid (DH-01)	ND	ND	ND	ND	ND	ND	<0.1
BP-CH_2OH (DH-02)	ND	ND	ND	ND	ND	ND	ND

	Incubation period (day)						
	0	14	30	60	120	180	180[1)]
BP-aldehyde (DH-03) & 4-TFMPM (DH-102)[2)]	ND	0.2	1.8	1.2	1.4	1.2	2.4
BP-2-OH (DH-04)	0.3	3.9	5.0	3.3	3.4	4.7	ND
BP-acid-2-OH (DH-05)	ND	ND	0.1	0.2	0.2	1.4	ND
BP-CH_2OH-2-OH (DH-06)	ND	ND	<0.1	0.3	0.9	0.6	ND
BP-aldehyde-2-OH (DH-07)	ND	ND	1.3	1.6	2.7	4.8	ND
BP-enaminealdehyde (DH-08)	ND	ND	ND	ND	ND	ND	1.3
4-TFMB (DH-101)	ND	ND	ND	<0.1	0.6	1.0	ND
BP-benzaldehyde (DH-103)	ND	ND	ND	ND	ND	ND	ND
Sum of others	ND	0.4	1.1	1.9	1.6	1.8	1.8

Notes:

ND: Not detected; Mean of duplicates.

[1] Sterilized.

[2] The values are sum of radioactivity of DH-03 and DH-102 because the spot of these degradates on TLC was close.

Table 17 Degradation of [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan in paddy soil under aerobic conditions, in percent of applied radioactivity[1)]

	Incubation period (day)						
	0	14	30	60	120	180	180[2)]
Recovery (Water + Soil)	97.6	94.1	93.9	92.4	91.9	88.3	88.2
Benzpyrimoxan	97.3	89.8	87.2	83.6	80.9	74.3	82.3
BP-acid (DH-01)	ND	ND	ND	ND	ND	<0.1	ND
BP-CH_2OH (DH-02)	ND	ND	ND	ND	ND	ND	ND
BP-aldehyde (DH-03)	ND	ND	ND	0.4	0.7	0.4	0.6
BP-2-OH (DH-04)	0.1	3.3	4.3	2.7	5.3	2.3	ND
BP-acid-2-OH (DH-05)	ND	ND	0.2	0.2	0.3	1.2	ND
BP-CH_2OH-2-OH (DH-06)	ND	ND	ND	0.5	1.0	1.1	ND
BP-aldehyde-2-OH (DH-07)	ND	0.6	1.2	1.8	1.2	3.1	ND
BP-enaminealdehyde (DH-08)	ND	ND	ND	ND	ND	ND	1.3
Sum of others[3)]	0.2	0.3	0.9	3.0	2.4	5.8	3.9

Notes:

ND: Not detected

[1] Mean of duplicates

[2] Sterilized

[3] Sum of TLC origin and unknown degradates

In a second study, the soil metabolism study was conducted in upland soil under aerobic conditions. The test system employed in the study consisted of a 50 g dry weight equivalent of soil in a vessel which was connected to a series of traps for organic volatiles and for alkaline volatiles including carbon dioxide using ethylene glycol and 20 percent ethanol amine, respectively. Following a 14-day pre-incubation, [Phenyl-U-^{14}C] and [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan were applied at 0.2 mg ai/kg dry soil, which was equivalent to the proposed maximum field application rate (200 g ai/ha) and incubated in the dark at 25 ± 2 ºC under aerobic conditions (Tanaka, 2017: E-37006). Table 18 shows the characteristics of the soil used in the study.

Table 18 Upland soil characteristics (Tanaka, 2017: E-37006)

Origin location	Kochi, Japan
Soil texture (USDA)	Loam
percent Sand	35.8
percent Silt	43.1
percent Clay	21.1
pH (KCl)	5.6 [21 °C]
pH (water)	6.7 [21 °C]
Organic carbon (percent)	1.97
Cation exchange capacity ($cmol_c/kg$)	15.4
Maximum water holding capacity (10 g/kg)	73.0
Bulk density (mg/cm^3)	1.06
Microbial biomass (mg/kg)	
At zero day (pre-incubation)	121
After 120 days	122
After 180 days (post-incubation)	78

Soil samples were analysed at 0, 14, 30, 60, 120 and 180 days after application and extracted with neutral extraction solvent (acetonitrile/distilled water (4/1), two acidic extraction solvents (acetonitrile/0.1 mol/L HCl (4/1) and acetonitrile/1.0 mol/L HCl (4/1 v/v)). Extracts and trap solutions thus obtained were subjected to liquid scintillation counting. Unextracted radioactivity obtained was subjected to combustion radioanalysis. The extracted fractions were analysed by TLC and HPLC. Additionally, characterization of radioactivity in 20 percent ethanolamine trap media (CO_2 fraction) and unextracted was conducted. Total recovery was determined as the sum of radioactivity in extracts, trap solution and unextracted and the results are shown in Table 19.

Radioactivity in unextracted was fractionated as follows: An aliquot of air-dried unextracted derived from 180 days sample and sterilized soil sample was re-extracted with 1 mol/L NaOH solution at about 50 ºC by shaking for 2 hours followed by centrifugation. The obtained precipitate (humin fraction) was weighed. An aliquot was subjected to combustion radioanalysis. The supernatant was acidified to pH ranged from 1–2 using.12 mol/L HCl, and resultant insoluble matter was removed by centrifugation. The supernatant and precipitate obtained by centrifuge were designated as fulvic acid and humic acid fractions, respectively. Fulvic acid fraction was filled up to 25 mL and then an aliquot was subjected to radioanalysis. An aliquot of humic acid fraction was subjected to combustion radioanalysis after reconstituting with 1 mol/L NaOH solution.

Table 19 Mass balance and distribution of benzpyrimoxan applied to upland soil, as percent of applied radioactivity[1)]

	Incubation period (day)						
	0	14	30	60	120	180	180[2)]
[Phenyl-U-^{14}C]							
Extracts	97.8	95.9	93.2	86.7	69.5	57.0	91.9
ACN/H_2O	94.1	80.1	69.8	60.9	44.2	33.2	78.2
ACN/0.1 mol/L HCl	3.8	12.9	18.7	19.7	18.0	15.8	11.7
ACN/1.0 mol/L HCl	ND	2.8	4.7	6.2	7.2	8.1	2.0
Unextracted	ND	1.1	3.1	6.6	16.7	25.1	3.0
Volatiles	N/A	<0.1	0.2	1.2	7.0	9.3	N/A
Ethylene glycol trap	N/A	ND	ND	ND	ND	ND	N/A

	Incubation period (day)						
	0	14	30	60	120	180	180[2)]
[Phenyl-U-^{14}C]							
Ethanolamine trap	N/A	<0.1	0.2	1.2	7.0	9.3	N/A
Total Recovery	97.8	97.0	96.6	94.5	93.2	91.4	94.9
[Pyrimidinyl-4(6)-^{14}C]							
Extracts	96.8	96.2	89.6	79.1	63.3	51.2	91.0
ACN/H_2O	93.3	80.7	66.1	55.4	40.2	30.7	77.9
ACN/0.1 mol/L HCl	3.6	12.6	18.3	17.8	17.3	14.5	11.0
ACN/1.0 mol/L HCl	ND	2.9	5.2	5.9	5.8	6.1	2.2
Unextracted	ND	1.1	5.1	9.6	14.8	25.2	4.5
Volatiles	N/A	<0.1	1.0	5.0	12.5	14.4	N/A
Ethylene glycol trap	N/A	ND	ND	ND	ND	ND	N/A
Ethanolamine trap	N/A	<0.1	1.0	5.0	12.5	14.4	N/A
Total Recovery	96.8	97.3	95.7	93.7	90.7	90.9	95.5

Notes:

N/A: Not applicable, ND: Not detected

[1] Mean of duplicates

[2] Sterilized

Tables 20 and 21 show the degradation data of benzpyrimoxan in upland soil. The total recoveries were more than 91.4 percent AR for [Phenyl-U-^{14}C]-benzpyrimoxan and more than 90.7 percent of AR for [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan. The radioactivity of extracted fraction was reduced to about 50 percent of AR at 180 days incubation, although the radioactivity of extracted fraction remained more than 90 percent of AR at 180 days for sterilized soil. Benzpyrimoxan was continuously degraded in soil throughout the incubation period, and reduced to about 30 percent of AR at 180 days. The laboratory half-life of benzpyrimoxan in upland soil under aerobic condition was 124 days.

Radioactivity in trap solution and unextracted accounted for a maximum of 14.4 and 25.2 percent of AR, respectively. Substantial mineralization ($^{14}CO_2$) was observed with both radiolabels, which indicated that benzpyrimoxan was steadily degraded in soil. Fractionation of unextracted revealed that radioactivity was mainly distributed in humin fraction.

Several identified and unknown degradates were detected, none of which accounted for more than 10 percent of AR. Metabolite DH-04 was the major degradate, which was formed at a maximum of 7.8 percent of AR after 14 days incubation. Benxpyrimoxan-CH_2OH (DH-02), benzpyrimoxan-aldehyde (DH-03), benzpyrimoxan-acid-2-OH (DH-05), benzpyrimoxan-aldehyde-2-OH (DH-07), benzpyrimoxan-enaminealdehyde (DH-08), 4-TFMB (DH-101) and 4-TFMPM (DH-102) were detected as minor degradates. In the case of sterilized condition, DH-03, DH-08 and DH-102 were detected, suggested that these degradates were derived from chemical reaction.

Table 20 Degradation of [Phenyl-U-^{14}C]-benzpyrimoxan in upland soil under aerobic conditions, percent of applied radioactiviy[1)]

	Incubation period (day)						
	0	14	30	60	120	180	180[2)]
Extract[3)]	97.8	95.9	93.2	86.7	69.5	57.0	91.9
Benzpyrimoxan	95.7	80.4	69.1	62.0	43.6	33.4	89.2
BP-acid (DH-01)	ND	ND	ND	ND	ND	ND	ND

	Incubation period (day)						
	0	14	30	60	120	180	180[2)]
BP-CH_2OH (DH-02)	ND	ND	ND	ND	ND	0.3	ND
BP-aldehyde (DH-03) & 4-TFMPM (DH-102)[4)]	0.4	ND	ND	1.8	2.8	4.3	0.5
BP-2-OH (DH-04)	ND	7.8	6.8	2.5	2.8	1.8	ND
BP-acid-2-OH (DH-05)	ND	0.8	7.4	8.9	6.8	3.9	ND
BP-CH_2OH-2-OH (DH-06)	ND	ND	ND	ND	ND	ND	ND
BP-aldehyde-2-OH (DH-07)	ND	6.2	5.3	2.1	1.6	1.0	ND
BP-enaminealdehyde (DH-08)	ND	ND	ND	ND	ND	ND	0.8
4-TFMB (DH-101)	ND	0.2	2.1	6.4	3.2	2.4	ND
BP-benzaldehyde (DH-103)	ND	ND	ND	ND	ND	ND	ND
Origin	1.7	0.6	2.5	2.8	3.6	5.7	1.1
Sum of others	ND	ND	ND	ND	4.9	4.2	0.2
Unextracted	ND	1.1	3.1	6.6	16.7	25.1	3.0
Organic volatile	N/A	ND	ND	ND	ND	ND	N/A
CO_2	N/A	<0.1	0.2	1.2	7.0	9.3	N/A
Total Recovery	97.8	97.0	96.6	94.5	93.2	91.4	94.9

Notes:

N/A: Not applicable, ND: Not detected

[1] Mean of duplicates

[2] Sterilized

[3] The sum of the extracted fraction (acetonitrile/distilled water, acetonitrile/0.1 mol/L HCl and acetonitrile/1.0 mol/L HCl).

[4] The values are sum of radioactivity of DH-03 and DH-102 because the spot of these degradates on TLC was close.

Table 21 Degradation of [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan in upland soil under aerobic conditions, percent of applied radioactiviy[1)]

	Incubation period (day)						
	0	14	30	60	120	180	180[2)]
Extract[3)]	96.8	96.2	89.6	79.1	63.3	51.2	91.0
Benzpyrimoxan	93.9	79.9	67.7	61.0	45.5	31.8	87.5
BP-acid (DH-01)	ND	ND	ND	ND	ND	ND	ND
BP-CH_2OH (DH-02)	ND	ND	ND	ND	ND	<0.1	ND
BP-aldehyde (DH-03)	<0.1	ND	ND	ND	<0.1	0.3	<0.1
BP-2-OH (DH-04)	ND	7.3	4.8	2.3	1.2	1.9	ND
BP-acid-2-OH (DH-05)	ND	1.1	7.1	8.4	8.1	4.8	ND
BP-CH_2OH-2-OH (DH-06)	ND	ND	ND	ND	ND	ND	ND
BP-aldehyde-2-OH (DH-07)	ND	7.0	5.4	2.1	1.5	1.0	ND
BP-enaminealdehyde (DH-08)	ND	ND	ND	ND	ND	ND	0.6
Origin	2.6	0.9	4.6	5.4	5.0	8.2	2.6
Sum of others	0.3	ND	ND	ND	1.9	3.1	0.2
Unextracted	ND	1.1	5.1	9.6	14.8	25.2	4.5
Organic volatile	N/A	ND	ND	ND	ND	ND	N/A
CO_2	N/A	<0.1	1.0	5.0	12.5	14.4	N/A
Total Recovery	96.8	97.3	95.7	93.7	90.7	90.9	95.5

Notes:

N/A: Not applicable, ND: Not detected

[1] Mean of duplicates

[2] Sterilized

[3] The sum of the extracted fraction (acetonitrile/distilled water, acetonitrile/0.1 mol/L HCl and acetonitrile/1.0 mol/L HCl).

In summary, benzpyrimoxan was degraded in aerobic soil under paddy and upland condition, following production of many degradates. Degradates were mainly produced by hydroxylation of pyrimidine ring, cleavage of cyclic acetal and oxidation of carbonyl group. In addition, pyrimidine ring part was cleaved or broken away. Degradates thus produced were further changed other degradates and took in bound residue, consequently mineralized to CO_2. The proposed methabolic pathway is shown in Figure 4.

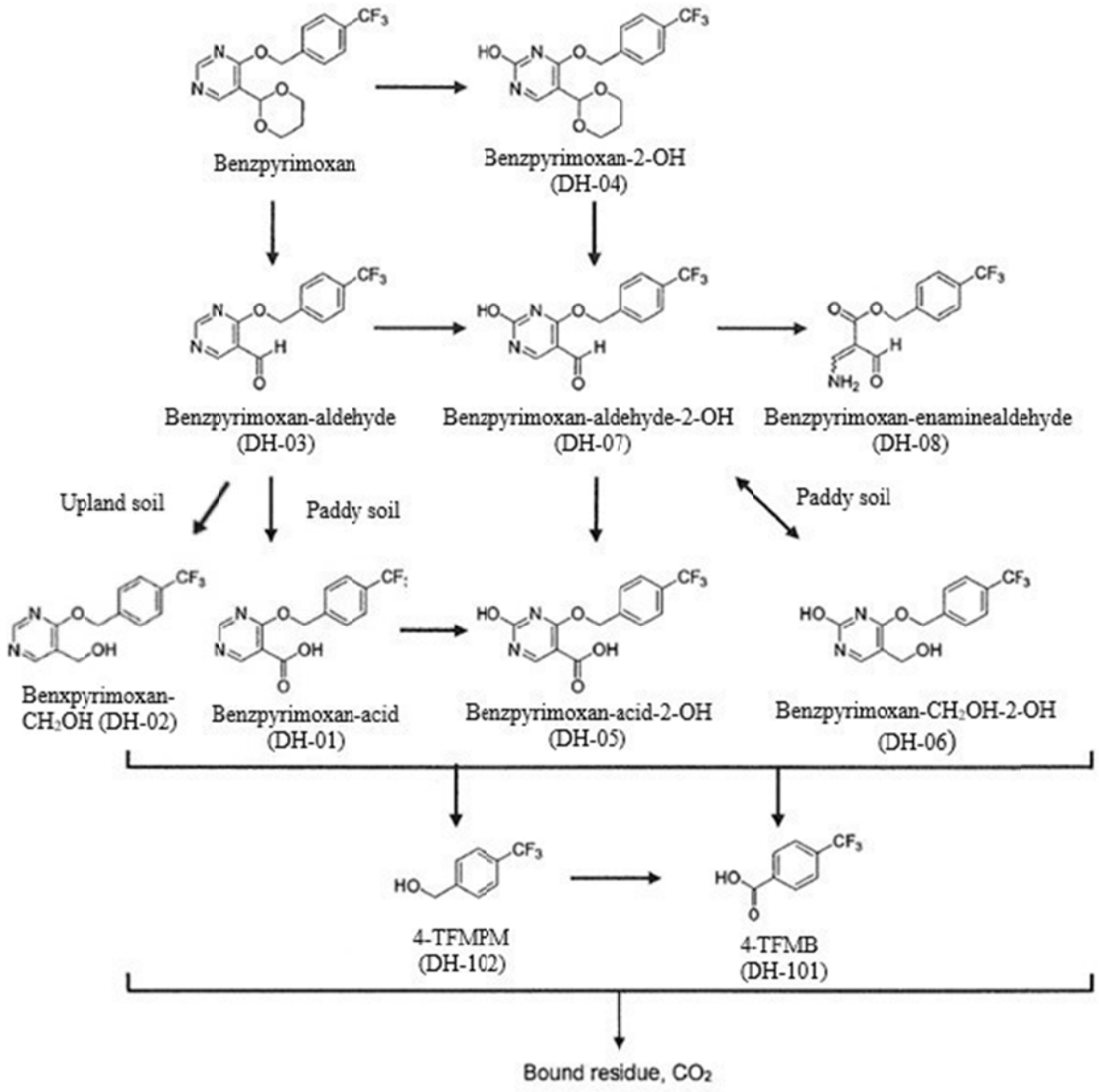


Figure 4 Metabolic pathway of benzpyrimoxan in aerobic soil

Hydrolysis

The hydrolysis of ^{14}C-benzpyrimoxan was studied in buffers solutions (Nishimura, 2017: E-37007).

For the preliminary test (Tier 1 test), hydrolysis of [Phenyl-U-^{14}C]-benzpyrimoxan was tested in aqueous buffer solutions at 50°C at different pHs (pH 4, 7 and 9), with a nominal concentration of 2 mg/L. The solutions were incubated in the dark under sterile conditions. Duplicate samples were taken after 0 and 5 days of inclubation and analysed by radio-HPLC.

After 5 days, 23.9, 98.1 and 98.2 percent AR were identified as unchanged benzpyrimoxan at pH 4, 7 and 9, respectively (Table 22). Benzpyrimoxan was considered to be hydrolytically stable at environmentally relevant temperatures under neutral and basic conditions, while benzpyrimoxan was considered to be hydrolytically unstable under acidic conditions.

Table 22 Proportions of radioactive components in aqueous solutions at pH 4, 7 and 9 at 50 °C after treatment of [Phenyl-U-^{14}C]-benzpyrimoxan, percent of applied radioactivity

Component	pH 4		pH 7		pH 9	
	0 days	5 days	0 days	5 days	0 days	5 days
Benzpyrimoxan	100.7	23.9	99.3	98.1	99.5	98.2
BP-aldehyde (DH-03)	-	4.6	-	-	-	-
BP-enaminealdehyde (DH-08)	-	37.0	-	-	-	-
4-TFMPM (DH-102)	-	20.0	-	-	-	0.5
Others[1)]	-	13.5	-	-	-	-
Total Recovery	100.7	99.1	99.3	98.1	99.5	98.7

Notes:

-: Not detected

[1)] Sum of minor unidentified components, each <5.8 percent AR

For the definitive test (Tier 2 test), hydrolysis of benzpyrimoxan separately labeled with ^{14}C in the phenyl and the pyrimidine rings was tested in buffer solutions at pH 4 at different temperatures (25 °C, 40 °C and 50 °C). Sampling times at 25 °C were 0, 9, 14, 17, 21, 24 and 30 days, at 40 °C were 0, 2, 6, 8, 10, 15 and 20 days, and at 50 °C were 0, 1, 2, 3, 4, 6 and 7 days. The results are shown in Tables 23 and 24.

At 25 °C, environmentally relevant temperature, the recovery during the study was >95 percent AR for all samples. After 30 days, 65.3 and 67.9 percent were identified as unchanged benzpyrimoxan in the [Phenyl-U-^{14}C] and [Pyrimidinyl-4(6)-^{14}C] label treated samples, respectively. The major hydrolysis product at pH 4 was benzpyrimoxan-enaminealdehyde (DH-08) with maximum amounts of 13.2 percent AR (24 DAT, average of both radiolabels). Two minor hydrolysis products were formed and identified, benzpyrimoxan-aldehyde (DH-03, representing a maximum 4.7 percent AR, 14 days, average of both radiolabels) and 4-TFMPM (DH-102, representing a maximum 8.6 percent AR, 30 days, ^{14}C-phenyl label). No other component represented greater than 5.4 percent AR (average of two replicates).

Table 23 Proportions of radioactive components in aqueous solutions at pH 4 at 25 °C after treatment of [Phenyl-U-^{14}C]-benzpyrimoxan, percent of applied radioactivity

Component	Sampling time (days)						
	0	9	14	17	21	24	30
Benzpyrimoxan	99.3	88.3	79.9	77.8	73.4	72.7	65.3
BP-aldehyde (DH-03)	-	3.4	4.6	3.9	3.4	3.6	3.3
BP-enaminealdehyde (DH-08)	-	3.3	8.4	8.3	10.4	13.2	12.6

Component	Sampling time (days)						
	0	9	14	17	21	24	30
4TFMPM (DH-102)	-	4.1	5.1	6.5	7.8	7.3	8.6
Unknown-01	-	3.6	3.9	3.0	3.6	3.7	3.4
Unknown-02	-	-	-	-	-	1.3	1.9
Unknown-03	-	-	-	1.1	-	1.5	1.5
Total Recovery	99.3	102.6	101.9	100.6	98.6	103.3	96.7

Note: -: Not detected

Table 24 Proportions of radioactive components in aqueous solutions at pH 4 at 25 °C after treatment of [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan, percent of applied radioactivity

Component	Sampling time (days)						
	0	9	14	17	21	24	30
Benzpyrimoxan	101.0	88.4	80.8	80.0	73.4	72.1	67.9
BP-aldehyde (DH-03)	-	4.5	4.8	4.7	4.7	4.9	3.5
BP-enaminealdehyde (DH-08)	-	4.0	8.1	8.3	10.3	13.2	12.8
Unknown-01	-	4.0	4.5	3.4	3.5	4.1	2.9
Unknown-02	-	-	-	-	-	1.4	-
Unknown-03	-	-	-	0.7	-	1.3	1.1
UP1[1)]	-	2.6	2.5	2.9	3.6	4.0	3.9
UP2[2)]	-	1.4	2.9	3.7	5.4	4.3	5.2
Total Recovery	101.0	104.8	103.5	103.7	100.8	105.3	97.3

Notes:

-: Not detected

[1)] includes DH-200 (NNI-1501-4-OH) and Pyr-Unk-01 (FW: 142, $C_5H_6N_2O_3$)

[2)] includes Pyr-Unk-02 (FW: 114, $C_4H_6N_2O_2$) and Pyr-Unk-03 (FW: 124, $C_5H_4N_2O_2$)

At 40 °C and 50 °C, the recovery during the study was >95 percent AR for all samples (data not shown). At the end of incubation period, benzpyrimoxan represented 16.7 and 14.5 percent AR, at 40 °C and 50 °C, respectively (average of both radiolabels). Three discrete hydrolytic products were formed and identified, namely DH-08 (representing a maximum 43.2 percent AR, 50 °C, 7 days, average of both radiolabels), DH-03 (representing a maximum 9.3 percent AR, 50 °C, 3 days, average of both radiolabels) and DH-102 (representing a maximum 30.7 percent AR, 40 °C, 20 days, ^{14}C-phenyl label). In the ^{14}C-pyrimidinyl label samples, two additional peaks (designated as UP1 and UP2) were detected, both representing a mixture of different compounds. The peak UP1 reached a maximum level of 8.2 percent AR (40 °C, 20 days). The peak UP1 consisted of benzpyrimoxan-4-OH (DH-200) and Pyr-Unk-01. The peak UP2 reached a maximum level of 20.8 percent AR (40 °C, 20 days). The peak UP2 consisted of Pyr-Unk-02 and Pyr-Unk-03.

The DT_{50} at pH 4 at 25 °C was 50.9 days (average of both radiolabels). Hydrolysis of benzpyrimoxan at pH 4 is highly dependent upon temperature (Table 25).

Table 25 Rate constants and DT_{50} values for the hydrolysis of benzpyrimoxan

Radiolabel	pH	Temperature (°C)	DT_{50} (days)
[Phenyl-U-^{14}C]	4	50	2.48
		40	7.56
		25	50.4
		20	97.3[1)]
	7	50	N/A[2)]
	9	50	N/A[2)]

Radiolabel	pH	Temperature (°C)	DT_{50} (days)
[Pyrimidinyl-4(6)-^{14}C]	4	50	2.53
		40	7.98
		25	51.4
		20	99.7[1)]

Notes:

[1)] The estimated values were calculated by using the rate constants at the other temperatures.

[2)] Less than 10 percent degradation was observed in the preliminary test (at 50 °C up to 5 days)

Photodegradation in buffer solutions

The photodegradation fate of [Phenyl-U-^{14}C] or [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan was studied in buffer solutions (Murata, 2017: E-37008). The test substances dissolved in buffer solution (pH 7), whose concentration was targeted as 2.0 mg/L (less than one half of its water solubility) were irradiated by artificial sunlight of a xenon arc lamp with filters blocking irradiation below 290 nm at 25.0 °C up to 25 days.

The test substances decreased to 84.6–85.0 percent AR after 25 days irradiation. No significant degradation was observed in the dark condition. The half-lives of the test substances were determined to be 121.6–154.4 days, which were estimated as 553.2–702.4 days (mean: 627.8 days) under natural sunlight in Tokyo spring.

In [Phenyl-U-^{14}C]-benzpyrimoxan samples, 4-TFMB (DH-101) and 4-TFMPM (DH-102) were identified, accounting for a maximum of 3.0 and 3.2 percent AR, respectively. In [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan samples, benzpyrimoxan-acid (DH-01) and benzpyrimoxan-4-OH (DH-200) were identified, accounting for a maximum of 0.4 and 6.0 percent AR, respectively. In both [Phenyl-U-^{14}C] and [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan samples, several minor degradates were detected, none of which accounted for greater than 10 percent AR through the study. In both [Phenyl-U-^{14}C] and [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan samples, organic volatile in the ethanolamine traps was accounted for 0.5–0.9 percent AR at the end of irradiation and was considered to be carbon dioxide.

Rotational crop studies

No rotational crop studies were submitted to the Meeting.

RESIDUE ANALYSIS

Analytical methods

Descriptions of analytical methods together with validation data for residues of benzpyrimoxan and its metabolites in plant and animal matrices were submitted to the Meeting. The methods rely on an initial extraction, usually with acetonitrile/water. After column clean-up, benzpyrimoxan and its metabolites are analysed by LC-MS/MS, at a LOQ of 0.01 mg/kg for both parent and metabolites.

Plant matrices (benzpyrimoxan and DH-04)

Method R-37002 was validated in husked rice, bran, polished rice, washing water and cooked rice. The pulverised rice grain, bran and straw samples were soaked with water. Except washing water, benzpyrimoxan and benzpyrimoxan-2-OH (DH-04) were sequentially extracted twice with acetonitrile/water (4/1) and twice with acetonitrile/0.1 mol/L HCl (4/1). The extract was cleaned-up by C_{18} cartridge, eluted with acetonitrile/water (3/2), and the eluate filtered prior to analysis by LC-MS/MS.

Washing water sample was cleaned-up by C_{18} cartridge, eluted with acetonitrile/water (3/2), the eluate filtered prior to analysis by LC-MS/MS.

In method A-37024, husked rice samples were hydrated with water and extracted with acetonitrile for 5 minutes in a grinder, followed by addition of a QuEChERs salt pouch containing magnesium sulphate, sodium chloride, sodium citrate dibasic sesquihydrate and sodium citrate tribasic dehydrate and extracting for 1 minute in a grinder. After centrifugation, aliquots were diluted into the calibration range with acetonitrile/water (1/1) and analysed by LC-MS/MS. The method was idepedently validated (Munford, 2020; A-35025).

Method validation data and MS/MS transitions for quantification and confirmation on plant matrices are summarized in Tables 26 (benzpyrimoxan) and 27 (benzpyrimoxan-2-OH, DH-04). Mean recoveries of benzpyrimoxan and DH-04 were whithin the range of 70–120 percent and RSD <20 percent. Linearity was obtained in the range of 0.03–2.0 µg/L per analyte using five calibration points, prepared in acetonitrile/water (3/2), and linear correlations were >0.99. LOQ was set at 0.01 mg/kg.

Table 26 Summary of validation data for benzpyrimoxan in rice matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Husked rice (MV)	341→159	0.01	5	89–94	91	2	R-37001
	Quantification	0.1	5	91–92	91	1	Nagata, 2016
	341→109	0.01	5	89–97	94	4	
	Confirmation	0.1	NA	NA	NA	NA	
Rice grain (MV)	341→159	0.01	6	82–97	88	6	R-37002
	Quantification	0.5	6	94–98	97	2	Morita, 2016
		5	6	91–96	94	2	
Husked rice (MV)	341→159	0.01	6	91–102	97	4	
	Quantification	0.5	6	86–91	90	2	
Rice straw (MV)	341→159	0.01	6	80–102	93	10	
	Quantification	0.5	6	85–90	88	3	
		10	6	90–98	94	3	
Husked rice (MV)	341→159	0.01	5	81–88	84	4	R-37021
	Quantification	0.10	5	89–93	91	2	Hamasaka, 2020
		0.50	5	96–98	97	1	
Bran (MV)	341→159	0.01	5	79–85	80	3	
	Quantification	0.10	5	90–93	90	2	
		3.50	5	82–87	86	2	
Polished rice (MV)	341→159	0.01	5	85–92	88	3	
	Quantification	0.10	5	93–100	96	3	
		0.20	5	94–96	95	1	
Washing water (MV)	341→159	0.01	5	93–97	95	2	
	Quantification	0.10	5	104–117	112	4	
Coocked rice (MV)	341→159	0.01	5	81–88	84	4	
	Quantification	0.10	5	90–97	94	4	
Husked rice (MV)	341→159	0.01	5	97–110	104	4.6	A-37024
	Quantification	0.10	5	85–98	93	5.8	Smith, 2020

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
	341→109	0.01	5	94–117	102	8.7	
	Confirmation	0.10	5	83–96	91	6.3	QuEChERS
Husked rice (ILV)	341→159	0.01	5	72–76	75	1.8	A-37025
	Quantification	0.10	5	70–73	72	1.5	Mumford, 2020
	341→109	0.01	5	68–73	72	2.7	
	Confirmation	0.10	5	69–72	70	1.4	QuEChERS

Notes:

MV: Method Validation, ILV: Independent Laboratory Validation, NA: Not Analysed

Table 27 Summary of validation data for DH-04 in rice matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Husked rice (MV)	357→159	0.01	5	92–104	98	4	R-37001
	Quantification	0.1	5	90–104	96	6	Nagata, 2016
	357→109	0.01	5	93–105	98	5	
	Confirmation	0.1	NA	NA	NA	NA	
Rice grain (MV)	357→159	0.01	6	89–101	94	5	R-37002
	Quantification	0.5	6	93–98	96	2	Morita, 2016
		1	6	95–99	98	2	
Husked rice (MV)	357→159	0.01	6	95–100	97	2	R-37004
	Quantification	0.5	6	100–103	101	1	Morita, 2017
Rice straw (MV)	357→159	0.01	6	101–115	106	5	
	Quantification	0.5	6	90–94	92	2	
		2	6	90–95	92	2	
		5	5	85–93	88	4	
Husked rice (MV)	357→159	0.01	5	96–105	99	4	R-37021
	Quantification	0.10	5	95–100	97	2	Hamasaka, 2020
		0.50	5	96–99	97	1	
Bran (MV)	357→159	0.01	5	77–89	84	5	
	Quantification	0.10	5	83–103	95	9	
		3.50	5	81–89	85	3	
Polished rice (MV)	357→159	0.01	5	87–89	88	1	
	Quantification	0.10	5	99–106	103	3	
		0.20	5	95–99	96	2	
Washing water (MV)	357→159	0.01	5	87–96	93	4	
	Quantification	0.10	5	102–113	109	4	
Coocked rice (MV)	357→159	0.01	5	86–97	92	5	
	Quantification	0.10	5	92–97	94	2	
Husked rice (MV)	357→159	0.01	5	89–110	98	7.3	A-37024
	Quantification	0.10	5	86–99	94	6.3	Smith, 2020
	357→299	0.01	5	94–103	99	3.6	
	Confirmation	0.10	5	86–97	92	5.3	QuEChERS

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Husked rice (ILV)	357→159	0.01	5	90–93	91	1.2	A-37025
	Quantification	0.10	5	90–95	92	2.0	Mumford, 2020
	357→299	0.01	5	85–92	89	3.0	
	Confirmation	0.10	5	89–91	90	1.1	QuEChERS

Notes:

MV: Method Validation, ILV: Independent Laboratory Validation, NA: Not Analysed

Animal matrices (benzpyrimoxan, DH-01, DH-02, DH-4, DH-05 and/or DH6)

Method R-37016 was validated in milk, cow muscle, liver, kidney and fat for benzpyrimoxan and metabolites benzpyrimoxan-acid (DH-01), benzpyrimoxan-CH_2OH (DH-02), benzpyrimoxan-2-OH (DH-04), benzpyrimoxan-acid-2-OH (DH-05) and benzpyrimoxan-CH_2OH-2-OH (DH-06). Milk and skim milk samples were weighed into a centrifuge tube, purified by PLS-2 SPE cartridge, eluted with acetonitrile, the eluate filtered and diluted prior to analysis for benzpyrimoxan, DH-04, and DH-05. Another aliquot of milk was treated with β-glucuronidase for 24 hours, cleaned up by PLS-2 SPE cartridge, eluted with acetonitrile, the eluate filtered and diluted prior to DH-06.analysis.

Tissue (muscle, liver and kidney) and milk cream samples were extracted twice with acetonitrile/water (1/1), an aliquot was cleaned up by C_{18} cartridge, eluted with acetonitrile, the eluate diluted and filtered prior to analysis for benzpyrimoxan, DH-01, DH-04, and DH-05. An additional aliquot was treated with β-glucuronidase, cleaned up by PLS-2 SPE cartridge, eluted with acetonitrile, the eluate filtered and diluted prior to analysis for DH-02 and DH-06.

Cow fat samples were extracted twice, first with hexane/acetone (4/1) and second with acetonitrile/water (1/1). The two phases were allowed to separate and the lower (acetonitrile/acetone) layer collected. The upper layer was extracted with acetonitrile and collected. The collected lower layers was adjusted, then an aliquot was cleaned up by C_{18} cartridge and eluted with acetonitrile. The eluate was then diluted and filtered prior to analysis for benzpyrimoxan, DH-01, DH-04, and DH-05. An additional aliquot was treated with β-glucuronidase, then cleaned up by PLS-2 SPE cartridge and eluted with acetonitrile. The eluate was filtered and diluted prior to analysis for DH-02 and DH-06. Analysis of all analytes was performed by LC-MS/MS, and the method validated at a LOQ of 0.01 mg/kg.

Method R-37017 was validated for egg and hen muscle and liver. Samples were extracted twice with acetonitrile/water (1/1). A small aliquot was purified by C_{18} cartridge, eluted with acetonitrile, the eluate was diluted and filtered prior to analysis for benzpyrimoxan, DH-02 and DH-05.

Hen fat samples were extracted twice, first with hexane/acetone (4/1) and second with acetonitrile/water (1/1). The extracts were combined in a separatory funnel, then collected the lower hydro-organic layer. The upper layer was extracted with acetonitrile and the acetonitrile combined with previously collected hydro-organic layer. A small aliquot was cleaned up by C_{18} cartridge, eluted with acetonitrile. The eluate was diluted and filtered prior to analysis for benzpyrimoxan, DH-02 and DH-05. Analysis of all analytes was performed by LC-MS/MS.

Validation data for methods R-37016 and R-37016 on animal matrices and mass transitions are summarized in Table 28 for benzpyrimoxan and Tables 29 to 32 for the metabolites.

Table 28 Method validation data for benzpyrimoxan in animal matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Milk	341→159	0.01	5	106–122	112	6.2	R-37016 VanMiddlesworth, 2018
	Quantification	0.1	5	101–113	105	4.5	
	341→109	0.01	5	104–120	111	7.7	
	Confirmation	0.1	5	101–108	106	2.8	
Cow muscle	341→159	0.01	5	88–99	93	4.6	
	Quantification	0.1	5	91–103	98	4.8	
	341→109	0.01	5	80–93	88	6.0	
	Confirmation	0.1	5	89–101	96	5.0	
Cow liver	341→159	0.01	5	83–93	88	4.1	
	Quantification	0.1	5	96–103	100	3.1	
	341→109	0.01	5	90–104	95	6.0	
	Confirmation	0.1	5	92–98	95	3.3	
Cow kidney	341→159	0.01	5	71–86	80	7.5	
	Quantification	0.1	5	73–87	81	7.1	
	341→109	0.01	5	69–82	76	7.0	
	Confirmation	0.1	5	76–85	81	4.0	
Cow fat	341→159	0.01	5	75–86	83	5.8	
	Quantification	0.1	5	80–89	86	4.3	
	341→109	0.01	5	76–85	82	4.1	
	Confirmation	0.1	5	83–89	86	2.7	
Egg	341→159	0.01	5	73–92	83	9.7	R-37017 VanMiddlesworth, 2018
	Quantification	0.1	5	80–118	94	15.3	
	341→109	0.01	5	76–98	87	12.0	
	Confirmation	0.1	5	79–119	94	15.8	
Hen muscle	341→159	0.01	5	86–108	100	9.2	
	Quantification	0.1	5	89–97	93	3.3	
	341→109	0.01	5	88–106	100	7.1	
	Confirmation	0.1	5	87–97	92	4.7	
Hen liver	341→159	0.01	5	84–119	97	14.2	
	Quantification	0.1	5	82–90	87	3.6	
	341→109	0.01	5	85–119	98	12.9	
	Confirmation	0.1	5	81–91	87	4.3	
Hen fat	341→159	0.01	5	91–113	101	8.6	
	Quantification	0.1	5	90–96	92	2.7	
	341→109	0.01	5	84–104	94	10.3	
	Confirmation	0.1	5	87–95	90	3.5	

Table 29 Validation data for DH-01 in animal matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Cow muscle	299→159	0.01	5	106–122	112	6.3	R-37016 VanMiddlesworth, 2018
	Quantification	0.1	5	100–112	108	4.6	
	299→109	0.01	5	86–119	100	12.9	
	Confirmation	0.1	5	100–113	107	5.1	
Cow liver *	299→159	0.01	5	61–69	65	5.9	
	Quantification			(89–101)	(93)	(5)	
		0.1	5	59–73	63	8.8	
				(112–117)	(114)	(2)	
	299→109	0.01	5	44–70	60	17.0	
	Confirmation			(70–127)	(89)	(28)	
		0.1	5	59–73	63	9.2	
				(109–113)	(112)	(2)	
Cow kidney	299→159	0.01	5	71–73	73	1.2	
	Quantification	0.1	5	103–111	108	2.9	
	299→109	0.01	5	76–85	81	4.1	
	Confirmation	0.1	5	103–113	109	3.5	
Cow fat	299→159	0.01	5	91–102	96	4.5	
	Quantification	0.1	5	85–99	93	6.9	
	299→109	0.01	5	91–103	97	4.7	
	Confirmation	0.1	5	89–104	98	6.3	

Notes:

* Upon fortification of DH-01 into liver, DH-05 can be measured in a quantitative conversion.

Total DH-01 were presented as DH-01 equivalent recoveries in parenthesis.

Table 30 Summary of validation data for DH-02 in animal matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Cow muscle	285→159	0.01	5	86–94	90	4.0	R-37016 VanMiddlesworth, 2018
	Quantification	0.1	5	94–100	97	3.0	
	285→109	0.01	5	87–103	94	6.9	
	Confirmation	0.1	5	89–99	96	4.4	
Cow liver	285→159	0.01	5	88–102	92	6.1	
	Quantification	0.1	5	88–97	92	4.0	
	285→109	0.01	5	91–113	104	8.1	
	Confirmation	0.1	5	88–95	92	3.0	
Cow kidney	285→159	0.01	5	92–101	97	4.0	
	Quantification	0.1	5	77–94	84	8.2	
	285→109	0.01	5	90–105	100	5.7	
	Confirmation	0.1	5	79–93	85	6.6	
Cow fat	285→159	0.01	5	95–107	101	5.1	
	Quantification	0.1	5	102–112	109	3.9	

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
	285→109	0.01	5	92–107	100	6.0	
	Confirmation	0.1	5	102–108	106	2.5	
Egg	285→159	0.01	5	86–107	97	8.4	R-37017 VanMiddlesworth, 2018
	Quantification	0.1	5	85–118	100	12.2	
	285→109	0.01	5	83–105	95	10.1	
	Confirmation	0.1	5	86–117	99	11.8	
Hen muscle	285→159	0.01	5	100–108	105	3.1	
	Quantification	0.1	5	98–112	106	5.1	
	285→109	0.01	5	101–111	106	3.4	
	Confirmation	0.1	5	100–112	106	4.5	
Hen liver	285→159	0.01	5	82–106	91	10.6	
	Quantification	0.1	5	80–89	86	4.1	
	285→109	0.01	5	80–104	90	10.0	
	Confirmation	0.1	5	80–86	85	3.1	
Hen fat	285→159	0.01	5	97–111	103	5.5	
	Quantification	0.1	5	98–106	103	2.9	
	285→109	0.01	5	97–109	104	4.6	
	Confirmation	0.1	5	99–109	104	3.6	

Table 31 Summary of method validation data for DH-04 in animal matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Milk	357→159	0.01	5	100–116	108	5.4	R-37016 VanMiddlesworth, 2018
	Quantification	0.1	5	96–106	102	3.9	
	357→109	0.01	5	103–119	109	6.1	
	Confirmation	0.1	5	97–107	104	4.1	
Cow muscle	357→159	0.01	5	87–99	92	5.8	
	Quantification	0.1	5	91–104	99	5.5	
	357→109	0.01	5	60–103	88	18.6	
	Confirmation	0.1	5	92–98	95	2.7	
Cow liver	357→159	0.01	5	91–95	93	1.7	
	Quantification	0.1	5	91–97	94	2.5	
	357→109	0.01	5	80–96	89	6.6	
	Confirmation	0.1	5	89–96	93	3.1	
Cow kidney	357→159	0.01	5	86–97	90	4.9	
	Quantification	0.1	5	79–89	85	4.5	
	357→109	0.01	5	74–98	87	12.6	
	Confirmation	0.1	5	77–91	84	7.0	
Cow fat	357→159	0.01	5	90–100	92	4.6	
	Quantification	0.1	5	91–98	95	3.3	
	357→109	0.01	5	87–91	89	2.1	
	Confirmation	0.1	5	89–99	94	4.4	

Table 32 Summary method validation data for DH-05 in animal matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Milk	315→159	0.01	5	81–99	89	7.7	R-37016 VanMiddlesworth, 2018
	Quantification	0.1	5	78–98	91	8.9	
	315→109	0.01	5	74–97	84	10.6	
	Confirmation	0.1	5	79–103	91	9.3	
Cow muscle	315→159	0.01	5	64–98	82	15.9	
	Quantification	0.1	5	89–102	98	5.3	
	315→109	0.01	5	91–109	100	6.6	
	Confirmation	0.1	5	97–108	102	4.0	
Cow liver	315→159	0.01	5	83–86	85	1.5	
	Quantification	0.1	5	95–100	98	2.1	
	315→109	0.01	5	80–115	97	15.8	
	Confirmation	0.1	5	91–106	97	5.6	
Cow kidney	315→159	0.01	5	67–90	80	13.5	
	Quantification	0.1	5	79–85	82	3.7	
	315→109	0.01	5	60–88	71	14.7	
	Confirmation	0.1	5	77–85	81	4.1	
Cow fat	315→159	0.01	5	87–98	95	4.8	
	Quantification	0.1	5	82–94	87	5.1	
	315→109	0.01	5	89–108	98	7.6	
	Confirmation	0.1	5	85–92	89	4.0	
Egg	315→159	0.01	5	50–71	64	13.2	R-37017 VanMiddlesworth, 2018
	Quantification	0.1	5	77–105	87	12.4	
	315→109	0.01	5	54–76	64	15.9	
	Confirmation	0.1	5	80–113	90	14.7	
Hen muscle	315→159	0.01	5	71–100	79	15.8	
	Quantification	0.1	5	82–88	86	2.9	
	315→109	0.01	5	71–94	77	12.6	
	Confirmation	0.1	5	81–89	85	3.4	
Hen liver	315→159	0.01	5	72–77	74	3.4	
	Quantification	0.1	5	70–82	77	5.9	
	315→109	0.01	5	67–82	75	7.8	
	Confirmation	0.1	5	70–77	73	4.1	
Hen fat	315→159	0.01	5	56–74	65	10.8	
	Quantification	0.1	5	77–84	82	3.7	
	315→109	0.01	5	57–85	67	16.6	
	Confirmation	0.1	5	77–83	80	3.2	

Table 33 Summary of validation data for DH-06 in animal matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Milk	301→159	0.01	5	95–120	113	8.9	R-37016 VanMiddlesworth, 2018
	Quantification	0.1	5	102–112	106	4.1	
	301→109	0.01	5	59–102	87	19.8	
	Confirmation	0.1	5	97–116	107	7.9	
Cow muscle	301→159	0.01	5	87–115	98	11.4	
	Quantification	0.1	5	96–106	101	3.8	
	301→109	0.01	5	79–101	88	10.5	
	Confirmation	0.1	5	98–106	103	2.9	
Cow liver	301→159	0.01	5	92–119	109	10.5	
	Quantification	0.1	5	98–105	101	3.1	
	301→109	0.01	5	68–111	96	17.2	
	Confirmation	0.1	5	99–106	103	2.5	
Cow kidney	301→159	0.01	5	82–118	100	13.3	
	Quantification	0.1	5	81–95	89	7.7	
	301→109	0.01	5	88–110	100	8.5	
	Confirmation	0.1	5	80–94	87	6.1	
Cow fat	301→159	0.01	5	88–109	98	8.0	
	Quantification	0.1	5	96–101	98	1.9	
	301→109	0.01	5	88–106	101	7.5	
	Confirmation	0.1	5	94–101	98	3.0	

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

The Meeting received data on the storage stability of benzpyrimoxan and its metabolites in samples for plant and animal commodities stored frozen.

Plant matrices

The storage stability of benzpyrimoxan and metabolite benzpyrimoxan-2-OH (DH-04) in rice grain, husked rice and rice straw was tested (Morita, 2016, 2017, 2018: R-37002, R-37004, R-37009). Homogenised samples were fortified with benzpyrimoxan and DH-04 in acetonitrile at 0.5 mg/kg, and the samples were stored in at -20 ± 6 °C. Duplicate samples were analysed at each sampling time. Benzpyrimoxan and DH-04 were analysed using method in rice residue studies by LC-MS/MS (LOQ of 0.01 mg/kg). The results are shown in Tables 34 and 35. Benzpyrimoxan and DH-04 were found to be stable upon frozen storage in rice commodities for at least 2.5 months (rice grain: 77 days, husked rice: 75 days, straw: 82 days).

Table 34 Recovery of benzpyrimoxan from stored fortified samples (0.5 mg/kg) of rice matrices

Storage interval	Procedural recovery	percent remaining	Mean	Reference
Rice grain				R-37002 Morita, 2016
66 days	90	91, 93	92	
77 days		91, 93	92	
Husked rice				
64 days	98	91, 96	94	

Storage interval	Procedural recovery	percent remaining	Mean	Reference
75 days		92, 92	92	
Rice straw				
67 days	86	91, 92	92	
78 days		92, 94	93	
Rice grain				R-37004
53 days	88	87, 90	88	Morita, 2017
Husked rice				
39 days	105	86, 91	88	
40 days	90	88, 92	90	
53 days	NA	90, 91	90	
75 days	NA	89, 94	92	
Rice straw				
46 days	90	93, 94	94	
47 days		89, 91	90	
60 days		88, 90	89	
82 days		85, 87	86	
Rice grain				R-37009
33 days	89	93, 95	94	Morita, 2018
62 days		85, 90	88	
Husked rice				
32 days	90	85, 90	88	
61 days		87, 89	88	
Rice straw				
34 days	99	97, 99	98	
63 days		93, 95	94	

Table 35 Recovery of benzpyrimoxan-2-OH (DH-04) from stored fortified rice samples at 0.5 mg/kg

Storage interval	Procedural recovery	percent remaining	Mean	Reference
Rice grain				R-37002
66 days	99	96, 99	98	Morita, 2016
77 days		98, 99	98	
Husked rice				
64 days	103	99, 99	99	
75 days		100, 102	101	
Rice straw				
67 days	84	89, 94	92	
78 days		91, 92	92	
Rice grain				R-37004
53 days	102	79, 93	86	Morita, 2017
Husked rice				
39 days	103	102, 115	108	
40 days		99, 101	100	

Storage interval	Procedural recovery	percent remaining	Mean	Reference
53 days		96, 96	96	
75 days		97, 100	98	
Rice straw				
46 days	97	85, 89	87	
47 days		82, 91	86	
60 days		88, 93	90	
82 days		89, 91	90	
Rice grain				R-37009 Morita, 2018
33 days	100	96, 97	96	
62 days		92, 92	92	
Husked rice				
32 days	101	94, 95	94	
61 days		94, 102	98	
Rice straw				
34 days	101	99, 100	100	
63 days		95, 97	96	

Animal matrices

The storage stability of benzpyrimoxan and metabolites benzpyrimoxan-acid (DH-01), benzpyrimoxan-CH_2OH (DH-02), benzpyrimoxan-2-OH (DH-04), benzpyrimoxan-acid-2-OH (DH-05) and benzpyrimoxan-CH_2OH-2-OH (DH-06) in cow milk and tissues was tested in feeding study (VanMiddlesworth, 2018: R-37016). Control samples of milk and tissues were fortified with benzpyrimoxan and metabolites at 0.1 mg/kg and stored under freezer conditions for at least as long as the longest storage interval (harvest to extraction) from the feeding study samples. The aged samples were extracted alongside freshly fortified control matrix samples. The conversion of DH-01 to DH-05 in aged liver, kidney and fat samples is measured as DH-05 and reported as DH-01 equivalent using the molecular weight ratio of DH-01 to DH-05 to calculate the equivalent residue of DH-01 in mg/kg and percent recovery. Benzpyrimoxan and metabolites were analysed using method in cow feeding study by LC-MS/MS (LOQ of 0.01 mg/kg). The results are shown in Tables 36 to 40.

Table 36 Recovery of benzpyrimoxan, DH-04, DH-05 and DH-06 from stored fortified samples of cow milk at 0.1 mg/kg (R-37016; VanMiddlesworth, 2018)

Interval, days	percent recovery*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining
	benzpyrimoxan		DH-04		DH-05		DH-06	
0	-	103, 104	-	108, 110	-	95, 95	-	109, 112
33	82,88	92, 92	86, 90	88, 92	82, 89	98, 104	91, 91	85, 86
98	86, 87	89, 95	89, 89	86, 89	88, 94	65, 66	88, 91	80, 83

Note: * Procedural recovery.

Table 37 Recovery of benzpyrimoxan, DH-01, DH-02, DH-04, DH-05 and DH-06 from stored fortified samples of cow muscle at 0.1 mg/kg (R-37016; VanMiddlesworth, 2018

Interval, days	percent recovery*	percent remaining	percent recovery*	percent remaining	percent reco-very*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining
	Benzpyrimoxan		DH-01		DH-02		DH-04		DH-05		DH-06	
0		97, 101		97, 98		94, 94		97, 98		98, 100		97, 97
30	83, 86	79, 80	89, 92	83, 86	86, 89	82, 86	90, 93	86, 94	91, 98	97, 102	86, 89	83, 86
90	80, 84	80, 80	88, 92	87, 88	98, 101	104, 107	92, 97	65, 92	88, 89	68, 70	98, 100	93, 99

Note: * Procedural recovery.

Table 38 Recovery of benzpyrimoxan, DH-01, DH-02, DH-04, DH-05 and DH-06 from stored fortified samples of cow liver at 0.1 mg/kg (R-37016 VanMiddlesworth, 2018)

Interval, days	percent recovery*	percent remaining	percent recovery*	percent remaining	percent recvery*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining
	Benzpyrimoxan		DH-01**		DH-02		DH-04		DH-05		DH-06	
0		95, 96		73, 77		93, 97		103, 107		99, 100		99, 103
32	81, 86	84, 88	42, 43	3, 7	88, 89	75, 80	85, 88	83, 89	100, 104	108, 112	91, 92	83, 86
85	85, 86	85, 93	57, 59	3, 9	90, 91	72, 78	88, 90	82, 90	85, 89	62, 66	97, 97	92, 93
0**				112, 113								
32**			119, 125	133, 137								
85**			91, 92	77, 81								

Notes:

* Procedural recovery.

** DH-01 was found to degrade quantitatively to DH-05 and was presented as DH-01 equivalent recoveries in parenthesis. The DH-05 residue can be converted to DH-01 equivalent residue through the ratio of the DH-01 to DH-05 molecular weights. The resulting residue can be summed with the measured DH-01 residue to give a DH-01 equivalent percent recovery.

Table 39 Recovery of benzpyrimoxan, DH-01, DH-02, DH-04, DH-05 and DH-06 from stored fortified samples of cow kidney at 0.1 mg/kg (R-37016 VanMiddlesworth, 2018)

Interval, days	percent recovery*	percent remaining	percent recovery*	percent remaining	percent reco-very*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining
	Benzpyrimoxan		DH-01**		DH-02		DH-04		DH-05		DH-06	
0		82, 85		94, 95		93, 97		98, 103		90, 91		97, 100
30	79, 83	65, 75	79, 82	0, 0	95, 95	76, 86	73, 75	59, 78	88, 88	83, 83	99, 99	83, 83
72	91, 91	64, 68	76, 82	0, 2	81, 93	68, 69	89, 92	77, 82	74, 78	37, 41	85, 90	62, 70
32**			-	102, 123								
85**			-	68, 69								

Notes:

* Procedural recovery.

** DH-01 was found to degrade quantitatively to DH-05 and were presented as DH-01 equivalent recoveries in parenthesis. The DH-05 residue can be converted to DH-01 equivalent residue through the ratio of the DH-01 to DH-05 molecular weights. The resulting residue can be summed with the measured DH-01 residue to give a DH-01 equivalent percent recovery.

Table 40 Recovery of benzpyrimoxan, DH-01, DH-02, DH-04, DH-05 and DH-06 from stored fortified samples of cow fat at 0.1 mg/kg (R-37016 VanMiddlesworth, 2018)

Interval, days	percent recovery*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining	percent recovery*	percent remaining
	Benzpyrimoxan		DH-01*		DH-02		DH-04		DH-05		DH-06	
0		82, 84		87, 89		93, 95		94, 97		103, 104		94, 97
33	68, 68	63, 65	62, 65	32, 41	70, 73	70, 72	76, 78	68, 73	91, 95	79, 80	70, 75	71, 71
77	77, 77	76, 86	73, 74	40, 45	76, 80	95, 115	87, 89	80, 94	96, 103	58, 72	80, 89	83, 86
33**			-	86, 96								
77**			-	87, 87								

Notes:

* Procedural recovery

** DH-01 was found to degrade quantitatively to DH-05 and were presented as DH-01 equivalent recoveries in parenthesis. The DH-05 residue can be converted to DH-01 equivalent residue through the ratio of the DH-01 to DH-05 molecular weights. The resulting residue can be summed with the measured DH-01 residue to give a DH-01 equivalent percent recovery.

The storage stability of benzpyrimoxan and metabolites benzpyrimoxan-CH_2OH (DH-02) and benzpyrimoxan-acid-2-OH (DH-05) in hen eggs was tested in feeding study (VanMiddlesworth, 2018: R-37017). Egg control samples were fortified with benzpyrimoxan and metabolites at 0.1 mg/kg and stored under freezer conditions for at least as long as the longest storage interval (harvest to extraction) from the feeding study samples. After the appropriate storage interval the aged samples are extracted alongside freshly fortified control matrix samples. Recoveries of both aged and freshly fortified samples are determined. Benzpyrimoxan, DH-02 and DH-05 were extracted from eggs with acetonitrile/water (1/1). Extracts were cleaned up on a C_{18} SPE cartridge, filtered and analysed by LC-MS/MS (LOQ of 0.01 mg/kg. The results are shown in Table 41

Table 41 Benzpyrimoxan, DH-02 and DH-05 from stored fortified hen egg samples at 0.1 mg/kg (R-37017 VanMiddlesworth, 2018)

Interval, days	percent procedural recovery	percent remaining	percent procedural recovery	percent remaining	percent procedural recovery	percent remaining
	benzpyrimoxan		DH-02		DH-05	
0	-	73, 77	-	77, 81	-	80, 85
37	80, 93	68, 69	87, 99	67, 71	97, 98	103, 103

USE PATTERN

Benzpyrimoxan is an insecticide having biological activity to rice plant hoppers (Hemiptera: Delphacidae). The Meeting received label for use in rice in Japan (Table 42).

Table 42 Registered use of benzpyrimoxan in rice by foliar spray in Japan

Crop	Formulation		Application					PHI, days
	Type	Conc. of ai	Method	Rate kg ai/hL	Rate Water L/ha	No. max	Interval, days	
Rice	SC	10 percent	Foliar	0.01	600-1500	3	Not indicated	7 days

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on benzpyrimoxan supervised field trials conducted in Japan using SC formulation on paddy rice and rice straw.

Conditions of the supervised residue trials were generally well reported. Most field reports provided data on the applicators used, plot size, field sample size and sampling date.

Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Date of analyses and duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except when residues were found in samples from control plots. Residue data are not corrected for percent recovery.

Each of the field trial sites generally consisted of untreated control plot and treated plot. Application rates and residue concentrations have generally been rounded to two significant figures. Residue values from the trials, which have been used for the estimation of maximum residue levels, STMRs and HRs, are underlined.

The residue concentrations are reported for benzpyrimoxan and benzpyrimoxan-2-OH (DH-04), expressed as parent compound equivalents (conversion factor = 0.955).

Total residues for estimation of maximum residues levels and STMRs are calculated by summing up the concentrations of benzpyrimoxan and DH-04, expressed as benzpyrimoxan, as illustrated below.

Benzpyrimoxan	DH-04	Total
<0.01	<0.01	<0.02 (0.01 + 0.01)
<0.01	0.025	0.035 (0.01+0.025)
0.019	0.058	0.077 (0.019+0.058)

Rice cereals

The Meeting received 8 decline trials on paddy conducted in Japan (Morita, 2016, 2017, 2018: R-37002, R-37004, R-37009). Applications were made between booting/heading and harvest with treatment intervals of 6–14 days. Mature rice plants were harvested at 7 days after last application (DALA) as well as 14 and 21 DALA and dried naturally for 3–7 days on a drying pole protected from rain. Rice grain (paddy rice) was collected at threshing. Samples of husked rice and hulls were collected upon husking. The samples were then transported under cooled conditions to the analytical laboratory within 1–2 days.

Samples were analysed for residues of benzpyrimoxan and benzpyrimoxan-2-OH (DH-04) following analytical method in study R-37002. The LOQs for both benzpyrimoxan and DH-04 were 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for benzpyrimoxan and DH-04 in

matrices were within 70–120 percent. Samples were stored deep frozen until extraction for a maximum of 61 days for rice grain and 56 days for husked rice. The results are shown in Table 43.

Table 43 Residues of benzpyrimoxan and DH-04 (expressed in parent equivalents) on rice from supervised trials in Japan using 3 applications of SC formulation

Year Location (variety)	Application			DALA, days	Commodity	Residues, mg/kg			Ref
	kg ai/hL	L/ha	Growth Stage[1)]			Parent	DH-04	Total[2)]	
2015 Fukui (Hanaechizen)	0.01 0.01 0.01	2000 2000 2000	Milk-ripe Dough-ripe Yellow-ripe (7, 7 days)	7	Grain	2.0	0.63	2.6 [3.9]	R-37002 Recovery for benzpyrimoxan: 90, 94 percent for grain and 85, 98 percent for husked rice at 0.1 mg/kg Recovery for DH-04: 99, 99 percent for grain and 90, 103 percent for husked rice at 0.1 mg/kg Sample arrival to analysis: 32-61 days for grain and 36-56 days for husked rice
					Husked rice	0.44	0.15	0.59 [0.89]	
			Full heading Milk-ripe Dough-ripe (7, 7 days)	14	Grain	1.7	0.57	2.2	
					Husked rice	0.36	0.15	0.51	
			Heading Full heading Milk-ripe (7, 7 days)	21	Grain	0.70	0.33	1.0	
					Husked rice	0.18	0.10	0.28	
2015 Kochi (Koshihikari)	0.01 0.01 0.01	2010 2010 2010	Milk-ripe Yellow-ripe Yellow-ripe (14, 7 days)	7	Grain	3.4	0.74	4.1 [5.6]	
					Husked rice	0.46	0.11	0.57 [0.79]	
			Milk-ripe Dough-ripe Yellow-ripe (8, 6 days)	14	Grain	1.8	0.42	2.2	
					Husked rice	0.32	0.10	0.42	
			Boot Milk-ripe Dough-ripe (7, 8 days)	21	Grain	0.68	0.19	0.87	
					Husked rice	0.15	0.07	0.22	
2016 Iwate (Hitomebore)	0.01 0.01 0.01	2000 2000 2000	Milk-ripe Dough-ripe Yellow-ripe (13, 8 days)	7	Husked rice	0.10	0.03	0.13 [0.19]	R-37004 Recovery for benzpyrimoxan: 88, 90 percent for grain and 90, 105 percent for husked rice at 0.1 mg/kg Recovery for DH-04: 98, 102 percent for grain and 101, 103 percent for husked rice at 0.1 mg/kg Sample arrival to analysis: 36 days for grain and 20-56 days for husked rice
			Milk-ripe End milk-ripe Dough-ripe (7, 6 days)	14	Husked rice	0.08	0.03	0.11	
			Early ripening Milk-ripe End milk-ripe (7, 7 days)	21	Husked rice	0.10	0.04	0.14	
2016 Niigata (Koshihikari BL)	0.01 0.01 0.01	2000 2000 2000	Milk-ripe Early hardening Just before harvest (14, 7 days)	7	Husked rice	0.24	0.10	0.34	
			Milk-ripe Dough-ripe Early hardening (7, 7 days)	14	Husked rice	0.32	0.12	0.44	
			Full heading Milk-ripe Dough-ripe (7, 7 days)	21	Husked rice	0.32	0.14	0.46 [0.74]	

Year Location (variety)	Application			DALA, days	Commodity	Residues, mg/kg			Ref
	kg ai/hL	L/ha	Growth Stage[1)]			Parent	DH-04	Total[2)]	
2016 Fukui (Hanaechizen)	0.01 0.01 0.01	2000 2000 2000	Milk-ripe Dough-ripe Yellow-ripe (7, 7 days)	7	Husked rice	0.26	0.10	0.36	
			Early of ear including Milk-ripe Dough-ripe (7, 7 days)	14	Husked rice	0.30	0.14	0.44 [0.72]	
			Heading Early of ear including Milk-ripe (7, 7 days)	21	Husked rice	0.26	0.13	0.39	
2016 Ibaraki (Koshihikari)	0.01 0.01 0.01	2100 2100 2100	Milk-ripe Ripening Ripening (14, 7 days)	7	Grain	1.6	0.40	2.0 [2.8]	
					Husked rice	0.18	0.07	0.25	
			Milk-ripe Dough-ripe Ripening (7, 7 days)	14	Grain	1.1	0.31	1.4	
					Husked rice	0.20	0.07	0.27 [0.41]	
			Early of rice ear hang down stage Milk-ripe Dough-ripe (7, 7 days)	21	Grain	0.46	0.21	0.67	
					Husked rice	0.11	0.06	0.17	
2017 Ibaraki (Koshihikari)	0.01 0.01 0.01	2030 2030 2030	Milk-ripe Dough-ripe Ripening (14, 7 days)	7	Grain	1.7	0.42	2.1	R-37009 Recovery for benzpyrimoxan: 89, 93 percent for grain and 89, 90 percent for husked rice at 0.1 mg/kg Recovery for DH-04: 97, 100 percent for grain and 100, 101 percent for husked rice at 0.1 mg/kg Sample arrival to analysis: 12-41 days for grain 4-33 days for husked rice
					Husked rice	0.17	0.08	0.25	
			Milk-ripe Dough-ripe Dough-ripe (8, 6 days)	14	Grain	1.9	0.59	2.5 [3.7]	
					Husked rice	0.33	0.14	0.47 [0.75]	
			Early of rice ear hang down Milk-ripe Dough-ripe (6, 8 days)	21	Grain	1.1	0.44	1.6	
					Husked rice	0.27	0.12	0.39	
	0.05 0.05 0.05	250 250 250	Milk-ripe Dough-ripe Ripening (14, 7 days)	7	Grain	1.5	0.43	1.9	
					Husked rice	0.11	0.06	0.17	
			Milk-ripe Dough-ripe Dough-ripe (8, 6 days)	14	Grain	1.7	0.57	2.3	
					Husked rice	0.16	0.08	0.24	
			Early of rice ear hang down Milk-ripe Dough-ripe (6, 8 days)	21	Grain	1.0	0.41	1.4	
					Husked rice	0.13	0.07	0.20	
2017	0.01	2000	Milk-ripe	7	Grain	1.2	0.15	1.4	

Year Location (variety)	Application			DALA, days	Commodity	Residues, mg/kg			Ref
	kg ai/hL	L/ha	Growth Stage[1)]			Parent	DH-04	Total[2)]	
Miyazaki (Hinohikari)	0.01 0.01	2000 2000	Dough-ripe Yellow-ripe (14, 8 days)					[1.7]	
					Husked rice	0.06	0.02	0.08	
			Milk-ripe Milk-ripe Dough-ripe (7, 7 days)	14	Grain	0.36	0.12	0.48	
					Husked rice	0.06	0.03	0.09 [0.15]	
			Heading Milk-ripe Milk-ripe (7, 7 days)	21	Grain	0.17	0.11	0.28	
					Husked rice	0.04	0.03	0.07	
	0.05 0.05 0.05	258 258 258	Milk-ripe Dough-ripe Yellow-ripe (14, 8 days)	7	Grain	1.4	0.11	1.6	
					Husked rice	0.05	0.02	0.07	
			Milk-ripe Milk-ripe Dough-ripe (7, 7 days)	14	Grain	0.19	0.08	0.27	
					Husked rice	0.04	0.02	0.06	
			Heading Milk-ripe Milk-ripe (7, 7 days)	21	Grain	0.09	0.06	0.15	
					Husked rice	0.02	0.01	0.03	

Notes:

[1)] Re-treatment interval is given in parenthesis.

[2)] [Total residue = parent + 3 × DH-04 expressed as parent]: the toxicity of DH-04 is three times higher than that of parent.

Rice straw

The Meeting received 8 decline trials on paddy rice conducted in Japan (Morita, 2016, 2017, 2018: R-37002, R-37004, R-37009). Applications were made between booting/heading and harvest with treatment intervals of 6–14 days. Mature rice plants were harvested at 7 DALA as well as 14 and 21 DALA and dried naturally for 3–7 days on a drying pole in a facility to prevent rain. Straw was collected at threshing. The samples were then transported under cooled conditions to the analytical laboratory within 1–2 days.

Samples were analysed for residues of benzpyrimoxan and benzpyrimoxan-2-OH (DH-04) following analytical method R-37002. The LOQs for both benzpyrimoxan and DH-04 were 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for benzpyrimoxan and DH-04 in matrices were within 70–120 percent. Samples were stored deep frozen until extraction for a maximum of 63 days. The results are shown in Table 44.

Table 44 Residues of benzpyrimoxan and DH-04 (expressed in parent equivalents) on rice straw from supervised trials in Japan using 3 foliar applications of SC formulation

Year Location (variety)	Application			DALA Days	Residues, mg/kg			Ref
	kg ai/hL	L/ha	Growth Stage[1)]		Parent	DH-04	Total	
2015 Fukui (Hanaechizen)	0.01 0.01 0.01	2000 2000 2000	Milk-ripe Dough-ripe Yellow-ripe	7	4.7	1.5	6.2	R-37002 Recovery for

Year Location (variety)	Application			DALA Days	Residues, mg/kg			Ref
	kg ai/hL	L/ha	Growth Stage[1)]		Parent	DH- 04	Total	
			(7, 7 days)					benzpyrimoxan: 86, 86 percent at 0.1 mg/kg Recovery for DH-04: 84, 86 percent at 0.1 mg/kg Sample arrival to analysis: 32-61 days
			Full heading Milk-ripe Dough-ripe (7, 7 days)	14	2.8	0.90	3.7	
			Heading Full heading Milk-ripe (7, 7 days)	21	0.57	0.44	1.0	
2015 Kochi (Koshihikari)	0.01 0.01 0.01	2010 2010 2010	Milk-ripe Yellow-ripe Yellow-ripe (14, 7 days)	7	7.2	1.7	9.0	
			Milk-ripe Dough-ripe Yellow-ripe (8, 6 days)	14	2.6	0.86	3.5	
			Boot Milk-ripe Dough-ripe (7, 8 days)	21	0.28	0.18	0.46	
2016 Iwate (Hitomebore)	0.01 0.01 0.01	2000 2000 2000	Milk-ripe Dough-ripe Yellow-ripe (13, 8 days)	7	4.2	1.3	5.5	R-37004 Recovery for benzpyrimoxan: 83, 90 percent at 0.1 mg/kg Recovery for DH-04: 94, 97 percent at 0.1 mg/kg Sample arrival to analysis: 27-63 days
			Milk-ripe End milk-ripe Dough-ripe (7, 6 days)	14	3.1	1.1	4.2	
			Early ripening Milk-ripe End milk-ripe (7, 7 days)	21	2.2	0.93	3.1	
2016 Niigata (Koshihikari BL)	0.01 0.01 0.01	2000 2000 2000	Milk-ripe Early hardening Just before harvest (14, 7 days)	7	8.1	2.5	11	
			Milk-ripe Dough-ripe Early hardening (7, 7 days)	14	2.7	1.7	4.4	
			Full heading Milk-ripe Dough-ripe (7, 7 days)	21	2.5	1.6	4.1	
2016	0.01	2000	Milk-ripe	7	7.8	1.9	9.7	

Year Location (variety)	Application			DALA Days	Residues, mg/kg			Ref
	kg ai/hL	L/ha	Growth Stage[1)]		Parent	DH- 04	Total	
Fukui (Hanaechizen)	0.01 0.01	2000 2000	Dough-ripe Yellow-ripe (7, 7 days)					
			Early of ear including Milk-ripe Dough-ripe (7, 7 days)	14	3.8	1.3	5.1	
			Heading Early of ear including Milk-ripe (7, 7 days)	21	1.4	0.78	2.2	
2016 Ibaraki (Koshihikari)	0.01 0.01 0.01	2100 2100 2100	Milk-ripe Ripening Ripening (14, 7 days)	7	5.6	1.6	7.2	
			Milk-ripe Dough-ripe Ripening (7, 7 days)	14	2.2	0.86	3.1	
			Early of rice ear hang down stage Milk-ripe Dough-ripe (7, 7 days)	21	0.59	0.29	0.88	
2017 Ibaraki (Koshihikari)	0.01 0.01 0.01	2030 2030 2030	Milk-ripe Dough-ripe Ripening (14, 7 days)	7	5.8	0.99	6.8	R-37009 Recovery for benzpyrimoxan : 85, 99 percent at 0.1 mg/kg Recovery for DH-04: 95, 101 percent at 0.1 mg/kg Sample arrival to analysis: 18-47 days
			Milk-ripe Dough-ripe Dough-ripe (8, 6 days)	14	4.7	1.0	5.7	
			Early of rice ear hang down Milk-ripe Dough-ripe (6, 8 days)	21	1.8	0.71	2.6	
	0.05 0.05 0.05	250 250 250	Milk-ripe Dough-ripe Ripening (14, 7 days)	7	3.5	0.55	4.0	
			Milk-ripe Dough-ripe Dough-ripe (8, 6 days)	14	2.5	0.63	3.1	
			Early of rice ear hang down Milk-ripe	21	0.68	0.28	0.96	

Year Location (variety)	Application kg ai/hL	L/ha	Growth Stage[1)]	DALA Days	Residues, mg/kg Parent	DH-04	Total	Ref
			Dough-ripe (6, 8 days)					
2017 Miyazaki (Hinohikari)	0.01 0.01 0.010	2000 2000 2000	Milk-ripe Dough-ripe Yellow-ripe (14, 8 days)	7	9.0	1.3	10	
			Milk-ripe Milk-ripe Dough-ripe (7, 7 days)	14	1.2	0.52	1.7	
			Heading Milk-ripe Milk-ripe (7, 7 days)	21	0.48	0.27	0.75	
	0.05 0.05 0.05	258 258 258	Milk-ripe Dough-ripe Yellow-ripe (14, 8 days)	7	6.7	0.75	7.5	
			Milk-ripe Milk-ripe Dough-ripe (7, 7 days)	14	0.68	0.26	0.94	
			Heading Milk-ripe Milk-ripe (7, 7 days)	21	0.46	0.18	0.64	

Note:

[1)] Re-treatment interval is given in parenthesis.

FATE OF RESIDUES IN STORAGE AND PROCESSING

The Meeting received information on high temperature hydrolysis of benzpyrimoxan and the fate of benzpyrimoxan residues during the processing of rice.

High temperature hydrolysis

The hydrolysis of [^{14}C]-benzpyrimoxan was studied in sterile buffered solutions of pH 4, 5 and 6 (Ihara, 2017: E-37004). [Phenyl-U-^{14}C] and [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan were incubated in aqueous buffered solutions at a nominal concentrations of 1.0 mg/L at three pH values. The conditions of incubation were as follows: pH 4 ± 0.1 at 90 ± 5 °C for 20 minutes, representing pasteurization; pH 5 ± 0.1 at 100 ± 5 °C for 60 minutes, representing brewing, baking and boiling; pH 6 ± 0.1 at 120 ± 5 °C for 20 minutes, representing sterilization.

At the end of the incubation periods, the solutions were measured for their total radioactivity by LSC for the recovery (mass valance) and analysed by normal phase TLC-RLG (radioluminography) to determine the proportions of benzpyrimoxan and any radiolabelled breakdown products. To confirm

identification of radioactive components, radioactivity in the samples were chromatographed on HPLC equipped with a fraction collector.

The total radioactivity recovery (based on the applied radioactivity percent AR) was 99.0–100.7 percent AR, 104.4–105.7 percent AR, and 96.0–99.6 percent AR for the pH 4, pH 5, and pH 6 hydrolysis samples, respectively (Table 45).

Table 45 Recoveries of radioactivity from processed buffered solutions

Conditions	Recovery of Applied Radioactivity [1.0 mg/L]	
	[Phenyl-U-^{14}C]	[Pyrimidinyl-4(6)-^{14}C]
pH 4, 90°C, 20min	100.7	99.0
pH 5, 100°C, 60 min	104.4	105.7
pH 6, 120°C, 20 min	99.6	96.0

The predominant residue was unchanged benzpyrimoxan. Benzpyrimoxan-aldehyde (DH-03), benzpyrimoxan-enamine-aldehyde (DH-08) and benzpyrimoxan-benzyl alcohol (DH-102) were identified with up to 7.4 percent TRR. The summary of the results are shown in Table 46

Table 46 Proportions of radioactive components in processed buffer solutions

	pH 4, 90 °C, 20min		pH 5, 100 °C, 60 min		pH 6, 120 °C, 20 min	
	mg eq/L	Percent TRR	mg eq/L	Percent TRR	mg eq/L	Percent TRR
[Phenyl-U-^{14}C]-benzpyrimoxan						
Benzpyrimoxan	0.96	95.2	1.01	96.4	0.99	99.8
Benzpyrimoxan-aldehyde (DH-03)	0.04	4.1	0.03	3.2	<0.01	0.2
Benzpyrimoxan-enamine-aldehyde (DH-08)	<0.01	0.3	<0.01	0.1	ND	ND
Benzpyrimoxan-benzyl alcohol (DH-102)	<0.01	0.4	<0.01	0.3	ND	ND
Unknown	ND	ND	ND	ND	ND	ND
Total	1.01	100	1.04	100	1.00	100
[Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan						
Benzpyrimoxan	0.94	95.0	0.98	92.4	0.96	99.5
Benzpyrimoxan-aldehyde (DH-03)	0.04	4.4	0.08	7.4	0.01	0.5
Benzpyrimoxan-enamine-aldehyde (DH-08)	<0.01	0.2	<0.01	0.2	ND	ND
Unknown	<0.01	0.3	ND	ND	ND	ND
Total	0.99	100	1.06	100	0.96	100

Notes:

ND: Not detected.

Rice processing

Two brown rice (husked rice) samples obtained from field trials in Ibaraki and Niigata (R-37004 and R-37009) were used as treated raw agricultural commodity (Hamasaka, 2020: R-37021). Since residues of benzpyrimoxan and benzpyrimoxan-2-OH (DH-04) in these samples were determined to be greater >LOQ of 0.01 mg/kg (10 times higher), samples were considered suitable for determination of rice processing factors.

Samples were processed through polishing and cooking. A schematic chart of processing procedure is illustrated in Figure 5.

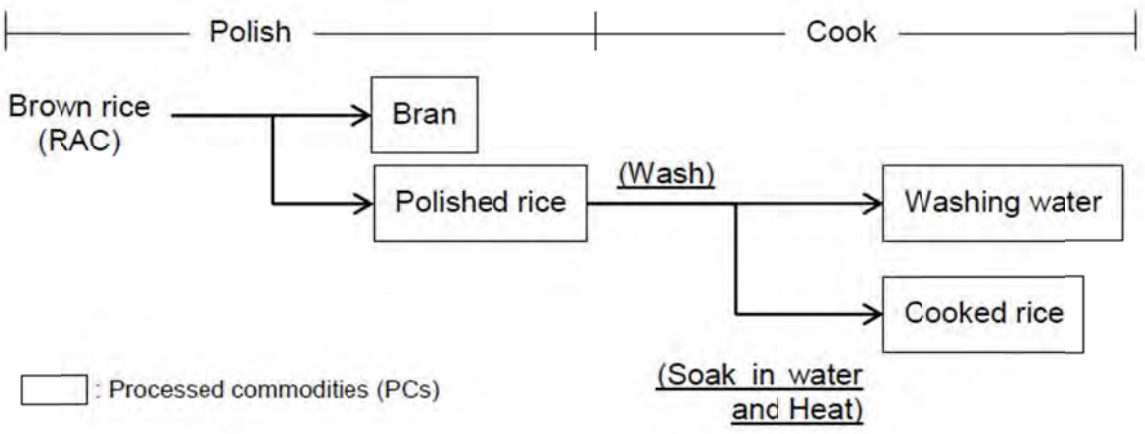


Figure 5 Processing of brown rice

For polishing, a small scale automated rice polishing machine was employed. The polished rice and the bran were stored frozen until analysis. For rice cooking, polished rice was rinsed three times with water. The cloudy washing water was brought to volume with acetonitrile and stored in a refrigerator until analysis. The washed rice was cooked in a small scale automatic rice cooker. The cooked polished rice was homogenised and stored in a freezer until analysis.

Husked rice, polished rice and bran samples were pulverised and soaked in distilled water before extraction, while the cooked rice was immediately subjected to extraction. The samples were extracted twice with acetonitrile/water (80/20) and the extracts combined. The remaining solid materials were extracted twice with acetonitrile/0.1 mol/L HCl (80/20), neutralised with 0.06 mol/L NaOH and acetonitrile was evaporated. Samples were dissolved in distilled water, cleaned-up on C18 cartridges (conditioned with acetonitrile) and the eluates were collected and brought to volume with acetonitrile. The purified samples were analysed by LC-MS/MS. Also, aliquots of the rice washing water were purified on C18 cartridges and subjected to LC-MS/MS analysis.

For method validation, untreated samples of husked rice, bran, polished rice, cooked rice and washing water were fortified with either benzpyrimoxan or DH-04 at levels of 0.01, 0.1, 0.2, 0.5 or 3.5 mg/kg. Mean recoveries were all between 70–120 percent, with RSDs <10 percent. Concurrent recoveries for both analytes in all processed matrices were 86–96 percent. The maximum storage interval from processing until extraction and analysis was 5–8 days.

The results are shown in Table 47.

Table 47 Residues of benzpyrimoxan and DH-04 in processed commodities of rice treated 3 times at 0.01 kg ai/hL in Japan

Year, Location (variety)	DALA	Commodity	Residues, mg/kg			Processing Factor
			Benzpyrimoxan	DH-04	Total*	
2016 Niigata (Koshihikari BL)	21	Husked rice	0.31	0.12	0.42	-
		Bran	2.9	1.1	4.0	9.5
		Polished rice	0.12	0.06	0.18	0.43
		Cooked polished rice	0.03	0.01	0.04	0.095
		Washing water	0.07	0.02	0.09	0.21

Year, Location (variety)	DALA	Commodity	Residues, mg/kg			Processing Factor
			Benzpyrimoxan	DH-04	Total*	
2017 Ibaraki (Koshihikari)	14	Husked rice	0.33	0.12	0.44	-
		Bran	2.7	1.2	3.8	8.6
		Polished rice	0.13	0.05	0.18	0.41
		Cooked polished rice	0.02	<0.01	0.03	0.068
		Washing water	0.06	0.02	0.08	0.18

Note:

* Total = benzpyrimoxan + DH-04 × conversion factor (0.955).

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

The Meeting received lactating dairy cow and laying hens feeding studies.

Lactating dairy cow

Fourteen Holstein dairy cows were treated orally with gelatin capsules fortified with benzpyrimoxan once daily for 28 consecutive days (VanMiddlesworth, 2018: R-37016) at four dose levels equal to 0 (Control, 2 cows), 8 (1×, 3 cows), 24 (3×, 3 cows), and 80 (10×, 6 cows) ppm diet (dry-weight basis. One control and three animals from the 10× dose group were randomly assigned for use in the depuration phase of the study.

During the acclimation and treatment periods, the cows were milked twice daily and samples were pooled to yield a single milk sample per day per animal. Milk samples were retained from the control and treated cows on Dose Days -1 (pre-dose) and days 1, 3, 7, 10, 14, 21 and 28. Additional samples were collected on 3 separate days throughout the depuration phase (Study days 31, 35 and 42). For each cow within a dose group, a daily composite was prepared from milk collected in the morning and evening on Days -1 through Day 28. Extra pooled milk on Days 14 and 28 were separated into cream and skim milk from a single control cow, three cows in the 1× dosing group, three cows in the 3× dosing group, and three cows in the 10× dosing group.

Upon completion of the 28-day dosing period, ten animals (one control, three 1×, three 3× and three 10×) were humanely terminated within 24 hours of receiving the final dose, on the morning of Day 29. Muscle (flank and loin), liver, kidney and fat (subcutaneous, omental and perirenal) were collected from each animal to yield samples of approximately 1 kg weight each, where available. All analytical samples were kept frozen for analysis.

Analytical methods for the analysis of benzpyrimoxan and the metabolites were validated prior to use in bovine milk, muscle, liver, kidney, and fat for benzpyrimoxan, DH-01, DH-02, DH-04, DH-05, and DH-06 at a LOQ of 0.01 mg/kg (Tables 28 to 32).

The longest period of milk storage prior to extraction was 42 days. For skim milk and cream the longest storage interval was 78 days. The longest period of storage for muscle, liver, kidney and fat was 41, 83, 69 and 68 days, respectively.

The results for milk, cream and skin milk are shown in Tables 48 and 49. There was a transfer of residue of benzpyrimoxan, DH-05 and DH-06 to milk, skim milk, and cream, during 28 days of consecutive dosing, reaching a plateau in milk after 3 days of dosing which remained for the remainder of the 28 day dosing schedule. After dosing is complete, DH-05 was found in the 3 day post dose sampling event at

80 ppm, but is not found in the 7 day or 14 day post dose sampling event. There is a very rapid decline after cessation of dosing.

Table 48 Benzpyrimoxan and metabolite residues in milk

		Study Day								Depuration phase		
		-1	1	3	7	10	14	21	28	31	35	42
Dose	Sample	Benzpyrimoxan (mg/kg)										
Control	1A	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	1B	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
8 ppm (1×)	2A	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	2B	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	2C	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
24 ppm (3×)	3A	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	3B	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	3C	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
80 ppm (10×)	4A	ND	ND	<0.01	ND	<0.01	<0.01	<0.01	ND	NA	NA	NA
	4B	ND	ND	<0.01	ND	ND	ND	<0.01	ND	NA	NA	NA
	4C	ND	ND	ND	ND	ND	ND	<0.01	ND	NA	NA	NA
	4D	ND	ND	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	NA	NA
	4E	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA
	4F	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
DH-04 (mg eq/kg)												
Control	1A	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	1B	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
8 ppm (1×)	2A	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	2B	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	2C	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
24 ppm (3×)	3A	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	3B	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	3C	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
80 ppm (10×)	4A	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	4B	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	4C	ND	ND	ND	ND	ND	ND	ND	<0.01	NA	NA	NA
	4D	ND	ND	ND	ND	ND	<0.01	ND	ND	ND	NA	NA
	4E	ND	ND	ND	ND	ND	ND	ND	<0.01	ND	ND	NA
	4F	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Mean	ND	ND	ND	ND	ND	<0.01	ND	<0.01	ND	ND	ND
DH-05 (mg eq/kg)												
Control	1A	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	1B	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
8 ppm (1×)	2A	ND	0.017	0.027	0.026	0.027	0.028	0.027	0.020	NA	NA	NA
	2B	ND	0.010	0.022	0.022	0.019	0.016	0.017	0.012	NA	NA	NA
	2C	ND	0.013	0.034	0.027	0.030	0.030	0.031	0.027	NA	NA	NA
	Mean	ND	0.013	0.028	0.025	0.025	0.025	0.025	0.019	NA	NA	NA
24 ppm (3×)	3A	ND	0.024	0.048	0.048	0.048	0.043	0.021	0.032	NA	NA	NA
	3B	ND	0.037	0.061	0.054	0.066	0.036	0.024	0.043	NA	NA	NA
	3C	ND	0.020	0.036	0.022	0.027	0.028	0.034	0.036	NA	NA	NA

		Study Day								Depuration phase		
		-1	1	3	7	10	14	21	28	31	35	42
	Mean	ND	0.027	0.048	0.041	0.047	0.036	0.026	0.037	NA	NA	NA
80 ppm (10×)	4A	ND	0.118	0.214	0.199	0.213	0.193	0.207	0.154	NA	NA	NA
	4B	ND	0.109	0.325	0.214	0.219	0.192	0.132	0.146	NA	NA	NA
	4C	ND	0.068	0.253	0.210	0.242	0.260	0.064	0.101	NA	NA	NA
	4D	ND	0.103	0.222	0.195	0.167	0.208	0.125	0.188	0.020	NA	NA
	4E	ND	0.054	0.328	0.455	0.379	0.421	0.290	0.230	0.012	ND	NA
	4F	ND	0.178	0.134	0.117	0.121	0.154	0.128	0.120	<0.01	ND	ND
	Mean	ND	0.105	0.246	0.232	0.223	0.238	0.158	0.157	0.014	ND	ND
DH-06 (free and conjugate) (mg eq/kg)												
Control	1A	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	1B	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
8 ppm (1×)	2A	ND	ND	ND	ND	<0.01	ND	ND	ND	NA	NA	NA
	2B	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	2C	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
	Mean	ND	ND	ND	ND	<0.01	ND	ND	ND	NA	NA	NA
24 ppm (3×)	3A	ND	ND	ND	ND	<0.01	<0.01	ND	ND	NA	NA	NA
	3B	ND	ND	<0.01	<0.01	<0.01	<0.01	ND	ND	NA	NA	NA
	3C	ND	ND	<0.01	<0.01	<0.01	<0.01	ND	ND	NA	NA	NA
	Mean	ND	ND	<0.01	<0.01	<0.01	<0.01	ND	ND	NA	NA	NA
80 ppm (10×)	4A	ND	0.016	0.028	0.031	0.025	0.030	0.027	0.010	NA	NA	NA
	4B	ND	0.012	0.023	0.021	0.016	0.016	0.012	<0.01	NA	NA	NA
	4C	ND	<0.01	0.020	0.022	0.014	0.018	<0.01	<0.01	NA	NA	NA
	4D	ND	0.023	0.033	0.033	0.027	0.042	0.022	0.015	ND	NA	NA
	4E	ND	<0.01	0.011	0.011	<0.01	0.011	<0.01	<0.01	ND	ND	NA
	4F	ND	0.012	0.016	0.015	0.017	0.017	0.014	<0.01	ND	ND	ND
	Mean	ND	0.014	0.022	0.022	0.018	0.022	0.016	0.011	ND	ND	ND

Notes:

ND = Not detected.

NA = Not analysed.

Conversion factor: 0.96 for DH-04, 1.08 for DH-05 and 1.13 for DH-06.

Table 49 Benzpyrimoxan and metabolite residues in cream and skim milk (mg eq/kg)

Dose	Sample	Day 14				Day 28			
		Parent	DH-04	DH-05	DH-06	Parent	DH-04	DH-05	DH-06*
Cream									
Control	1A	ND	ND	ND	ND	ND	ND	ND	ND
8 ppm (1×)	2A	ND	ND	0.015	ND	ND	ND	0.017	ND
	2B	ND	ND	0.017	ND	<0.01	ND	0.018	ND
	2C	ND	ND	0.023	ND	ND	ND	0.015	ND
	Mean	ND	ND	0.018	ND	<0.01	ND	0.016	ND
24 ppm (3×)	3A	ND	ND	0.040	<0.01	ND	ND	0.024	ND
	3B	0.016	ND	0.032	<0.01	ND	ND	0.035	<0.01
	3C	ND	ND	0.024	<0.01	ND	ND	0.037	ND
	Mean	<0.01	ND	0.032	<0.01	ND	ND	0.032	<0.01
80 ppm (10×)	4A	0.018	ND	0.191	0.029	<0.01	ND	0.134	<0.01
	4B	0.017	ND	0.205	0.019	<0.01	ND	0.118	<0.01
	4C	0.010	ND	0.209	0.014	0.011	ND	0.083	<0.01
	Mean	0.015	ND	0.201	0.021	0.010	ND	0.112	<0.01
Skim milk									
Control	1A	ND	ND	ND	ND	ND	ND	ND	ND

Dose	Sample	Day 14				Day 28			
		Parent	DH-04	DH-05	DH-06	Parent	DH-04	DH-05	DH-06*
Cream									
8 ppm (1×)	2A	ND	ND	0.024	ND	ND	ND	0.017	ND
	2B	ND	ND	0.020	ND	ND	ND	0.014	ND
	2C	ND	ND	0.035	ND	ND	ND	0.035	ND
	Mean	ND	ND	0.026	ND	ND	ND	0.022	ND
24 ppm (3×)	3A	ND	ND	0.060	ND	ND	ND	0.028	ND
	3B	ND	ND	0.049	<0.01	ND	ND	0.050	ND
	3C	ND	ND	0.037	<0.01	ND	ND	0.045	<0.01
	Mean	ND	ND	0.049	<0.01	ND	ND	0.041	<0.01
80 ppm (10×)	4A	0.018	ND	0.259	0.024	ND	ND	0.156	<0.01
	4B	ND	ND	0.268	0.016	ND	ND	0.173	<0.01
	4C	ND	ND	0.272	0.016	ND	ND	0.126	<0.01
	Mean	<0.01	ND	0.266	0.019	ND	ND	0.152	<0.01

Notes:

ND = Not detected.

Conversion factor = 0.96 for DH-04, 1.08 for DH-05 and 1.13 for DH-06.

* Free and conjugate.

The results for tissues are shown in Table 50. There was no appreciable residue transfer or preferential accumulation to the bovine muscle. For liver and kidney, residues of benzpyrimoxan and metabolites were detected on the first day after cessation of dosing (day 28 sampling event) from cows administered at the 1×, 3× and 10× dosing levels. Benzpyrimoxan and DH-05 were found in the liver of the 10× dosing level in the 3 day post dose sampling event during the depuration phase, but not in the subsequent sampling events. For perirenal, omental and subcutaneous fat, residues of benzpyrimoxan, DH-02, DH-04 and DH-05 were detected on the first day after cessation of dosing (day 28 sampling event) from cows administered at the 1×, 3×, and 10× dosing levels. Fat residue in the 10× dosing level was <0.01 mg/kg by the 3 day post dose sampling event.

Table 50 Benzpyrimoxan and metabolite residues in liver, kidney, muscle and fat (mg eq/kg)

Dose rate	Parent	DH-01	DH-02*	DH-04	DH-05	DH-06*
Liver						
Control	ND	ND	ND	ND	ND	ND
8 ppm (1×)	0.011, <0.01, 0.013 (0.011)	ND, ND, ND (ND)	0.011, <0.01, <0.01 (0.010)	ND, ND, ND (ND)	0.027, 0.011, 0.011 (0.016)	ND, ND, ND (ND)
24 ppm (3×)	0.047, 0.042, 0.035 (0.041)	ND, ND, ND Mean ND	0.016, 0.018, 0.050 (0.028)	ND, ND, ND (ND)	0.049, 0.033, 0.050 (0.044)	ND, <0.01, 0.028 (0.013)
80 ppm (10×)	0.149, 0.122, 0.153 (0.141)	ND, ND, ND (ND)	0.103, 0.064, 0.091 (0.086)	ND, ND, ND (ND)	0.253, 0.141, 0.148 (0.181)	0.015, <0.01, <0.01 (0.012)
Kidney						
Control	ND	ND	ND	ND	ND	ND
8 ppm (1×)	ND, ND, <0.01 (<0.01)	ND, ND, <0.01 (<0.01)	0.029, 0.025, 0.011 (0.022)	ND, ND, ND (ND)	0.062, 0.032, 0.035 (0.043)	ND, <0.01, <0.01 (<0.01)
24 ppm (3×)	<0.01, ND, ND (<0.01)	<0.01, 0.014, <0.01 (0.011)	0.021, 0.028, 0.036 (0.028)	ND, 0.012, ND (<0.01)	0.039, 0.092, 0.075 (0.069)	<0.01, <0.01, <0.01 (<0.01
80 ppm (10×)	<0.01, <0.01, 0.031 (0.017)	0.032, 0.015, <0.01 (0.019)	0.109, 0.101, 0.239 (0.150)	<0.01, <0.01, 0.020 (0.013)	0.297, 0.151, 0.224 (0.224)	0.034, 0.020, 0.025 (0.026)
Flank muscle						

Dose rate	Parent	DH-01	DH-02*	DH-04	DH-05	DH-06*
Control	ND	ND	ND	ND	ND	ND
8 ppm (1×)	<0.01, ND, ND Mean <0.01	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)
24 ppm (3×)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)
80 ppm (10×)	ND, ND, <0.01 (<0.01)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)
Loin muscle						
Control	ND	ND	ND	ND	ND	ND
8 ppm (1×)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)
24 ppm (3×)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)
80 ppm (10×)	ND, ND, <0.01 (<0.01)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, <0.01 (<0.01)	ND, ND, ND (ND)	ND, ND, ND (ND)
Omental fat						
Control	ND	ND	ND	ND	ND	ND
8 ppm (1×)	ND, ND, <0.01 (<0.01)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)
24 ppm (3×)	ND, <0.01, <0.01 (<0.01)	ND, ND, ND (ND)	ND, 0.012, ND (<0.01)	ND, ND, ND (ND)	ND, <0.01, <0.01 (<0.01)	ND, ND, ND (ND)
80 ppm (10×)	0.044, 0.026, 0.013 (0.028)	ND, ND, ND (ND)	<0.01, <0.01, <0.01 (<0.01)	ND, ND, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	ND, ND, ND (ND)
Perirenal fat						
Control	ND	ND	ND	ND	ND	ND
8 ppm (1×)	ND, ND, ND (ND)	ND, ND, ND (ND)	<0.01, ND, ND (<0.01)	ND, ND, ND (ND)	0.013, ND, <0.01 (<0.01)	ND, ND, ND (ND)
24 ppm (3×)	ND, ND, <0.01 (<0.01)	ND, ND, ND (ND)	<0.01, ND, <0.01 (<0.01)	ND, ND, ND (ND)	0.010, <0.01, 0.012 (0.011)	ND, ND, ND (ND)
80 ppm (10×)	0.031, 0.021, 0.014 (0.022)	ND, ND, <0.01 (<0.01)	0.010, 0.014, 0.115 (0.046)	ND, ND, 0.012 (<0.01)	0.042, 0.037, 0.184 (0.088)	ND, ND, <0.01 (<0.01)
Subcutaneous fat						
Control	ND	ND	ND	ND	ND	ND
8 ppm (1×)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)	ND, ND, ND (ND)
24 ppm (3×)	ND, <0.01, ND (<0.01)	ND, ND, ND (ND)	ND, <0.01, <0.01 (<0.01)	ND, ND, ND (ND)	ND, 0.044, ND (0.015)	ND, ND, ND (ND)
80 ppm (10×)	0.019, 0.013, <0.01 (0.014)	ND, ND, ND (ND)	<0.01, <0.01, <0.01 (<0.01)	ND, ND, <0.01 (<0.01)	<0.01, 0.021, 0.021 (0.017)	ND, ND, ND (ND)

Notes:

ND = not detected.

Conversion factor = 1.14 for DH-01, 1.20 for DH-02, 0.96 for DH-04, 1.08 for DH-05 and 1.13 for DH-06.

* Free and conjugate.

Laying hen

ISA Husked (Gold Star) laying hens (*Gallus gallus domesticus*) were treated orally with gelatin capsules fortified with benzpyrimoxan once daily for 29 consecutive days (VanMiddlesworth, 2018: R-37017). Sixty egg laying hens were dosed at four dose levels equal to 0 ppm (Control,), 3 ppm (1×), 9 ppm (3×), and 30 ppm (10×) diet (dry-weight basis). Birds chosen for the study were randomly assigned to one of five

treatment groups: control (12 hens in 3 subgroups of 4 hens each: 1A, 1B, and 1C), 1× (12 hens in 3 subgroups of 4 hens each: 2A, 2B, and 2C), 3× (12 hens in 3 subgroups of 4 hens each: 3A, 3B, and 3C) and 10× (24 hens in 6 subgroups of 4 hens each: 4A, 4B, 4C, 4D, 4E, 4F). One control subgroup (subgroup 1C) and three subgroups from the 10× dose group (subgroups 4D, 4E, and 4F) were assigned for use in the depuration phase of the study.

During the acclimation and treatment periods, the eggs were collected twice daily and the samples were pooled to yield a single sample per day per subgroup. Egg samples were retained from the control and treated hens on Dose Days -1 (pre-dose) and days 1, 3, 7, 10, 14, 17, 21, 24 and 28. Additional samples were collected on 3 separate days throughout the depuration phase (Study days 32, 36 and 43). For each subgroup within a dose group, a daily composite was prepared from eggs collected in the morning and evening on Days -1 through Day 28. The pooled egg samples on Days 14 and 28 were separated into egg yolk and egg white from a single control (group 1, subgroup 1C), three subgroups in the 1× dosing group (group 2, subgroups 2A, 2B, and 2C), three subgroups in the 3× dosing group (group 3, subgroups 3A, 3B, and 3C), and three subgroups in the 10× dosing group (group 4, subgroup 4D, 4E, and 4F).

Upon completion of the 29-day dosing period, eleven subgroups (two controls: subgroups 1A and 1B, three 1×: subgroups 2A, 2B, and 2C, three 3×: subgroups 3A, 3B, 3C, and three 10×: subgroups 4A, 4B, 4C) were humanely terminated at the in-life facility within 6 hours of receiving the final dose. 100–150 g of muscle (thigh and breast combined), the entire liver, and 50 g of fat (subcutaneous and abdominal combined) were collected from each bird to yield samples. All analytical samples were kept frozen until analysis.

Analytical methods for the analysis of benzpyrimoxan and the metabolites were validated at a LOQ of 0.01 mg/kg for benzpyrimoxan, benzpyrimoxan-CH_2OH (DH-02), and benzpyrimoxan-acid-2-OH (DH-05). Average recoveries for both quantitation and confirmation ion transitions were in the acceptable range for all analytes (Tables 28, 30 and 32). The longest storage period for egg was 36 days. Tissue samples of muscle, liver, and fat were analysed within 30 days.

The results for eggs, egg yolk and whites are shown in Tables 51 and 52. There was transfer of residue of benzpyrimoxan, DH-02, and DH-05 to egg, egg yolk, or egg white during 29 days of consecutive dosing, but rapidly declined within 3 days following the final dose. There were no residues found in whole eggs when benzpyrimoxan is delivered in the feed at the anticipated livestock dietary exposure levels.

Table 51 Benzpyrimoxan and metabolites residues in whole eggs (mg/kg)

		Study Day									
Dose	Sample	-1	1	3	7	10	14	17	21	24	28
Benzpyrimoxan											
Control	1A	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	1B	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	1C	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3 ppm (1×)	2A	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	2B	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	2C	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	Mean	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
9 ppm (3×)	3A	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	3B	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	3C	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	Mean	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
30 ppm	4A	ND	ND	ND	ND	<0.01	ND	ND	ND	ND	ND

		Study Day									
Dose	Sample	-1	1	3	7	10	14	17	21	24	28
(10×)	4B	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	4C	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	4D	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	4E	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	4F	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	Mean	ND	ND	ND	ND	<0.01	ND	ND	ND	ND	ND
DH-02											
Control	1A	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	1B	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	1C	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	Mean	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
3 ppm (1×)	2A	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	2B	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	2C	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	Mean	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
9 ppm (3×)	3A	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	3B	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	3C	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	Mean	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
30 ppm (10×)	4A	ND	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	4B	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	4C	ND	ND	ND	<0.01	ND	<0.01	<0.01	<0.01	<0.01	<0.01
	4D	ND	ND	<0.01	<0.01	<0.01	NA	<0.01	<0.01	<0.01	NA
	4E	ND	ND	<0.01	<0.01	<0.01	NA	<0.01	<0.01	<0.01	NA
	4F	ND	ND	ND	<0.01	<0.01	NA	<0.01	<0.01	<0.01	NA
	Mean	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
DH-05											
Control	1A	<0.01	ND	ND	ND	ND	ND	ND	ND	ND	ND
	1B	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	1C	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	Mean	<0.01	ND	ND	ND	ND	ND	ND	ND	ND	ND
3 ppm (1×)	2A	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	2B	ND	ND	ND	ND	<0.01	NA	ND	ND	ND	NA
	2C	ND	ND	ND	ND	ND	NA	ND	ND	ND	NA
	Mean	ND	ND	ND	ND	<0.01	NA	ND	ND	ND	NA
9 ppm (3×)	3A	ND	ND	ND	<0.01	<0.01	NA	<0.01	ND	<0.01	NA
	3B	ND	ND	ND	<0.01	<0.01	NA	<0.01	ND	ND	NA
	3C	ND	ND	ND	ND	<0.01	NA	ND	ND	ND	NA
	Mean	ND	ND	ND	<0.01	<0.01	NA	<0.01	ND	<0.01	NA
30 ppm (10×)	4A	ND	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	4B	ND	ND	<0.01	0.010	0.011	0.011	<0.01	0.011	<0.01	0.011
	4C	ND	ND	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	4D	ND	ND	ND	<0.01	<0.01	NA	<0.01	<0.01	<0.01	NA
	4E	ND	ND	ND	<0.01	<0.01	NA	<0.01	<0.01	0.010	NA
	4F	ND	ND	ND	<0.01	<0.01	NA	<0.01	<0.01	<0.01	NA
	Mean	ND	ND	<0.01	0.010	0.010	0.010	<0.01	0.010	0.010	0.010

Dose	Sample	Benzpyrimoxan (mg/kg)			DH-02 (mg eq/kg)			DH-05 (mg eq/kg)		
		Depuration phase			Depuration phase			Depuration phase		
		31	35	42	31	35	42	31	35	42
Control	1C	ND	ND	ND	ND	ND	ND	ND	ND	ND
30 ppm (10×)	4D	ND	NA	NA	ND	NA	NA	<0.01	NA	NA
	4E	ND	ND	NA	<0.01	ND	NA	<0.01	ND	NA
	4F	ND	ND	ND	<0.01	ND	ND	<0.01	ND	ND

		Study Day									
Dose	Sample	-1	1	3	7	10	14	17	21	24	28
	Mean	ND	ND	ND	<0.01	ND	ND	<0.01	ND	ND	

Notes:

ND = Not detected, NA = not analysed

Conversion factor = 1.20 for DH-02 and 1.08 for DH-05.

Table 52 Benzpyrimoxan and metabolite residues in egg yolk and whites (mg eq/kg)

		Day 14			Day 28		
Dose	Sample	Parent	DH-02	DH-05	Parent	DH-02	DH-05
Egg yolk							
Control	1C	ND	ND	ND	ND	ND	ND
3 ppm (1×)	2A	ND	ND	ND	ND	ND	ND
	2B	ND	ND	ND	ND	ND	ND
	2C	ND	ND	ND	ND	ND	ND
	Mean	ND	ND	ND	ND	ND	ND
9 ppm (3×)	3A	ND	ND	<0.01	ND	<0.01	<0.01
	3B	ND	ND	<0.01	ND	ND	<0.01
	3C	ND	ND	<0.01	ND	<0.01	<0.01
	Mean	ND	ND	<0.01	ND	<0.01	<0.01
30 ppm (10×)	4D	ND	<0.01	0.011	ND	0.012	0.014
	4E	ND	0.011	0.011	ND	<0.01	0.012
	4F	ND	<0.01	0.011	<0.01	<0.01	0.012
	Mean	ND	0.010	0.011	<0.01	0.011	0.013
Egg whites							
Control	1C	ND	ND	ND	ND	ND	ND
3 ppm (1×)	2A	ND	ND	ND	ND	ND	ND
	2B	ND	ND	ND	ND	ND	ND
	2C	0.015	ND	ND	ND	ND	ND
	Mean	<0.01	ND	ND	ND	ND	ND
9 ppm (3×)	3A	ND	ND	ND	ND	ND	ND
	3B	ND	ND	ND	ND	ND	ND
	3C	ND	ND	ND	ND	ND	ND
	Mean	ND	ND	ND	ND	ND	ND
30 ppm (10×)	4D	ND	ND	<0.01	ND	ND	ND
	4E	ND	ND	ND	ND	ND	ND
	4F	ND	ND	ND	ND	ND	ND
	Mean	ND	ND	<0.01	ND	ND	ND

Notes:

ND = Not detected.

Conversion factor = 1.20 for DH-02 and 1.08 for DH-05.

Table 53 shows the rsults for hen tissues. There was no appreciable residue transfer or preferential accumulation to the hen muscle. For liver and fat, the residues of benzpyrimoxan (fat), DH-02 (liver and fat), and DH-05 (liver) were found the last day of dosing from hens administered at the 1× (liver), 3× (liver and fat), and 10× (liver and fat) dosing level. After cessation of the dose, there were no residues measured in the liver or fat.

Table 53 Benzpyrimoxan and metabolite residues in liver, muscle and fat (mg eq/kg)

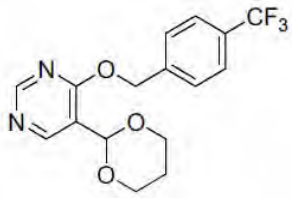
Dose rate	Parent		DH-02		DH-05	
Liver						
Control	ND, ND	Mean ND	ND, ND	Mean ND	ND, ND	Mean ND
3 ppm (1×)	ND, ND, ND	Mean ND	0.013, <0.01, 0.019	Mean 0.014	<0.01, 0.012, 0.016	Mean 0.013
9 ppm (3×)	ND, ND, ND	Mean ND	0.046, <0.01, 0.020	Mean 0.025	0.043, 0.017, 0.041	Mean 0.034
30 ppm (10×)	ND, ND, ND	Mean ND	0.029, 0.018, 0.045	Mean 0.031	0.034, 0.041, 0.067	Mean 0.048
Muscle						
Control	ND, ND	Mean ND	ND, ND	Mean ND	ND, ND	Mean ND
3 ppm (1×)	ND, ND, ND	Mean ND	ND, ND, ND	Mean ND	ND, ND, ND	Mean ND
9 ppm (3×)	ND, ND, ND	Mean ND	ND, ND, ND	Mean ND	<0.01, ND, <0.01	Mean <0.01
30 ppm (10×)	ND, ND, ND	Mean ND	ND, ND, <0.01	Mean <0.01	<0.01, <0.01, <0.01	Mean <0.01
Fat						
Control	ND, ND	Mean ND	ND, ND	Mean ND	ND, ND	Mean ND
3 ppm (1×)	ND, ND, ND	Mean ND	<0.01, ND, ND	Mean <0.01	ND, ND, ND	Mean ND
9 ppm (3×)	<0.01, <0.01, <0.01	Mean <0.01	0.014, <0.01, 0.013	Mean 0.012	ND, ND, <0.01	Mean <0.01
30 ppm (10×)	0.013, 0.012, 0.015	Mean 0.013	0.015, 0.012, 0.022	Mean 0.017	<0.01, ND, <0.01	Mean <0.01

APPRAISAL

Benzpyrimoxan is an insecticide (insect growth regulator) with activity on juvenile stages of the rice plant hopper (Hemiptera: Delphacidae). Benzpyrimoxan is highly active against nymphal stages of rice plant hoppers without any adulticidal activity. It is registered for the control of sap sucking insects on rice.

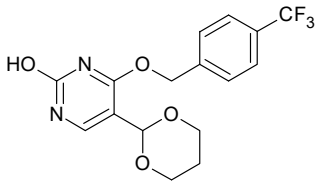
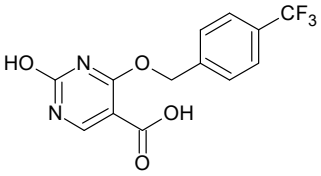
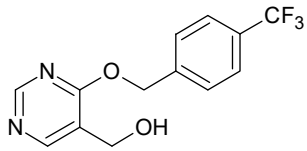
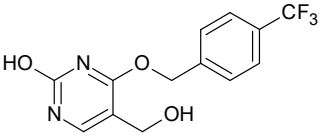
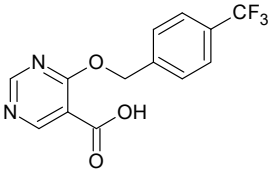
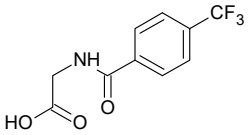
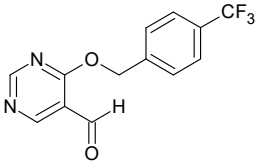
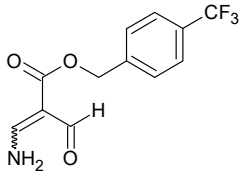
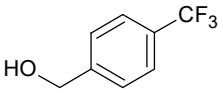
At the Fifty-first Session of the CCPR (2019), it was scheduled for the evaluation as a new compound in 2020 and rescheduled to the 2022 JMPR. The Meeting received information on identity, physical and chemical properties, animal and plant metabolism, rotational crop study, environmental fate, analytical methods, GAP information, storage stability, processing, supervised residue trials and farm animal feeding study.

The IUPAC name for benzpyrimoxan is 5-(1,3-dioxan-2-yl)-4-[4-(trifluoromethyl)benzyloxy]pyrimidine.



The following abbreviations are used for the major metabolites discussed in Table 1 below.

Table 54 Metabolites and their codes referred to in the appraisal

Code	Name and Matrix	Structure
Benzpyrimoxan-2-OH DH-04	5-(1,3-dioxan-2-yl)-4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidin-2-ol Rice, Soil	
Benzpyrimoxan-acid-2-OH DH-05	2-hydroxy-4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidine-5-carboxylic acid Goat, Hen	
Benzpyrimoxan-CH_2OH DH-02	(4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidin-5-yl)methanol Goat, Hen	
Benzpyrimoxan-CH_2OH-2-OH DH-06	5-(hydroxymethyl)-4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidin-2-ol Goat, Hen	
Benzpyrimoxan-acid DH-01	4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidine-5-carboxylic acid Goat	
Benzpyrimoxan-benzoyl-glycine DH-402	N-[4-(trifluoromethyl)benzoyl]glycine Goat	
Benzpyrimoxan-aldehyde DH-03	4-{[4-(trifluoromethyl)phenyl] methoxy}pyrimidine-5-carbaldehyde High temperature hydrolysis	
Benzpyrimoxan-enamine-aldehyde DH-08	[4-(trifluoromethyl)phenyl] methyl (2E)-3-amino-2-formylprop-2-enoate High temperature hydrolysis	
4-TFMPM DH-102	[4-(trifluoromethyl)phenyl] methanol High temperature hydrolysis	

Physical and chemical properties

Benzpyrimoxan is not volatile. It has a higher solubility in organic solvents (up to 178 g/L in dichloromethane) in comparison to water (5 mg/L). The octanol/water partition coefficient log P_{ow} of 3.4

for benzpyrimoxan suggests a potential to partition into fat. Benzpyrimoxan is hydrolytically stable under natural and basic conditions while it is considered to be moderately persistent under acidic conditions (pH 4), with DT_{50} value at 25 °C of about 50 days. It is stable to photolysis.

Plant metabolism

The Meeting received plant metabolism studies for after foliar application on paddy rice with benzpyrimoxan labelled at [Phenyl-U-^{14}C] and [Pyrimidinyl-4(6)-^{14}C].

[^{14}C]-benzpyrimoxan was applied to rice grown pots under paddy conditions in a greenhouse at a rate of 0.20 kg ai/ha (1× GAP). In one experiment, the plant was sprayed three times at 7 day intervals, at heading stage (BBCH 55) and milk stage (BBCH 61–65 and 73–75). In a second experiment, the plant was sprayed twice with an interval of 7 days, at heading stage (BBCH 55) and milk stage (BBCH 61–65), with the third application at BBCH 87–89, 4 weeks after the second application. Samples were taken at 7 days after treatment (DAT). Samples of panicle, foliage and root were collected at milk stage. At ripe stage, panicle, straw and root were collected and then panicle was separated into grain and hulls after 14 days of air drying.

Total radioactive residues (TRR) in panicle and foliage at milk stage were 1.2–1.4 and 1.8–2.4 mg eq/kg, respectively. The TRRs in hull and straw at ripe stage were 2.8–4.7 and 3.3-3.7 mg eq/kg, respectively. Relatively small amounts of radioactive residues were detected in grain (0.10–0.25 mg eq/kg) and root (0.05–0.09 mg eq/kg). At least 77 percent TRR of [Pyrimidinyl-4(6)-14C] label) were recovered from the samples in the surface rinse with acetonitrile (15–49 percent TRR), acetonitrile/water (45–86 percent TRR) and acetonitrile/0.1 mol/L HCl (1.4–9.4 percent TRR) in all the plant parts at both stages. The post-extraction solids (PES) of all samples were characterized by enzyme, and acidic or alkaline hydrolyses, which released an additional 2–19 percent TRR.

Benzpyrimoxan was the predominant residue in all samples at milk and ripe stages, at levels of 0.05–0.14 mg/kg (48–57 percent TRR) in grain, 1.7–2.8 mg/kg (60–61 percent TRR) in hull and 1.6–1.9 mg/kg (48–51 percent TRR) in straw. Benzpyrimoxan-2-OH (DH-04) was the major metabolite in all samples at both stages, at 0.02–0.04 mg eq/kg (15–17 percent TRR) in grain, 0.14–0.24 mg eq/kg (5.0–5.1 percent TRR) in hull and 0.34–0.39 mg eq/kg (9.3–12 percent TRR) in straw.

Minor (< 10 percent TRR) metabolites were also detected. Benzpyrimoxan-CH_2OH (DH-02) conjugate was identified in hulls (3.1–3.8 percent TRR; 0.11–0.15 mg eq/kg) and straw (2.6–4.4 percent TRR, 0.09–0.16 mg eq/kg). Benzpyrimoxan-acid-2-OH (DH-05) (free and conjugated) was identified in hulls (1.1–1.7 percent TRR, 0.05 mg eq/kg) and straw (4.4–4.8 percent TRR, 0.15–0.18 mg eq/kg). Benzpyrimoxan-CH_2OH-2-OH (DH-06) (free and conjugated) was found in hulls (1.2 percent TRR, 0.03 mg eq/kg) and straw (3.4–3.5 percent TRR, 0.11–0.13 mg eq/kg).

Conclusions

Parent benzpyrimoxan is the main residue in rice. The potential metabolic pathway in rice is hydroxylation of a pyrimidine ring to benzpyrimoxan-2-OH (DH-04), followed by hydrolysis of an acetal moiety to form a hydroxymethyl moiety and carboxylic acid moiety.

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens where animals were dosed with [^{14}C]-benzpyrimoxan. The metabolism and distribution of benzpyrimoxan in farm animals were investigated using the [Phenyl-U-^{14}C] and the [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan.

Rats

The metabolism of benzpyrimoxan in rats was reviewed in the framework of the toxicological evaluation by the WHO Core Assessment Group of the 2022 JMPR.

Lactating goats

Lactating goats received daily oral dosing of [^{14}C]-benzpyrimoxan for 5 consecutive days at 14 ppm in the diet for [Phenyl-U-^{14}C]-benzpyrimoxan and at 15 ppm in the diet for [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan. The goats were sacrificed 6 hours after the last dose for the phenyl label and 8 hours after the last dose for the pyrimidinyl label.

TRR were highest in the liver (0.22–0.69 mg eq/kg) and kidney (0.18–0.25 mg eq/kg), followed by muscle (0.008–0.010 mg eq/kg) and fat (0.008–0.015 mg eq/kg). The concentration of radioactivity in the milk reached a plateau of 0.052–0.095 mg eq/kg by day 3–5.

Liver and kidney were extracted with acetonitrile/water and acetonitrile, fat with hexane/acetone and acetone and milk (Day 5) with acetonitrile and acetonitrile/water. Liver, kidney and milk were further extracted with acetonitrile/0.1 mol/L HCl. Muscle was not analysed due to low concentration (< 0.01 mg eq/kg). Most of radioactive residues (78–93 percent TRR) in milk and tissues were extracted (neutral solvent: 57–93 percent TRR, ACN/HCl: < 1.2–3.8 percent TRR).

Benzpyrimoxan was not detected in any sample except in liver (2.3 percent TRR, 0.014 mg/kg). Benzpyrimoxan-acid-2-OH (DH-05) was found as a predominant metabolite in kidney (40–47 percent TRR, 0.074–0.11 mg eq/kg) and milk (59–86 percent TRR, 0.048–0.062 mg eq/kg) and as a major metabolite in liver (6.4–11 percent TRR, 0.025–0.039 mg eq/kg).

Benzpyrimoxan-CH_2OH (DH-02) (free and conjugated) was found as a major metabolite in liver (3.1–22 percent TRR, 0.019–0.051 mg eq/kg) and kidney (5.9–16 percent TRR, 0.011–0.039 mg eq/kg). Benzpyrimoxan-CH_2OH-2-OH (DH-06) (free and conjugated) was the major metabolite in liver (4.8–15 percent TRR, 0.029–0.036 mg eq/kg) and benzpyrimoxan-acid (DH-01) in kidney (16–20 percent TRR, 0.038 mg eq/kg).

Benzpyrimoxan-2-OH (DH-04) was identified as a minor metabolite in liver (2.6–3.1 percent TRR, 0.006–0.019 mg eq/kg) and benzpyrimoxan-benzoyl-glycine (DH-402) in kidney (7.9 percent TRR, 0.019 mg eq/kg).

Laying hens

Laying hens received daily oral dosing for 7 consecutive days of [Phenyl-U-^{14}C]-benzpyrimoxan at a rate equivalent to 11 ppm in the diet and of [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan at 13 ppm. The hens were sacrificed 6 hours after the last dose.

TRRs in tissues were highest in liver (0.066–0.19 mg eq/kg) and subcutaneous fat (0.065–0.18 mg eq/kg), followed by abdominal fat (0.042–0.063 mg eq/kg) and leg and breast muscle (0.003–0.014 mg eq/kg). Residue levels in egg white reached a plateau of 0.006–0.009 mg eq/kg by days 4–7 and those in egg yolk reached a plateau of 0.13–0.20 mg eq/kg by day 5–7.

Liver, muscle and egg were extracted with acetonitrile/water and acetonitrile, and fat with hexane/acetone and acetone. Liver, muscle and egg were further extracted with acetonitrile/0.1 mol/L HCl. Good extractability was achieved for fat samples (95–96 percent TRR). Over 50 percent TRR was extracted from liver and muscle samples (neutral solvent: 54–79 percent TRR, ACN/HCl: 1.7–2.2 percent

TRR), and 34–73 percent TRR from eggs (neutral solvent: 31–67 percent TRR, ACN/HCl: 2.9–6.7 percent TRR).

Benzpyrimoxan was the predominant component in muscle (43 percent TRR, 0.006 mg/kg) and fat (71 percent TRR, 0.043–0.089 mg/kg), a minor residue in eggs (5.7–13 percent TRR, 0.002–0.004 mg/kg) and was not detected in liver.

DH-05 was identified as a major metabolite in liver (20–28 percent TRR, 0.012–0.050 mg eq/kg). DH-02 was a major metabolite in liver (6.1–22 percent TRR, 0.011–0.013 mg eq/kg), fat (9.8–14 percent TRR, 0.006–0.018 mg eq/kg)and was identified as a major metabolite in muscle (14 percent TRR) and egg (13–27 percent TRR), but at levels below 0.01 mg eq/kg. DH-06 was found as a minor component in liver (3.3 percent TRR, 0.002–0.006 mg eq/kg). No other metabolites were identified.

Conclusions

Parent benzpyrimoxan is the minor residue or no residue in animal commodities except hen muscle and fat. The major metabolites of benzpyrimoxan in farm animals are formed by hydrolysis of the acetal moiety to form a hydroxymethyl moiety (DH-02 and DH-06) and its resulting carboxylic acid moiety (DH-01 and DH-05), followed by conjugation to glucuronides. Hydroxylation of a pyrimidine ring are also observed.

Environmental fate

The Meeting received aerobic soil (paddy and upland) metabolism, aqueous hydrolysis and aqueous photolysis studies for benzpyrimoxan.

In the aerobic paddy soil metabolism study conducted in a vessel with phenyl and pyrimidinyl radiolabelled benzpyrimoxan, benzpyrimoxan was degraded in paddy soil very gradually with a DT_{50} of > 1 year at 25 °C. Benzpyrimoxan was detected with 73–95 percent AR during 180 days incubation period. Several identified and unknown degradates were detected, however, none of which accounted for more than 10 percent of AR throughout the study period for both radiolabels.

In the aerobic upland soil metabolism study conducted in a vessel with phenyl and pyrimidinyl radiolabelled benzpyrimoxan, benzpyrimoxan was gradually degraded in upland soil with a DT_{50} of 124 days at 25 °C. Benzpyrimoxan was detected as the major component with 33 percent AR after 180 days incubation. Several identified degradants were detected but accounted for less than 10 percent of AR and did not accumulate

In conclusion, benzpyrimoxan was gradually degraded in soil, and the breakdown products also moderately degraded to form unextracted residue and CO_2. Benzpyrimoxan is persistent in soil.

In the aqueous hydrolysis study, benzpyrimoxan was hydrolytically stable at pH 7 and 9 at 50 °C but decomposed at pH 4 with a DT_{50} of 50–51 days at 25 °C. The hydrolysis product was benzpyrimoxan-enamine aldehyde (DH-08) with maximum amounts of 13 percent AR. Several identified and unknown hydrolysis products accounted for less than 10 percent of AR. Hydrolysis is unlikely to be a major route of environmental degradation.

In the aqueous photolysis study, benzpyrimoxan was photolytically stable at 25 °C for 25 days. Photolysis in water is unlikely to be a major route of environmental degradation.

Rotational crop metabolism

No rotational crop studies were provided to the Meeting.

The Meeting noted that residue of benzpyrimoxan may be taken up by follow-on and rotational crops since this compound is persistent in soil,

The Meeting considered that information on the agricultural practice for paddy rice cultivation and International Harmonised Guidelines (OECD TG504) indicated potential crop rotation for paddy rice.

The Meeting could not conclude on the residues related to benzpyrimoxan in rotational crops.

Methods of analysis

The Meeting received information on analytical methods for benzpyrimoxan and its metabolites in plant and animal matrices.

Plant matrices

In the method for determination of benzpyrimoxan and benzpyrimoxan-2-OH (DH-04) in rice matrices (grain, husked rice, polished rice, cooked rice, bran and straw), samples were extracted with acetonitrile/water and acetonitrile/0.1 mol/L HCl (. After SPE clean-up, residues were determined by LC-MS/MS. The method was validated with an LOQ of 0.01 mg/kg for each analyte.

A QuEChERS method was also validated for benzpyrimoxan and DH-04 in husked rice with an LOQ of 0.01 mg/kg for each analyte.

The Meeting concluded that the presented methods were sufficiently validated and are suitable to measure benzpyrimoxan and DH-04 in rice commodities.

Animal matrices

In the method for determination of benzpyrimoxan, benzpyrimoxan-acid (DH-01), benzpyrimoxan-CH_2OH (DH-02), benzpyrimoxan-2-OH (DH-04), benzpyrimoxan-acid-2-OH (DH-05) and benzpyrimoxan-CH_2OH-2-OH (DH-06), cow tissues (muscle, liver, kidney) and cream samples were extracted with acetonitrile/water and cleaned up by SPE. Fat samples were extracted with hexane/acetone and acetonitrile/water, the hexane layer extracted with acetonitrile followed by SPE clean-up. Milk and skin milk samples were centrifuged and provided to SPE. An aliquot of each sample was treated with -glucuronidase. The residues were determined by LC-MS/MS .

The method recoveries of DH-01 in liver were < 70 percent due its conversion to DH-05, although the sum of DH-01 and DH-05 recovery was within the acceptable range.

In the method for the analysis of benzpyrimoxan, DH-02 and DH-05 in hen commodities, egg, muscle and liver samples were extracted twice with acetonitrile/water and purified by SPE. Fat samples were extracted with hexane/acetone and acetonitrile/water, the lower layer collected, the hexane layer extracted with acetonitrile and the hydro-organic layer combined. The extract was cleaned up by SPE. The residues were analysed by LC-MS/MS at a LOQ of 0.01 mg/kg

The Meeting concluded that the presented methods were sufficiently validated at a LOQ of 0.01 mg/kg and are suitable to measure benzpyrimoxan, DH-01 (except liver), DH-02 (free and conjugated), DH-04, DH-05 and DH-06 (free and conjugated) in animal commodities. DH-4 and DH-06 (free and conjugated) were not validated in hen commodities.

Stability of pesticide residues in stored analytical samples

The Meeting received information on storage stability of benzpyrimoxan and benzpyrimoxan-2-OH (DH-04) in rice grain (paddy rice), brown rice and straw fortified at 0.5 mg/kg and stored at -20 °C.

Benzpyrimoxan and DH-04 were stable for at least 2.5 months in rice grain, husked rice and straw, which covered the residue sample storage intervals used in the field trials considered by the current Meeting.

The Meeting received information on storage stability of benzpyrimoxan, DH-01, DH-02, DH-04, DH-05 and/or DH-06 in animal commodities fortified at 0.1 mg/kg and stored at -20 °C.

Benzpyrimoxan, DH-04 and DH-06 were stable for at least 3 months in milk, muscle and liver, at least 2.5 months in fat, and at least 1 month in kidney, DH-05 was stable for at least 1 month in milk, muscle, liver, kidney, fat and egg and DH-02 was stable for at least 3 months in muscle and liver, for at least 2.5 months in fat, and for at least 1 month in kidney and egg.

The Meeting noted that DH-01 was unstable in liver, kidney and fat, quantitatively degrading to DH-05 within 1 month of frozen storage. DH-01 was stable for at least 3 months in muscle.

Definition of the residue

Plant commodities

In plant metabolism studies on benzpyrimoxan in rice, benzpyrimoxan (48–61 percent TRR) was a major component in all rice commodities (grains, hull and straw). DH-04 (5.0–17 percent TRR) was identified at 0.02–0.39 mg eq/kg in all rice commodities. DH-02 conjugate, DH-05 (free and conjugated), DH-06 (free and conjugated) were detected at > 0.01 mg eq/kg and not greater than 4.5 percent TRR in feed commodities (panicle, foliage, hull and straw).

The Meeting decided that the suitable analyte for enforcement purposes is parent benzpyrimoxan in rice commodities.

In deciding which compounds should be included in the residue definition for dietary risk assessment, the Meeting considered the toxicological properties of the candidate DH-04 (benzpyrimoxan-2-OH). The Meeting agreed that DH-04 was approximately 3 times more toxic than parent.

The Meeting decided that the suitable analytes for dietary risk assessment are benzpyrimoxan and benzpyrimoxan-2-OH.

Animal commodities

In animal metabolism studies, benzpyrimoxan was the predominant component in hen muscle (43 percent TRR, 0.006 mg eq/kg) and hen fat (71 percent TRR, 0.043–0.089 mg eq/kg), but the minor residue in goat liver (2.3 percent TRR, 0.014 mg/kg) and eggs (5.7–13 percent TRR, 0.002–0.004 mg/kg), and not detected in hen liver, goat kidney, goat muscle, goat fat and milk.

Benzpyrimoxan-acid-2-OH (DH-05) was the predominant component of the residue in goat kidney (40–47 percent TRR) and milk (59–86 percent TRR), and the major residue in liver (goat: 6.4–11 percent TRR, hen: 20–28 percent TRR).

Benzpyrimoxan-acid (DH-01) was only found in goat kidney (16–20 percent TRR) and it may be converted to DH-05 in liver, kidney and fat during storage and/or analysis. The Meeting considered that interconversion of DH-01 to DH-05 may have occurred in the matrices due to the storage interval between sampling and extraction of 24–50 days in animal metabolism studies.

The Meeting decided that the suitable analytes for enforcement purposes are parent benzpyrimoxan, DH-01 and DH-05 in animal commodities.

The metabolism and feeding studies for ruminant show that total residues of benzpyrimoxan, DH-01 and DH-05 in skim milk are 1.5 times higher than in cream. Laying hen metabolism study shows that total residues in fat are higher than in muscle, but the ratio does not show a clear fat solubility.

The Meeting considered the residues of benzpyrimoxan not to be fat-soluble.

In deciding which compounds should be included in the residue definition for dietary risk assessment, the Meeting considered the likely occurrence of the compound and the toxicological properties of the candidates DH-01, DH-02 (free and conjugated), DH-05 and DH-06 (free and conjugated).

DH-02 (free and conjugated) was a major metabolite in only goat liver (3.1–22 percent TRR, 0.019–0.051 mg eq/kg) and kidney (5.9–16 percent TRR, 0.011–0.039 mg eq/kg) and DH-06 (free and conjugated) only in goat liver (4.8–15 percent TRR, 0.029–0.036 mg eq/kg).

Since liver and kidney contributes little to the total dietary exposure and an ARfD for benzpyrimoxan was not considered necessary, the Meeting concluded that these metabolites should not be included in the residue definition for dietary risk assessment.

The Meeting concluded that the toxicities of DH-01 (benzpyrimoxan-acid) and DH-05 (benzpyrimoxan-acid-2-OH) were covered by the health-based guidance values for parent compound.

The Meeting decided to define the residue for dietary risk assessment for animal commodities as the sum of benzpyrimoxan, benzpyrimoxan-acid and benzpyrimoxan-acid-2-OH, expressed as benzpyrimoxan.

The Meeting recommended the following residue definitions for benzpyrimoxan:

Definition of the residue for compliance with the MRL for rice commodities: *Benzpyrimoxan*

Definition of the residue for dietary risk assessment for rice commodities: *Sum of benzpyrimoxan and 3 × benzpyrimoxan-2-OH, expressed as benzpyrimoxan*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *Sum of benzpyrimoxan, benzpyrimoxan-acid and benzpyrimoxan-acid-2-OH, expressed as benzpyrimoxan*

The Meeting considers the residue not to be fat-soluble.

Results of supervised residue trials on crops

Supervised trials were available for the use of benzpyrimoxan on rice.

Product labels were available from Japan.

Total residues for estimation of dietary exposure from food commodities are calculated by summing up the concentrations of benzpyrimoxan and 3 × benzpyrimoxan-2-OH (expressed as benzpyrimoxan equivalents), because benzpyrimoxan-2-OH is three times higher toxicity than parent benzpyrimoxan. The method of calculation is illustrated below.

Example of the method for calculation of total residues (mg/kg) for dietary exposure

Benzpyrimoxan	Benzpyrimoxan-2-OH (DH-04)	Total
< 0.01	< 0.01 × 3	< 0.04
< 0.01	0.025 × 3	0.085

For the purpose to calculating the livestock animal dietary burden, no factor of 3 is applied to the residue levels of the metabolite. Total residues are benzpyrimoxan + benzpyrimoxan-2-OH, expressed as benzpyrimoxan.

As no studies on residues in follow-on and rotational crops were provided to the Meeting, no estimations for maximum residue levels, STMR and median residues levels in annual crops sown/planted after rice cultivation could be made. The supervised residue trials with direct application and studies on rotational crops will be evaluated together at a future meeting when all results will be available.

Rice

The critical GAP for rice in Japan allows three spray applications of 0.01 kg ai/hL with a PHI of 7 days. Data were available from supervised trials on rice in Japan matching Japanese GAP.

Benzpyrimoxan residues in rice grain with husk were (n=5): 1.2, 1.6, 1.9, 2.0 and 3.4 mg/kg.

Total residues in rice grain were (n=5): 1.7, 2.8, 3.7, 3.9 and 5.6 mg/kg.

For the purposes to calculate the livestock animal dietary burden residues (benzpyrimoxan plus DH-04, expressed as benzpyrimoxan) in rice grain were (n=5): 1.4, 2.0, 2.5, 2.6 and 4.1 mg/kg.

As indicated no recommendation could be made for rice. Benzpyrimoxan residues in husked rice were (n=8): 0.06, 0.10, 0.20, 0.30, 0.32, 0.33, 0.44 and 0.46 mg/kg.

Total residues in husked rice were (n=8): 0.15, 0.19, 0.41, 0.72, 0.74, 0.75, 0.79 and 0.89 mg/kg.

As indicated no recommendation could be made for rice, husked.

Residues in animal feeds

Rice, hay and/or straw

The critical GAP for straw of rice in Japan allows three spray applications of 0.01 kg ai/hL with a PHI of 7 days. Data were available from supervised trials on rice in Japan matching the GAP.

Benzpyrimoxan residues in rice straw were (n=8): 4.2, 4.7, 5.6, 5.8, 7.2, 7.8, 8.1 and 9.0 mg/kg on dry weight basis.

Total residues (benzpyrimoxan plus DH-04, expressed as benzpyrimoxan) in rice straw were (n=8): 5.5, 6.2, 6.8, 7.2, 9.0, 9.7, 10 and 11 mg/kg on dry weight basis.

No recommendation could be made for rice straw until the contribution of residues from direct application as well as uptake through the soil can be assessed.

Fate of residues during processing

High temperature hydrolysis

The hydrolysis of [^{14}C]-benzpyrimoxan was studied in sterile buffered solutions of pH 4, 5 and 6. [Phenyl-U-^{14}C] and [Pyrimidinyl-4(6)-^{14}C]-benzpyrimoxan were incubated at 1.0 mg/L in aqueous buffered solutions to simulate common processing practices (pasteurization, baking/boiling and sterilization).

At pH 4, 5 and 6 with heating, the predominant residue was parent benzpyrimoxan (92–100 percent AR). Some minor degradation products (benzpyrimoxan-aldehyde (DH-03), benzpyrimoxan-enamine-aldehyde (DH-08) and benzpyrimoxan-benzyl alcohol (DH-102)) were identified with up to 7.4 percent AR.

Benzpyrimoxan residue is stable during processing and no DH-04 residue is expected.

Residues in processed commodities

The Meeting received information on the fate of total residues of benzpyrimoxan and benzpyrimoxan-2-OH (DH-04) during processing in rice, husked. Calculated processing factors are summarised in the following table.

Table 55 Processing factors for rice, husked and STMR-P values

Raw commodity	Processed commodity	Calculated processing factor[#] [best estimate]
Rice, husked = Brown rice	Bran	8.6, 9.5 [9.1]
	Polished rice	0.41, 0.43 [0.42]
	Cooked polished rice	0.068, 0.095 [0.082]

[#] Each value represents a separate study. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

Residues in animal commodities

Farm animal feeding studies

The Meeting received a lactating dairy cow and a laying hen feeding studies, which provided information on likely residues resulting in animal commodities, milk and eggs from benzpyrimoxan in the animal diet.

Lactating dairy cows

Holstein dairy cows were dosed with benzpyrimoxan for 28 days at the equivalent of 8, 24 and 80 ppm in the diet. Residues of benzpyrimoxan, and benzpyrimoxan-acid-2-OH (DH-05) in milk, and residues of benzpyrimoxan, benzpyrimoxan-acid (DH-01) and DH-05 in tissues (liver, kidney, muscle and fat) were determined.

For whole milk, no benzpyrimoxan residue was detected at all feeding levels. DH-05 residue was detected at all feeding level and achieved a plateau concentration of 0.012–0.034 mg/kg at the 8 ppm (1 ×) feeding level after 3 days of dosing.

For liver, residues of benzpyrimoxan and DH-05 were detected at all feeding levels (benzpyrimoxan: < 0.01–0.15 mg/kg, DH-05: 0.011–0.25 mg/kg). No residue of DH-01 was found at any feeding level.

For kidney, benzpyrimoxan residues were found at the 80 ppm feeding level (1 cow: 0.031 mg/kg). DH-01 residue was detected with < 0.01–0.032 mg/kg at the 24 and 80 ppm feeding levels. DH-05 residue was detected with 0.032–0.30 mg/kg at all feeding levels.

For fat, benzpyrimoxan residue was detected with < 0.01–0.031 mg/kg at the 80 ppm feeding level. DH-05 residue was detected with < 0.01–0.18 mg/kg at all feeding levels. No DH-01 residue was found at any feeding level.

For muscle, no residues (< 0.01 mg/kg) were found at any feeding level.

Laying hens

Laying hens were dosed with benzpyrimoxan for 29 days at the equivalent of 3, 9 and 30 ppm in the diet. Residues of benzpyrimoxan and DH-05 were determined in eggs, liver, muscle and fat.

For eggs, DH-05 residue was found (< 0.01–0.011 mg/kg) at the 30 ppm feeding level. Benzpyrimoxan residue was below the LOQ (< 0.01 mg/kg) at any feeding level.

For liver, DH-05 residue was detected with < 0.01–0.067 mg/kg at all feeding levels. No benzpyrimoxan residue was found at any feeding level.

For fat, benzpyrimoxan residue was detected with 0.012–0.015 mg/kg at the 30 ppm feeding level. No DH-05 residue was found at any feeding level.

For muscle, no residues (< 0.01 mg/kg) were found at any feeding level.

Farm animal dietary burden

The Meeting noted that the studies on residues in follow-on and rotational crops were not provide to the Meeting, and decided not to estimate maximum residue levels and STMRs on annual crops that may lead to animal feeds. The estimations will be made in the future when the studies are available.

RECOMMENDATIONS

Definition of the residue for compliance with the MRL for plant commodities: *Benzpyrimoxan*

Definition of the residue for dietary risk assessment for plant commodities: *Sum of benzpyrimoxan and benzpyrimoxan-2-OH, expressed as benzpyrimoxan*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *Sum of benzpyrimoxan, benzpyrimoxan-acid and benzpyrimoxan-acid-2-OH, expressed as benzpyrimoxan*

The residue is not fat-soluble.

FUTURE WORK OR INFORMATION

Desirable information:

- Submission of existing soil dissipation studies.
- Information on residues in rotational crops (confined rotational crop study and/or field residue trials on rotational crops)

DIETARY RISK ASSESSMENT

The Meeting could not make any recommendations for residue levels in crops since information on the residues in follow-on and rotational crops was not provided to the Meeting. Furthermore, no dietary exposure assessment was conducted.

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BIFENTHRIN (178)

First draft prepared by Dr G Ye, Ministry of Agriculture and Rural Affairs, Beijing, Republic of China

EXPLANATION

Bifenthrin is a pyrethroid insecticide and miticide. It was first evaluated for residues and toxicology by the JMPR in 1992. Bifenthrin was evaluated under periodic review programme in 2009 (T) and 2010 (R), and subsequently evaluated in 2015 and 2019 for additional MRLs.

An ADI of 0–0.01 mg/kg bw and an ARfD of 0.01 mg/kg bw were established by the 2009 JMPR. The residue definition for compliance with the MRL and for estimation of dietary intake (for animal and plant commodities) is bifenthrin (sum of isomers). The residue is fat-soluble.

Bifenthrin was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional MRLs by the 2021 JMPR, which was delayed to the 2022 JMPR.

The Meeting received information on GAPs and supervised residue trials for apple, peach, avocado, pomegranate, peppers (bell, non-bell), melon, spinach, and peanut, as well as the processing studies on apple and peanut.

METHODS OF RESIDUE ANALYSIS

A number of analytical methods (for enforcement and data collection) for plant and animal matrices were evaluated by the 2010, 2015 and 2019 Meeting. Residue analytical methods consisted of extraction with acetone or hexane, cleaning-up with SPE, and determination by GC-ECD and GC-MSD. Typical LOQs achieved for plant and animal commodities fall in the range of 0.01–0.05 mg/kg.

The Meeting received descriptions and concurrent recovery data for analytical methods for residues of bifenthrin used in supervised trials. The methods used in trials are either similar to or slight modification of methods evaluated by previous JMPRs. These methods are characterized by acetone extraction, partitioning of residues into hexane, clean-up using Florisil, and analysis by GC-ECD

Method P-3526 (Culligan J.F. 2001, report no. P-526): Samples were homogenised and extracted by shaking in 15 mL of acetone for 15 minutes, and partitioned twice with hexane. An aliquot was cleaned up with silica gel solid phase extraction column. Bifenthrin was quantified by GC-ECD. The LOQ was of 0.05 mg/kg for bifenthrin in the apple matrix (fruit, juice and pomace), peach fruit, avocado fruit and pomegranate.

Method P-2132M (Ridler, J.E., 1989, P-2132M P.42): Samples were homogenised and extracted with acetone, and partitioned twice with hexane, cleaned-up with Florisil solid phase extraction column. Bifenthrin is quantified by GC-ECD. The LOQs were of 0.1, 0.055, 0.048 and 0.05 mg/kg for bifenthrin in cantaloupe, bell pepper, non-bell pepper and spinach.

Method P-2715 (Kim, I., 1992): Homogenised samples were extracted twice with acetone, partitioned with hexane and were cleaned up with a Florisil solid phase extraction column. Bifenthrin is quantified by GC-ECD. The LOQ of 0.05 mg/kg was validated for bifenthrin in spinach.

Method P-3457(Chen A., 2000): Homogenised samples were extracted with acetone and sodium chloride solution, partitioned with hexane, concentrated under nitrogen and were cleaned up with Florisil solid phase extraction column. Bifenthrin is quantified by GC-MSD. The LOQ was of 0.05 mg/kg for bifenthrin in peanut meat.

Method P-0130 (Winkler, D, A, 1992, MRID # 41492605; evaluated by the 2010 JMPR): Samples were homogenised and extracted by ultrasonicating with acetone, partitioned with cyclohexane and were cleaned up with a silica gel solid-phase extraction column. Bifenthrin is quantified by GC-ECD. The LOQ was of 0.05 mg/kg for bifenthrin in peanut meat and hay, 1.0 mg/kg in peanut vine and 2.0 mg/kg in peanut hay.

Method P-2763 (Kim, I., 1992, P-2763; evaluated by 2010 JMPR): Nutmeat and meal samples of processed peanut were extracted with acetone followed by ultrasonication, and further extracted with acetonitrile. Oil samples were extracted with acetonitrile, partitioned with cyclohexane and were cleaned up with a silica gel cartridge solid phase extraction. Bifenthrin is quantified by GC-ECD. The LOQ was of 0.05 mg/kg for bifenthrin in peanut nutmeat, meal and refined oil.

Concurrent recovery data for the methods used for determination of bifenthrin residues in plant commodities for which supervised trial data were submitted to the current Meeting are summarized below.

Table 1 The concurrent recoveries of analytical method for bifenthrin in related commodities

Crop	Fortification (mg/kg)	n	Range Recovery (percent)	Mean recovery (percent)	CV percent	Reference
Apple, fruit	0.05 0.125 0.5 5	10 1 14 3	69-84 86 73-85 76-77	75 84 79 77	4 4	P-3526
Apple, juice	0.05 0.5 5	8 4 3	71-89 74-90 76-81	78 80 79	7 7 2	P-3526
Apple, pomace	0.05 0.5 5	6 3 3	71-86 78-85 70-80	79 81 77	7 4 6	P-3526
Peach, fruit	0.05 0.5	6 7	82-102 71-99	96 88	8 12	P-3526
Avocado, fruit	0.05 0.5	6 2	69-82 83-97	75 90	6	P-3526
Pomegranate	0.05 0.5	4 2	77-84 96-90	81 93	3	P-3526
Cantaloupe	0.1	3	97-99	98	0.9	P-213M
Bell pepper	0.055 0.012	9 2	74-88 88-94	84 91	6.1	P-213M
Non-bell pepper	0.048 0.55 0.12	6 7 2	77-98 77-92 75-84	88 86 80	7 5	P-213M
Spinach	0.05	5	83-103	96	12	P-213M
Spinach	0.05 0.25 0.5 1 2	2 2 2 2 1	101-103 100-107 91-99 93-107 97	102 104 95 100 97		P-2715
Peanut, nutmeat	0.05	3	86-114	97	15	P-3457
Peanut, nutmeat	0.05 0.5	7 1	94-105 82	101 82	4	P-0130
Peanut, vine	2 5	1 3	91 73-87	91 79	 7	

Crop	Fortification (mg/kg)	n	Range Recovery (percent)	Mean recovery (percent)	CV percent	Reference
Peanut, hay	1 5 10 15	1 3 7 1	108 81-105 69-96 85	108 90 79 85	 13 11	
Peanut hulls	0.05 0.25 0.5	4 3 1	88-123 71-91 76	105 82 76	15 10	
Peanut, nutmeat	0.05 0.1	1 1	68 78			P-2763
Peanut, meal	0.05 0.1	1 1	90 68			P-2763
Peanut, refined oil	0.05 0.1	1 1	85 69			P-2763

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

The maximum storage intervals in the residue trials were covered by storage stability of at least 18 months in high acid, 49 months in high water, 36 months in high oil and high starch, and 15 months in high protein commodities stored at -18 ˚C concluded by 2010 and 2015 JMPR.

USE PATTERNS

Bifenthrin is registered for use in avocado, pome fruit, peach, pomegranate, melon, spinach, peanut, and okra in the United States. Registered use patterns are summarised in Table 2.

Table 2 Registration information on foliar applications of bifenthrin (EC formulation) in the United States

Crop	Formulation	Application				PHI (days)	Remark
	kg ai/L	Rate (kg ai/ha)	Water (L/ha)	RTI (min, days)	Max no.		
Pome fruit*	0.24	0.0448-0.224	1870 (ground dilute spray)/ 467 (ground conc. spray)/ 93(aerial)	30	3	14	Do not apply more than 0.56 kg ai./ha per year with no more than 0.51 kg ai/ha applied after petal fall (BBCH 69). Do not graze livestock in treated orchards or cut treated cover crops for feed
Peach subgroup**	0.24	0.0448-0.224		30	3	14	
Avocado	0.111 g/kg (bifenthrin) + 0.037 g/kg (zeta cypermethrin)	0.062 (bifenthrin) 0.021(zeta-cyp)	884 (ground)/93 (air)	14	5	1	Do not apply more than 0.31 (bifenthrin）+0.104(zeta-cypermethrin) kg ai/ha per year
Pomegranate	0.24	0.112-0.224	467	14	3	14	Do not apply more

Crop	Formulation	Application				PHI (days)	Remark
	kg ai/L	Rate (kg ai/ha)	Water (L/ha)	RTI (min, days)	Max no.		
							than 0.561 kg ai./ha per year
Melon	0.24	0.0448-0.112	187(ground)/ 47 (air)	7	3	3	Do not apply more than 0.336 kg ai./ha per year no more than 2 applications after bloom
Pepper/eggplant (subgroup 8-10B)***	0.24	0.0336-0.112	93 (ground/ 19 (air)	7	2	7	Do not apply more than 0.224 kg ai/ha per season
Spinach	0.24	0.0336-0.112	47-470 (air)/93-47-0(ground)	7	4	40	Do not apply more than 0.45 kg ai/ha per year
Peanut	0.24	0.0336-0.112	93 (ground/ 19 (air)	14	5	14	Do not apply more than 0.56 kg ai/ha per year Do not feed green immature plants and peanut hay to livestock

Notes:

*Pome fruit (US group 11-10) includes: Apple, Azarole, Crabapple, Loquat, Mayhaw, Medlar, Pear, Pear, Asian, Quince, Tejocote.

**Peach subgroup ((US subgroup 12-12B) includes: Peach; Nectarine.

***Pepper/eggplant (US subgroup 8-10B) includes: African eggplant; bell pepper; eggplant; martynia; non-bell pepper; okra; pea eggplant; pepino; roselle; scarlet eggplant.

Supervised field residues trials

The Meeting received information on supervised field trials for bifenthrin on pome fruit (apple and pear), peach, avocado, pomegranate, melon, bell and non-bell pepper, spinach and peanut

Crop	Table No.
Apple	3
Peach.	4
Avocado	5
Pomegranate	6
Melon	7
Bell pepper	8
Non-bell pepper	9
Spinach,	10, 11
Peanut,	12, 13
Peanut vine, hay, hull	14

Trials were generally documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Unless stated otherwise, residue data are recorded unadjusted for recovery.

Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

Conditions of the supervised residue trials were generally included in field reports. Trial designs used non-replicated plots. Field reports provided data on the sprayers used, plot size, field sample size and sampling date.

In trials where replicate field samples were taken from a single plot and analysed separately, or where duplicate analyses of the same sample were made, the average of residue values from the trials conducted according to the ± 25 percent of maximum total seasonal application rate according to the GAP has been used for the estimation of maximum residue levels. Those results included in the tables are underlined.

Pome Fruits (002)

Apple

Twelve supervised trials on apple were conducted in the United States during 2013-2014 growing seasons (Samoil, K.S., 2015a, IR-4 PR No. 11016). Apples were treated with 3 foliar applications of 25 percent bifenthrin EC, retreatment interval of 19–23 days, except one trial (C001) with 4 foliar applications. The first application targeted a rate of 0.11 kg ai/ha and the second and third applications targeted a rate of 0.22 kg ai/ha. All applications were made using appropriate spray equipment. Adjuvants were used in the application. Duplicate samples were harvested 13-15 days after the last application (DALA), and for decline trials 7, 10–11, 17–18, and 21 DALA. The maximum storage intervals for field-treated samples were 469 days (fruit), 482 days (juice), and 466 days (pomace), which were covered by the storage stability studies. Residues of bifenthrin were determined using Method P-3526. Results of the trials are summarized in Table 3.

Table 3 Residues of bifenthrin from supervised trials on apples treated with 25 percent EC in the United States (IR-4 PR No. 11016)

Location, Country, year (Variety)	Application/ treatment			Total/ season kg ai/ha	DALA	Commodity	Residue, mg/kg
	kg ai/ha	L water/ha	No (RTI, days)				
GAP, United States	0.0448-0.224	1870 (grd dilute spray)/ 467 (grd conc. spray)/ 93(aerial)	3(30,30)	0.561	14		
Parlier, CA, United States, 2013 (Granny Smith)	0.114 0.225 0.225	701 692 701	3 (21, 21)	0.565	14	Fruit	0.286 (0.237, 0.334)

Location, Country, year (Variety)	Application/ treatment			Total/ season kg ai/ha	DALA	Commodity	Residue, mg/kg
	kg ai/ha	L water/ha	No (RTI, days)				
Hotchkiss, CO, United States, 2013* (Gala)	0.112 0.111 0.112 0.224	561 552 561 561	4 (21, 1, 19)	0.559	14	Fruit	0.435 (0.422, 0.448)
Twin Falls, ID, United States, 2013 (Red Delicious)	0.112 0.224 0.226	935 935 944	3 (20, 20)	0.562	15	Fruit	0.238 (0.207, 0.268)
Prosser, WA, United States, 2013 (Gala)[1]	0.113 0.241 0.224	832 832 795	3 (21, 21)	0.578	13	Fruit	0.105 (0.100, 0.110)
Prosser, WA, United States, 2013 (Fuji)[2]	0.111 0.224 0.227	1814 1795 1832	3 (20, 21)	0.562	7 10 14 17 21	Fruit	0.125 (0.164, 0.0860) 0.131 (0.0987, 0.164) 0.133 (0.158, 0.107) 0.110 (0.105, 0.115) 0.0882 (0.0743, 0.102)
Prosser, WA, United States, 2013 (Ginger Gold)[3]	0.112 0.226 0.226	1458 1458 1458	3 (19, 23)	0.565	13	Fruit	0.0912 (0.118, 0.0644)
Fennville, MI, United States, 2013 (Golden Smoothie)[4]	0.112 0.224 0.225	729 748 720	3 (21, 21)	0.561	14	Fruit	0.422 (0.446, 0.397)
Fennville, MI, United States, 2013 (Jonamac)[5]	0.111 0.225 0.223	972 972 944	3 (21, 21)	0.559	14	Fruit	0.268 (0.288, 0.248)
Cream Ridge, NJ, United States, 2013 (Rome)[6]	0.111 0.230 0.224	935 926 944	3 (22, 20)	0.565	14	Fruit	0.162 (0.156, 0.167)
Cream Ridge, NJ, United States, 2013 (Red Delicious)[7]	0.115 0.227 0.229	1954 1917 1898	3 (22, 21)	0.571	14	Fruit	0.201 (0.215, 0.186)

Location, Country, year (Variety)	Application/ treatment			Total/ season kg ai/ha	DALA	Commodity	Residue, mg/kg
	kg ai/ha	L water/ha	No (RTI, days)				
Cream Ridge, NJ, United States, 2014 (Red Delicious)	0.114 0.226 0.226	841 879 907	3 (21, 21)	0.567	7 11 14 18 21	Fruit	0.121 (0.120, 0.122) 0.199 (0.219, 0.179) 0.192 (0.207, 0.177) 0.115 (0.120, 0.110) 0.162 (0.208, 0.116)
Lansing, NY, United States, 2013 (Acey Mac)	0.112 0.224 0.226	654 654 664	3 (21, 21)	0.562	15	Fruit	0.102 (0.125, 0.0794)

Notes:

[1] Prossar (Gala) application date: 2013-06-26, 07-17, 08-07.

[2] Prossar (Fuji) application date: 2013-08-14, 09-03, 09-24.

[3] Prossar (Ginger gold) application date: 2013-07-10, 07-29, 09-03.

[4] Fennville, (Golden Smoothie) application date: 2013-08-09, 08-30, 09-20.

[5] Fennville (Jonamac) application date: 2013-08-09, 08-30, 09-20.

[6] Cream Ridge (Rome) application date:2013-08-07, 08-29, 09-18.

[7] Cream Ridge (Red Delicious) application date: 2013-07-29, 08-20, 09-10.

* The second application was mistakenly made at 0.111 g ai/ha; an additional application at 0.112 g ai/ha was made one day after the second. A fourth application was made 19 days after the third at the correct rate of 0.224 g ai/ha.

Stone fruit (003)

Peach

Twelve supervised trials on peach were conducted in the United States during 2013-2014 growing seasons (Samoil, K.S., 2015b, IR-4 PR No. 11017). Peaches were treated with 3 foliar applications of 25 percent bifenthrin EC, retreatment interval of 20–23 days except one trial (AR09) with 4 foliar applications. The first application targeted a rate of 0.11 kg ai/ha and the second and third applications targeted a rate of 0.22 kg ai/ha. All applications were made using appropriate spray equipment. Adjuvants were used in the application. Samples were harvested 10–15 DALA, and in the decline trial at 6, 10–11, 17 and 21 DALA. The maximum storage interval for field-treated samples (12 months) was covered by the storage stability studies. Residues of bifenthrin were determined using Method P-3526. Results of the trials are summarized in Table 4.

Table 4 Residues of bifenthrin from supervised trials on peach following application 25 percent EC (IR-4 PR No. 11017)

City, State/Region, Country/ Year (variety)	Application				Matrix	DALA	Average residues (mg/kg) (individual values)
	kg ai/ha	L water/ha	No (RTI, days)	Total rate (kg ai/ha)			
GAP, United States	0.0448-0.224	1870 (grd dilute spray)/ 467 (grd conc. spray)/ 93 (aerial)	1-3(30,30)	0.561		14	
Clarksville, AR, United States, 2013 (Cresthaven)	0.113 0.226 0.222 0.222	1178 1178 1197 1225	4 (20, 23, 20)	0.783 (>25 percent)	Fruit without pits	10 (>25 percent)	0.116 (0.117, 0.114)
Parlier, CA, United States, 2013 (June Flame)[1]	0.113 0.222 0.225	477 477 467	3 (21, 21)	0.560	Fruit without pits	14	0.199 (0.205, 0.193)
Parlier, CA, United States, 2013 (Cresthavent/Henry II)[2]	0.112 0.223 0.226	1103 1113 1113	3 (22, 20)	0.561	Fruit without pits	14	0.381 (0.298, 0.463)
Winters, CA, United States, 2013 (O'Henry)	0.115 0.230 0.239	1103 1103 1141	3 (21,21)	0.584	Fruit without pits	6	0.224 (0.204, 0.243)
						10	0.292 (0.290, 0.294)
						14	0.257 (0.283, 0.231)
						17	0.231 (0.237, 0.225)
						21	0.190 (0.233, 0.246)
Winters, CA, United States, 2014 (Lori-May)	0.119 0.224 0.218	795 748 729	3 (21, 22)	0.561	Fruit without pits	13	0.122 (0.119, 0.124)
Fennville, MI, United States, 2013 (Red Haven)	0.112 0.223 0.221	963 972 954	3 (21, 21)	0.556	Fruit without pits	15	0.116 (0.105, 0.127)
Clayton, NC, United States, 2013 (Contender)	0.111 0.224 0.226	729 748 748	3 (22, 22)	0.561	Fruit without pits	14	0.299 (0.323, 0.275)
Jackson Springs, NC, United States, 2013 (Contender)	0.114 0.232 0.230	589 598 589	3 (22, 22)	0.576	Fruit without pits	14	0.410 (0.326, 0.494)
Cream Ridge, NJ, United States, 2013 (John Boy)[3]	0.112 0.225 0.225	720 776 767	3 (21, 21)	0.562	Fruit without pits	14	0.221 (0.229, 0.213)
Cream Ridge, NJ, United States, 2013 (Suncrest)[4]	0.112 0.226 0.226	1692 1776 1823	3 (21, 20)	0.565	Fruit without pits	14	0.238 (0.269, 0.206)
Cream Ridge, NJ, United States, 2014 (John Boy)	0.113 0.225 0.226	823 804 804	3 (21, 21)	0.565	Fruit without pits	13	0.168 (0.155, 0.180)
Fredericksburg, TX, United States, 2013 (Redskin)	0.112 0.225 0.224	654 636 654	3 (10, 20)	0.561	Fruit without pits	14	0.199 (0.204, 0.193)

Notes:

[1] Parlier (June Flame) application date: 2013-05-08, 05-29, 06-19.

[2] Parlier (Cresthavent/Henry II) application date: 2013-06-10, 07-02, 07-22.

[3] Cream Ridge (John Boy) application date: 2013-06-04, 06-25, 07-16.

[4] Cream Ridge (Suncrest) application date: 2013-06-20, 07-11, 07/31.

Assorted Tropical and Sub-Tropical Fruits – Inedible Peel (006)

Avocado

Six supervised trials on avocado were conducted in the United States during 2013 growing seasons (Samoil, K.S., 2015c, IR-4 PR No. 10578). Avocados were treated with 5 foliar applications of 25 percent bifenthrin EC, retreatment interval of 14-17 days. The application rates were 0.081–0.091 kg ai/ha. All applications were made using appropriate spray equipment. Adjuvants were used in the application. Duplicate samples were harvested 1 DALA and at 0, 1, 3, 6 and 10 DALA in the decline trials. The maximum storage interval for field-treated samples (17 month) was covered by storage stability studies. Residues of bifenthrin were determined using Method P-3526. Results of the trials are summarized in Table 5.

Table 5 Residues of bifenthrin from supervised trials on avocado in the United States following the application of 25 percent EC (IR-4 PR No. 10578)

City, State/Region, Country/Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues ppm (mg/kg) (individual values)
	Rate (kg ai/kg)	Spray volume L/ha	No (RTI, days)				
GAP, United States	0.062	888 (ground) 93 (air)	1-5 (14)	0.31		1	
Irvine, CA, United States, 2013 (Hass)[1]	0.084 0.084 0.084 0.084 0.084	897 897 888 888 897	5 (15, 14, 14, 14)	0.421	Fruit without stem and pit	0	0.257 (0.268, 0.245)
						1	0.249 (0.246, 0.251)
						3	0.295 (0.313, 0.276)
						6	0.304 (0.295, 0.312)
						10	0.150 (0.159, 0.140)
Irvine, CA, United States, 2013 (Hass)[2]	0.085 0.084 0.084 0.084 0.084	1234 1225 1225 1225 1225	5 (14, 14, 14, 14)	0.421	Fruit without stem and pit	1	0.138 (0.122, 0.153)
Exeter, CA, United States, 2013 (Hass)	0.085 0.085 0.085 0.085 0.085	1122 1094 1122 1131 1141	5 (15, 16, 14, 15)	0.426	Fruit without stem and pit	1	0.122 (0.111, 0.133)

City, State/Region, Country/Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues ppm (mg/kg) (individual values)
	Rate (kg ai/kg)	Spray volume L/ha	No (RTI, days)				
Homestead, United States, 2013 (Hass)	0.081 0.081 0.081 0.082 0.081	907 907 907 907 907	5 (14, 15, 13, 16)	0.404	Fruit without stem and pit	1	0.264 (0.284, 0.243)
Miami, FL, United States, 2013 (Hass)	0.085 0.085 0.083 0.083 0.083	1178 1178 1150 1150 1150	5 (17, 14, 14, 14)	0.419	Fruit without stem and pit	1	0.0761 (0.0706, 0.0816)
Juana Diaz, PR, United States, 2013(Hass)	0.091 0.086 0.089 0.089 0.085	1309 1243 1262 1281 1234	5 (15, 14, 14, 14)	0.439	Fruit without stem and pit	1	0.0800 (0.0765, 0.0835)

Notes:

[1]: Irvine (Hass) application date: 2013-05-13, 05-28, 06-11, 06-25, 07-09.

[2]: Irvine (Hass) application date: 2013-05-22, 06-05, 06-19, 07-03, 07-17.

Pomegranate

Four supervised trials on pomegranate were conducted in the United States during 2014 growing season (Samoil, K.S., 2016, IR-4 PR No. 11249). The pomegranates were treated with 3 foliar applications of 25 percent bifenthrin EC, retreatment interval of 14 days. The first application targeted a rate of 0.11 kg ai/ha and the second and third applications targeted a rate of 0.22 kg ai/ha. All applications were made using appropriate spray equipment. Adjuvants were used in the application. Duplicate samples were harvested 14 DALA or at 6, 11, 14, 18 and 21 DALA for the decline trials. The maximum storage interval for field-treated samples (13 months) was covered by storage stability studies. Residues of bifenthrin were determined using Method P-3526. Results of the trials are summarized in Table 6.

Table 6 Residues of bifenthrin from supervised trials on pomegranate in the United States following the application of 25 percent EC (IR-4 Study No. 11249)

Location, Country/Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues (mg/kg) (individual values)
	Rate kg ai/ha	Water L/ha	No (RTI, days)				
GAP, United States	0.0112-0.224	467	(14)	0.561		14	
Parlier, CA, United States, 2014 (Wonderful)[1]	0.113 0.226 0.227	916 897 907	3 (14, 14)	0.567	Fruit	6	0.212 (0.171, 0.253)
						11	0.218 (0.244, 0.191)
						14	0.177 (0.214, 0.140)

Location, Country/Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues (mg/kg) (individual values)
	Rate kg ai/ha	Water L/ha	No (RTI, days)				
						18	0.180 (0.199, 0.160)
						21	0.140 (0.178, 0.102)
Parlier, CA, United States, 2014 (Wonderful)[2]	0.113 0.231 0.231	495 505 505	3 (14, 14)	0.575	Fruit	14	0.177 (0.216, 0.138)
Yuba City, CA, United States, 2014 (Wonderful)	0.112 0.223 0.221	608 608 598	3 (14, 14)	0.556	Fruit	14	0.109 (0.109, 0.109)
Davis, CA, United States, 2014 (Wonderful)	0.119 0.227 0.238	645 617 645	3 (14, 14)	0.584	Fruit	14	0.163 (0.156, 0.169)

Notes:

[1] Application date: 2014-09-04, 09-18, 10-02, with no adjuvant.

[2] Application date: 2014-09-03, 09-17, 10-01, with adjuvant.

Cucurbits – inedible peel, melons, except watermelon (011B)

Melon (cantaloupe)

Seven supervised trials on cantaloupe were conducted in the United States during 1990 growing seasons (Biehn, W.L., 1996, IR-4 PR No. 4151). Cantaloupe were treated with 3 foliar applications at a rate of 0.112 kg ai/ha of 25 percent bifenthrin EC The second applications were conducted 30-39 days after first application except one trial (Test 05, 52 days), and the third applications 7-8 days after second application except 38 days in one trial (Test 07). All applications were made using appropriate spray equipment. No adjuvants were used in the application. Duplicate samples were harvested 0, 3 and 7–8 days after the last application. The maximum storage intervals for field-treated samples (17 months) were covered by storage stability studies. Residues of bifenthrin were determined using Method P-213M. Results of the trials are summarized in Table 7.

Table 7 Residue of bifenthrin in melon following application 25 percent EC (IR-4 Report No. 4151)

Location, Country/Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues (mg/kg) (individual values)
	Rate kg ai/ha	Water L/ha	No (RTI, days)				
GAP, United States	0.0448-0.112	140 (grd) 47 (air)	1-3(7)	0.337		3	
Holtville, CA, United States, 1990 (Topmark)	0.112 0.112 0.112	47 47 47	3 (39, 7)	0.336	Whole Fruit	0	<0.1 (<0.1, <0.1)
						3	<0.1 (<0.1, <0.1)
						8	<0.1 (<0.1, <0.1)
	0.112	171	3 (39, 7)	0.336	Whole Fruit	0	<0.1 (<0.1, <0.1)

Location, Country/Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues (mg/kg) (individual values)
	Rate kg ai/ha	Water L/ha	No (RTI, days)				
	0.112 0.112	171 171					
						3	0.11 (0.12, <0.1)
						8	<0.1 (<0.1, <0.1)
					Peel	0	0.13 (0.16, <0.1)
						3	0.18 (0.15, 0.20)
						8	0.12 (0.13, <0.1)
					flesh	0	<0.1 (<0.1, <0.1)
						3	<0.1 (<0.1, <0.1)
						8	<0.1 (<0.1, <0.1)
Uvalde, TX, United States, 1990 (Tam Uvalde)	0.112 0.112 0.112	47 47 47	3 (36, 8)	0.336	Whole Fruit	3	<0.1 (<0.1, <0.1)
						7	<0.1 (<0.1, <0.1)
	0.112 0.112 0.112	191 191 191	3 (36, 8)	0.336	Whole Fruit	0	<0.1 (<0.1, <0.1)
						3	<0.1 (<0.1, <0.1)
						7	<0.1 (<0.1, <0.1)
					Peel	0	<0.1 (<0.1, <0.1)
						3	0.17 (<0.1, 0.24)
						7	0.17 (0.11, 0.22)
					Flesh	0	<0.1 (<0.1, <0.1)
						3	<0.1 (<0.1, <0.1)
						7	<0.1 (<0.1, <0.1)
Oaktown, IN, United States, 1990 (Super Star)	0.112 0.112 0.112	178 178 178	3 (52, 7)	0.336	Whole Fruit	3	0.12 (0.12, 0.11)
						7	<0.1 (<0.1, <0.1)
Sparks, GA, United States, 1990 (Edisto 47)	0.112 0.112 0.112	228 228 228	3 (28, 7)	0.336	Whole Fruit	3	0.32* (0.35, 0.29)
						7	<0.1 (<0.1, <0.1)
Eagle MI, United States, 1990 (Burpee Hybrid)	0.112 0.112 0.112	254 174 158	3 (30, 38)	0.336	Whole Fruit	3	<0.1 (<0.1, <0.1)
						7	<0.1 (<0.1, <0.1)

Notes:

* Fruits were harvested that were not mature and were only 2-4 inches in diameter (not marketable).

Fruiting vegetables, other than Cucurbits, peppers (012A)

Pepper, sweet

The following trials on bell pepper were previously reported by the 2010 JMPR. Five supervised trials on pepper, sweet (bell pepper) were conducted in the United States during 1994 growing seasons (Samoil, K.S., 1999a, IR-4 PR No. 05281). Bell peppers were treated with 2 foliar applications at rates of 0.097–0.119 kg ai/ha of 25 percent bifenthrin EC, retreat interval of 7-8 days. All applications were made using appropriate spray equipment. No adjuvants were used in the application. Duplicate samples were harvested 7–8 days after the last application. The maximum storage intervals for field-treated samples (3 months) were covered by storage stability studies. Residues of bifenthrin were determined using Method P-213M. Results of the trials are summarized in Table 8.

Table 8 Residues in bell pepper following application of bifenthrin 2EC (IR-4 Study No. 05281)

Location, Country/Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues (mg/kg) (individual values)
	Rate (kg ai/ha)	**Water** L/ha	No (RTI, days)				
GAP, United States	0.0336-0.112		1-2(7)	0.225		7	
Charleston, SC, United States, 1994 (Keystone Resistance Giant)	0.112 0.112	189 189	2 (8)	0.224	Fruit	7	0.14 (0.14, 0.13)
Weslaco, TX, United States, 1994 (Grande Rio #66)	0.112 0.112	280 280	2 (7)	0.224	Fruit	7	0.10 (0.10, 0.09)
Gainesville, FL, United States, 1994 (Capistrano)	0.112 0.112	280 280	2 (7)	0.244	Fruit	7	0.17 (0.24, 0.09)
Holtville, CA, United States, 1994 (Valiant)	0.097 0.119	475 484	2 (7)	0.216	Fruit	6	0.06 (0.07, <0.055)
Bridgeton, NJ, United States, 1994 (Bell Captain)	0.112 0.112	536 532	2 (8)	0.224	Fruit	7	<0.055 (<0.055, <0.055)

Pepper, chilli

Trials on pepper, chilli (non-bell pepper) were previously reported by the 2010 JMPR. Seven supervised trials on non-bell pepper were conducted in the United States during 1994 growing seasons (Samoil, K.S., 1999b, IR-4 PR No. 05280). Non-bell peppers were treated with 2 foliar applications at rates of 0.084–0.112 kg ai/ha of 25 percent bifenthrin EC, retreat interval of 6–8 days. All applications were made using appropriate spray equipment. No adjuvants were used in the application. Duplicate samples were harvested 6–7 days after the last application. The maximum storage intervals for field-treated samples (4 months) were covered by storage stability studies. Residues of bifenthrin were determined using Method P-213M with the LOQ of 0.05 mg/kg. Results of the trials are summarized in Table 9.

Table 9 Residues from non-bell pepper trials following application of bifenthrin 2EC

City, State/Region, Country/Year (variety)	Application			Total rate (kg ai/ha)	Commodity	DALA	Average residues ppm (mg/kg) (individual values)
	Rate (kg ai/kg)	Water (L/ha.)	No (RTI, days)				
GAP, United States	0.0336-0.112	93(grd) 19(air)	1-2(7)	0.225		7	
Non-bell peppers (IR-4 Study No. 05280)							
Charleston, SC, United States, 1994 (Conquistador)	0.112 0.112	189 189	2 (8)	0.224	Fruit	7	0.29 (0.31, 0.27)
Weslaco, TX, United States, 1994 (TAM Jalapeno Mild)	0.112 0.112	280 280	2 (7)	0.224	Fruit	7	0.15 (0.21, 0.09)
Gainesville, FL, United States, 1994 (Habanero)	0.112 0.112	280 280	2 (7)	0.244	Fruit	7	0.14 (0.17, 0.10)
Ripon, CA, United States, 1994 (Sonora)	0.112 0.112	274 278	2 (6)	0.224	Fruit	7	0.10 (0.09, 0.11)
Bridgeton, NJ, United States, 1994 (Jalapeno)	0.112 0.112	536 532	2 (7)	0.224	Fruit	7	0.08 (0.07, 0.09)
Baton Rouge, LA, United States, 1994 (TAM Mild #1)	0.084 0.084	258 258	2 (7)	0.168	Fruit	7	<0.05 (<0.05, <0.05)
Clinton, NC, United States, 1994 (Cayenne)	0.112 0.112	184 192	2 (8)	0.224	Fruit	6	0.18 (0.23, 0.12)

Leafy Vegetables (including brassica leafy vegetables) (013)

Spinach

The following trials on spinach were previously reported by the 2015 JMPR. Seven supervised trials on spinach were conducted in the United States during 1999 growing seasons (Samoil, K.S., 2001, IR-4 PR No. 07088). Spinach was treated with 1 foliar application at rates of 0.448–467 kg ai/ha of 25 percent bifenthrin EC All applications were made using appropriate spray equipment. No adjuvants were used in the application. Duplicate samples were harvested 37–41 days after application. The maximum storage intervals for field treated samples (2 months) were covered by storage stability studies. Residues of bifenthrin were determined using Method P-2132M with the LOQ of 0.05 mg/kg. Results of the trials are summarized in Table 10.

Table 10 Residues of bifenthrin in spinach following application 25 percent EC (IR-4 Study No. 07088)

City, State/Region, Country,/Year (variety)	Application			Commodity	DALA	Average residues ppm (mg/kg) (individual values)[c]
	Rate (kg ai/ha)	Water (L/ha)	No			
GAP, United States	0.0336-0.112	467	1-4(7)		40	Less than 0.45kg ai/ha per year

City, State/Region, Country,/Year (variety)	Application			Commodity	DALA	Average residues ppm (mg/kg) (individual values)[c]
	Rate (kg ai/ha)	Water (L/ha)	No			
Weslaco, TX, United States, 1999 (Fall Green)[1]	0.448	283	1	Leaves	41	<0.05 (<0.05, <0.05)
Weslaco, TX, United States, 1999 (Olympia)[2]	0.448	366	1	Leaves	39	<0.05 (<0.05, <0.05)
Weslaco, TX, United States, 1999 (Olympia)[3]	0.467	375	1	Leaves	39	<0.05 (<0.05, <0.05)
Salisbury, MD, United States, 1999 (Vienna)	0.454	267	1	Leaves	37	<0.05 (<0.05, <0.05)
Bridgeton, NJ, United States, 1999 (Melody)	0.448	344	1	Leaves	36	<0.05 (<0.05, <0.05)
Yuma, AZ, United States, 1999 (St. Helens)	0.11		4	Leaves	20	0.11 (0.89, 1.4)
					40	0.15 (0.14, 0.16)
Imperial, CA, United States, 1999 (St. Helens)	0.11 (aerial)		4	Leaves	20	0.47 (0.44, 0.50)
	0.11 (ground)		4	Leaves	20	1.0 (1.0, 1.1)
					39	0.050 (0.040, 0.060)

Notes:

[1] Weslaco (Fall Green) application date 1999-10-26.

[2] Weslaco (Olympia) application date 1999-03-04.

[3] Weslaco (Olympia) application date 1999-03-25.

Three supervised trials on spinach were conducted in the United States during 1992-1993 growing seasons (Kim, I., 1993a, FMC Study No. 182SPI92R1). Spinach was treated with 4 foliar applications at rates of 0.112 kg ai/ha of 25 percent bifenthrin EC, retreat interval of 4–12 days All applications were made using appropriate spray equipment. No adjuvants were used in the application. Duplicate samples were harvested 20–40 days after the last application. The maximum storage intervals for field-treated samples (5months) were covered by storage stability studies. Residues of bifenthrin were determined using Method P-2715 with the LOQ of 0.05 mg/kg. Results of the trials are summarized in Table 11. Residues of 4'-hydroxy-bifenthrin were also analysed but all residues were below LOQ (< 0.05 mg/kg).

Table 11 Residues of bifenthrin in spinach following 4 applications 25 percent EC (FMC Study No. 182SPI92R1)

Location Country/Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues (mg/kg) (individual values)
	Rate (kg ai/kg)	Water (L/ha)	No (RTI, days)				
GAP, United States	0.0336-0.112	467	1-4(7)	0.448		40	
Yuma, AZ, United States, 1992/1993 (St. Helens)	0.112 0.112 0.112 0.112	93 93 93 93	4 (12, 7, 13)	0.479	Leaves	20	1.13 (1.37, 0.89)
	0.112 0.112 0.112 0.112	93 93 93 93	4 (10, 7, 4)	0.479	Leaves	40	0.15 (0.16, 0.14)
Imperial, CA, United States, 1992/1993 (St. Helens)	0.112 0.112 0.112 0.112	47 47 47 47	4 (8, 6, 5)	0.448	Leaves	20	0.47 (0.50, 0.44)
Imperial, CA, United States, 1992/1993 (St. Helens)	0.112 0.112 0.112 0.112	98 98 98 98	4 (8, 6, 5)	0.457	Leaves	20	1.04 (1.06, 1.01)
	0.112 0.112 0.112 0.112	98 98 98 98	4 (7, 7, 4)	0.457	Leaves	39	0.05 (0.06, 0.05)

Oilseed (023)

Peanut

Four supervised trials on peanut were conducted in the United States during 2003 growing seasons (Morris, R.T., 2004, FMC Study No. 182PNT03R1). Peanut was treated with a soil application at rate of 0.28 kg ai/ha with 1.15G and a foliar application at rate of 0.28 kg ai/ha of bifenthrin 2EC 7days after soil application. All applications were made using appropriate spray equipment. No adjuvants were used in the application. Duplicate samples were harvested 14 days after the last application in 3 trials and 3, 7, 15 and 17 days in one trial. The maximum storage intervals for field-treated samples (6months) were covered by storage stability studies. Residues of bifenthrin were determined using Method P-3457 with the LOQ of 0.05 mg/kg. Results of the trials are summarized in Table 12.

Table 12 Residue in peanut following a soil application and a foliar application of bifenthrin 2EC (FMC Study No. 182PNT03R1)

Location, State/Region, Country/Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA (days)	Average residues (mg/kg) (individual values)
	Rate (kg ai/ha)	Water (L/ha)	No*(RTI, days)				
GAP, United States	0.0336-0.112	93 (grd)/ 19 (air)	1-5(14)	0.561		14	
Madison, FL, United States, 2003 (Georgia Green)	0.28(soil)+ 0.28(foliar)	116	2 (7)	0.56	Nutmeat	14	<0.05 (<0.05, <0.05)

Location, State/Region, Country/Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA (days)	Average residues (mg/kg) (individual values)
	Rate (kg ai/ha)	Water (L/ha)	No*(RTI, days)				
Ashburn, GA, United States, 2003 (Georgia Green)	0.28(soil)+ 0.28(foliar)	116	2 (7)	0.56	Nutmeat	14	<0.05 (<0.05, <0.05)
Sparks, GA, United States, 2003 (Georgia Green)	0.28(soil)+ 0.28(foliar)	140	2 (7)	0.538	Nutmeat	3	<0.05 (<0.05, <0.05)
						7	<0.05 (<0.05, <0.05)
						15	<0.05 (<0.05, <0.05)
						17	<0.05 (<0.05, <0.05)
Columbia, AL, United States, 2003 (Georgia Green)	0.28(soil)+ 0.28(foliar)	115	2 (7)	0.56	Nutmeat	14	<0.05 (<0.05, <0.05)

Notes:

* One soil treatment of 0.28 kg ai/ha plus 0.28 kg ai/ha at the interval of 7 days.

Four supervised trials on peanut were conducted in the United States during 1992 growing seasons (Kim, I., 1993b, FMC Study No. 182PNT92R1). Peanut was treated with 5 foliar applications at rate of 0.112 kg ai/ha of bifenthrin 2EC, at premature seedling, early bloom, pegging, early nut maturity and mature nuts, with retreat interval of 9–35 days. All applications were made using appropriate spray equipment. No adjuvants were used in the application. Duplicate samples were harvested 0 and 7days after the last application. The maximum storage intervals for field-treated samples (6 months) were covered by storage stability studies. Residues of bifenthrin were determined using Method P-0130 with the LOQ of 0.05 mg/kg. Results of the trials are summarized in Table 13.

Table 13 Residues in peanut following application of bifenthrin EC (FMC Study No. 182PNT92R1)

Location, Country, Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues (mg/kg) (individual values)[c] Bifenthrin
	Rate (kg ai/ha)	Water (L/ha)	No (RTI, days)				
GAP, United States	0.0336-0.112	93 (grd)/ 19 (air)	1-5(14)	0.561		14	
Gaston, NC, United States, 1992 (NC6)	0.112 0.112 0.112 0.112 0.112	88 88 88 88 88	5 (21, 21, 35, 35)	0.56	Nutmeat	0 7	<0.05 (ND, <0.05) <0.05 <0.05, <0.05)
Eakly, OK, United States, 1992 (Spanco)	0.112 0.112 0.112 0.112 0.112	120 120 120 120 120	5 (17, 11, 59, 25)	0.56	Nutmeat	0 7	<0.05 (<0.05, <0.05) <0.05 (<0.05, <0.05)
Pearsall, TX, United States, 1992 (Birdsong GK-7)	0.112 0.112 0.112 0.112 0.112	110 110 110 110 110	5 (25, 34, 51, 9)	0.56	Nutmeat	0 7	<0.05 (<0.05, <0.05) <0.05 <0.05, <0.05)

Location, Country, Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues (mg/kg) (individual values)[c] Bifenthrin
	Rate (kg ai/ha)	Water (L/ha)	No (RTI, days)				
Emporia, VA, United States, 1992 (NC6)	0.112 0.112 0.112 0.112 0.112	120 120 120 120 120	5 (21, 21, 35, 35)	0.56	Nutmeat	1 8	<0.05 (<0.05, <0.05) <0.05 (<0.05, <0.05)

Animal feed

Four supervised trials on peanut were conducted in the United States during 1992 growing seasons (Kim, I., 1993b, FMC Study No. 182PNT92R1). Peanut was treated with 5 foliar applications at rate of 0.112kg ai/ha of bifenthrin 2EC, at premature seedling, early bloom, pegging, early nut maturity and mature nuts, with retreat interval of 9-35 days. All applications were made using appropriate spray equipment. No adjuvants were used in the application. Duplicate samples were harvested 1, 7, 10, 14 and 21 days after the last application The maximum storage intervals for field-treated samples (6 months) were covered by storage stability studies. Residues of bifenthrin were determined using Method P-0130 with the LOQ of 0.05 mg/kg. Results of the trials are summarized in Table 14.

Table 14 Residues in peanut animal feed commodities following application of bifenthrin EC (FMC Study No. 182PNT92R1)

Location, Country, Year (variety)	Application			Total rate (kg ai/ha)	Matrix	DALA	Average residues (mg/kg) (individual values)[c] Bifenthrin
	Rate (kg ai/ha)	Water (L/ha)	No (RTI, days)				
GAP, United States	0.0336-0.112	93 (grd)/ 19 (air)	1-5(14)	0.561		14	
Gaston, NC, United States, 1992 (NC6)	0.112 0.112 0.112 0.112 0.112	88 88 88 88 88	5 (21, 21, 35, 35)	0.56	Vine	0	3.60 (3.50, 3.70)
					Hay	7	7.13 (6.26, 8.00)
						14	11.3 (9.66, 13.0)
						21	3.97 (4.50, 3.43)
					Hulls	0	0.15 (0.13, 0.17)
						7	0.09 (0.10, 0.07)
Eakly, OK, United States, 1992 (Spanco)	0.112 0.112 0.112 0.112 0.112	120 120 120 120 120	5 (17, 11, 59, 25)	0.56	Vine	0	3.83 (2.60, 5.05)
					Hay	7	6.77 (6.17, 7.37)
						14	6.40 (6.91, 5.89)
						21	5.05 (5.28, 4.81)
					Hulls	0	0.06 (<0.05, 0.07)
						7	0.09 (0.09, 0.09)

	Application						
Location, Country, Year (variety)	Rate (kg ai/ha)	Water (L/ha)	No (RTI, days)	Total rate (kg ai/ha)	Matrix	DALA	Average residues (mg/kg) (individual values)[c] Bifenthrin
Pearsall, TX, United States, 1992 (Birdsong GK-7)	0.112 0.112 0.112 0.112 0.112	110 110 110 110 110	5 (25, 34, 51, 9)	0.56	Vine	0	5.26 (5.11, 5.40)
					Hay	7	3.50 (3.41, 3.59)
						14	2.87 (2.34, 3.40)
						21	4.21 (3.92, 4.50)
					Hulls	0	0.05 (0.05, <0.05)
						7	0.07 (0.08, <0.05)
Emporia, VA, United States, 1992 (NC6)	0.112 0.112 0.112 0.112 0.112	120 120 120 120 120	5 (21, 21, 35, 35)		Vine	1	4.37 (3.94, 4.79)
					Hay	8	8.65 (8.02, 9.27)
						15	8.85 (8.34, 9.36)
						22	4.53 (5.14, 3.92)
					Hulls	1	0.13 (0.13, 0.12)
						8	0.08 (0.07, 0.08)

Fate of residues during processing

The Meeting received trials on the processing of apple and peanut.

Apple

Apples treated with three foliar applications of bifenthrin at approximately rate of 2.80 kg ai/ha/season, an adjuvant was added to the spray mixture. Apple samples were harvested 15 days after the last application at commercial maturity. Samples were shipped under ambient conditions to the processing facility where samples were processed to juice and pomace (Samoil, K.S. 2015a, IR-4 PR No. 11016).

Apples were tub washed in cold tap water for 5 minutes, were crushed by a hammermill to a unform pulp consistency. The crushed apple pulps were placed in a swept surface kettle and heated with low pressure steam until the temperature reaches 40–50 °C. The pectic enzyme treated apple pulp was pressed using a hydraulic style apple press. The collected fresh juice was filtered through US#40 screen prior to packaging a fresh juice sample and/or further refining processes of pasteurized, clarified and/or concentrated apple juice samples. The wet pomace collected from the pressing process was analysed for moisture content and sampled as the wet pomace fraction. The raw juice was reheated to 93 °C for 15–30 seconds to deactivate the pectic enzymes and was then placed in the cooler to allow the solids to settle overnight. The clear juice was racked and the solids were discarded. The juice was vacuum filtered through diatomaceous earth to improve clarification. The filtered juice is pasteurized by heating to 93$\pm$3 °C and packed in cans while hot. The cans were sealed and inverted, then cooled using cold water. Residues of bifenthrin were determined using Method P-3526 with the LOQ of 0.05 mg/kg. Results of the trials are summarized in Table 15.

Table 15 Residues of bifenthrin in apple and its processed commodities following application of bifenthrin WSB (IR-4 PR No. 11016)

Location, Country/Year (variety)	Matrix	Application rate (kg ai/ha)	DALA (days)	Residues (mg/kg) (individual values)	Processing factor
Twin Falls, ID, United States, 2013 (Red Delicious)	Fruit	0.561, 1.11 and 1.12	15	1.07 (1.22, 0.928)	--
	Juice			<0.05 (<0.05, <0.05)	<0.048
	Pomace			2.66 (3.77, 1.55)	2.5

Residues of bifenthrin are not concentrated in juice but are concentrated in pomace.

Peanut

Peanuts treated with three foliar applications at rate of 0.336 kg ai/ha (total approximately 1.008 kg ai/ha/season) bifenthrin EC, no adjuvant was added to the spray mixture. Peanut samples were collected 13 days after the last application at commercial maturity (Culligan, J.F. 1996, FMC Study No. 182PNT95R2). The samples were processed following the below scheme. The residues of bifenthrin were determined using Method P-2814 with the LOQ of 0.05mg/kg. Results of the trials are summarized in Table 16.

As residues in nutmeat (raw commodity) and all the processed commodities were below the LOQ (except that one value for refined oil was at the LOQ), processing factors could not be calculated.

Table 16 Residues of bifenthrin in peanut and its processed commodities following application of bifenthrin WSB (FMC Study No. 182PNT95R2)

Location, Country/Year (variety)	Application rate (kg ai/ha)	DALA	Matrix	Residues (mg/kg)	Processing factor
Sunsweet, GA, United States, 1995 (Florunner)	3×0.336	13	Nutmeat, field	<0.05, <0.05, <0.05, <0.05	--
			Nutmeat, pre-processing	<0.05, <0.05, <0.05, <0.05	--
			Meal	<0.05, <0.05, <0.05, <0.05	NC
			Refined oil	<0.05, <0.05, 0.05, <0.05	NC

Notes:

NC: Not Calculated because average residues in RAC and processed commodity were <LOQ.

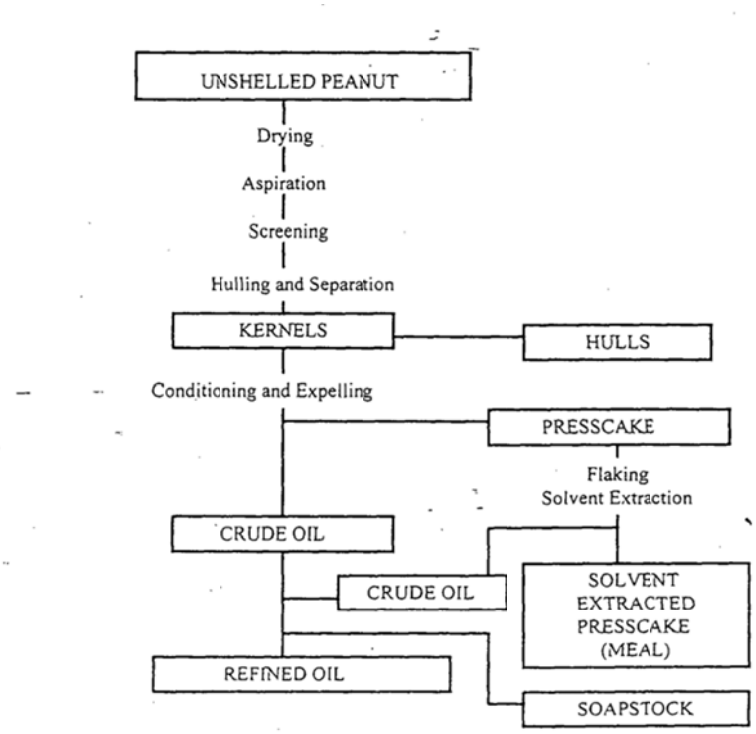


Figure 1 Flow scheme for processing peanut nutmeat into processed parts

APPRAISAL

Bifenthrin is a pyrethroid insecticide and miticide. It was first evaluated for residues and toxicology by the JMPR in 1992. Bifenthrin was evaluated under the periodic review programme in 2009 (T) and 2010 (R), and subsequently evaluated in 2015 and 2019 for additional MRLs.

An ADI of 0–0.01 mg/kg bw and an ARfD of 0.01 mg/kg bw were established by the 2009 JMPR. The definition of the residue for compliance with the MRL and for dietary risk assessment for animal and plant commodities is *bifenthrin* (sum of isomers). The residue is fat-soluble.

Bifenthrin was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2021 Extra JMPR and was re-scheduled to the 2022 JMPR.

The current Meeting received information on new GAPs and supervised residue trials for apple, peach, avocado, pomegranate, pepper (bell, non-bell), melon, spinach, and peanut, as well as processing studies on apple and peanut.

Methods of analysis

The Methods used in residue trials were similar to or slight modifications to the method evaluated by previous Meetings. In general, the data generation methods considered by this Meeting involved extraction with acetone and cleaning-up with SPE. Final determination was achieved using GC-ECD or GC-MSD. The methods were considered suitable for analysis of bifenthrin in trials.

Stability of pesticide residues in stored analytical samples

The 2010 and 2015 JMPR evaluated the stability of bifenthrin in analytical samples stored under frozen conditions and concluded that bifenthrin is stable for at least 18 months in high acid, 49 months in high water, 36 months in high oil and high starch, and 15 months in high protein commodities under frozen conditions. The maximum frozen storage intervals in the supervised trials provided to the current Meeting were shorter than the storage intervals indicated above.

Results of supervised residue trials on crops

The Meeting received information on supervised trials of bifenthrin on apple, peach, avocado, pomegranate, melon, peppers (sweet and chili), spinach and peanut.

Pome fruit, except Japanese persimmon

Apple

The GAP for bifenthrin is in the United States for the US pome fruit group, consisting of foliar applications at 0.224 kg ai/ha with re-treatment intervals (RTIs) of not less 30 days and a PHI of 14 days, no more than 3 applications and a total application rate not more than 0.56 kg ai/ha per year with no more 0.51 kg ai/ha applied after petal fall (BBCH 69). Given the annual rate limitation, the cGAP is an initial application at 0.112 kg ai/ha followed by 2 applications, each at 0.224 kg ai/ha.

In independent trials approximating the cGAP but with retreatment intervals of 20 rather than 30 days, residues of bifenthrin in fruits were (n=8): 0.10, 0.11, 0.13, 0.19, 0.20, 0.24, 0.29, and 0.42 mg/kg.

Noting that the residue decline studies on apple and peach showed no significant changes in concentration between 7 and 21 days after the final application, the Meeting agreed that the difference in retreatment intervals between the trials and the cGAP would not affect residues by more than 25 percent and agreed to use the data from the trials to estimate a maximum residue level.

The Meeting estimated a maximum residue level of 0.7 mg/kg, an STMR of 0.195 mg/kg and an HR of 0.45 mg/kg (highest individual result) for bifenthrin in apple. Noting that the registration is for the US pome fruit group, which does not include Japanese persimmon, the Meeting agreed to extrapolate the estimates for apple to the group of pome fruit, except persimmon, Japanese.

The Meeting noted that acute dietary exposure assessment for apple and pear exceeded the ARfD of 0.01 mg/kg bw (*apple: 250 percent for children in China; pear: 310 percent for children in Canada*). No alternative GAP was available.

Peach

The critical GAP for bifenthrin is in United States for the US Peach subgroup 12-12B, consisting of foliar applications at 0.22 kg ai/ha with RTI of not less 30 days and a PHI of 14 days, no more than 3 application and the total application rate less than 0.56 kg ai/ha per year with no more 0.51 kg ai/ha applied after petal fall (BBCH 69). Given the annual rate limitation, the cGAP is an initial application at 0.12 kg ai/ha followed by 2 applications, each at 0.22 kg ai/ha.

In independent trials approximating the cGAP, residues of bifenthrin in fruit without stone were (n=11): 0.12, 0.12, 0.17, 0.20(2), 0.22, 0.24, 0.26, 0.30, 0.38 and 0.41 mg/kg.

Noting that the residue decline studies on apple and peach showed no significant changes in concentration between 7 and 21 days after the final application, the Meeting agreed that the difference in retreatment intervals between the trials and the cGAP would not affect residues by more than 25 percent.

Furthermore, the 2017 JMPR concluded that for stone fruit, based on the weight of the stone relative to the whole fruit, residues measured in fruit without stones would overestimate whole-fruit residues by about 10 percent and that correcting for this factor would lead to the same maximum residue level estimation. Therefore, the Meeting agreed to use the data from the trials to estimate a maximum residue level.

The Meeting estimated a maximum residue level of 0.8 mg/kg, an STMR of 0.22 mg/kg and an HR of 0.49 mg/kg (highest individual result) for bifenthrin in peach and agreed to extrapolate to the subgroup of peaches.

The Meeting noted that acute dietary exposure assessment showed that residues in peach, apricot, and nectarine exceeded the ARfD of 0.01 mg/kg bw (*peach: 230 percent for children in Japan; apricot: 110 percent for children in Germany; nectarine: 210 percent for children in The Netherlands (Kingdom of the*)). No alternative GAP was available.

Avocado

The critical GAP for bifenthrin on avocado in the United States is foliar applications at 0.062 kg ai/ha with an RTI of not less 14 days and a PHI of 1 day, no more than 5 applications.

Five independent trials were conducted on avocado in the United States with 5 foliar applications at rates of 0.081–0.091 kg ai/ha, and RTIs of 13–17 days, the total application rates of 0.40–0.44 kg ai/ha and a PHI of 1 day. The residues of bifenthrin in fruits (without stone) were (n=5): 0.076, 0.08, 0.11, 0.26 and 0.30 mg/kg. The scaled residues using the scaling factors of 0.73–0.77 (last application rate in trial/GAP) were (n=5): 0.056, 0.058, 0.089, 0.20 and 0.22 mg/kg.

Based on the scaled residue data, the Meeting estimated a maximum residue level of 0.5 mg/kg (assuming the pit constitutes 15 percent of the whole fruit weight), an STMR of 0.089 mg/kg and an HR of 0.23 mg/kg (highest individual result) for bifenthrin in avocado.

Pomegranate

The critical GAP for bifenthrin on pomegranate in the United States is foliar applications at 0.22 kg ai/ha with RTI of not less 14 days and a PHI of 14 days, no more than 3 application and the total application rate less than 0.56 kg ai/ha per year. Given the annual rate limitation, the cGAP is an initial application at 0.12 kg ai/ha followed by 2 applications, each at 0.22 kg ai/ha.

In independent trials matching the cGAP, residues of bifenthrin in pomegranate were (n=4): 0.11, 0.16, 0.17 and 0.18 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg, a STMR of 0.165 mg/kg and an HR of 0.22 mg/kg (highest individual result) for bifenthrin in pomegranate.

Melon (cantaloupe)

The critical GAP for bifenthrin on melon in the United States is 3 foliar applications at 0.11 kg ai/ha at RTI of not less 7 days and a PHI of 3 days, with no more than two applications after bloom.

In independent trials matching the cGAP, residues of bifenthrin in fruits were (n=4): < 0.1(2), 0.11 and 0.12 mg/kg.

The Meeting agreed that four trials were insufficient to make a recommendation for melon.

Peppers

The critical GAP for bifenthrin in the United States is for the US crop group covering use on peppers and eggplant and consists of 2 foliar applications at 0.11 kg ai/ha with RTI of not less 7 days and a PHI of 7 days, no more than 2 applications.

Five independent trials on pepper, sweet were conducted in the United States approximating the GAP. The residues of bifenthrin in fruits were (n=5): < 0.055, 0.06, 0.10, 0.14 and 0.17mg/kg.

Seven independent trials on pepper, chilli were conducted in the United States approximating the GAP. The residues of bifenthrin in fruits were (n=7): < 0.05, 0.08, 0.10 0.14, 0.15, 0.18 and 0.29 mg/kg.

The ranked order of the combined bifenthrin residues in sweet pepper and pepper, chilli were (n=12): < 0.05, < 0.055, 0.06, 0.08, 0.10(2), 0.14(2), 0.15, 0.17, 0.18, and 0.29 mg/kg.

The Meeting noted that the provided trials were previously evaluated by the 2010 JMPR under a registered use on peppers and that the current registration is for peppers and eggplant. The Meeting estimated a maximum residue level of 0.4 mg/kg, an STMR of 0.12 mg/kg, and an HR of 0.31 mg/kg (from a single sample) for bifenthrin in the Subgroup of peppers (except okra, martynia and roselle) to replace its previous recommendation. The Meeting agreed to extrapolate those estimates to the Subgroup of eggplants.

For estimating residues in dried chili peppers, the Meeting used the data from chili peppers and a default processing factor of 7. On that basis, the Meeting estimated a maximum residue level of 4 mg/kg an STMR of 0.98 mg/kg, and an HR of 2.2 mg/kg for bifenthrin in chili pepper, dried to replace its previous recommendation of 5 mg/kg.

Spinach

The critical GAP for bifenthrin on spinach in the United States is 4 foliar applications at 0.11 kg ai/ha at RTI of not less 7 days and a PHI of 40 days.

In independent trials on spinach approximating the cGAP, residues of bifenthrin in spinach were (n=4): 0.05 (2) and 0.15 mg/kg (2).

The Meeting agreed that four trials were insufficient to make a recommendation for spinach.

Peanut

The critical GAP for bifenthrin on peanut in the United States is five foliar applications at 0.11 kg ai/ha with RTI of not less 14 days and a PHI of 14 days.

In four trials involving one soil application (0.28 kg ai/ha) and one foliar application (0.28 kg ai/ha), with harvest 3–17 DALA, the residues of bifenthrin in nutmeat were (n=4): < 0.05 (4) mg/kg.

In four trials with 5 foliar applications at 0.11 kg ai/ha, with RTIs between last two sprays of 9–35-days and harvest 0–8 DALA, residues of bifenthrin in nutmeat were (n=4) < 0.05 (4) mg/kg.

In a trial to obtain samples for processing studies, 3 foliar applications were made at 0.34 kg ai/ha, and RTIs of 11–47 days, with harvest 13 DALA, the bifenthrin residue in nutmeat was < 0.05 mg/kg.

None of the available trials matched the cGAP. Noting the residues from all trials, including the trials with exaggerated rates, were < 0.05 mg/kg, the Meeting agreed to estimate a maximum residue level of 0.05(*) and an STMR of 0.05 mg/kg for bifenthrin in peanut.

Residues in animal feeds

Peanut vine, hay and hull

The only GAP provided for peanuts was from the United States. There is a label restriction in the United States which excludes the feeding of green immature plants and peanut hay to livestock. The Meeting did not make new estimates for residues in animal commodities and confirmed its previous recommendation.

Fate of residues during processing

The Meeting received processing studies for apple and peanut.

Estimated processing factors for apple considered at this Meeting are summarised below.

Table 17 Processing factors for estimation of STMR

RAC	Processed commodity	Median or best estimate processing factor	STMR-P =$STMR_{RAC}$ × PF (mg/kg)
Apple (STMR = 0.2 mg/kg)	Juice	< 0.048	0.0096
	Wet pomace	2.5	0.5

For peanut, four nutmeat samples were obtained from a trial with three applications of bifenthrin at 0.336 kg ai/ha. Residues in all samples of nutmeat and meal were < 0.05 mg/kg. In oil, bifenthrin residues were < 0.05 mg/kg in three samples and 0.05 mg/kg in one sample. A processing factor for oil could not be calculated due to the non-quantifiable residue in the nutmeat.

Noting that the dosing in the trial was at a 1.8× rate and that when scaled to a cGAP rate the expected residues in meal and refined oil would be < 0.05 mg/kg, the Meeting agreed to estimate the median-P for meal at 0.05 mg/kg and the STMR-P for refined oil at 0.05 mg/kg.

Farm animal dietary burden

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the JMPR. The dietary burdens, estimated using the 2018 OECD Feed diets listed in Appendix XIV Electronic attachments to the 2016 Edition of the FAO manual, are presented in Annex 6.

The maximum total dietary burdens calculated in 2019 were 8.3 ppm (beef cattle), 7.4 ppm (dairy cattle), 0.59 ppm (poultry broiler) and 2.0 ppm (poultry layer). The only animal feed evaluated by the current Meeting is apple pomace. Maximum total dietary burdens calculated by the current Meeting using the OECD diets were unchanged or slightly less than those derived by the 2019 Meeting. The Meeting therefore confirmed its previous recommendations for residue levels in animal products.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Table 18 Residue levels suitable for establishing maximum residue limits and for IEDI and IESTI assessments

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR mg/kg
		New	Previous		
FI 0326	Avocado	0.5		0.089	0.23

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR mg/kg
		New	Previous		
VO 20046	Eggplant, Subgroup of	0.4		0.12	0.31
SO 0697	Peanut	0.05*		0.05	
HS 0444	Pepper, chilli, dry	4	5	0.98	2.2
FS 2001	**Peaches, Subgroup of #**	0.8#		0.22	0.49
VO 0051	Peppers, Subgroup of (except okra, martynia and roselle)	0.4	0.5	0.12	0.31
FI 0355	Pomegranate	0.5		0.165	0.22
FP 0009	Pome fruits, Group of (except persimmon, Japanese)#	0.7#		0.195	0.45
JF 0226	Apple juice			0.0096	
OR 0697	Peanut oil, edible			0.05	
	Apple, wet pomace	--	--	0.5	--

Notes:

On the basis of information provided to the JMPR it was concluded that the estimated acute dietary exposure to residues of bifenthrin for the consumption of Peaches, Subgroup of and Pome fruit, Group of (except Japanese persimmon) may present a public health concern.

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for bifenthrin is 0–0.01 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for bifenthrin were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report. The IEDIs ranged from 10–40 percent of maximum ADI of 0.01 mg/kg bw. The Meeting concluded that the long-term dietary exposure to residues of bifenthrin from uses considered by the current Meeting is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for bifenthrin is 0.01 mg/kg bw, the International Estimate of Short-Term Intakes (IESTIs) was calculated for food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR report.

The IESTIs were less than 100 percent of the ARfD, except for apple (up to 230 percent for children in China), pear (up to 310 percent for children in Canada), peach (up to 260 percent for children in Japan), apricot (up to 110 percent for children in Germany), and nectarine (up to 210 percent for children in the Netherlands). The Meeting concluded that acute dietary exposure to residues of bifenthrin may present a public health concern for those commodities.

REFERENCES

Reference Number	Author(s)	Year	Study Title
Report Number IR-4 PR No. 11016	Samoil, K.S.	2015a.	Bifenthrin: Magnitude of the residue on apple. IR-4 Project Rutgers, The State University of New Jersey. GLP: Yes. Unpublished.

Reference Number	Author(s)	Year	Study Title
Report Number IR-4 PR No. 11017.	Samoil, K.S.	2015b	Bifenthrin: Magnitude of the residue on peach. IR-4 Project Rutgers, The State University of New Jersey. GLP: Yes. Unpublished.
Report Number IR-4 PR No. 10578	Samoil, K.S.	2015c	Bifenthrin: Magnitude of the residue on avocado. IR-4 Project Rutgers, The State University of New Jersey.. GLP: Yes. Unpublished.
Report Number IR-4 PR No. 11249.	Samoil, K.S.	2016	Bifenthrin: Magnitude of the residue on pomegranate. IR-4 Project Rutgers, The State University of New Jersey. GLP: Yes. Unpublished.
Report Number IR-4 PR No. 4151	Biehn, W.L.	1996	Bifenthrin: Magnitude of the residue on cantaloupe. IR-4 Northcentral Region Analytical Laboratory. , FMC Study No. 182CAN90R1. GLP: Yes. Unpublished
Report Number IR-4 PR No. 05281	Samoil, K.S.	1999a	Bifenthrin: Magnitude of the residue on pepper (bell). IR-4 Project, Center for Minor Crop Pest Management, Technology Centre of New Jersey. . GLP: Yes. Unpublished.
Report Number IR-4 PR No. 05280.	Samoil, K.S.	1999b	Bifenthrin: Magnitude of the residue on pepper (Non-bell). IR-4 Project, Center for Minor Crop Pest Management, Technology Centre of New Jersey. GLP: Yes. Unpublished.
Report Number P-2839, FMC Study No. 182SPI92R1	Kim, I-Y.	1993a	Magnitude of the residue of bifenthrin and 4'-Hydroxy bifenthnn in/on spinach treated with Capture 2EC. FMC Corporation.. GLP: Yes. Unpublished.
Report Number IR-4 PR No. 07088	Samoil, K.S.	2001	Bifenthrin: Magnitude of the residue on spinach. IR-4 Project, Center for Minor Crop Pest Management, The Technology Centre of New Jersey. GLP: Yes. Unpublished.
Report P-2814, FMC Study Number 182PNT92R1	Kim, I-Y	1993b	Magnitude of the residue of Bifenthrin in/on peanuts treated with Capture 2 EC. FMC Corporation Agricultural Chemical Group, Residue Chemistry. GLP: Yes. Unpublished.
Report Number P-3694, FMC Study No. 182PNT03R1.	Morris, R.T	2004	Magnitude of the residues of bifenthrin in/on peanut nutmeat from peanuts treated with Capture 1.15 g and Capture 2EC Insecticide/Miticide. FMC Corporation Agricultural Products Group Environmental Sciences. GLP: Yes. Unpublished.
Report Number P-3161. FMC Study No.182LET03R1.	Culligan, J.F.	1996	Magnitude of the residue of bifenthrin in/on peanut processed parts following treatment with three applications of Capture 2 EC each at a rate of 0.3 pounds active ingredient per acre. FMC Corporation. GLP: Yes. Unpublished.
Report No.: P-3526	Culligan JF	2001	Magnitude of the Residue of Bifenthrin in/on Potatoes and Potato Processed Parts Following Treatment with Capture 1.15G and Capture 2EC Insecticide-Miticide. FMC Corporation. Unpublished.
P-213M	Ridler, J.E.	1989	Analytical method for the determination of bifenthrin in/on various crops and soils, FMC corporation, Ag. Chemical Group, Princeton, NJ.
P-2715	Kim, I	1992	Magnitude of the residue of bifenthrin and 4'-Hydroxy bifenthrin in/on head lettuce treated with Capture 2EC, FMC Corporation, Agricultural Chemical Group, Princeton, NJ.
P-3457 MRID No.45350906	Chen, A.	2000	Magnitude of the residue of bifenthrin in/on grapefruit treated with Bridge WSB or Capture 2EC Insecticide at a rate of 0.5lb ai/A, FMC Corporation, Agricultural Products Group, Princeton, NJ
P-0130 MRID # 41492605)	(Winkler, D, A,	1992,	Method Validation for the Determination of Bifenthrin in/on Pecans and Walnuts", PC-0 130 Revised Unpublished report prepared by EN-CAS Analytical Laboratories for FMC Corporation.
P-2763	Kim, I	1992	Residue analytical method for the determination of bifenthrin in/on peanut processed parts, FMC Corporation, Agricultural products group, Princeton, NJ

BROFLANILIDE (326)

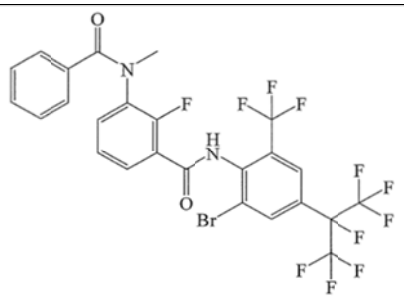
First draft prepared by Dr J Heidler, Federal Institute for Risk Assessment, Berlin, Germany

EXPLANATION

Broflanilide is a meta-diamide insecticide for the control of chewing-insect pests. It is the pro-insecticide to its active form desmethyl broflanilide, which acts by binding to the GABA receptor, resulting in a block of inhibitory neurotransmission and death of target insects. At the Fifty-first Session of the CCPR, broflanilide was scheduled for evaluation as a new compound in 2020 and rescheduled to the 2022 JMPR.

The Meeting received information on identity, physicochemical properties, metabolism (plant, confined rotational crops and animals), environmental fate, field rotational crops, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials in bulb vegetables (green onion, leek), brassica vegetables (cabbage, Chinese cabbage), tomato, root vegetables (radish, Japanese radish, turnip, potato), cereals (wheat, barley), maize, sweet corn and coffee, fate of residues in processing, and livestock feeding studies.

IDENTITY

ISO common name	Broflanilide
IUPAC name	*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-3-(*N*-methylbenzamido)benzamide
CA nomenclature	3-(benzoylmethylamino)-*N*-[2-bromo-4-[1,2,2,2-tetrafluoro-1-(trifluoromethyl)ethyl]-6-(trifluoromethyl)phenyl]-2-fluorobenzamide
Synonyms	BAS 450 I, MCI-8007, MLP-8607, Reg.No. 5672774, LS 5672774, LSP5672774
CAS No.	1207727-04-5
CIPAC No.	994
Structural formula	
Molecular formula	$C_{25}H_{14}BrF_{11}N_2O_2$
Molecular mass	663.3 g/mol

Physical and chemical properties

Table 1 Physical and chemical properties of broflanilide

Property	Method	Test material batch and purity	Results	Reference
Melting point, freezing point or solidification point	EEC A.1 OECD 102 OCSPP 830.7200 Metal block method	Pure active substance 089-100112-1 99.67 percent	154.0 to 155.5 °C	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755

Property	Method	Test material batch and purity	Results	Reference
Boiling point	EEC A.2 OECD 103 OCSPP 830.7220 Siwoloboff method	Pure active substance 089-100112-1 99.67 percent	Not determinable; decomposes above approximately 180 °C	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
Temperature of decomposition or sublimation	EEC A.2 OECD 103 OCSPP 830.7220 Siwoloboff method	Pure active substance 089-100112-1 99.67 percent	Decomposes above approximately 180 °C	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
Relative density	EEC A.3 OECD 109 OCSPP 830.7300 Using a pycnometer	Pure active substance 089-100112-1 99.67 percent	1.66 (D^{23}_{4}) at 23 °C	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
Vapour pressure	EEC A.4 OECD 104 OCSPP 830.7950 Using a vapour pressure balance	Pure active substance 089-100112-1 99.67 percent	Vapour pressure 9×10^{-9} Pa at 25 °C	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
Henry's law constant	Calculated from water solubility and vapour pressure data	Not relevant	3.0×10^{-6} $Pa.m^3.mol^{-1}$	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
Colour and physical state	Physical state: Visual assessment Colour: Munsel colour system under normal daylight	Pure active substance 089-100112-1 99.67 percent	Colour: N 9.5/90.0 percent R (white) Physical State: Solid (powder) at 20 °C	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
Odour	Organoleptic assessment	Pure active substance 089-100112-1 99.67 percent	Odour: None discernible	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
UV/VIS	OECD 101 OCSPP 830 7050	Pure active substance 089-100112-1 99.67 percent	Purified water (pH 7.2)[a] ε = 17200 at λ_{max} of 239 ε = 5000 at λ_{max} of 274 ε = 4090 at λ_{max} of 282 0.1 M aqueous HCl (pH 1.4) ε = 17000 at λ_{max} of 239 ε = 4980 at λ_{max} of 274 ε = 4120 at λ_{max} of 282 0.1 M aqueous NaOH (pH 13.0) ε = 17600 at λ_{max} of 248 ε = 5560 at λ_{max} of 293	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755

Property	Method	Test material batch and purity	Results	Reference
IR	OECD 101 OCSPP 830 7050	Pure active substance 089-100112-1 99.67 percent	The IR spectrum was consistent with the assigned structure of broflanilide.	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
NMR	OECD 101 OCSPP 830 7050	Pure active substance 089-100112-1 99.67 percent	The proton and carbon-13 NMR spectra were consistent with the assigned structure of broflanilide.	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
MS	OECD 101 OCSPP 830 7050	Pure active substance 089-100112-1 99.67 percent	The mass spectra were consistent with the assigned structure of broflanilide.	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
Wavelengths at which UV/VIS molecular extinction occurs, max > 290 nm	OECD 101 OCSPP 830 7050	Pure active substance 089-100112-1 99.67 percent	See UV/VIS	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
Solubility of parent	EEC A.6, OECD 105 OCSPP 830.7840 Column elution method	Pure active substance 089-100112-1 99.67 percent	At 20 °C, 0.71 mg/L in purified water 0.28 mg/L in pH 4 buffer 0.51 mg/L in pH 7 buffer 3.6 mg/L in pH 10 buffer	P Sydney 2017b BROFLAN_003 MCAG identifier MUY0011 BASF DocID 2017/7008727
Solubility of metabolite DM-8007	EEC A.6, OECD 105 OCSPP 830.7840 Column elution method	173-150602-1 98.84 percent	At 20 °C, 6.63 µg/L in purified water 5.36 µg/L in pH 4 buffer 5.61 µg/L in pH 7 buffer 9.73 µg/L in pH 10 buffer	Y Ota 2017a BROFLAN_004 MCAG identifier 84490 BASF DocID 2017/7008733
Solubility of metabolite S(PFP-OH)-8007	EEC A.6, OECD 105 OCSPP 830.7840 Column elution method	kub150713 99.02 percent	At 20 °C, 61.1 µg/L in purified water 52.5 µg/L in pH 4 buffer 44.4 µg/L in pH 7 buffer 1710 µg/L in pH 10 buffer	Y Ota 2017b BROFLAN_005 MCAG identifier 84493 BASF DocID 2017/7008728
Solubility of metabolite DC-DM-8007	OECD 105 OCSPP 830.7840 Flask shake method	173-160128-1 99.83 percent	At 20 °C, 1603 µg/L in purified water 1271 µg/L in pH 4 buffer 1139 µg/L in pH 7 buffer 8000 µg/L in pH 10 buffer	L Panter 2017a BROFLAN_006 MCAG identifier 034811 BASF DocID 2017/7016494
Solubility in organic solvents	EEC A.6 Flask shake method	Pure active substance 089-100112-1 99.67 percent	At 20 °C, Heptane: 0.096 g/L Xylene: 6.0 g/L 1,2-Dichloroethane: 110 g/L Acetone: > 250 g/L Methanol: > 250 g/L n-Octanol: 7.4 g/L Ethyl acetate: >250 g/L	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755

Property	Method	Test material batch and purity	Results	Reference
n-Octanol/water partition coefficient of parent	EEC A.8 OECD 107 OCSPP 830.7550 Flask method	Pure active substance 089-100112-1 99.67 percent	At 20 °C, At pH 4: logP_{ow} = 5.2 At pH 7: logP_{ow} = 5.2 At pH 10: logP_{ow} = 4.4	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
n-Octanol/water partition coefficient of metabolite DM-8007	EEC A.8 OECD 107 OCSPP 830.7550 Shake flask method	173-150602-1 98.84 percent	At 25 °C, logP_{ow} = 5.66 in purified water logP_{ow} = 5.72 in pH 4 buffer logP_{ow} = 5.75 in pH 7 buffer logP_{ow} = 5.35 in pH 10 buffer	Y Ota 2016a BROFLAN_007 MCAG identifier 84491 BASF DocID 2016/7011899
n-Octanol/water partition coefficient of metabolite S(PFP-OH)-8007	EEC A.8 OECD 107 OCSPP 830.7550 Shake flask method	kub150713 99.02 percent	At 25 °C, logP_{ow} = 5.91 in purified water logP_{ow} = 5.84 in pH 4 buffer solution logP_{ow} = 5.06 in pH 7 buffer logP_{ow} = 4.80 in pH 10 buffer	Y Ota 2016b BROFLAN_008 MCAG identifier 84494 BASF DocID 2016/7012759
n-Octanol/water partition coefficient of metabolite DC-DM-8007	OECD 107 OCSPP 830.7550 Shake flask method	173-160128-1 99.83 percent	At 25 °C, logP_{ow} = 4.34 in purified water logP_{ow} = 4.38 in pH 4 buffer logP_{ow} = 4.47 in pH 7 buffer logP_{ow} = 3.80 in pH 10 buffer	L Panter 2017b BROFLAN_009 MCAG identifier 034812 BASF DocID 2017/7007421
Hydrolysis rate at pH 4, 7 and 9 under sterile and dark conditions	OECD 111 OPPTS 835.2120 JMAFF 8147	[B-ring-^{14}C]MCI-8007 Lot No. CFQ42036 Radiochemical purity: 98.11 percent	<10 percent degradation after 5 days at 50°C incubated at pH 4, 7, and 9 Broflanilide is considered hydrolytically stable.	M Schick 2016a BROFLAN_010 MCAG identifier 2499W-1 BASF DocID 2016/7012757
Direct phototransformation in sterile water using artificial light	OECD 316 OCSPP 835.2240 JMAFF 8147	[B-ring-^{14}C]MCI-8007 Lot No. CFQ42036 Radiochemical purity: 100 percent [C-ring-^{14}C]MCI-8007 Lot No. CFQ42037 Radiochemical purity: 100 percent	In pH 7 buffer solution at 25 °C DT_{50} = 845-1216 hours (69-89 OECD days, 79-123 US-EPA days, 222-287 JMAFF days) Identified metabolites: S(PFP-OH)-8007: up to 6.2% AR AB-oxa: up to 7.2% AR	M Ponte 2017a BROFLAN_011 MCAG identifier 2579W BASF DocID 2017/7016803

Property	Method	Test material batch and purity	Results	Reference
Direct phototransformation in sterile water using artificial light	OECD 316 OCSPP 835.2240 JMAFF 8147	[B-ring-^{14}C]MCI-8007 Lot No. CFQ42036 Radiochemical purity: 100 percent [C-ring-^{14}C]MCI-8007 Lot No. CFQ42037 Radiochemical purity: 100 percent	In pH 5 buffer solution at 25 °C DT_{50} = 136-204 hours (14-20 OECD days, 15 -22 US-EPA days, 44-64 JMAFF days) Identified metabolites: S(PFP-OH)-8007: up to 6.6% AR S(Br-OH)-8007: up to 16.1% AR DBr-8007: up to 3.8% AR AB-oxa: up to 7.9% AR In pH 9 buffer solution at 25 °C DT_{50} = 39-73 hours (3-6 OECD days, 4-7 US-EPA days, 11-21 JMAFF days) Identified metabolites: S(PFP-OH)-8007: up to 8.6% AR S(Br-OH)-8007: up to 5.5% AR S(F-OH)-8007: up to 6.9% AR DBr-8007: up to 6.5% AR AB-oxa: up to 40.5% AR	V Ponte 2017a BROFLAN_012 MCAG identifier 2914W BASF DocID 2017/7016650
Quantum yield of direct photo-transformation	OECD 316 OCSPP 835.2240 JMAFF 8147	[B-ring-^{14}C]MCI-8007 Lot No. CFQ42036 Radiochemical purity: 100 percent [C-ring-^{14}C]MCI-8007 Lot No. CFQ42037 Radiochemical purity: 100 percent	At 25 °C 1.09 x 10-3 in pH 5 buffer solution 4.42 × 10-4 in pH 7 buffer solution 6.83 × 10-3 in pH 9 buffer solution	M Ponte 2017a BROFLAN_011 MCAG identifier 2579W BASF DocID 2017/7016803 and V Ponte 2017 BROFLAN_012 MCAG identifier 2914W BASF DocID 2017/7016650
Dissociation in water of purified active substance	OECD 112 OCSPP 830.7370 Spectrophotometric method	Pure active substance 089-100112-1 99.67 percent	pKa = 8.8 at 20 °C	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
Dissociation in water of metabolite DM-8007	OECD 112 OCSPP 830.7370 Spectrophotometric method	173-150602-1 98.84 percent	pK_a = 8.78 at 20 °C	Y Ota 2016c BROFLAN_013 MCAG identifier 84492 BASF DocID 2016/7012758
Dissociation in water of metabolite S(PFP-OH)-8007	OECD 112 OCSPP 830.7370 Spectrophotometric method	kub150713 99.02 percent	pK_a = 11.17 at 20 °C	Y Ota 2016d BROFLAN_014 MCAG identifier 84495 BASF DocID 2016/7011896
Dissociation in water of metabolite DC-DM-8007	OCSPP 830.7370 Computational estimation	Not relevant	Estimated pK_a = 10.40	P. Miner 2017a BROFLAN_015 MCAG identifier 034813 BASF DocID 2017/7008692

Property	Method	Test material batch and purity	Results	Reference
Estimated photochemical oxidative degradation	Computational estimation	Not relevant	Hydroxyl radical rate constant: 4.32×10^{-12} $cm^3.molecule^{-1}.sec^{-1}$ Tropospheric half-life = 30 hours (Hydroxyl radical concentration of 1.5 × 10^6 per cm^3 and a 12-hour day)	P Sydney 2017a BROFLAN_001 2017d BROFLAN_002 MCAG identifier MUY0026 BASF DocID 2020/2109751 2020/2109755
Flammability including auto-flammability			No study submitted.	
Flash point			Not applicable to solids.	
Explosive properties			No study submitted.	
Surface Tension			No study submitted.	
Oxidizing properties			No study submitted.	
pH			No study submitted.	
Stability			See storage stability section	
Storage stability	OCSPP 830.6317 OCSPP 830.6320	Technical grade active substance 201211-001 98.67 percent	Stable for at least 1 year at ambient temperature	P Sydney 2017c BROFLAN_016 MCAG identifier MUY0028 BASF DocID 2017/7012222
Stability (temperature, metals)			No study submitted.	
Other/special studies			Not relevant.	

Notes:

[a.] ε is molar absorption coefficient (dm3.mol-1.cm-1). λmax is absorption wavelength maxima (nm). Maxima below 220 nm were disregarded as these are below the low wavelength cut-off of the solvents employed.

Formulations

Broflanilide is applied formulated alone or in combination with other active substances. It is formulated as aerosol dispenser (AE), bait (RB), emulsifiable concentrate (EC), flowable concentrate for seed treatment, (FS), suspension concentrate (SC), water dispersible granules (WG), and smoke generator (FU) products.

Table 2 Examples of formulations registered containing broflanilide as active ingredient

Formulation	Broflanilide content	Formulation	Broflanilide content
AE	0.0045% w/w	EC	1.5% w/w
AE	0.125% w/w	FS	300 g/L
AE	0.2% w/w (with insecticide)	FS	16.7 g/L (with fungicides)
RB	0.005% w/w	FU	10% w/w
RB	0.02% w/w	SC	100 g/L
RB	0.025% w/w	SC	5% w/w
RB	0.25% w/w	SC	12 % w/w
EC	5% w/w	SC	20 % w/w
EC	0.5% w/w	WG	5% w/w

METABOLISM AND ENVIRONMENTAL FATE

Metabolism studies were conducted using ^{14}C-labelled broflanilide. The position of the label for the test substances is presented in Figure 1:

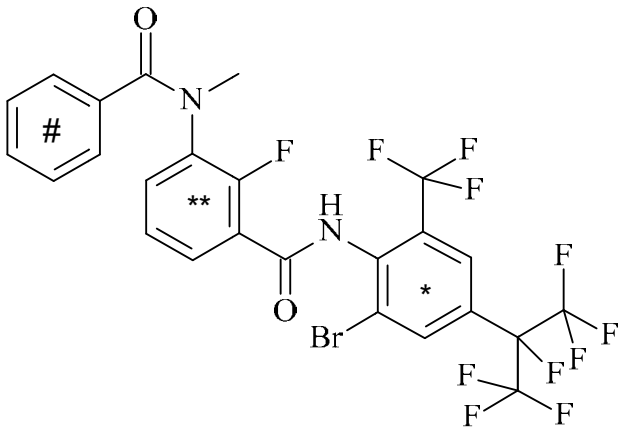


Figure 1 Structure of broflanilide and position of radiolabels

Notes:

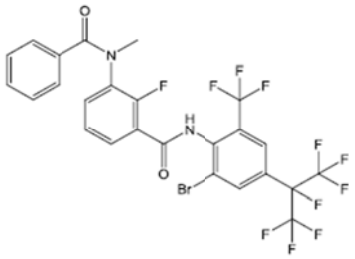
* Denotes [B-ring-U-^{14}C] radiolabelled material.

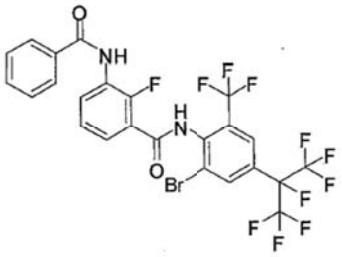
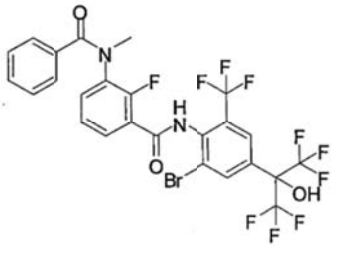
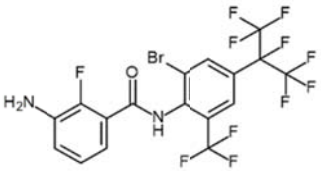
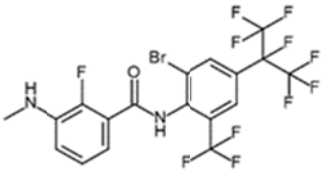
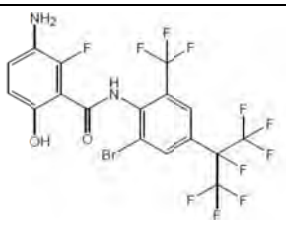
# Denotes [C-ring-U-^{14}C] radiolabelled material.

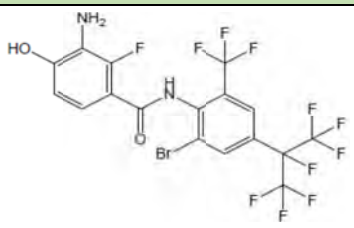
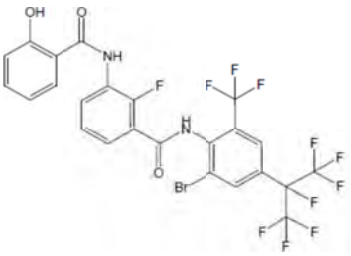
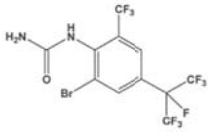
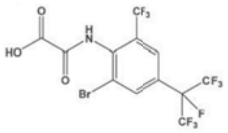
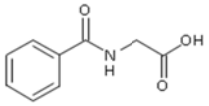
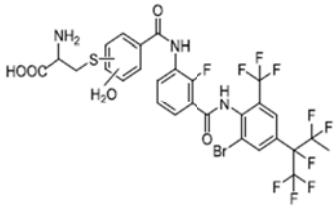
** Denotes [A-ring-U-^{14}C] radiolabelled material.

Chemical names, structures and code names of metabolites and degradation products of broflanilide are shown in Table 3.

Table 3 Known metabolites of broflanilide

Code Names	Chemical Names (IUPAC)	Structure	Where found
Broflanilide MCI-8007, BAS 450 I, MLP-8607, LS 5672774 LSP5672774 (Reg. No 5672774))	N-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-3-(*N*-methylbenzamido)benzamide	 Molar mass: 663.3 g/mol	Plants (cabbage (leaves), tomato (fruit & leaves), Japanese radish (leaves), soya bean (forage & hay), rice (foliage, brown rice, hulls & straw), tea (leaves)) Animals (goat (muscle, skim milk, milk fat, fats, liver, kidney), hen (egg white), fish) Soil (degradation & photolysis) Confined rotational crop (wheat (forage, hay & straw), lettuce (mature & immature), radish (leaves))

Code Names	Chemical Names (IUPAC)	Structure	Where found
DM-8007 M11 MLP-8473 (Reg. No 5856361)	3-benzamido-*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluorobenzamide	 Molar mass: 649.3 g/mol	Plants (cabbage (leaves), tomato (fruit & leaves), Japanese radish (leaves), soya bean (forage & hay), rice (foliage, brown rice, hulls & straw), tea (leaves)) Animals (goat (muscle, skim milk, milk fat, fats, liver, kidney), hen (muscle, fats, egg yolk, egg white, liver), fish) Soil (degradation & photolysis) Confined rotational crop (wheat (hay & straw), radish (leaves)
S(PFP-OH)-8007 M8 (Reg. No 5959598)	*N*-[2-bromo-4-(1,1,1,3,3,3-hexafluoro-2-hydroxypropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-3-(*N*-methylbenzamido)benzamide	 Molar mass: 661.3 g/mol	Plants (cabbage (leaves), tomato (fruit & leaves), Japanese radish (leaves), soya bean (forage & hay), rice (foliage, brown rice, hulls & straw), tea (leaves)) Soil (degradation)
DC-DM-8007 Reg.No. 5936906	3-amino-*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluorobenzamide	 Molecular mass: 545.1 g/mol	Animals (goat (muscle, skim milk, milk fat, fats, liver, kidney), hen, (muscle, fats, egg yolk, egg white, liver)) Soil
DC-8007 Reg.No. 5936907	*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-3-(methylamino)benzamide	 Molecular mass: 559.2 g/mol	Soil
DC-DM-(A4-OH)-8007	3-amino-*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-6-hydoxybenzamide		Animals (goat (liver, kidney)

Code Names	Chemical Names (IUPAC)	Structure	Where found
DC-DM-(A6-OH)-8007	3-amino-*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-4-hydoxybenzamide		Animals (goat (liver, kidney)
DM-(C2-OH)-8007	*N*-2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-3-(2-hydoxybenamido)benzamide		Animals (goat (liver, kidney)
B-urea (Reg. No 6065386)	[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]urea	 Molar mass: 451.1 g/mol	Confined rotational crop (wheat (forage, hay & straw), lettuce (immature & mature), radish (leaves))
B-oxam-acid (Reg. No 6066332)	*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]oxamic acid	 Molar mass: 480.1 g/mol	Confined rotational crop (wheat (forage, hay & straw), radish (leaves))
Hippuric acid	N-benzoylglycine		Animals (goat skim milk, kidney, liver)
H-U27B (and related but not fully elucidated H-U27C)	Hydroxyl cysteine conjugate of DM-8007		Animals (hen liver)

Plant metabolism

The metabolic fate in plants was investigated following application of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide to cabbage, tomato, Japanese radish, soya bean, rice, wheat and tea.

Cabbage

A metabolism study with cabbage (variety "Copenhagen Market") was performed outdoors with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide (Estigoy, 2017, BROFLAN_017). Cabbage plants received two foliar applications at nominal rates of 0.025 kg ai/ha each. The first application occurred on immature cabbage (BBCH 45) and the second application 7 days later (BBCH 46). Immature cabbage (BBCH 46) was harvested 6 days after application 1, while mature cabbage (BBCH 49) was harvested 21 days after application 2.

Upon arrival in the laboratory, the outer leaves of the cabbage plants were separated from the inner leaves. Only the outer leaves received a surface rinse with acetonitrile, before all samples were homogenized by blending with dry ice. The radioactive content in the samples was determined by combustion, followed by liquid scintillation counting (LSC). The radioactive content in the surface rinse was directly determined by LSC. Portions of the rinsed outer leaves and the inner leaves were subjected to extraction with acetonitrile (twice) and acetonitrile:water (1+1) (once). Radioactive components in the acetonitrile rinses and solvent extracts were determined by LSC and characterized/identified by co-chromatography with authentic reference standards using reverse phase HPLC, LC-MS and TLC.

Table 4 shows similar TRR levels for both labels with the majority of the radioactivity found in the outer cabbage leaves.

Table 4 Total radioactive residues in cabbage after two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Matrix	DAT	TRR determined (values are from the rinse + combustion of inner and outer leaves of cabbage plants [mg/kg]	TRR calculated [mg/kg] [1]
[B-ring-U-^{14}C]-broflanilide			
Cabbage (inner and outer leaves)	6 DAT1	0.352	0.306
Cabbage (inner and outer leaves)	21 DAT2	0.181	0.146
[C-ring-U-^{14}C]-broflanilide			
Cabbage (inner and outer leaves)	6 DAT1	0.304	0.264
Cabbage (inner and outer leaves)	21 DAT2	0.305	0.266

Notes:

[1] Sum of the residues in the surface rinses, acetonitrile:water extracts and PES.

The radioactivity extracted from homogenized cabbage is presented in Table 5. Inner leaves taken 21 DAT 2 were not extracted due to their low radioactivity (0.000-0.005 mg eq/kg). Extracted radioactivity was similar for both labels ranging between 92–95 percent TRR, while the PES accounted for 6–8 percent TRR (Table 5).

Table 5 Extractability of radioactive residues from cabbage samples after two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

	Immature cabbage (6 DAT1)		Mature cabbage (21 DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
TRR[1]	0.352		0.181	
Surface rinse	0.167	54.6	0.097	66.4
Extracts of rinsed outer leaves	0.075	24.5	0.038	26
PES of rinsed outer leaves	0.013	4.2	0.011	7.5
Extracts of inner leaves	0.047	15.4	n/a	n/a
PES of inner leaves	0.004	1.3	n/a	n/a
Sum ERR	0.289	94.5	0.135	92.4
Sum PES	0.017	5.5	0.011	7.5
TRR[2]	0.306	100	0.146	99.9

	Immature cabbage (6 DAT1)		Mature cabbage (21 DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR[2]/TRR[1]	86.9		80.7	
[C-ring-U-^{14}C]-broflanilide				
TRR[1]	0.304		0.305	
Surface rinse	0.125	47.3	0.168	63.2
Extracts of rinsed outer leaves	0.044	16.7	0.076	28.6
PES of rinsed outer leaves	0.008	3.0	0.017	6.4
Extracts of inner leaves	0.08	30.3	n/a	n/a
PES of inner leaves	0.007	2.7	0.005[3]	1.9[3]
Sum ERR	0.249	94.3	0.244	91.8
Sum PES	0.015	5.7	0.022	8.3
TRR[2]	0.264	100	0.266	100.1
TRR[2]/TRR[1]	86.8		87.2	

Notes:

[1] TRR determined (values are from the rinse + combustion of inner and outer layers of cabbage plants).

[2] TRR was calculated as the sums of the residues in the surface rinses, acetonitrile:water extracts and PES.

[3] Samples were not extracted (values are based on the combustion data).

The distribution of radioactivity in cabbage is presented in Table 6. Parent broflanilide was the major identified residue in immature and mature cabbage accounting for 66–84 percent TRR (0.10–0.25 mg eq/kg). Additionally two minor metabolites were identified, namely S(PFP-OH)-8007 and DM-8007 accounting for 3.8–7.6 percent TRR (0.01–0.012 mg eq/kg) and 2.9–7.9 percent TRR (0.009–0.021 mg eq/kg), respectively. The unextracted residue was not further characterized.

Table 6 Summary of identified/characterized residues in cabbage after two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	Immature cabbage (6 DAT1)		Mature cabbage (21 DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
TRR	0.306		0.145	
Surface rinse	0.167	54.6	0.097	66.9
Broflanilide	0.14	45.8	0.074	51.0
S(PFP-OH)-8007	0.006	2.0	0.007	4.8
DM-8007	0.003	1.0	0.005	3.4
Others[1]	0.018	5.9	0.01	6.9
Extract of outer leaves	0.075	24.5	0.038	26.2
Broflanilide	0.064	20.9	0.028	19.3
S(PFP-OH)-8007	0.004	1.3	0.004	2.8
DM-8007	0.004	1.3	0.005	3.4
Others[1]	0.003	1.0	0.001	0.7
Extract of inner leaves	0.047	15.4	n/a	n/a
Broflanilide	0.041	13.4	n/a	n/a
S(PFP-OH)-8007	0.002	0.7	n/a	n/a
DM-8007	0.002	0.7	n/a	n/a
Others[1]	0.002	0.7	n/a	n/a
Sum of broflanilide	0.245	80.1	0.102	70.3
Sum of S(PFP-OH)-8007	0.012	3.9	0.011	7.6
Sum of DM-8007	0.009	2.9	0.01	6.9

Fraction	Immature cabbage (6 DAT1)		Mature cabbage (21 DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
Total identified	0.266	86.9	0.123	84.8
Total characterized	0.023	7.5	0.011	7.6
Unextracted	0.017	5.5	0.011	7.5
Total	0.306	99.9	0.145	99.9
[C-ring-U-^{14}C]-broflanilide				
TRR	0.264		0.266	
Surface rinse	0.125	47.3	0.168	63.2
Broflanilide	0.109	41.3	0.119	44.7
S(PFP-OH)-8007	0.005	1.9	0.008	3.0
DM-8007	0.002	0.8	0.013	4.9
Others[2]	0.01	3.8	0.028	10.5
Extract of outer leaves	0.044	16.7	0.076	28.6
Broflanilide	0.039	14.8	0.057	21.4
S(PFP-OH)-8007	0.002	0.8	0.004	1.5
DM-8007	0.002	0.8	0.008	3.0
Others[2]	n/d		0.007	2.6
Extract of inner leaves	0.08	30.3	n/a	n/a
Broflanilide	0.073	27.7	n/a	n/a
S(PFP-OH)-8007	0.003	1.1	n/a	n/a
DM-8007	0.005	1.9	n/a	n/a
Others[2]	n/d		n/a	n/a
Sum of broflanilide	0.221	83.7	0.176	66.2
Sum of S(PFP-OH)-8007	0.01	3.8	0.012	4.5
Sum of DM-8007	0.009	3.4	0.021	7.9
Total identified	0.24	90.9	0.209	78.6
Total characterized	0.01	3.8	0.035	13.2
Unextracted	0.015	5.7	0.022	8.3
Total	0.265	100.4	0.266	100.0

Notes:

[1] 1-16 metabolites, none > 0.003 mg eq/kg (2.1 percent of TRR).

[2] 2-14 metabolites, none > 0.005 mg eq/kg (1.9 percent of TRR).

Tomato

A metabolism study with tomato (variety "Marglobe") was performed outdoors with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide (Estigoy, 2017, BROFLAN_018). Rain shelters constructed from galvanized pipe, lumber and clear greenhouse plastic were placed over the planter boxes for the duration of the study to protect them from thunderstorms. Tomato plants received two foliar applications at nominal rates of 0.025 kg ai/ha each. The first application occurred at the pre-bud stage (approx. BBCH 49-50) and the second application 83 days later at the beginning ripening stage (approx. BBCH 79-81). Immature tomatoes and leaves (BBCH 75) were harvested 71 days after application 1, while mature tomatoes and leaves (~BBCH 88) were harvested 10 days after application 2.

Upon arrival in the laboratory, samples were immersed in acetonitrile to remove surface residues. The rinsed tomatoes and leaves were homogenized by blending with dry ice and the radioactive content in

the samples was determined by combustion, followed by liquid scintillation counting (LSC). Portions of the tomato leaves from harvest 2 were subjected to extraction with acetonitrile (twice) and acetonitrile:water (1+1) (once). Radioactive components in the acetonitrile rinses and solvent extracts were determined by LSC and characterized/identified by co-chromatography with authentic reference standards using reverse phase HPLC, LC-MS and TLC. The PES of the tomato leaves from harvest 2 was further characterized by treatment with 0.01M Na2-EDTA solution, followed by incubation in a water bath (30 °C) for 23 hours (pectin extraction) and with dimethyl sulfoxide followed by incubation in a water bath (80 °C) for 48 hours (lignin extraction). Tomato fruits from both harvests and immature tomato leaves from harvest 1were not extracted, due to the low amount of radioactivity present (< 0.001 mg eq/kg).

TRR was very low or non-detectable in samples taken at harvest 1 (Table 7), while in samples from harvest 2 similar TRR levels were found for both labels, with the majority of the radioactivity detected in the leave surface rinse samples.

Table 7 Total radioactive residues in tomato fruits and leaves after two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	Residue at Harvest 1 (71DAT1) mg eq/kg	Residue at Harvest 2 (10DAT2) mg eq/kg
[B-ring-U-^{14}C]-broflanilide		
Leaves Surface Rinse	0.000	1.057
Rinsed Leaves	0.001	0.539
Leaves total	0.001	1.596
Fruit Surface Rinse	n/d	0.007
Rinsed Fruits	n/d	0.003
Fruit total	n/d	0.010
[C-ring-U-^{14}C]-broflanilide		
Leaves Surface Rinse	0.000	0.678
Rinsed Leaves	0.000	0.226
Leaves total	0.000	0.904
Fruit Surface Rinse	n/d	0.008
Rinsed Fruits	n/d	0.002
Fruit total	n/d	0.010

The radioactivity extracted from homogenized tomato fruits and leaves taken at harvest 2 is presented in Table 8. Extracted radioactivity was similar for both labels ranging between 96–99 percent TRR in tomato leaves and 70–80 percent TRR in tomato fruit. The PES in tomato fruit accounted for 20–30 percent TRR, but was < 0.003 mg eq/kg in absolute concentration.

Table 8 Extractability of radioactive residues from tomato fruits and leaves of harvest 2 after two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	Tomato Leaves (10DAT2)		Tomato Fruits (10DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
TRR[1]	1.596		0.01	
Surface rinse	1.057	70.2	0.007	70.0
Extracts	0.388	25.8	n/a	n/a
PES	0.06	4.0	0.003	30.0
Sum ERR	1.445	96.0	0.007	70.0

Fraction	Tomato Leaves (10DAT2)		Tomato Fruits (10DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR[2]	1.505	100	0.01	100
TRR[2]/TRR[1]	94		100	
[C-ring-U-^{14}C]-broflanilide				
TRR[1]	0.904		0.01	
Surface rinse	0.678	79.7	0.008	80
Extracts	0.162	19.0	n/a	n/a
PES	0.011	1.3	0.002	20.0
Sum ERR	0.84	98.7	0.008	80
TRR[2]	0.851	100	0.01	100
TRR[2]/TRR[1]	94		100	

Notes:

[1] TRR determined (values are from the rinse + combustion of tomato fruits and leaves).

[2] TRR was calculated as the sums of the residues in the surface rinses, acetonitrile:water extracts and PES.

The distribution of radioactivity in tomato fruits and leaves is presented in Table 9. Parent broflanilide was the major identified residue, accounting for 87–89 percent TRR (0.76–1.3 mg eq/kg) in tomato leaves and 60–68 percent TRR (0.006–0.007 mg eq/kg) in tomato fruit. Additionally, metabolites S(PFP-OH)-8007 and DM-8007 were identified in tomato leaves and fruit at minor levels of 3.0–3.4 percent TRR (0.0003–0.051 mg eq/kg) and 3.4–4.0 percent TRR (0.0004–0.060 mg eq/kg), respectively. Further characterization of the PES from tomato leaves treated with [B-ring-U-^{14}C]-labelled broflanilide allocated 0.3 percent TRR to pectin and 2.5 percent TRR to lignin.

Table 9 Summary of identified/characterized residues in tomato fruits and leaves after two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	Tomato Leaves (10DAT2)		Tomato Fruits (10DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
TRR	1.505		0.01	
Surface rinse	1.057	70.2	0.007	70.0
Broflanilide	0.971	64.5	0.006	60.0
S(PFP-OH)-8007	0.032	2.1	n/d	
DM-8007	0.035	2.3	0.0004[3]	4.0[3]
Others[1]	0.019	1.3	0.001	10.0
Extract	0.388	25.8	n/a	n/a
Broflanilide	0.334	22.2	n/a	n/a
S(PFP-OH)-8007	0.019	1.3	n/a	n/a
DM-8007	0.025	1.7	n/a	n/a
Others[1]	0.01	0.7	n/a	n/a
PES	0.06	4.0	0.003	30.0
Pectin extraction	0.0048	0.3	n/a	n/a
Lignin extraction	0.0377	2.5	n/a	n/a
Sum of broflanilide	1.305	86.7	0.006	60.0
Sum of S(PFP-OH)-8007	0.051	3.4	n/d	0.0
Sum of DM-8007	0.06	4.0	0.0004	4.0

Fraction	Tomato Leaves (10DAT2)		Tomato Fruits (10DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
Total identified	1.416	94.1	0.0064	64.0
Total characterized	0.0715	4.8	0.001	10.0
Unextracted	0.0176	1.2	0.003	30.0
Total	1.505	100.0	0.010	104.0
[C-ring-U-^{14}C]-broflanilide				
TRR	0.851		0.01	
Surface rinse	0.678	79.7	0.008	80.0
Broflanilide	0.622	73.1	0.0068	68.0
S(PFP-OH)-8007	0.02	2.4	0.0003	3.0
DM-8007	0.018	2.1	0.0004[3]	4.0[3]
Others[2]	0.018	2.1	0.0009	9.0
Extract	0.162	19.0	n/a	n/a
Broflanilide	0.139	16.3	n/a	n/a
S(PFP-OH)-8007	0.009	1.1	n/a	n/a
DM-8007	0.011	1.3	n/a	n/a
Others[2]	0.004	0.5	n/a	n/a
Sum of broflanilide	0.761	89.4	0.0068	68
Sum of S(PFP-OH)-8007	0.029	3.4	0.0003	3.0
Sum of DM-8007	0.029	3.4	0.0004	4.0
Total identified	0.819	96.2	0.0075	75.0
Total characterized	0.022	2.6	0.0009	9.0
Unextracted	0.011	1.3	0.002	20.0
Total	0.852	100.1	0.010	104.0

Notes:

[1] 1-14 metabolites, none >0.007 mg eq/kg (0.5 percent of TRR).

[2] 2-4 metabolites, none >0.006 mg eq/kg (0.7 percent TRR).

[3] DM-8007 metabolite was characterized by normal phase TLC analysis, but not detected in HPLC analysis.

Japanese radish

A metabolism study with Japanese radish (variety Karayoshi, *Raphanus sativus* L. *var. longipinnatus*) was performed indoors (glass green house) with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide (Hayashi, 2017, BROFLAN_019). The first treatment was applied to the soil at a rate of 0.4 kg ai/ha immediately after seeding, and a second treatment was applied foliar 41 days later at a rate of 0.225 kg ai/ha, 29 days before the final harvest. Plants (leaves and root) were collected at three sampling points: 40DAT1 (intermediate harvest-1), 14DAT2 (intermediate harvest-2) and 29DAT2 (final harvest).

Upon arrival in the laboratory, the leaves were surface-rinsed with acetonitrile (except the intermediate harvest-1) and the root was further separated into peel and flesh. All samples were homogenized by blending with dry ice. Portions of the samples were subjected to extraction with acetonitrile:water (8+2) (twice) and acetonitrile:0.1M HCl (8+2) (once). The extract were pooled and the radioactive content determined by LSC, while the PES was combusted first and then the radioactivity determined by LSC. The leaf extract at the intermediate harvest-2 and the final harvest contained feasible amounts of radioactivity for further characterization/identification. These extracts were fractionated by solid phase extraction (SPE), followed by quantification of the radioactive components by co-

chromatography with authentic reference standards using reverse phase HPLC and TLC. All other samples were not further analysed.

The TRR was calculated as the sum of the rinse/extracts and the PES for each matrix, rather than by direct combustion (Table 10). TRR was generally < 0.01 mg eq/kg for both labels in samples taken at the intermediate harvest 1. In samples from the intermediate harvest-2 and from the final harvest, the TRR was at least two orders in magnitude higher in the leaves compared to the root, but similar among each matrix for both time points and labels.

Table 10 Total radioactive residues in radish leaves and roots after treatment with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Matrix	Intermediate harvest-1 (40DAT1) mg eq/kg	Intermediate harvest-2 (14DAT2) mg eq/kg	Final harvest (29DAT2) mg eq/kg
[B-ring-U-^{14}C]-broflanilide			
Leaves	0.0059	3.868	4.178
Roots	0.0038	0.0113	0.0036
[C-ring-U-^{14}C]-broflanilide			
Leaves	0.0069	4.443	3.608
Roots	0.0075	0.0112	0.0119

The radioactivity extracted from homogenized radish leaves ranged between 95–99 percent TRR, except for leaves from intermediate harvest 1 for the C-ring label with 70 percent TRR (Table 11). In these samples the radioactivity in the PES accounted for 30 percent TRR, but was < 0.0021 mg eq/kg in absolute concentration. In radish roots, the sum of the radioactivity found in peel and flesh extracts ranged between 54–96 percent TRR, while 4.3–47 percent TRR remained in the PES. However, absolute concentrations in the PES were throughout < 0.0056 mg eq/kg.

Table 11 Extractability of radioactive residues from radish leaves after treatment with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Radish leaves	Intermediate harvest-1 (40DAT1)		Intermediate harvest-2 (14DAT2)		Final harvest (29DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide						
TRR	0.0059	100	3.868	100.0	4.178	100.0
Surface Rinse	n/a	n/a	3.571	92.3	3.806	91.1
Extract	0.0056	94.6	0.249	6.4	0.314	7.5
PES	0.0003	5.4	0.048	1.3	0.058	1.4
Sum ERR	0.0056	94.6	3.820	98.8	4.120	98.6
[C-ring-U-^{14}C]-broflanilide						
TRR	0.0069	100	4.443	100.0	3.608	100.0
Surface Rinse	n/a	n/a	4.125	92.9	3.252	90.2
Extract	0.0048	69.7	0.274	6.2	0.304	8.4
PES	0.0021	30.3	0.044	1.0	0.053	1.5
Sum ERR	0.0048	69.7	4.398	99.0	3.555	98.5

Table 12 Extractability of radioactive residues from radish roots after treatment with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Radish roots	Intermediate harvest-1 (40DAT1)		Intermediate harvest-2 (14DAT2)		Final harvest (29DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide						
TRR	0.0038	100.0	0.0113	100.0	0.0036	100.0
Extracts of peel	0.0019	50.7	0.0085	75.5	0.0031	84.4
PES of peel	0.0001	2.5	0.0005	4.2	0.0003	9.6
Extracts of flesh	0.0017	45.0	0.0021	18.7	< 0.0001	<LOD
PES of flesh	0.0001	1.8	0.0002	1.7	0.0002	6.0
Sum ERR	0.0036	95.7	0.0106	94.2	0.0031	84.4
Sum PES	0.0002	4.3	0.0007	5.8	0.0005	15.6
[C-ring-U-^{14}C]-broflanilide						
TRR	0.0075	100.0	0.0112	100.0	0.0119	100.0
Extracts of peel	0.0034	45.0	0.0064	57.1	0.0033	26.4
PES of peel	0.0004	5.0	0.0005	4.6	0.0001	1.1
Extracts of flesh	0.0026	34.6	0.0029	25.7	0.003	27.2
PES of flesh	0.0012	15.5	0.0014	12.7	0.0055	45.3
Sum ERR	0.006	79.5	0.0093	82.8	0.0063	53.6
Sum PES	0.0016	20.5	0.0019	17.3	0.0056	46.5

The distribution of radioactivity in radish leaves is presented in Table 13. Parent broflanilide was the major identified residue, accounting for 77–82 percent TRR (2.8–3.6 mg eq/kg). Additionally, metabolites S(PFP-OH)-8007 and DM-8007 were identified at minor levels of 1.7–2.9 percent TRR (0.067–0.12 mg eq/kg) and 2.5–3.3 percent TRR (0.11–0.13 mg eq/kg), respectively. The radioactive residue in radish roots was not further characterized.

Table 13 Summary of identified/characterized residues in radish leaves after treatment with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	Leaves (intermediate harvest 2, 14DAT2)		Leaves (final harvest, 29DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
TRR	3.868		4.178	
Surface rinse	3.571	92.3	3.806	91.1
Broflanilide	2.967	76.7	3.147	75.3
S(PFP-OH)-8007	0.056	1.5	0.107	2.6
DM-8007	0.098	2.5	0.115	2.8
Extract	0.249	6.4	0.314	7.5
Broflanilide	0.153	3.9	0.178	4.3
S(PFP-OH)-8007	0.011	0.3	0.013	0.3
DM-8007	0.008	0.2	0.018	0.4
Sum of broflanilide	3.120	80.7	3.325	79.6
Sum of S(PFP-OH)-8007	0.067	1.7	0.121	2.9

Fraction	Leaves (intermediate harvest 2, 14DAT2)		Leaves (final harvest, 29DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
Sum of DM-8007	0.106	2.7	0.133	3.2
Total identified	3.293	85.1	3.579	85.7
Characterized by HPLC	0.490[1]	12.7	0.484[2]	11.6
Unextracted	0.027	0.7	0.031	0.7
Total	3.810	98.5	4.094	98.0
[C-ring-U-^{14}C]-broflanilide				
TRR	4.443		3.608	
Surface rinse	4.125	92.8	3.252	90.1
Broflanilide	3.495	78.7	2.613	72.4
S(PFP-OH)-8007	0.097	2.2	0.080	2.2
DM-8007	0.095	2.1	0.101	2.8
Extract	0.274	6.2	0.304	8.4
Broflanilide	0.147	3.3	0.150	4.2
S(PFP-OH)-8007	0.006	0.1	0.008	0.2
DM-8007	0.017	0.4	0.017	0.5
Sum of broflanilide	3.642	82.0	2.763	76.6
Sum of S(PFP-OH)-8007	0.103	2.3	0.089	2.5
Sum of DM-8007	0.112	2.5	0.118	3.3
Total identified	3.856	86.8	2.969	82.3
Characterized by HPLC	0.452[3]	10.2	0.500[4]	13.8
Unextracted	0.071	1.6	0.063	1.7
Total	4.380	98.6	3.531	97.9

Notes:

[1] 14 metabolites, none >0.129 mg eq/kg (3.3 percent of TRR).

[2] 15 metabolites, none >0.151 mg eq/kg (3.6%TRR).

[3] 7 metabolites, none >0.144 mg eq/kg (3.2 percent of TRR).

[4] 15 metabolites, none >0.128 mg eq/kg (3.5 percent of TRR).

Soya bean

A metabolism study with soya bean (variety "Woodruff") was performed outdoors with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide (Estigoy, 2017, BROFLAN_020). Rain shelters constructed from galvanized pipe, lumber and clear greenhouse plastic were placed over the planter boxes for the duration of the study to protect them from thunderstorms. Soya plants received two foliar applications at nominal rates of 0.025 kg ai/ha each. The first application occurred at bud formation (approx. BBCH 49–51) and the second application 77 days later at the beginning of the pod and seed ripening stage (approximately BBCH 79–81). Soya bean forage and hay samples were harvested 21DAT1 (BBCH 69) and 35DAT1 (BBCH 74), respectively. The mature soya bean seeds were harvested 12DAT2.

Upon arrival in the laboratory, soya bean forage and hay were surface-rinsed with acetonitrile and the radioactivity determined by LSC. Subsequently all samples were homogenized by blending with dry ice and their radioactive content determined by combustion, followed by LSC. Portions of soya bean forage and hay were subjected to extraction with acetonitrile (twice) and acetonitrile:water (1+1) (once). The extract were pooled and the radioactive content determined by LSC. The extracts were further

characterized/identified by co-chromatography with authentic reference standards using reverse phase HPLC, LC-MS and TLC. The PES of soya bean hay were further characterized by treatment with 0.01M Na2-EDTA solution, followed by incubation in a water bath (30 °C) for 23 hours (pectin extraction) and with dimethyl sulfoxide followed by incubation in a water bath (80 °C) for 48 hours (lignin extraction). Soya bean seeds were not further analysed due to the low amount of radioactivity present.

Table 14 shows similar TRR levels for both labels with the highest levels found in soya bean forage. In soya bean seeds, the detected radioactivity was < 0.01 mg eq/kg for both labels.

Table 14 Total radioactive residues (mg eq./kg) in soya bean forage, hay and seeds after treatment with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Matrix	DAT	TRR measured (rinse + combustion)	TRR calculated [1]
[B-ring-U-^{14}C]-broflanilide			
Forage	21 DAT1	0.460	0.451
Hay	35 DAT1	0.261	0.263
Seed	12 DAT2	0.008	n/a
[C-ring-U-^{14}C]-broflanilide			
Forage	21 DAT1	0.433	0.426
Hay	35 DAT1	0.287	0.284
Seed	12 DAT2	0.008	n/a

Notes:

[1] Sum of the residues in the surface rinses, acetonitrile:water extracts and PES.

The radioactivity extracted from homogenized soya bean forage and hay is presented in Table 15. Extracted radioactivity was similar for both labels ranging between 92–93 percent TRR in soya bean forage and 89–91 percent TRR in soya bean hay.

Table 15 Extractability of radioactive residues from soya bean forage and hay after two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	Soya bean forage (21DAT1)		Soya bean hay (35DAT1)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
TRR[1]	0.46		0.261	
Surface rinse	0.309	68.5	0.138	52.5
Extracts	0.111	24.6	0.1	38
PES	0.031	6.9	0.025	9.5
Sum ERR	0.42	93.1	0.238	90.5
TRR[2]	0.451	100	0.263	100
TRR[2]/TRR[1]	98.0		100.8	
[C-ring-U-^{14}C]-broflanilide				
TRR[1]	0.433		0.287	
Surface rinse	0.287	67.4	0.151	53.2
Extracts	0.106	24.9	0.102	35.9
PES	0.033	7.7	0.031	10.9
Sum ERR	0.393	92.3	0.253	89.1
TRR[2]	0.426	100	0.284	100
TRR[2]/TRR[1]	98.4		99.0	

Notes:

[1] TRR determined (values are from the direct determination of rinse + combustion).

[2] TRR was calculated as the sums of the residues in the surface rinses, extracts and PES.

The distribution of radioactivity in soya bean forage and hay is presented in Table 16. Parent broflanilide was the major identified residue, accounting for 75–76 percent TRR (0.32–0.34 mg eq/kg) in soya bean forage and 67–71 percent TRR (0.19 mg eq/kg) in soya bean hay. Additionally, metabolites S(PFP-OH)-8007 and DM-8007 were identified in soya bean forage and hay at minor levels of 3.8–5.6 percent TRR (0.010–0.021 mg eq/kg) and 5.1–8.3 percent TRR (0.022–0.023 mg eq/kg), respectively. Further characterization of the PES from soya bean hay treated with [C-ring-U-^{14}C]-labelled broflanilide allocated 3.0 percent TRR to pectin and 4.3 percent TRR to lignin (Table 16).

Table 16 Summary of identified/characterized residues in soya bean forage and hay after two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	Soya bean forage (21DAT1)		Soya bean hay (35DAT1)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
TRR	0.45		0.265	
Surface rinse	0.309	68.7	0.139	52.5
Broflanilide	0.271	60.2	0.116	43.8
S(PFP-OH)-8007	0.013	2.9	0.006	2.3
DM-8007	0.014	3.1	0.008	3.0
Others[1]	0.011	2.4	0.009	3.4
Extract	0.141	31.3	0.126	47.5
Broflanilide	0.067	14.9	0.072	27.2
S(PFP-OH)-8007	0.008	1.8	0.004	1.5
DM-8007	0.009	2.0	0.014	5.3
Others[1]	0.026	5.8	0.011	4.2
Sum of broflanilide	0.338	75.1	0.188	70.9
Sum of S(PFP-OH)-8007	0.021	4.7	0.01	3.8
Sum of DM-8007	0.023	5.1	0.022	8.3
Total identified	0.382	84.9	0.22	83.0
Total characterized	0.037	8.2	0.02	7.5
Unextracted	0.031	6.9	0.025	9.4
Total	0.45	100.0	0.265	100.0
[C-ring-U-^{14}C]-broflanilide				
TRR	0.427		0.284	
Surface rinse	0.288	67.4	0.151	53.2
Broflanilide	0.262	61.4	0.13	45.8
S(PFP-OH)-8007	0.012	2.8	0.008	2.8
DM-8007	0.012	2.8	0.01	3.5
Others[2]	0.002	0.5	0.003	1.1
Extract	0.139	32.6	0.133	46.8
Broflanilide	0.062	14.5	0.059	20.8
S(PFP-OH)-8007	0.007	1.6	0.008	2.8
DM-8007	0.01	2.3	0.013	4.6
Others[2]	0.027	6.3	0.022	7.7

Fraction	Soya bean forage (21DAT1)		Soya bean hay (35DAT1)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
PES	0.033	7.7	0.031	10.9
Pectin extraction	n/a		0.008	3.0
Lignin extraction	n/a		0.012	4.3
Sum of broflanilide	0.324	75.9	0.189	66.5
Sum of S(PFP-OH)-8007	0.019	4.4	0.016	5.6
Sum of DM-8007	0.022	5.2	0.023	8.1
Total identified	0.365	85.5	0.228	80.3
Total characterized	0.029	6.8	0.046	16.1
Unextracted	0.033	7.7	0.010	3.7
Total	0.427	100.0	0.284	100.0

Notes:

[1] 2-12 metabolites, none >0.005 mg eq/kg (1.9 percent of TRR).

[2] 1-10 metabolites, none >0.009 mg eq/kg (2.1%TRR).

Rice

A metabolism study with rice (variety Koshihikari, *Oryza sativa* L.) was performed indoors (glass green house) with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide (Hayashi, 2017, BROFLAN_021). The first treatment was applied to the flooding water at a rate of 0.3 kg ai/ha immediately after transplantation of the rice seedlings, and a second treatment was applied foliar at a rate of 0.15 kg ai/ha 73 days later. The rice plants were collected at 13DAT2 (intermediate harvest: foliage) and 32DAT2 (the final harvest: brown rice, hulls, straw and root).

Upon arrival in the laboratory, only the foliage from the intermediate harvest was surface-rinsed with acetonitrile, before all samples were homogenized by blending with dry ice. Portions of the foliage, brown rice, straw and hulls were subjected to extraction with acetonitrile:water (8+2) (twice). The extract were pooled and the radioactive content determined by LSC, while the radioactivity in the PES was determined by combustion analysis. Further characterization of the extracts was accomplished by SPE fractionation, followed by identification of the radioactive components by co-chromatography with authentic reference standards using reverse phase HPLC, TLC and LC-TOF-MS. The PES of brown rice was further characterized by washing with a 50 mM KH_2PO_4/NaOH buffer (pH 6.9), followed by enzymatic treatment with α-amylase (starch fraction) and protease (protein fraction) and acidic hydrolysis with 6 N H_2SO_4.The PES of hulls and straw was further characterized by sequential extraction with 0.1 M HCl solution, 1 percent Na2-EDTA (pectin fraction), dimethylsulfoxide (lignin fraction), 24 percent KOH solution (hemicellulose fraction) and 72 percent sulfuric acid solution (cellulose fraction).

The TRR was calculated as the sum of the rinse (foliage only), extracts and the PES for each matrix, except for root, where the radioactivity was directly determined via combustion Table 17. TRR was generally similar for both labels with the highest levels found in rice hulls and straw. In brown rice levels were at least one order in magnitude lower and levels differed significantly between labels (B-ring label about 1/5 of C-ring label). The higher radioactivity in the brown rice using the C-ring label was explained by the possible generation of $^{14}CO_2$, followed by incorporation through photosynthesis.

Table 17 Total radioactive residues (mg eq./kg) in rice forage, brown rice, hulls, straw and root after treatment with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Matrix	DAT	TRR measured	TRR calculated[1]
[B-ring-U-^{14}C]-broflanilide			
Foliage	13DAT2	n/a	1.1491
Brown rice	32DAT2	n/a	0.0207
Hulls		n/a	5.5093
Straw		n/a	4.8864
Root		1.6821	n/a
[C-ring-U-^{14}C]-broflanilide			
Foliage	13DAT2	n/a	1.9096
Brown rice	32DAT2	n/a	0.1114
Hulls		n/a	6.7494
Straw		n/a	4.1665
Root		0.7560	n/a

Notes:

[1] TRR calculated as the sum of surface rinse (foliage only), extracts and PES.

The extracted radioactivity from rice forage, brown rice, hulls and straw ranged between 85–98 percent TRR, except for brown rice for the C-ring label with 18 percent TRR (Table 18). In these samples the radioactivity in the PES accounted for 82 percent TRR. Since the absolute measured radioactivity in the extracts was similar for both labels, it was assumed that the higher radioactivity in the PES from C-ring label was due to incorporation of $^{14}CO_2$, into the plant matrix.

Table 18 Extractability of radioactive residues from rice forage, brown rice, hulls and straw after treatment with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

	Foliage (13DAT2)		Brown rice (32DAT2)		Hulls (32DAT2)		Straw (32DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide								
TRR	1.149	100.0	0.021	100.0	5.509	100.0	4.886	100.0
Surface Rinse	0.929	80.8	n/a		n/a		n/a	
Extract	0.198	17.2	0.018	85.4	5.376	97.6	4.787	98.0
PES	0.022	2.0	0.003	14.6	0.134	2.4	0.099	2.0
Sum ERR	1.127	98.1	0.018	85.4	5.376	97.6	4.787	98.0
[C-ring-U-^{14}C]-broflanilide								
TRR	1.910	100.0	0.111	100.0	6.749	100.0	4.167	100.0
Surface Rinse	1.260	66.0	n/a		n/a		n/a	
Extract	0.567	29.7	0.020	18.1	6.455	95.6	4.026	96.6
PES	0.083	4.3	0.091	81.9	0.295	4.4	0.140	3.4
Sum ERR	1.827	95.7	0.020	18.1	6.455	95.6	4.026	96.6

The distribution of radioactivity in rice forage, brown rice, hulls and straw is presented in Table 19. Parent broflanilide was the major identified residue, accounting for 84-87 percent TRR (1.0-1.6 mg eq/kg) in rice foliage, 83–90 percent TRR (4.6–8.1 mg eq/kg) in hulls and 85–87 percent TRR (3.6–4.1 mg eq/kg) in straw. In brown rice relative amounts of parent broflanilide differed significantly

between the two labels, accounting for 64 percent TRR using the B-ring label and 13 percent TRR using the C-ring label. However, absolute numbers were comparable at 0.013–0.014 mg eq/kg. Additionally, metabolites S(PFP-OH)-8007 and DM-8007 were identified in all matrices at minor levels of 1.0–8.5 percent TRR (0.002–0.28 mg eq/kg) and 0.8–5.4 percent TRR (0.001–0.26 mg eq/kg), respectively (Table 19).

Table 19 Summary of identified/characterized residues in rice forage, brown rice, hulls and straw after treatment with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	Foliage (13DAT2)		Brown rice (32DAT2)		Hulls (32DAT2)		Straw (32DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide								
TRR	1.149	100.0	0.021	100.0	5.509	100.0	4.886	100.0
Surface rinse	0.929	80.8	n/a		n/a		n/a	
Broflanilide	0.820	71.4	n/a		n/a		n/a	
S(PFP-OH)-8007	0.034	2.9	n/a		n/a		n/a	
DM-8007	0.032	2.8	n/a		n/a		n/a	
Extract	0.198	17.2	0.018	85.4	5.376	97.6	4.787	98.0
Broflanilide	0.176	15.3	0.013	63.6	4.568	82.9	4.133	84.7
S(PFP-OH)-8007	0.011	1.0	0.002	8.5	0.251	4.6	0.229	4.7
DM-8007	0.011	1.0	0.001	5.0	0.214	3.9	0.264	5.4
Sum of broflanilide	0.996	86.7	0.013	63.6	4.568	82.9	4.133	84.7
Sum of S(PFP-OH)-8007	0.045	3.9	0.002	8.5	0.251	4.6	0.229	4.7
Sum of DM-8007	0.043	3.8	0.001	5.0	0.214	3.9	0.264	5.4
Characterized by HPLC	0.045[1]	3.9	<LOD		0.256[4]	4.6	0.162[6]	3.3
PES	0.022	2.0	0.003	14.6	0.134	2.4	0.099	2.0
Buffer rinse	n/a		<LOD		n/a		n/a	
Starch fraction	n/a		0.001	2.9	n/a		n/a	
Protein fraction	n/a		<LOD		n/a		n/a	
6 N H2SO4 reflux	n/a		<LOD		n/a		n/a	
0.1 M HCl extract	n/a		n/a		0.007	0.1	0.003	0.1
Pectin fraction	n/a		n/a		0.003	0.1	0.001	0.0
Lignin fraction	n/a		n/a		0.087	1.6	0.036	0.7
Hemicellulose fraction	n/a		n/a		0.039	0.7	0.031	0.6
Cellulose fraction	n/a		n/a		<LOD	<LOD	0.006	0.1
Total identified	1.084	94.3	0.016	77.2	5.033	91.4	4.626	94.7
Total characterized	0.045	3.9	0.001	2.9	0.391	7.1	0.240	4.9
Unextracted	0.022	2.0	0.001	4.8	0.020	0.4	0.010	0.2
Total	1.151	100.1	0.018	84.9	5.444	98.8	4.875	99.8
[C-ring-U-^{14}C]-broflanilide								
TRR	1.910	100.0	0.111	100.0	6.749	100.0	4.167	100.0
Surface rinse	1.260	66.0	n/a		n/a		n/a	
Broflanilide	1.106	57.9	n/a		n/a		n/a	
S(PFP-OH)-8007	0.046	2.4	n/a		n/a		n/a	
DM-8007	0.047	2.4	n/a		n/a		n/a	
Extract	0.567	29.7	0.020	18.1	6.455	95.6	4.026	96.6
Broflanilide	0.491	25.7	0.014	12.5	6.088	90.2	3.634	87.2

Fraction	Foliage (13DAT2)		Brown rice (32DAT2)		Hulls (32DAT2)		Straw (32DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
S(PFP-OH)-8007	0.030	1.6	0.002	1.4	0.276	4.1	0.166	4.0
DM-8007	0.019	1.0	0.001	0.8	0.184	2.7	0.194	4.7
Sum of broflanilide	1.598	83.7	0.014	12.5	6.088	90.2	3.634	87.2
Sum of S(PFP-OH)-8007	0.075	3.9	0.002	1.4	0.276	4.1	0.166	4.0
Sum of DM-8007	0.066	3.5	0.001	0.8	0.184	2.7	0.194	4.7
Characterized by HPLC	0.093[2]	4.9	0.002[3]	1.5	0.059[5]	0.9	0.087[7]	2.1
PES	0.083	4.3	0.091	81.9	0.295	4.4	0.140	3.4
Buffer rinse	n/a		0.003	2.9	n/a		n/a	
Starch fraction	n/a		0.024	21.4	n/a		n/a	
Protein fraction	n/a		0.008	7.2	n/a		n/a	
6 N H2SO4 reflux	n/a		0.046	41.6	n/a		n/a	
0.1 M HCl extract	n/a		n/a		0.014	0.2	0.005	0.1
Pectin fraction	n/a		n/a		0.007	0.1	0.002	0.1
Lignin fraction	n/a		n/a		0.133	2.0	0.040	1.0
Hemicellulose fraction	n/a		n/a		0.065	1.0	0.041	1.0
Cellulose fraction	n/a		n/a		0.042	0.6	0.023	0.5
Total identified	1.739	91.1	0.016	14.7	6.549	97.0	3.993	95.8
Total characterized	0.093	4.9	0.083	74.7	0.319	4.7	0.198	4.8
Unextracted	0.083	4.3	0.009	8.3	0.047	0.7	0.022	0.5
Total	1.915	100.3	0.109	97.7	6.916	102.5	4.214	101.1

Notes:

[1] 8 metabolites, none >0.008 mg eq/kg (0.7 percent of TRR).
[2] 9 metabolites, none >0.025 mg eq/kg (1.3%TRR).
[3] 4 metabolites, none >0.001 mg eq/kg (0.7%TRR).
[4] 11 metabolites, none >0.040 mg eq/kg (0.7%TRR).
[5] 2 metabolites, none >0.042 mg eq/kg (0.6%TRR).
[6] 6 metabolites, none >0.054 mg eq/kg (1.1%TRR).
[7] 4 metabolites, none >0.041 mg eq/kg (1.0%TRR).

Wheat

A metabolism study in wheat (variety Thasos) after seed treatment was performed with [B-ring-U-^{14}C]-labelled broflanilide indoors (Rosenbaum, 2017, BROFLAN_022). Wheat seeds were coated with broflanilide in an FS formulation prior sowing at a nominal application rate of 10 g ai/100 kg seeds. The actual applied amount of the test item corresponds to an actual application rate of 0.022 kg ai/ha. Immature wheat plants (wheat forage) were collected at growth stage BBCH 39 (77 DAT). Half of the forage was allowed to dry for 8 days at room temperature (wheat hay). Mature wheat plants were harvested at growth stage BBCH 89 (154 DAT) and were separated into straw and grains.

Upon arrival in the laboratory, samples were homogenized by blending with dry ice and their radioactive content determined by combustion, followed by LSC. Portions of wheat straw and grains were subjected to extraction with acetonitrile:water (1+1) (twice), followed by acetonitrile (once). The extract were pooled and the radioactive content determined by LSC. The straw extracts were further characterized by liquid-liquid partitioning with ethyl acetate, followed by fractionation using SPE. Identification of the radioactivity present in the straw extracts was accomplished by co-chromatography with authentic reference standards using reverse phase HPLC and LC-MS. The PES of wheat straw and

grains were further characterized by enzyme solubilization using macerozyme, tyrosinase and amylase. Wheat forage and hay were not further analysed due to the low amount of radioactivity present.

TRR levels in wheat matrices were generally low, with the highest level measured in wheat straw at 0.029 mg eq/kg. A summary of the radioactive residues found in present in Table 20.

Table 20 Total radioactive residues (mg eq./kg) in wheat matrices after seed treatment with [B-ring-U-^{14}C]-labelled broflanilide

Matrix	DAT	TRR measured	TRR calculated[1]
[B-ring-U-^{14}C]-broflanilide			
Forage	77	0.002	n/a
Hay		0.006	n/a
Straw	154	0.029	0.029
Grain		0.011	0.010

Notes:

[1] Sum of extracts and post-extraction residue (PES).

The radioactivity found in the extracts and in the unextracted remainder is presented in Table 21. Overall extractability was higher in straw at 79 percent TRR compared to grains at 29 percent TRR.

Table 21 Extractability of radioactive residues from wheat straw and grains after seed treatment with [B-ring-U-^{14}C]-labelled broflanilide

Fraction	Straw (154DAT)		Grain (154DAT)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
Acetonitrile/water extract	0.023	78.7	0.003	26.9
Additional extract[1]	n/a		< 0.001	2.3
PES	0.006	21.3	0.007	70.8
Sum ERR	0.023	78.7	0.003	29.2
TRR	0.029	100	0.010	100

Notes:

[1] Corresponds to the re-dissolved precipitate from acetonitrile/water extract.

The distribution of radioactivity in wheat straw and grains is presented in Table 22. No individual components could be identified in either matrix. In wheat straw one unknown component was detected accounting for 14 percent TRR, but was detected at < 0.01 mg eq/kg in absolute numbers. The overall fraction of characterized radioactivity accounted for 81 percent TRR in wheat straw and 53 percent TRR in wheat grain.

Table 22 Summary of identified/characterized residues in wheat straw and grains after seed treatment with [B-ring-U-^{14}C]-labelled broflanilide

Designation	Wheat straw 154 DAT		Wheat grain 154 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
TRR calculated	0.029	100.0	0.010	100.0
Identified in ERR	n.a.	n.a.	n.a.	n.a.
Ethyl acetate phase				

Designation	Wheat straw 154 DAT		Wheat grain 154 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
Unknown at 27 min	0.004	14.0	n.a.	n.a.
Additionally characterized by HPLC	0.008	29.2	n.a.	n.a.
Total characterized by HPLC in ERR	0.012	43.2	n.a.	n.a.
Water phase	0.008	26.4	n.a.	n.a.
Sum of evaporator condensate, evaporation flask residue and centrifugation pellet	0.001	4.9	n.a.	n.a.
ACN/H_2O-extracts	n.a.	n.a.	0.003	29.2
Total characterized in ERR	0.021	74.4	0.003	29.2
PES	0.006	21.3	0.007	70.8
Macerozyme solubilizate	0.001	4.2	0.001	9.3
Tyrosinase solubilizate	< 0.001	1.7	< 0.001	0.4
Amylase solubilizate	< 0.001	0.8	0.002	14.5
Total characterized in PES	0.002	6.7	0.003	24.2
Total characterized in ERR and PES	0.023	81.1	0.006	53.4
Unextracted	0.004	14.7	0.004	40.1
Total	0.027	95.8	0.010	93.5

Tea

A metabolism study with tea (variety Japanese small leaf tea) was performed outdoors with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide (Fleischmann, 2017, BROFLAN_023). Tea plants received two foliar applications at nominal rates of 0.1 kg ai/ha each with a RTI of 14 days. Tea leaves were harvested at 7DAT2 and 14DAT2.

Upon arrival in the laboratory, tea leaves were surface-rinsed with acetonitrile and the radioactivity in the rinse determined by LSC. Subsequently, the samples were homogenized by blending with dry ice and their radioactive content determined by combustion, followed by LSC. Portions of the tea leaves were subjected to extraction with acetonitrile (twice) and acetonitrile:water (1+1) (once). The extracts were pooled and the radioactive content determined by LSC. Further characterization/identification of the extract was accomplished by co-chromatography with authentic reference standards using reverse phase HPLC, LC-HRMS and TLC. The PES was not further characterized.

The TRR in tea leaves was calculated as the sum of the rinse, extracts and the PES (Table 23). The TRR was generally similar for both labels ranging between 15–20 mg eq/kg.

Table 23 Total radioactive residues (mg eq./kg) calculated[1] in tea leaves after two foliar application with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

DAT	[B-ring-U-^{14}C]-broflanilide	[C-ring-U-^{14}C]-broflanilide
7DAT2	19.359	20.289
14DAT2	17.016	15.000

Notes:

[1] TRR calculated as the sum of surface rinse, extracts and PES.

The extracted radioactivity from tea leaves ranged between 99–100 percent TRR, with most of the radioactive being removed by the acetonitrile rinse (Table 24).

Table 24 Extractability of radioactive residues from tea leaves after two foliar application with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	Tea leaves (7DAT2)		Tea leaves (14DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
Acetonitrile rinse	19.027	98.3	16.537	97.2
Acetonitrile/water extract	0.259	1.3	0.375	2.2
PES	0.073	0.4	0.104	0.6
Sum ERR	19.286	99.6	16.912	99.4
TRR	19.359	100.0	17.016	100.0
[C-ring-U-^{14}C]-broflanilide				
Acetonitrile rinse	19.895	98.1	14.542	97.0
Acetonitrile/water extract	0.296	1.5	0.352	2.4
PES	0.098	0.5	0.106	0.7
Sum ERR	20.191	99.5	14.894	99.3
TRR	20.289	100.0	15.000	100.0

The distribution of radioactivity in tea leaves is presented in Table 25. Parent broflanilide was the major identified residue, accounting for 96–97 percent TRR (14–19 mg eq/kg). Additionally, metabolites S(PFP-OH)-8007 and DM-8007 were identified at minor levels of 1.0–1.4 percent TRR (0.14–0.27 mg eq/kg) and 0.0–1.0 percent TRR (0.007–0.20 mg eq/kg), respectively (Table 25).

Table 25 Summary of identified/characterized residues in tea leaves after two foliar application with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	Tea leaves (7DAT2)		Tea leaves (14DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide				
TRR	19.359	100.0	17.016	100.0
Surface rinse	19.027	98.3	16.537	97.2
Broflanilide	18.648	96.3	16.143	94.9
S(PFP-OH)-8007	0.263	1.4	0.195	1.2
DM-8007	n/d		0.136	0.8
Unknown RT 52 min	0.116	0.6	n/d	
Others[1]	n/d		0.063	0.4
Extract	0.259	1.3	0.375	2.2
Broflanilide	0.210	1.1	0.287	1.7
S(PFP-OH)-8007	0.006	0.0	0.005	0.0
DM-8007	0.007	0.0	n/d	
Unknown RT 52 min	n/d		0.004	0.0
Others[1]	0.035	0.2	0.078	0.5
Sum of broflanilide	18.858	97.4	16.430	96.6
Sum of S(PFP-OH)-8007	0.269	1.4	0.200	1.2
Sum of DM-8007	0.007	0.0	0.136	0.8

Fraction	Tea leaves (7DAT2)		Tea leaves (14DAT2)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
Total identified	19.134	98.9	16.766	98.5
Total characterized	0.151	0.8	0.145	0.9
Unextracted	0.073	0.4	0.104	0.6
Total	19.358	100.0	17.015	100.0
[C-ring-U-^{14}C]-broflanilide				
TRR	20.289	100.0	15.000	100.0
Surface rinse	19.895	98.1	14.542	97.0
Broflanilide	19.262	94.9	14.176	94.5
S(PFP-OH)-8007	0.255	1.3	0.143	1.0
DM-8007	0.189	0.9	0.093	0.6
Unknown RT 52 min	0.129	0.6	0.057	0.4
Others[2]	0.060	0.3	0.074	0.5
Extract	0.296	1.5	0.352	2.4
Broflanilide	0.236	1.2	0.253	1.7
S(PFP-OH)-8007	0.005	0.0	n/d	
DM-8007	0.009	0.0	0.015	0.1
Unknown RT 52 min	0.005	0.0	n/d	
Others[2]	0.042	0.2	0.083	0.6
Sum of broflanilide	19.498	96.1	14.429	96.2
Sum of S(PFP-OH)-8007	0.260	1.3	0.143	1.0
Sum of DM-8007	0.198	1.0	0.108	0.7
Total identified	19.956	98.4	14.680	97.9
Total characterized	0.236	1.2	0.214	1.4
Unextracted	0.098	0.5	0.106	0.7
Total	20.290	100.0	15.000	100.0

Notes:

[1] 1-15 metabolites <1%TRR.

[2] 1-9 metabolites <1.05%TRR.

In order to investigate the translocation of broflanilide in plants, the radioactivity in leaves on a stem above and below a treated leaf was determined. Results showed that 92–97 percent of the TRR remained on the treated leaf. Distribution of the radioactive residues on leaves above the treated leaf were 0.1–3.3 percent TRR and on leaves below the treated leaf were between 2.3–5.0 percent TRR.

Summary plant metabolism

In all plant metabolism studies, broflanilide was moderately degraded into DM-8007 via demethylation or into S(PFP-OH)-8007 via oxidative defluorination (substitution of fluorine with hydroxy group). Parent broflanilide was the major identified component in all matrices, while both metabolites were detected at minor amounts only.

The proposed metabolic pathway of broflanilide is shown in Figure 2.

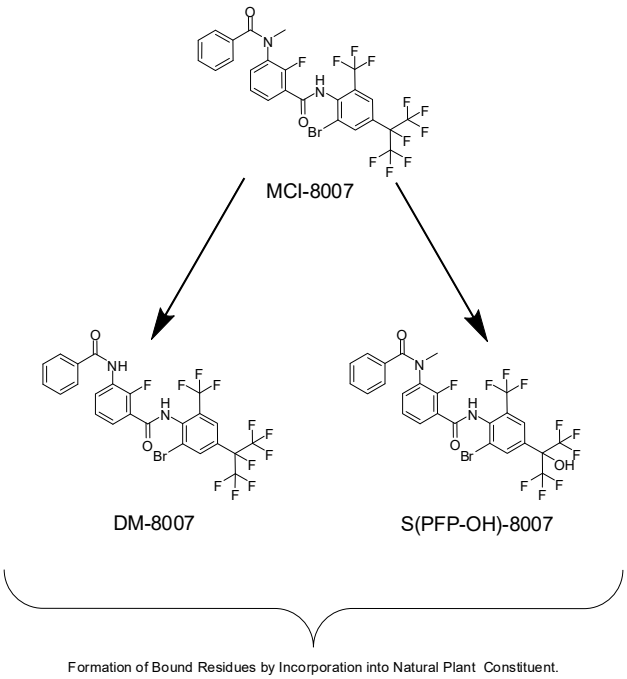


Figure 2 Proposed metabolic pathway of broflanilide in primary crops (cabbage, tomato, Japanese radish, soya bean, rice, tea)

Animal metabolism

Metabolism studies were provided for lactating goats and laying hens using [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide.

Laboratory animals

The evaluation of the metabolism studies in laboratory animals was carried out by the WHO Core Assessment Group.

Lactating goats

A metabolism study with lactating goats was performed with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide (Estigoy, 2017, BROFLAN_024). The compound was administered orally once daily (after morning milking) for 10 consecutive days to one lactating goat per label. Administered doses were 19 ppm (0.62 mg/kg bw day) and 20 ppm (0.73 mg/kg bw) for the B- and C-label, respectively. Animals were milked twice daily, with urine and faeces being collected twice daily. Tissue samples were collected after sacrifice, which occurred 12 hr after the last dose for the [B-ring-U-^{14}C]-labelled broflanilide and 8 hours after the last dose for the [C-ring-U-^{14}C]-labelled broflanilide. Samples collected at sacrifice included flank and loin muscles, omental, subcutaneous, and renal fats, liver, kidney, as well as whole blood, bile, the gastrointestinal tract.

The total radioactivity in milk, bile, urine, and cage wash was directly measured by LSC. Faeces, gastrointestinal tract, and whole blood samples were subjected to combustion prior to the determination of total radioactivity by LSC. Muscles (flank and loin), milk, fat (omental, subcutaneous and renal), liver, and kidney were dissolved in tissue solubiliser followed by LSC.

Tissues were homogenized in the presence of dry ice followed by sequential solvent extraction:

Samples of flank and loin muscles were extracted twice with acetonitrile/water (1+1) and then once with 100 mL of acetonitrile. The extracts were pooled and the radioactive content determined by LSC. Characterization/identification of the extract was accomplished by co-chromatography with authentic reference standards using reverse phase HPLC and TLC analysis. The PES were combusted and radioactive content measured by LSC.

Milk was separated into milk fat and skim milk by centrifugation. Subsequently, the skim milk was extracted twice with acetone:water (1+1) and once with acetone. The corresponding milk fat was extracted twice with 1:4 acetone/hexane (1+4) and then once with acetone. The extracts were pooled and the radioactive content determined by LSC. The milk fat was concentrated and partitioned with acetonitrile and the acetonitrile layers were measured by LSC. The hexane layer, which contained the fat, stuck to the separating funnel and was not measured. Characterization/identification of the extract was accomplished by co-chromatography with authentic reference standards using reverse phase HPLC, TLC and LC-MS analysis. The PES were combusted and radioactive content measured by LSC.

Fat samples were extracted identically to the milk fat. Liver and kidney samples were twice with acetonitrile/water (1+1) and then once with acetonitrile. The radiocarbon content of each extract was measured by LSC. The PES was combusted, and radiocarbon measured by LSC. The acetonitrile/water extracts were combined, concentrated and centrifuged prior to LSC analysis. To improve concentration recoveries, the solid precipitates from the concentration were rinsed with either acetonitrile or acetonitrile:water (1+1). The rinses were radioassayed by LSC, combined with the concentrated extract and analysed by HPLC, TLC and/or LC-MS analysis. Samples of liver (B and C-ring label) and kidney (B-ring label only) containing high PES were re-extracted using the SPEX Sample Prep (Geno/ Grinder) to improve extractability of bound radioactive residues. The PES from these samples was further characterized by enzyme solubilization using protease and lipase, followed by incubations with 1N HCl and 1 N NaOH. The extracts and solubilizates were also treated with β-glucuronidase in order to cleave conjugates to their respective aglycones.

The total recovery of the administered radioactivity was good for both labels ranging between 87–92 percent. The majority of the radioactivity was found in faeces at 51–75 percent AR. In urine, the recovered radioactivity of [C-ring-U-^{14}C]-labelled broflanilide was at 24 percent AR, while only 0.7 percent AR was found of [B-ring-U-^{14}C]-labelled broflanilide. A higher fraction of metabolites from [C-ring-U-^{14}C]-labelled broflanilide excreted via urine was suggested. A summary of the recovered radioactivity is presented in Table 26.

Table 26 Recovered radioactive residues after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide for 10 consecutive days to lactating goats

Matrix	[B-ring-U-^{14}C]-broflanilide		[C-ring-U-^{14}C]-broflanilide	
	% AR	mg eq/kg	% AR	mg eq/kg
Flank Muscle	0.0	0.300	0.0	0.370
Loin Muscle	0.0	0.216	0.0	0.228
Skim Milk [1]	0.0	0.017	0.0	0.028
Milk Fat [1]	0.7	2.967	0.6	1.628
Whole Milk [2]	(0.7)	(0.254)	(0.7)	(0.269)
Omental Fat	0.9	3.411	0.8	3.422
Subcutaneous Fat	0.1	2.598	0.1	2.830
Renal Fat	0.2	3.065	0.3	3.290
Liver	0.7	2.197	0.1	0.457
Kidney	0.0	0.250	0.0	0.265

Matrix	[B-ring-U-^{14}C]-broflanilide		[C-ring-U-^{14}C]-broflanilide	
	% AR	mg eq/kg	% AR	mg eq/kg
Blood	0.0	0.095	0.0	0.071
Urine	0.7	0.068	23.6	4.187
Bile	0.0	6.511	0.0	1.122
Faeces	75.4	19.154	51.0	9.057
Cage wash	0.0	0.045	0.8	0.513
Gastrointestinal Tract	13.0	3.181	9.6	2.795
Total	91.7		87.0	

Notes:

[1] Levels were calculated based on the average dpm/g of Day 1 pm to Day 10 pm samples.

[2] Total recovered in Skim Milk and Milk Fat.

In milk the total radioactivity both labels increased rapidly after the first administration and ranged over the course of the study between 0.123-0.381 mg eq/kg for [B-ring-U-^{14}C]-labelled broflanilide and between 0.130-0.429 mg eq/kg for [C-ring-U-^{14}C]-labelled broflanilide. Residue levels reached a plateau after approximately 6 days and 2 days [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide, respectively (Figure 3). In milk fat, levels of broflanilide were for both labels ~2 orders in magnitude higher compared to skim milk, reaching a level of up to 4.1 mg eq/kg. The results are summarised in the following Table 27.

Table 27 Recovered radioactive residues in milk after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide for 10 consecutive days to lactating goats

Timing of sampling	[B-ring-U-^{14}C]-broflanilide			[C-ring-U-^{14}C]-broflanilide		
	Skim Milk	Milk Fat	Whole Milk	Skim Milk	Milk Fat	Whole Milk
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Day 1 AM	0	0	0	0	0	0
Day 1 PM	0.007	1.580	0.150	0.017	1.106	0.252
Day 2 AM	0.009	1.336	0.123	0.014	0.651	0.164
Day 2 PM	0.012	1.606	0.157	0.030	1.608	0.357
Day 3 AM	0.013	2.050	0.176	0.024	1.139	0.262
Day 3 PM	0.017	3.206	0.301	0.036	2.002	0.429
Day 4 AM	0.027	2.592	0.222	0.023	1.396	0.285
Day 4 PM	0.020	3.882	0.326	0.038	2.172	0.371
Day 5 AM	0.018	3.300	0.258	0.026	0.568	0.130
Day 5 PM	0.026	4.101	0.381	0.035	1.383	0.293
Day 6 AM	0.017	3.401	0.264	0.026	1.069	0.191
Day 6 PM	0.018	3.513	0.298	0.028	1.489	0.240
Day 7 AM	0.014	3.297	0.254	0.025	2.389	0.200
Day 7 PM	0.020	3.207	0.310	0.027	2.348	0.329
Day 8 AM	0.015	2.619	0.218	0.027	2.197	0.244
Day 8 PM	0.015	3.345	0.308	0.037	1.928	0.292
Day 9 AM	0.015	3.072	0.244	0.030	2.071	0.271
Day 9 PM	0.017	3.577	0.316	0.029	2.058	0.346
Day 10 AM	0.016	3.188	0.244	0.027	2.294	0.270
Day 10 PM	0.019	3.488	0.279	0.036	1.066	0.193
Average	0.017	2.967	0.254	0.028	1.628	0.269

Figure 3 shows the time course of the concentrations of [^{14}C]- broflanilide in milk.

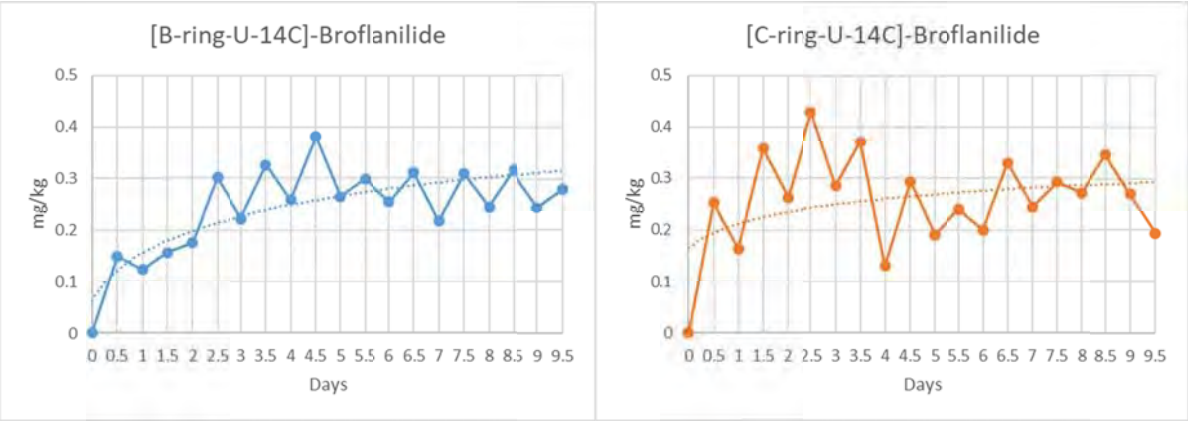


Figure 3 Time course of the concentrations of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide in milk

Extraction solvents released at least 88 percent TRR from most matrices, except for liver and kidney at 42-68 percent TRR (both labels) and 76 percent TRR (B-ring label), respectively. A summary of the results is presented in Table 28 and Table 29.

Table 28 Extractability of residues from muscle, liver and kidney after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide to a lactating goat

	Flank muscle		Loin muscle		Liver		Kidney	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide								
TRR[1]	0.300		0.216		2.197		0.250	
ACN/H_2O extract	0.301	91.8	0.179	87.7	0.887	41.8	0.185	75.8
PES	0.027	8.2	0.025	12.3	1.237	58.2	0.059	24.2
TRR[2]	0.328	100.0	0.204	100.0	2.124	100.0	0.244	100.0
TRR[2]/TRR[1]	109.3		94.4		96.7		97.6	
[C-ring-U-^{14}C]-broflanilide								
TRR[1]	0.370		0.228		0.457		0.265	
ACN/H_2O extract	0.336	97.7	0.234	98.3	0.308	68.1	0.261	95.6
PES	0.008	2.3	0.004	1.7	0.144	31.9	0.012	4.4
TRR[2]	0.344	100.0	0.238	100.0	0.452	100.0	0.273	100.0
TRR[2]/TRR[1]	93.0		104.4		98.9		103.0	

Notes:

[1] TRR determined by combustions.

[2] TRR was calculated as the sum of the residues in the extract and PES.

Table 29 Extractability of residues from fats and skim milk after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide to a lactating goat

	Omental fat		Subcutaneous fat		Renal fat		Milk fat		Skim milk	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide										
TRR[1]	3.411		2.598		3.065		4.101		0.026	
Hexane/acetone extract	3.370	99.6	2.571	99.7	3.042	99.4	4.184	99.8	n/a	
Acetone/water extract	n/a		n/a		n/a		n/a		0.027	100.0
PES	0.015	0.4	0.008	0.3	0.019	0.6	0.007	0.2	0.000	0.0
TRR[2]	3.385	100.0	2.579	100.0	3.061	100.0	4.191	100.0	0.027	100.0
TRR[2]/TRR[1]	99.2		99.3		99.9		102.2		103.8	
[C-ring-U-^{14}C]-broflanilide										
TRR[1]	3.422		2.830		3.290		2.348		0.027	
Hexane/acetone extract	3.441	99.9	2.820	99.9	3.346	99.8	2.118	99.8		
Acetone/water extract	n/a		n/a		n/a		n/a		0.026	100.0
PES	0.003	0.1	0.002	0.1	0.007	0.2	0.004	0.2		
TRR[2]	3.444	100.0	2.822	100.0	3.353	100.0	2.122	100.0	0.026	100.0
TRR[2]/TRR[1]	100.6		99.7		101.9		90.4		96.3	

Notes:

[1] TRR determined by combustions.

[2] TRR was calculated as the sum of the residues in the extract and PES.

The distribution of radioactivity in lactating goats is presented in Tables 30 to 32. Parent broflanilide was only detected as a minor residue in muscle, kidney and liver at 0.5–6.7 percent TRR (0.005–0.022 mg eq/kg) and was not detected in all other matrices. A major identified metabolite using both labels was metabolite DM-8007 in muscle, milk (skim and fat), fats, liver (only C-label) and kidney at 21–100 percent TRR (0.01–3.4 mg eq/kg). In the B-label treated goat only, metabolite DC-DM-8007 was detected at major proportions in muscle, milk (skim and fat), fats, liver and kidney at 29–67 percent TRR (0.017–2.3 mg eq/kg), while in the C-label only, hippuric acid was detected in skim milk, liver and kidney at 19–69 percent TRR (0.018–0.13 mg/kg). Also, hydroxylated and conjugated DC-DM-(A4-OH)-8007, DC-DM-(A6-OH)-8007 and DM-(C2-OH)-8007 were identified in liver and kidney (B-label only), accounting for up to 15 percent TRR (0.32 mg eq/kg), 11 percent TRR (0.24 mg eq/kg) and 17 percent TRR (0.078 mg eq/kg). The proposed metabolic pathway of broflanilide in lactating goat is shown in Figure 4.

Table 30 Summary of identified/characterized residues in muscle, skim milk and milk fat after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Residue component	Flank muscle		Loin muscle		Skim milk		Milk fat	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
[B-ring-U-^{14}C]-broflanilide								
TRR, calculated	100.0	0.328	100.0	0.204	100.0	0.028	100.0	4.191
Extract	91.8	0.301	87.7	0.179	100.0	0.027	99.8	4.184

Residue component	Flank muscle		Loin muscle		Skim milk		Milk fat	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
[B-ring-U-^{14}C]-broflanilide								
Broflanilide	1.5	0.005	2.5	0.005	ND	ND	ND	ND
DC-DM-8007	55.8	0.183	48.0	0.098	63.0	0.017	53.9	2.259
DM-8007	33.8	0.111	37.3	0.076	37.0	0.010	45.9	1.925
Total identified	91.2	0.299	87.7	0.179	100.0	0.027	99.8	4.184
Total characterized	0.6	0.002	0.0		0.0		0.0	
Unextracted	8.2	0.027	12.3	0.025	0.0		0.2	0.007
Total	100.0	0.328	100.0	0.204	100.0	0.027	100.0	4.191
[C-ring-U-^{14}C]-broflanilide								
TRR, calculated	100.0	0.344	100.0	0.238	100.0	0.026	100.0	2.122
Extract	97.7	0.336	98.3	0.234	100.0	0.026	99.8	2.118
Broflanilide	5.2	0.018	6.7	0.016	ND	ND	ND	ND
DM-8007	92.4	0.318	89.5	0.213	26.9	0.007	99.8	2.118
Hippuric acid	ND	ND	ND	ND	69.2	0.018	ND	ND
Total identified	97.7	0.336	96.2	0.229	96.2	0.025	99.8	2.118
Total characterized	ND	ND	2.1	0.005	3.9	0.001	0.0	0.0
Unextracted	97.7	0.336	98.3	0.234	100.0	0.026	99.8	2.118
Total	2.3	0.008	1.7	0.004	ND	ND	0.2	0.004

Table 31 Summary of identified/characterized residues in kidney and fats after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

residue component	Kidney		Omental fat		Subcutaneous fat		Renal fat	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
[B-ring-U-^{14}C]-broflanilide								
TRR (calculated)	-	-	100.0	3.368	100.0	2.575	100.0	3.054
Extract	-	-	99.6	3.370	99.7	2.571	99.4	3.042
Broflanilide	-	-	ND	ND	ND	ND	ND	ND
DC-DM-8007	-	-	60.2	2.039	66.6	1.717	64.6	1.977
DM-8007	-	-	39.3	1.331	33.1	0.854	34.8	1.065
Total identified	-	-	99.6	3.370	99.7	2.571	99.4	3.042
Total characterized	-	-	0.0		0.0		0.0	
Unextracted	-	-	0.4	0.015	0.3	0.008	0.6	0.019
Total	-	-	100.0	3.385	100.0	2.579	100.0	3.061
[C-ring-U-^{14}C]-broflanilide								
TRR (calculated)	100.0	0.273	100.0	3.434	100.0	2.820	100.0	3.330
Extract	95.6	0.261	99.9	3.441	99.9	2.820	99.8	3.346
Broflanilide	2.9	0.008	ND	ND	ND	ND	ND	ND
DM-8007	44.7	0.122	99.9	3.441	99.9	2.820	99.8	3.346
Hippuric acid	48.0	0.131	ND	ND	ND	ND	ND	ND
Total identified	95.6	0.261	99.9	3.441	99.9	2.820	99.8	3.346
Total characterized	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Unextracted	4.4	0.012	0.1	0.003	0.1	0.002	0.2	0.007
Total	100.0	0.273	100.0	3.444	100.0	2.822	100.0	3.353

Table 32 Summary of identified/characterized residues in liver and kidney after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide

Fraction	[B-ring-U-^{14}C]-broflanilide				[C-ring-U-^{14}C]-broflanilide	
	Liver		Kidney		Liver	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	2.181		0.248		0.455	
ACN/H_2O extract	0.902	41.3	0.180	72.6	0.309	67.8
Broflanilide	0.011	0.5	n/d		0.022	4.8
DC-DM-8007	0.282	12.9	0.100	40.2	n/a	
DM-8007	0.098	4.5	0.053	21.3	0.161	35.3
DC-DM-(A4-OH)-8007[1]	0.060	2.8	0.005	1.9	n/a	
DC-DM-(A6-OH)-8007[1]	0.174	8.0	0.010	3.9	n/a	
DM-(C2-OH)-8007[1]	0.089	4.1	0.007	2.7	0.078	17.1
Hippuric acid	n/a		n/a		0.042	9.3
Unknowns	0.187[2]	8.5	0.006	2.5	0.006	1.3
Protease	0.374	17.1	0.019	7.7	0.043	9.4
DC-DM-8007	0.041	1.9	0.005	2.2	n/a	
DC-DM-(A4-OH)-8007[1]	0.057	2.6	0.004	1.6	n/a	
DC-DM-(A6-OH)-8007[1]	n/d		0.002	0.8	n/a	
Hippuric acid	n/a		n/a		0.029	6.4
Unknowns	0.276[3]	12.8	0.008	3.1	0.014	3.1
Lipase	0.536	24.6	0.030	12.1	0.060	13.2
DC-DM-8007	0.135	6.2	n/d		n/a	
DC-DM-(A4-OH)-8007[1]	0.203	9.3	0.010	4.0	n/a	
Hippuric acid	n/a		n/a		0.016	3.5
Unknowns	0.197[4]	9.1	0.020	8.1	0.044	9.7
1N HCl	0.134	6.1	0.009	3.6	0.017	3.7
DC-DM-8007	0.134	6.1	n/d		n/a	
Unknowns			0.009	3.6	0.017	3.7
1N NaOH	0.238	10.9	0.010	4.0	0.027	5.9
DC-DM-8007	0.046	2.1	n/d		n/a	
DC-DM-(A6-OH)-8007[1]	0.062	2.8	n/d		n/a	
Unknowns	0.130[5]	6.0	0.010	4.0	0.027	5.9
Sum of broflanilide	0.011	0.5	n/d		0.022	4.8
Sum of DC-DM-8007	0.593	29.2	0.105	42.4	n/a	
Sum of DM-8007	0.098	4.5	0.053	21.3	0.161	35.3
Sum of DC-DM-(A4-OH)-8007[1]	0.320	14.7	0.019	7.5	n/a	
Sum of DC-DM-(A6-OH)-8007[1]	0.236	10.8	0.010	3.9	n/a	
Sum of DM-(C2-OH)-8007[1]	0.089	4.1	0.007	2.7	0.078	17.1
Sum of hippuric acid	n/a		n/a		0.087	19.2
Total identified	1.393	63.8	0.195	78.6	0.348	76.4
Total characterized	0.791	36.4	0.053	21.3	0.108	23.7
Unextracted	0	0.0	0	0.0	0	0.0
Total	2.184	100.2	0.248	99.9	0.456	100.1

Notes:

[1] Detected as glucuronide.
[2] Sum of metabolites, none >0.0658 mg/kg, 3.0%TRR.
[3] Sum of metabolites, none >0.0733 mg/kg, 3.4%TRR.
[4] Sum of metabolites, none >0.0733 mg/kg, 3.4%TRR.
[5] Sum of metabolites, none >0.1064 mg/kg, 4.9%TRR.

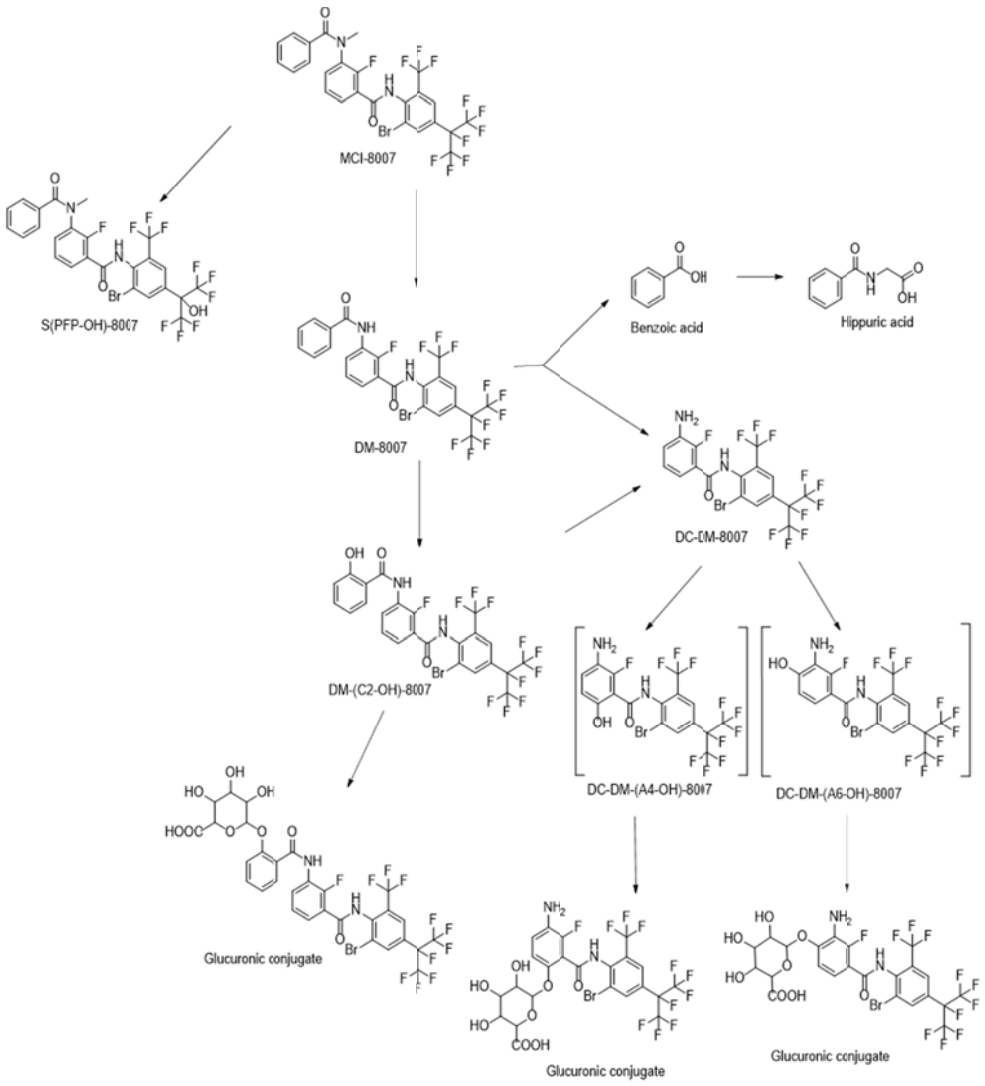


Figure 4 Proposed metabolic pathway of broflanilide in lactating goat

Laying hens

A metabolism study with laying hens was performed with [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide (Estigoy, 2017, BROFLAN_025). The compound was administered orally in cellulose-filled gelatin capsules once daily after the morning collection of eggs and excreta for 14 consecutive days to 10 laying hens per label. Two additional hens per label received a single dose only, used for blood kinetic analysis. Administered doses were 14 ppm (0.86 mg/kg bw day) and 15 ppm (0.84 mg/kg bw) for the B- and C-label, respectively. Eggs and excreta samples were collected twice daily, at approximately 12 hour intervals. Samples of breast muscle, thigh (leg) muscle, abdominal and subcutaneous fat, liver and the entire gastrointestinal tract were collected after sacrifice, which occurred 6 hr after the last dose. Blood

samples were collected for blood kinetic analysis at nine intervals (0.5, 1, 2, 4, 6, 8, 10, 12, and 24 hours) after the first dose.

The total radioactivity in cage wash was directly measured by LSC. Excreta, gastrointestinal tract, and whole blood samples were subjected to combustion prior to the determination of total radioactivity by LSC. Egg white, egg yolk, fats (abdominal and subcutaneous), muscles (breast and thigh), and liver were dissolved in tissue solubiliser followed by LSC.

Tissues were homogenized in the presence of dry ice followed by sequential solvent extraction. Egg white, egg yolk, and muscle samples were extracted twice with acetonitrile/water (1+1) and then once with acetonitrile. The extracts (except thigh muscle) were pooled and cleaned up by solid phase extraction on a silica cartridge to remove fat.

Fat samples were extracted twice with 50 mL acetone/hexane (1:4, v:v) then once with 50 mL acetone. The extracts were pooled, the acetone removed by evaporation, followed by liquid-liquid portioning of the remaining hexane phase with acetonitrile.

Liver samples were initially extracted three times with acetonitrile:water (1:1, v:v) and then once with acetonitrile. For some liver samples additional extractions were performed with weak base (1 mol/L aqueous sodium hydroxide) and strong base (25 percent aqueous sodium hydroxide). The PES of hen liver from both radiolabels (B and C-ring) were further incubated and extracted with protease, followed by lipase enzyme. After enzymatic extraction, the post extracted solids were further extracted with weak acid (1 mol/L hydrochloric acid) and weak base (1mol/L aqueous sodium hydroxide).

The radioactive content is all extracts/fractions was determined by LSC. Characterization/identification of the extract was accomplished by co-chromatography with authentic reference standards using reverse phase HPLC, TLC and/or HPLC/MS analysis. The PES were combusted and radioactive content measured by LSC.

The total recovery of the administered radioactivity was good for both labels ranging between 73–79 percent and 92–95 percent if normalized for percent equivalent mass weight of muscle and fat. The majority of the radioactivity was found in excreta at 56–65 percent AR. In edible tissues radioactivity was highest in fat at 15–19 mg eq/kg, followed by egg yolk at 3.4–3.6 mg eq/kg and liver at 1.8–2.6 mg eq/kg. A summary of the recovered radioactivity is presented in Table 33.

Table 33 Recovered radioactive residues after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide for 14 consecutive days to laying hens

Matrix	[B-ring-U-^{14}C]-broflanilide		[C-ring-U-^{14}C]-broflanilide	
	% AR	mg eq/kg	% AR	mg eq/kg
Liver	0.5	2.631	0.4	1.843
Thigh (Leg) Muscle	0.8	2.140	0.5	1.397
Breast Muscle	0.1	0.330	0.1	0.240
Muscle[1]	3.6[1]		2.5[1]	
Abdominal Fat	3.9	19.132	3.4	15.770
Subcutaneous Fat	1.5	18.549	1.0	14.579
Fat[1]	21.6[1]		17.9[1]	
Egg White	0.0	0.014	0.0	0.012
Egg Yolk	4.1	3.605	2.8	3.365
Excreta	56.0	4.782	65.0	5.490
GI Tract	6.1	6.581	6.0	6.715
Cage wash	0.1	0.139	0.1	0.152

Matrix	[B-ring-U-^{14}C]-broflanilide		[C-ring-U-^{14}C]-broflanilide	
	% AR	mg eq/kg	% AR	mg eq/kg
Total	73.1 (92.0)[1]		79.4 (94.7)[1]	

Notes:

[1] Normalized value based on percent equivalent mass weight of muscle and fat in the administered animals (taken from *New Zealand Journal of Agricultural Research, 1998, Vol. 41: 555-559*).

Incorporation of radioactivity into egg whites reached steady state within 3–4 days, while no plateau was reached in egg yolks. The concentration ratio of between egg yolk and egg white increased over the time course by a factor >400. A summary of the recovered radioactivity is presented in Table 34.

Table 34 Recovered radioactive residues in eggs after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide for 14 consecutive days to laying hens

Application day	[B-ring-U-^{14}C]-broflanilide				[C-ring-U-^{14}C]-broflanilide			
	Egg white		Egg yolk		Egg white		Egg yolk	
	% AR	mg eq/kg	% AR	mg eq/kg	% AR	mg eq/kg	% AR	mg eq/kg
1	0.0	0.000	0.0	0.000	0.0	0.000	0.0	0.000
2	0.0	0.009	0.0	0.008	0.0	0.005	0.0	0.021
3	0.0	0.016	0.03	0.727	0.0	0.019	0.02	0.592
4	0.0	0.025	0.08	2.003	0.0	0.020	0.07	1.685
5	0.0	0.027	0.12	3.671	0.0	0.020	0.11	3.252
6	0.0	0.034	0.19	5.120	0.0	0.023	0.17	4.597
7	0.0	0.028	0.26	6.838	0.0	0.025	0.21	6.621
8	0.0	0.029	0.27	8.670	0.0	0.021	0.20	7.213
9	0.0	0.030	0.37	8.949	0.0	0.035	0.25	7.677
10	0.0	0.033	0.89	10.129	0.0	0.029	0.33	9.267
11	0.0	0.036	0.41	10.663	0.0	0.026	0.31	9.235
12	0.0	0.032	0.50	11.464	0.0	0.031	0.38	10.988
13	0.0	0.032	0.50	12.709	0.0	0.023	0.32	10.441
14	0.0	0.032	0.46	12.772	0.0	0.029	0.45	12.530
Total	0.0	0.363	4.1	93.723	0.0	0.306	2.8	84.120

Extraction with solvents released at least 89 percent TRR from all matrices, except for liver where 65–72 percent TRR (both labels) were released. Additional sequential treatments with enzymes, hydrochloric acid and sodium hydroxide, or treatment with sodium hydroxide alone resulted in overall extraction efficiency for liver of 99–100 percent. A summary of the results is presented in Table 35 and Table 36.

Table 35 Extractability of residues from egg white, egg yolk, thigh muscle and breast muscle after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide for 14 consecutive days to laying hens

	Egg white		Egg yolk		Thigh (Leg) muscle		Breast muscle		Abdominal fat		Subcutaneous fat	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
[B-ring-U-^{14}C]-broflanilide												
TRR[1]	0.017		7.270		2.140		0.330		19.132		18.549	
Acetonitrile: water extract	0.017	89.5	6.063	96.0	2.094	96.2	0.345	94.3	n/a	n/a	n/a	n/a

	Egg white		Egg yolk		Thigh (Leg) muscle		Breast muscle		Abdominal fat		Subcutaneous fat	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Hexane: Acetone extract	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	19.382	100.0	18.506	99.9
PES	0.002	10.5	0.256	4.0	0.083	3.8	0.021	5.7	0.006	0.0	0.014	0.1
TRR[2]	0.019	100.0	6.319	100.0	2.177	100.0	0.366	100.0	19.39	100.0	18.52	100.0
TRR[2]/TRR[1]	111.8		86.9		101.7		110.9		101.3		99.8	
[C-ring-U-^{14}C]-broflanilide												
TRR[1]	0.018		7.096		1.397		0.240		15.77		14.58	
Acetonitrile: water extract	0.016	88.9	6.549	96.9	1.523	97.4	0.267	95.7	n/a	n/a	n/a	n/a
Hexane: Acetone extract	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	15.81	100.0	14.867	100.0
PES	0.002	11.1	0.211	3.1	0.040	2.6	0.012	4.3	0.006	0.0	0.005	0.0
TRR[2]	0.018	100	6.76	100	1.563	100	0.279	100	15.82	100.0	14.87	100.0
TRR[2]/TRR[1]	100.0		95.3		111.9		116.3		100.3		102.0	

Notes:

[1] TRR measured.

[2] TRR was calculated as the sum of the residues in the extract and PES.

Table 36 Extractability of residues from liver (using two different extraction regimes) after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide for 14 consecutive days to laying hens

	[B-ring-U-^{14}C]-broflanilide				[C-ring-U-^{14}C]-broflanilide			
	Liver		Liver (original extraction)		Liver		Liver (original extraction)	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR[1]	2.631		2.631		1.843		1.843	
Acetonitrile: water extract	1.607	64.5	1.752	66.5	1.317	71.6	1.441	72.4
Protease	0.254	10.6	n/a	n/a	0.164	8.9	n/a	n/a
Lipase enzyme	0.265	10.6	n/a	n/a	0.143	7.8	n/a	n/a
Water rinse	0.038	1.5	n/a	n/a	0.016	0.9	n/a	n/a
1mol/L hydrochloric acid	0.022	0.9	n/a	n/a	0.016	0.9	n/a	n/a
Water rinse	0.066	2.7	n/a	n/a	0.039	2.1	n/a	n/a
1mol/L sodium hydroxide	0.183	7.4	0.626	23.8	0.113	6.1	0.378	19.0
Water rinse	0.045	1.8	n/a	n/a	0.032	1.7	n/a	n/a
25 percent sodium hydroxide	n/a	n/a	0.232	8.8	n/a	n/a	0.157	7.9
Totally extracted	2.490	100.0	2.610	99.1	1.840	100.0	1.976	99.3
PES	0.0	0.0	0.025	0.9	0.0	0.0	0.014	0.7
TRR[2]	2.490	100.0	2.635	100.0	1.840	100.0	1.990	100.0
TRR[2]/TRR[1]	94.6		100.2		99.8		108.0	

Notes:

[1] TRR measured.

[2] TRR was calculated as the sum of the residues in the extracts and PES.

The distribution of radioactivity in laying hens is presented in Table 37 to Table 39. Parent broflanilide was only tentatively identified in egg white from the B-label at 2.1 percent TRR (0.0004 mg eq/kg). The predominant identified residue for both labels was metabolite DM-8007 in all

matrices at 57–100 percent TRR (0.013–19 mg eq/kg). As a minor metabolite only occurring with the B-label, DC-DM-8007 was detected in all matrices, accounting for up to 3 percent TRR (0.56 mg eq/kg) in subcutaneous fat (in egg white the TRR was with 16 percent higher, but absolute amount with 0.003 mg eq/kg lower). Additionally in liver only, H-U27B (B-label), a hydroxyl cysteine conjugate of DM-8007 and the similar, but structurally not fully elucidated compound H-U27C (C-label), were identified at levels of 5.3 percent TRR (0.131 mg eq/kg) and 3.3 percent TRR (0.061 mg eq/kg).

Table 37 Summary of identified/characterized residues in muscle and fat after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide for 14 consecutive days to laying hens

Residue component	Breast muscle		Thigh (leg) muscle		Abdominal fat		Subcutaneous fat	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
[B-ring-U-^{14}C]-broflanilide								
Extract	94.3	0.345	96.2	2.094	100.0	19.382	99.9	18.506
Broflanilide	ND	ND	ND	ND	ND	ND	ND	ND
DC-DM-8007	(0.8)	(0.003) [1]	1.1	0.025	2.5	0.485	3.0	0.555
DM-8007	91.3	0.334	94.3	2.052	97.5	18.897	96.9	17.951
DM-(C4-OH)-8007	ND	ND	ND	ND	ND	ND	ND	ND
Total identified	91.3	0.334	95.4	2.077	100.0	19.382	99.9	18.506
Total characterized	3.0	0.011 [2]	0.8	0.017 [3]	0.0	- [4]	0.0	- [4]
Unextracted	5.7	0.021	3.8	0.083	0.0	0.006	0.1	0.014
Total	100.0	0.366	100.0	2.177	100.0	19.388	100.0	18.520
[C-ring-U-^{14}C]-broflanilide								
Extract	95.7	0.267	97.4	1.523	100.0	15.812	100.0	14.867
Broflanilide	ND	ND	ND	ND	ND	ND	ND	ND
DM-8007	93.5	0.261	95.3	1.489	100.0	15.812	99.3	14.763
DM-(C4-OH)-807	ND	ND	ND	ND	ND	ND	ND	ND
S(Br-OH)-8007	ND	ND	ND	ND	ND	ND	ND	ND
S(PFP-OH)-8007	ND	ND	ND	ND	ND	ND	ND	ND
Total identified	93.5	0.261	95.3	1.489	100.0	15.812	99.3	14.763
Total characterized	2.2	0.006 [5]	2.2	0.034 [6]	0.0	- [4]	0.7	0.104 [7]
Unextracted	4.3	0.012	2.6	0.040	0.0	0.006	0.0	0.005
Total	100.0	0.279	100.1	1.563	100.0	15.818	100.0	14.872

Notes:

[1] This was detected in TLC analysis (0.92 percent X 0.345 mg/kg = 0.003 mg/kg).

[2] 2 metabolites, none >0.006 mg/kg, 1.6%TRR.

[3] 1 metabolite, 0.017 mg/kg, 0.8%TRR.

[4] No other metabolite.

[5] 1 metabolite, 0.006 mg/kg, 2.2%TRR.

[6] 1 metabolite, 0.034 mg/kg, 2.2%TRR.

[7] 3 metabolites, none >0.059 mg/kg 0.4%TRR.

Table 38 Summary of identified/characterized residues in egg yolk and egg white after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide for 14 consecutive days to laying hens

Residue component	Egg yolk		Egg white	
	% TRR	mg eq/kg	% TRR	mg eq/kg
[B-ring-U-^{14}C]-broflanilide				
Extract	96.0	6.063	89.5	0.017
Broflanilide	ND	ND	(2.1)	(0.0004)[1]
DC-DM-8007	2.1	0.133	15.8	0.003
DM-8007	89.3	5.645	68.4	0.013
DM-(C4-OH)-8007	ND	ND	ND	ND
Total identified	91.4	5.778	84.2	0.016
Total characterized	4.5	0.285 [2]	5.3	0.001 [3]
Unextracted	4.1	0.256	10.5	0.002
Total	100.0	6.319	100.0	0.019
[C-ring-U-^{14}C]-broflanilide				
Extract	96.9	6.549	88.9	0.016
Broflanilide	ND	ND	ND	ND
DM-8007	93.2	6.300	84.4	0.015
DM-(C4-OH)-8007	ND	ND	ND	ND
S(Br-OH)-8007	ND	ND	ND	ND
S(PFP-OH)-8007	ND	ND	ND	ND
Total identified	93.2	6.300	84.4	0.0151
U2-3	ND	ND	2.2	0.0004
HPLC charc.	ND	ND	2.2	0.0004 [5]
Total characterized	3.7	0.249 [4]	4.4	0.0004
Unextracted	3.1	0.211	11.2	0.002
Total	100.0	6.760	100.0	0.018

Notes:

[1] This was detected in TLC analysis (2.2 percent X 0.017 mg/kg = 0.0004 mg/kg).

[2] 3 metabolites, none >0.139 mg/kg, 2.2%TRR.

[3] 2 metabolites, none >0.001 mg/kg, 5.3%TRR.

[4] 3 metabolites, none >0.151 mg/kg, 2.2%TRR.

[5] 2 metabolites, none >0.0002 mg/kg, 1.1%TRR.

Table 39 Summary of identified/characterized residues in liver after oral administration of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide for 14 consecutive days to laying hens

Fraction	[B-ring-U-^{14}C]-broflanilide		[C-ring-U-^{14}C]-broflanilide	
	Liver		Liver	
	mg eq/kg	% TRR	mg eq/kg	% TRR
ACN/H_2O extract	1.607	64.5	1.317	71.6
Broflanilide	ND	ND	ND	ND
DC-DM-8007	0.043	1.7	n/a	n/a
DM-8007	1.424	57.2	1.271	69.1
Unknowns	0.140[2]	5.6	0.046	2.5
Protease (aqueous + organic)	0.264	10.6	0.164	8.9

Fraction	[B-ring-U-^{14}C]-broflanilide		[C-ring-U-^{14}C]-broflanilide	
	Liver		Liver	
	mg eq/kg	% TRR	mg eq/kg	% TRR
H-U27B[1] (B-label) or H-U27C[7] (C-label)	0.068	2.7	0.045	2.4
Unknowns	0.196[3]	7.9	0.119[8]	6.5
Lipase (aqueous + organic)	0.303	12.2	0.159	8.7
H-U27B[1] (B-label) or H-U27C[7] (C-label)	0.036	1.4	0.013	0.72
Unknowns	0.267[4]	10.8	0.146[9]	7.9
1N HCl	0.088	3.6	0.055	3.0
DC-DM-8007	0.019	0.8	ND	ND
DM-8007	0.015	0.6	ND	ND
H-U27B[1]	0.020	0.8	ND	ND
Unknowns	0.034[5]	1.4	0.055[10]	3.0
1N NaOH	0.228	9.2	0.145	7.9
DC-DM-8007	0.022	0.9	ND	ND
DM-8007	0.008	0.3	0.017	0.9
H-U27B[1] (B-label) or H-U27C[7] (C-label)	0.007	0.3	0.003	0.2
Unknowns	0.190[6]	7.7	0.125[11]	6.8
Sum of broflanilide	ND	ND	ND	ND
Sum of DC-DM-8007	0.084	3.4	n/a	n/a
Sum of DM-8007	1.447	58.1	1.288	70.0
Sum of H-U27B[1] (B-label) or H-U27C[7] (C-label)	0.131	5.3	0.061	3.3
Total identified	1.662	66.8	1.349	73.3
Total characterized	0.829	33.4	0.492	26.7
Unextracted	0	0	0	0
Total	2.490	100.0	1.840	100.0

Notes:
[1] Identified as hydroxyl cysteine conjugate of DM-8007.
[2] Unknowns U34 + U40 + others, none >0.061 mg/kg, 2.4%TRR.
[3] Unknowns U30 + U32 + U34 + others, none >0.048 mg/kg, 1.9%TRR.
[4] Unknowns U30 + U32 + U34 + others, none >0.016 mg/kg, 0.7%TRR.
[5] Other, none >0.034 mg/kg, 1.4%TRR.
[6] Unknowns U30 + others, none >0.040 mg/kg, 1.6%TRR.
[7] Mass spectroscopic investigations confirmed the structural similarity to H-U27B.
[8] Unknowns U30 + others, none >0.019 mg/kg, 1.0%TRR.
[9] Other, 1 metabolite 0.0940 mg/kg, 5.10%TRR, included TRR in the rinse after Lipase enzyme (0.016 mg/kg, 0.9%TRR).
[10] No other metabolites.
[11] Unknowns U30 + others, none >0.041 mg/kg, 2.2%TRR.

Summary livestock metabolism

In lactating goats the transfer of radioactivity into milk and tissues was low. In edible tissue, highest the TRRs were found in fat and milk fat. Parent broflanilide was only detected in minor amounts in muscle, kidney and liver, demonstrating extensive metabolism. The main transformation products of Broflanilide in milk and tissues were metabolites DM-8007, DC-DM-8007 and glucuronide conjugates. Glucuronide conjugation was observed for metabolite DM-(C2-OH)-8007, which was previously formed by hydroxylation of metabolite DM-8007. Conjugation was also observed for metabolites DC-DM-(A4-OH)-8007 and DC-DM-(A6-OH)-8007, which were previously formed by hydroxylation of metabolite DC-DM-

8007. Additionally, broflanilide was also further metabolized to hippuric acid which was detected in skim milk, liver, kidney

In laying hens transfer of radioactivity into tissues and eggs was moderate. In edible tissues, highest TRR levels were found in fat, followed by egg yolk. Parent broflanilide was only tentatively identified in egg white demonstrating as well extensive metabolism. The main transformations of broflanilide in the hen tissues were to metabolites DM-8007, DC-DM-8007 and hydroxyl cysteine conjugate of DM-8007 (H-U27).

In all studies, TRR levels in fat were at least one order in magnitude higher compared to meat, indicating fat solubility.

The proposed metabolic pathway of broflanilide in laying hens is shown in Figure 5.

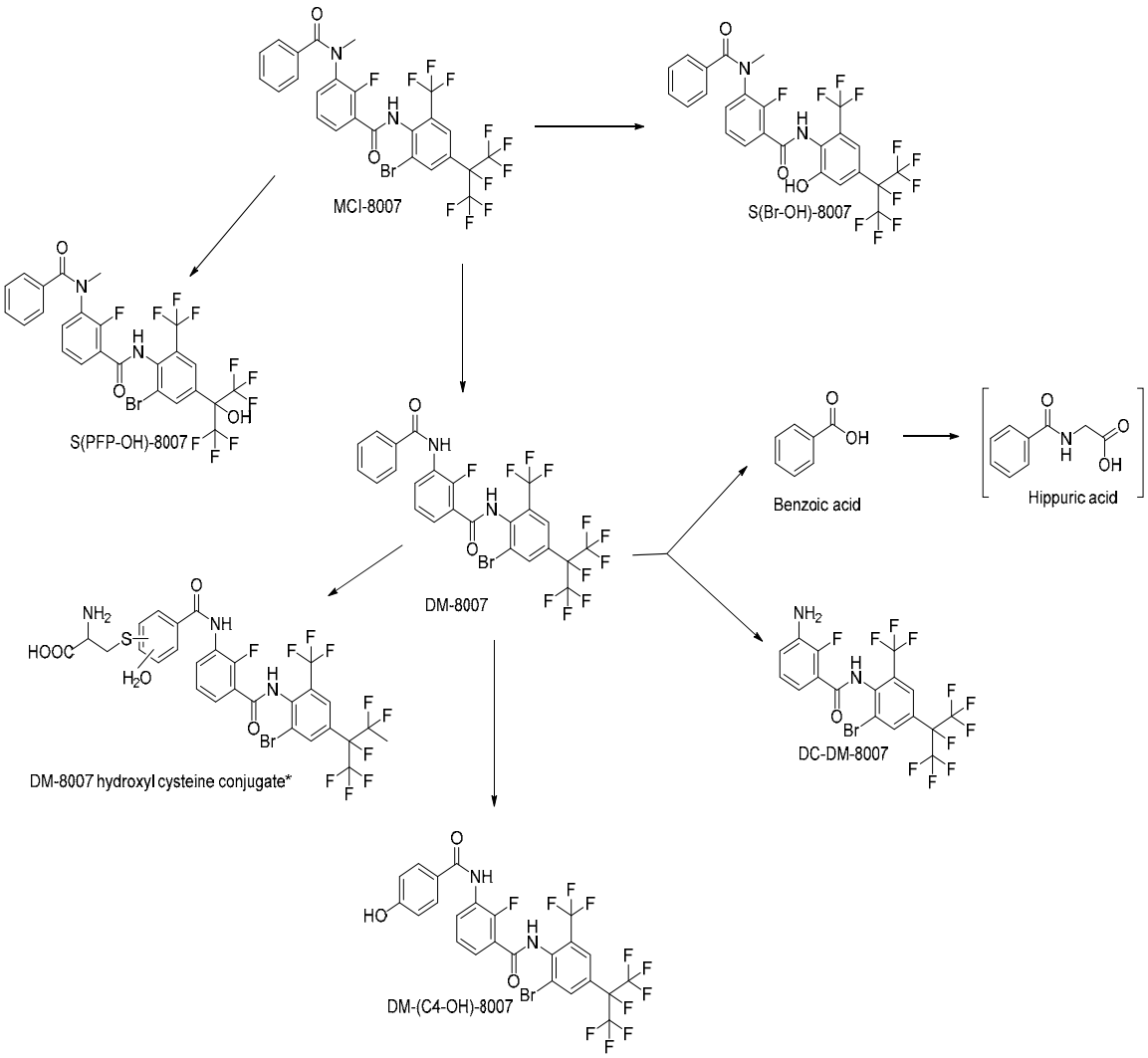


Figure 5 Proposed metabolic pathway of broflanilide in laying hens

Bioaccumulation in fish

A bioaccumulation study in rainbow trout (*Oncorhynchus mykiss*) was performed with [B-ring-U-^{14}C]-labelled broflanilide (Dodd, 2017, BROFLAN_026). Fish were exposed in a flow through system to broflanilide at nominal concentrations of 1.0 µg/L (mean measured: 0.9 µg/L) and 10 µg/L (mean measured: 8.6 µg/L) over a 28 day exposure period, followed by 10 days depuration. Water samples were taken daily and there was no notable degradation of broflanilide in tank water over the course of the study. Fish were sampled at day 0, 1, 3, 7, 14, 21 and 27 during the exposure phase and day 1, 3, 7 and 10 during the depuration phase. The fish were separated into fillet (edible), skeleton and viscera (non-edible) stored at ≤-15 °C until taken for analysis.

The radioactive content in the samples was determined by combustion, followed by LSC. Homogenized samples were subjected to extraction with acetonitrile (twice) and acetonitrile:water (1+1). The lipid content was determined by sequential extraction with chloroform/methanol (1:2), chloroform and water. The chloroform layer was evaporated to dryness and the weight of the dried lipid residue determined. Radioactive components in the solvent extracts were determined by LSC and characterized/identified by co-chromatography with authentic reference standards using reverse phase HPLC and TLC.

After an initial accumulation of radioactive residues to day 7 (low level) or day 3 (high level), steady state was considered to have been achieved. Mean concentrations of radioactivity were highest for both exposure level in the non-edible fish portion. Mean concentrations of radioactivity in the edible portion at steady state were 0.188-0.297 mg eq/kg for the low level and 1.39-1.94 mg eq/kg for the high level. The corresponding steady state measured bioconcentration factors for total radioactivity were for the edible fish portion 241 (low level), 174 (high level), for the non-edible fish portion 470 (low level), 345 (high level) and for the whole fish 366 (low level), 267 (high level). Depuration was rapid for both exposure levels with ~50 percent of the radioactivity eliminated after 1 day and >95 percent elimination after 7 days (Table 40).

Table 40 Mean concentration of radioactivity (mg eq./kg ± sd) and bioconcentration factors (BCF; steady state) for the radioactivity during exposure of fish in water treated with ^{14}C-Broflanilide (B-label) at 1 or 10 mg/L and percent depuration

	Day	Edible (BCF or % depuration)	Non-edible (BCF or % depuration	Whole fish (BCF or % depuration
1 mg/L exposure	1	0.020 ± 0.003 (21)	0.040 ± 0.003 (43)	0.031 ± 0.002 (33)
	3	0.102 ± 0.045 (109)	0.178 ± 0.078 (189)	0.141 ± 0.063 (150)
	7	0.165 ± 0.017 (176)	0.383 ± 0.047 (407)	0.282 ± 0.028 (300)
	14	0.204 ± 0.036 (213)	0.455 ± 0.178 (474)	0.352 ± 0.122 (367)
	21	0.297 ± 0.110 (313	0.503 ± 0.098 (529)	0.406 ± 0.094 (427)
	27	0.188 ± 0.042 (198)	0.388 ± 0.144 (408)	0.289 ± 0.089 (304)
	Mean (14-27)	0.230 ± 0.059 (241)	0.449 ± 0.058 (470)	0.349 ± 0.059 (366)
Depuration	1	0.109 ± 0.083 (52.6%)	0.218 ± 0.114 (51.4%)	0.176 ± 0.073 (51.3%)
	3	0.081 ± 0.041 (64.8%)	0.144 ± 0.063 (67.9%)	0.115 ± 0.054 (67.0%)
	7	0.006 ± 0.001 (97.4%)	0.014 ± 0.003 (96.9%)	0.010 ± 0.002 (97.1%)
	10	0.013 ± 0.010 (94.3%)	0.005 ± 0.002 (98.9)	0.008 ± 0.006 (97.7%)
10 mg/L	1	0.662 ± 0.079 (70)	1.23 ± 0.129 (131)	0.950 ± 0.052 (101)

	Day	Edible (BCF or % depuration)	Non-edible (BCF or % depuration	Whole fish (BCF or % depuration
exposure	3	1.19 ± 0.297 (127)	2.63 ± 0.638 (280)	1.99 ± 0.418 (212)
	7	1.39 ± 0.468 (146)	3.28 ± 0.718 (345)	2.45 ± 0.663 (258)
	14	1.94 ± 0.300 (204)	3.38 ± 1.100 (356)	2.78 ± 0.646 (293)
	21	1.71 ± 0.940 (180)	3.43 ± 0.464 (361)	2.60 ± 0.648 (274)
	27	1.58 ± 0.404 (166)	3.00 ± 0.493 (316)	2.30 ± 0.411 (242)
	Mean (7-27)	1.66 ± 0.231 (174)	3.27 ± 0.192 (345)	2.53 ± 0.205 (267)
Depuration	1	0.714 ± 0.336 (57.0%)	1.62 ± 0.487 (50.5%)	1.21 ± 0.420 (52.2%)
	3	0.271 ± 0.071 (83.7%)	0.603 ± 0.260 (81.6%)	0.449 ± 0.158 (82.3%)
	7	0.066 ± 0.024 (96.0%)	0.111 ± 0.020 (96.6%)	0.093 ± 0.020 (96.3%)
	10	0.020 ± 0.023 (98.8%)	0.074 ± 0.039 (97.7%)	0.049 ± 0.032 (98.1%)

Parent broflanilide accounted in the edible portion at steady state for 31.4–36.8 percent (low level), 31.5–40.2 percent (high level) and in the non-edible portion 29.9–33.0 percent (low level), 33.4–38.5 percent (high level). DM-8007 was the only additional compound identified, accounting at steady state for 53.4–62.6 percent (low level), 55.7–64.8 percent (high level) and in the non-edible portion 56.7–63.3 percent (low level), 57.9–62.8 percent (high level) (Table 41).

Table 41 Summary of identified components in fish (percent sample radioactivity)

Compound	Day	1 mg/L		10 mg/L	
		Edible (%)	Non-edible (%)	Edible(%)	Non-edible (%)
Broflanilide (Exposure)	1	25.6	40.2	53.7	53.4
	3	46.5	40.3	45.1	43.7
	7	41.7	37.1	40.1	36.9
	14	36.8	32.5	31.5	33.4
	21	32.8	33.0	40.2	38.5
	27	31.4	29.9	31.7	35.0
Broflanilide (Depuration)	1	14.4	14.1	12.5	11.8
	3	2.8	nd	4.1	3.0
	7	-	-	-	-
	10	-	-	-	-
DM-8007 (Exposure)	1	42.9	54.0	35.1	40.1
	3	47.8	55.9	50.7	52.4
	7	52.7	56.7	54.1	59.7
	14	53.4	61.5	64.8	62.8
	21	62.6	58.4	55.7	57.9
	27	61.3	63.3	63.8	61.0
DM-8007 (Depuration)	1	79.0	78.3	78.1	84.4
	3	83.8	93.2	83.6	91.4
	7	-	-	-	-
	10	-	-	-	-

At the low exposure level, mean concentrations of broflanilide at steady state were 0.077 mg/kg (edible), 0.143 mg/kg (non-edible) and 0.112 mg/kg (whole fish). The corresponding steady state bioconcentration factors for broflanilide were 84 (edible), 157 (non-edible) and 123 (whole fish). At the high exposure level, mean concentrations of broflanilide at steady state were 0.589 mg/kg (edible), 1.18 mg/kg (non-edible) and 0.893 mg/kg (whole fish). The corresponding steady state bioconcentration factors for broflanilide were 68 (edible), 135 (non-edible) and 102 (whole fish) (Table 42).

For metabolite DM-8007 at the low exposure level, mean concentrations at steady state were 0.137 mg eq/kg (edible), 0.273 mg eq/kg (non-edible) and 0.209 mg eq/kg (whole fish). At the high exposure level, mean concentrations of DM-8007 at steady state were 0.994 mg eq/kg (edible), 1.975 mg eq/kg (non-edible) and 1.508 mg eq/kg (whole fish) (Table 42). Mean steady state bioconcentration factors for broflanilide were 68-84 (edible), 135-157 (non-edible) and 102-123 (whole fish).

There was no apparent increase in the lipid content of the fish during the course of the study. Whole fish lipid content expressed as percent tissue weight was 2.97 percent at Day 0, 2.62 percent at the termination of exposure and 2.66 percent at termination of the depuration phase

Table 42 Concentrations of [^{14}C]-broflanilide and metabolite DM-8007 (mg eq/kg) in fish tissues during exposure and depuration phase. BFC= bioconcentration factor

Compound	Day	1 mg/L			10 mg/L		
		Edible	Non-edible	Whole fish	Edible	Non-edible	Whole fish
Broflanilide (Exposure)	1	0.005	0.016	0.011	0.355	0.657	0.505
	3	0.047	0.072	0.059	0.537	1.15	0.853
	7	0.069	0.142	0.109	0.557	1.21	0.898
	14	0.075	0.148	0.117	0.611	1.13	0.909
	21	0.097	0.166	0.131	0.687	1.32	0.984
	27	0.059	0.116	0.089	0.501	1.05	0.782
Mean at steady-state (BCF)		0.077 (84)	0.143 (157)	0.112 (123)	0.589 (68)	1.18 (135)	0.893 (102)
Broflanilide (Depuration)	1	0.016	0.031	0.025	0.089	0.191	0.144
	3	0.002	-	0.001	0.011	0.018	0.015
DM-8007 (Exposure)	1	0.009	0.022	0.016	0.232	0.493	0.362
	3	0.049	0.100	0.074	0.603	1.38	1.01
	7	0.087	0.217	0.158	0.752	1.96	1.39
	14	0.109	0.280	0.206	1.26	2.12	1.76
	21	0.189	0.294	0.238	0.952	1.99	1.45
	27	0.115	0.246	0.184	1.01	1.83	1.43
Mean at steady-state		0.137	0.273	0.209	0.994	1.975	1.508
DM-8007 (Depuration)	1	0.086	0.171	0.139	0.558	1.37	0.994
	3	0.067	0.134	0.104	0.227	0.551	0.400

The calculation of the lipid-normalized bioconcentration factors (steady state) for total radioactivity during exposure to tank water treated with [^{14}C]-Broflanilide is shown in Table 43. Lipid normalized steady state bioconcentration factors were 517-720 for total radioactivity and 198-242 for broflanilide.

Table 43 Lipid-normalized bioconcentration factors (steady state) for total radioactivity in whle fish during exposure to tank water treated with [^{14}C]-Broflanilide. Results are normalized to 5 percent lipid content

Exposure Day	1 mg/L		10 mg/L	
	Total radioactivity	Broflanilide	Total radioactivity	Broflanilide
1	60	23	182	99
3	300	134	424	204
7	564	244	485	207
14	675	228	539	195
21	905	316	581	231
27	580	183	462	160
Mean	720[1]	242[1]	517[2]	198[2]

Notes:
[1] Mean from 14-27 days
[2] Mean from 7-27 says

Table 44 Uptake and depuration rate constants and bioconcentration factors (BCF) for total radioactive residues of broflanilide in whole fish, edible tissue and non-edible tissue based on measured and calculated data

	1 mg/L			1 mg/L		
	Edible	Non-edible	Whole fish	Edible	Non-edible	Whole fish
Ln (5 percent lipid normalization factor) *(based on mean lipid content of 6 fish at the end of uptake)*	2.91	1.47	1.91	2.91	1.47	1.91
k_1 (growth corrected uptake rate constant, day^{-1})	72.6	26.7	185	74.8	167	118
k_{2g} (growth-corrected depuration rate constant, day^{-1})	0.94	0.145	1.51	1.17	1.25	1.20
C_{Fss}, (steady-state fish concentration, µg/kg) *(mean (days 14-27 low exposure, days 7-27 high exposure ± standard deviation)*	$2.3x10^{-4}$ ±0.059	$4.49x10^{-4}$ ±0.058	$3.49x10^{-4}$ ±0.059	$1.66x10^{-3}$ ±0.231	$3.27x10^{-3}$ ±0.192	$2.53x10^{-3}$ ±0.205
C_W (concentration in water, µg/L) *(mean (days 0-27) ± standard deviation)*	--	--	0.9±0.09	--	--	8.6±0.5
BCF_{ss} (steady-state BCF) (mean *(days 14-27 low exposure, days 7-27 high exposure)*	84	157	123	68	135	102
BCF_{ssL} (lipid normalized BCF_{ss}) *(mean (days 14-27 low exposure, days 7-27 high exposure))*	--	--	242	--	--	198
BCF_{Kg} (growth corrected kinetic BCF)	77.4	184	122	64.1	134	98.4
BCF_{KLG} (lipid normalized BCF_{Kg}) [1)]	232	271	234	192	198	189
$t_{1/2g}$ (growth-corrected half-life; days)	0.7	4.8	0.5	0.6	0.6	0.6

Environmental fate

For the investigation of the environmental fate of broflanilide, the Meeting received studies on aerobic soil metabolism and degradation, soil photolysis and on the behaviour in confined and field rotational crops.

Aerobic soil degradation

The rate of degradation of broflanilide was studied in one fresh aerobic soil (moisture adjusted to 50 percent maximum water holding capacity) using [A-ring-U-^{14}C]-, [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide at a nominal application rate of 0.2 mg/kg, corresponding to 50 g ai/ha (Ta, 2017, BROFLAN_027). Soil characteristics are shown in Table 45.

Table 45 Soil characteristics (BROFLAN_027)

Soil Name	Texture	Soil series	pH (water)	Cation exchange capacity [meq/100g]	Maximum Water Holding Capacity (g/100 g dry soil)	Organic C [percent]
Soil Ca	Sandy Clay Loam	Centerville clay	7.6	23.2	46.5	0.87

The test system was maintained in the dark at a nominal temperature of 20 2 C for 365 days. Volatile organics and CO_2 were trapped with ethylene glycol and 1 mol/L NaOH, respectively. Samples were taken at 0, 7, 14, 30, 59, 91, 120, 177, 269, and 365 days after application.

The soil samples were sequentially extracted with methanol, followed by two extractions with methanol/water (7/3). The 365 DAT samples were further extracted with ethyl acetate, hexane, and dioxane. Extracts were analysed by HPLC against reference standards to identify parent and metabolites. Confirmation of the identity of the metabolites was done for selected samples by LC/MS. The radioactivity in extracts and traps was quantified by LSC. The soil remaining after the extraction was combusted followed by LSC.

Broflanilide declined from 102–108 percent AR to 75–86 percent AR over the study time. The only identified metabolite were DM-8007 and S(PFP-OH)-8007, accounting for up to 1.6 percent AR and 1.1 percent AR, respectively (Table 46). The radioactivity in the bound residues increased over time from 0.9–1.1 percent AR to 5.7–8.0 percent AR.

Table 46 Biotransformation of radiolabelled broflanilide, expressed as of applied radioactivity percentage (mean from duplicate samples)

	Sampling time (days)									
	0	7	14	30	59	91	120	177	269	365
[A-ring-U-^{14}C]-broflanilide										
Broflanilide	102.3	99	97	94.3	95.6	94.8	91.1	90.4	83.9	80.1
DM-8007	ND	ND	<LOQ	0.5	ND	ND	<LOQ	0.8	1	0.9
S(PFP-OH)-8007	1.1	0.9	0.9	0.7	1	0.9	0.6	1	ND	ND
Unknown (51.1-51.5 min)	ND	ND	ND	ND	<LOQ	<LOQ	<LOQ	1.1	2	3
Unknown (51.9 min)	ND	ND	ND	ND	<LOQ	ND	ND	ND	0.6	2.3
Others	0.6	ND	ND	ND	0.5	<LOQ	<LOQ	ND	<LOQ	0.9
Total extracted	104	99.9	98.4	95.9	98.1	96.2	92.8	93.3	88.2	88.5
CO_2	n/a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.5	0.8	1.1
Volatiles organics	n/a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Unextracted	0.9	1.3	1.7	1.8	2.5	3.1	3.3	5.1	7.2	8
Total	104.9	101.2	100	97.7	100.6	99.3	96.2	98.8	96.3	97.7
[B-ring-U-^{14}C]-broflanilide										
Broflanilide	104.8	94.3	95.1	95.2	94.5	90.1	90.3	88.8	82.8	86.1
DM-8007	0.9	1.2	0.7	1.2	0.5	1.3	1.2	1.3	1.3	1.1

	Sampling time (days)									
	0	7	14	30	59	91	120	177	269	365
S(PFP-OH)-8007	0.8	0.7	0.7	0.6	<LOQ	0.6	1	0.6	ND	ND
Unknown (51.3-51.4 min)	ND	ND	ND	ND	ND	ND	ND	1.5	2.3	1.6
Unknown (51.8-51.9 min)	ND	ND	ND	ND	ND	ND	ND	ND	1.4	1.4
Others	ND	ND	ND	ND	ND	ND	1	ND	1.2	ND
Total extracted	106.5	96.2	96.7	97.1	95.7	92	93.7	92.3	89.1	91.4
CO_2	n/a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Volatiles organics	n/a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Unextracted	1.1	1.4	1.3	1.6	2.2	3.2	3.1	4.5	8.1	7.3
Total	107.6	97.5	98	98.7	98	95.3	96.8	96.7	97.2	98.7
[C-ring-U-^{14}C]-broflanilide										
Broflanilide	107.7	94.9	90.4	91.8	89.5	79.8	85.4	85.3	80	74.7
DM-8007	ND	0.6	0.9	<LOQ	<LOQ	1.6	1	0.8	<LOQ	<LOQ
S(PFP-OH)-8007	1	0.8	<LOQ	1	<LOQ	<LOQ	0.7	ND	ND	ND
Unknown (3.2 min)	ND	ND	ND	ND	ND	1.9	ND	ND	ND	ND
Unknown (50.5-50.8 min)	ND	ND	<LOQ	0.9	0.9	ND	1.1	0.8	0.6	1
Unknown (51.2-51.6 min)	ND	ND	<LOQ	0.6	1.6	1.2	1	<LOQ	2.8	2.4
Unknown (51.6-51.9 min)	ND	ND	<LOQ	<LOQ	ND	1	ND	<LOQ	1.9	2.8
Others	ND	ND	0.8	<LOQ	ND	1.4	1.2	<LOQ	0.6	2
Total extracted	108.7	96.4	93.8	95.7	93.1	87.3	90.3	88.4	86.5	84.4
CO_2	n/a	<LOQ	<LOQ	0.7	1.1	1.5	2	2.8	4	4.7
Volatiles organics	n/a	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Unextracted	0.9	1.4	2.6	2.5	3.1	3.4	3.3	4.5	6.5	5.7
Total	109.6	97.8	96.6	98.9	97.3	92.3	95.6	95.7	97	94.8

Notes:

LOQ= 0.3 percent AR (for CO_2 and volatiles organics); 0.5 percent AR (all others).

Degradation endpoints were derived from the best-fit kinetic models for broflanilide. The degradation results are given in Table 47.

Table 47 Half-life calculation for broflanilide in soil under aerobic conditions (BROFLAN_028)

Data set	Kinetic model	Estimated parameter [d^{-1}]	DT_{50} [d]
Full 365 day	SFO	k: 0.0006	1126 [a]
Truncated 120 day	SFO	k: 0.0012	596

Notes:

[a] If DT_{50} > 1000 days, KinGUII does not output estimated value. In these cases, DT_{50} was back-calculated according to ln(2)/k, where k was the SFO rate constant optimized during the kinetic fitting.

In a second study, the degradation of broflanilide was investigated in three fresh aerobic soils (moisture adjusted to 50 percent maximum water holding capacity) using [B-ring-U-^{14}C]-labelled broflanilide at a nominal application rate of 0.2 mg/kg, corresponding to a proposed maximum field application rate of 50 g ai/ha (Ta, 2017, BROFLAN_028). Soil characteristics are shown in Table 48. “Processed soil” was sieved through a 2 mm sieve before use, while intact soil cores were directly placed in Plexiglas boxes. The moisture content of the soils were maintained at approximately 50 percent maximum water holding capacity (MWHC).

Table 48 Soil characteristics

Soil Name	Texture	Soil series	pH (water)	Cation exchange capacity [meq/100g]	Maximum Water Holding Capacity (g/100 g dry soil)	Organic matter [percent]
Soil IL	Silty clay	Drummer silty clay loam	4.8	22.5	54.4	0.87
Soil NC	Sandy loam	Norfolk	5.9	4.7	31.3	1.7
Soil TN	Silt loam	Falaya silt loam	7.0	5.7	34.5	1.3

Both test systems were kept in the dark at nominal temperature of 25 2 C for 365 days. CO2 was trapped with 1 mol/L NaOH. Duplicate samples were taken at 0, 15, 30, 58, 86, 120, 177, 259 and 365 days after application.

The soil samples were sequentially extracted with acetonitrile, acetonitrile:water (1/1), and methanol:water (1/1) with the exception of 259 DAT samples of replicate 2 and 365 DAT samples of both replicates. The 259 DAT samples of replicate 2 and 365 DAT samples of both replicates were extracted with methanol, methanol:water (7/3), and methanol:water (7/3). Further, the "processed" 365 DAT samples were extracted with ethyl acetate, hexane, and dioxane. Extracts were analysed by two different HPLC methods (method 1 & method 2) against reference standards to identify parent and metabolites. The radioactivity in extracts and traps was quantified by LSC. The soil remaining after the extraction was combusted followed by LSC.

The mass balances of broflanilide in intact soil cores and processed soil are presented in Tables 49 and 50. In all soils, the extracted radioactivity declined from 97.5–99.6 percent AR to 59–90 percent AR, while the unextracted radioactive increased from <LOQ-1.2 percent AR to 8–41 percent AR. Further analysis of the bound residues from 365 DAT showed that fulvic acid, humic acid and humin, accounted for 1.2–6.6 percent AR, 3.9–23 percent AR and 1.7–6.8 percent AR, respectively. The fraction detected as CO_2 accounted for up to 4 percent AR.

Table 49 Biotransformation of radiolabelled broflanilide n intact soil cores, expressed as percentage of applied radioactivity (mean from duplicate samples)

	Sampling time (days)								
	0	15	30	58	86	120	177	259	365
IL soil									
Total extracted radioactivity	97.5	97.7	96.1	92.2	91.9	80.7	90.8	86.2	83.7
CO_2	NA	0.4	0.6	0.8	0.9	1.1	1.4	1.5	1.6
Unextracted	<LOQ	3	3.5	5.4	5	16.5	8.5	11.6	12.1
Total	97.5	101.1	100.2	98.4	97.8	98.3	100.5	99.3	97.4
NC soil									
Total extracted radioactivity	98.5	98.2	96.1	85.9	86.4	79.6	86.6	61.5	58.7
CO_2	NA	0.6	1	1.6	1.8	1.8	2.7	3.9	4
Unextracted	<LOQ	2.2	4.8	12.8	13.3	17.9	11.7	29.4	40.6
Total	98.5	101	101.8	100.3	101.6	99.4	101	95.6	103.3
TN soil									
Total extracted radioactivity	99	94.8	91.8	80.5	79.2	78.1	81.8	73.6	64.5
CO_2	NA	0.3	0.5	1	1.3	1.8	2.5	2.5	2.7
Unextracted	<LOQ	3.2	7.3	17.5	22.3	18.2	16.7	24.1	26.2
Total	99	98.4	99.5	98.9	102.9	98.2	101	100.3	93.5

Notes:

LOQ= 0.3 percent AR (for CO_2); 1.3 percent AR.

Table 50 Biotransformation of radiolabelled broflanilide in processed soil cores, expressed as percentage (mean from duplicate samples) of applied radioactivity

	Sampling time (days)								
	0	15	30	58	86	120	177	259	365
IL soil									
Total extracted radioactivity	98.8	95.6	95	94.1	92.9	97.1	90.3	89	88.9
CO_2	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Unextracted	1.2	2.1	2.8	3.7	4.1	4.7	5	7.6	8
Total	100	97.7	97.8	97.8	97	101.8	95.3	96.6	96.9
NC soil									
Total extracted radioactivity	99.6	97.2	95.9	93.5	91.9	90	89.3	85.4	83.6
CO_2	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.5
Unextracted	0.4	1.4	2.1	5.2	5.5	8.1	7.6	12.3	12.9
Total	100	98.6	98	98.7	97.4	98.1	96.9	97.8	97
TN soil									
Total extracted radioactivity	99.5	97.6	96.3	95.7	93.3	94.7	90.6	91.4	90
CO_2	NA	<LOQ	<LOQ	<LOQ	0.4	0.4	0.6	0.8	1.1
Unextracted	0.5	2.4	3.1	3.6	4.9	6	7.3	7	10.3
Total	100	100	99.3	99.3	98.6	101.1	98.5	99.1	101.5

Notes:

LOQ= 0.3 percent AR (for CO_2); 1.3 percent AR.

The fraction of identified broflanilide and its metabolites is presented in Tables 51 to 62. For all soils (intact and processed) and methods, parent broflanilide declined from 95–99 percent to 52–87 percent over the study time. Identified metabolites were S(PFP)-OH-8007 at up to 1.2 percent AR, DC-DM-8007 at up to 2.3 percent AR, DM-8007 at up to 4.9 percent AR, DC-8007 at up to 0.5 percent AR and unknowns/others at up to 5.5 percent AR.

Table 51 HPLC quantitation of broflanilide and its metabolites from intact IL soil extract (method 1) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP)-OH-8007	DC-DM-8007	UNK 2	UNK 3	Broflanilide	DM-8007	UNK 4	Others
0	1.1	<LOQ	<LOQ	<LOQ	95.6	1.1	<LOQ	NA
15	1.0	<LOQ	<LOQ	<LOQ	94.8	2.3	<LOQ	NA
30	1.1	<LOQ	<LOQ	<LOQ	94.0	1.2	<LOQ	NA
58	0.8	<LOQ	<LOQ	<LOQ	90.2	1.3	<LOQ	NA
86	<LOQ	<LOQ	<LOQ	<LOQ	89.2	1.7	<LOQ	<LOQ
120	0.6	<LOQ	<LOQ	<LOQ	77.3	1.5	<LOQ	NA
177	0.7	<LOQ	<LOQ	0.7	86.5	1.9	<LOQ	<LOQ
259	0.7	<LOQ	<LOQ	1.2	80.1	1.7	0.7	NA
365	<LOQ	<LOQ	0.9	<LOQ	78.7	1.6	<LOQ	<LOQ

Table 52 HPLC quantitation of broflanilide and its metabolites from intact IL soil extract (method 2) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP)-OH-8007	UNK 4	DC-DM-8007	Broflanilide	DM-8007	UNK6	UNK 7
0	0.6	<LOQ	<LOQ	97.2	<LOQ	<LOQ	<LOQ
15	<LOQ	<LOQ	<LOQ	97.0	0.9	<LOQ	<LOQ
30	<LOQ	<LOQ	<LOQ	95.4	1.1	<LOQ	<LOQ
58	<LOQ	0.7	0.7	87.2	1.0	2.0	0.7
86	<LOQ	<LOQ	<LOQ	89.6	1.6	<LOQ	<LOQ
120	<LOQ	<LOQ	<LOQ	79.1	0.5	<LOQ	<LOQ
177	<LOQ	<LOQ	<LOQ	89.6	<LOQ	<LOQ	<LOQ
259	<LOQ	<LOQ	<LOQ	83.4	1.3	<LOQ	<LOQ
365	<LOQ	<LOQ	<LOQ	80.6	0.9	0.6	<LOQ

Table 53 HPLC quantitation of broflanilide and its metabolites from intact NC soil extract (method 1) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP)-OH-8007	UNK1	DC-DM-8007	UNK2	UNK3	UNK4	UNK5	Broflanilide	DM-8007	UNK7	UNK8	UNK9	UNK 10
0	1.2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	95.4	1.3	<LOQ	<LOQ	<LOQ	<LOQ
15	0.9	<LOQ	<LOQ	<LOQ	<LOQ	0.6	<LOQ	94.3	1.6	<LOQ	<LOQ	<LOQ	<LOQ
30	0.9	1.0	<LOQ	<LOQ	<LOQ	1.3	<LOQ	89.1	2.7	0.9	<LOQ	<LOQ	<LOQ
58	<LOQ	2.5	<LOQ	0.5	<LOQ	<LOQ	0.7	78.8	2.4	<LOQ	<LOQ	<LOQ	<LOQ
86	<LOQ	2.6	<LOQ	1.1	<LOQ	1.0	<LOQ	79.8	2.1	<LOQ	<LOQ	<LOQ	<LOQ
120	<LOQ	1.8	<LOQ	1.5	<LOQ	0.5	<LOQ	73.5	1.7	<LOQ	<LOQ	<LOQ	<LOQ
177	<LOQ	<LOQ	<LOQ	0.9	1.8	<LOQ	<LOQ	80.3	0.8	<LOQ	<LOQ	<LOQ	<LOQ
259	<LOQ	<LOQ	<LOQ	<LOQ	1.4	<LOQ	<LOQ	54.1	0.9	0.9	0.6	1.0	0.9
365	0.5	<LOQ	0.5	1.8	<LOQ	<LOQ	<LOQ	52.2	2.9	<LOQ	<LOQ	<LOQ	<LOQ

Table 54 HPLC quantitation of broflanilide and its metabolites from intact NC soil extract (method 2) in percent of applied radioactivity (mean from duplicate samples)

DAT	UNK 1	DC-DM-8007	UNK 3	DC-8007	Broflanilide	UNK 5	DM-8007	UNK 6	UNK 7	UNK 8	UNK 9	Others
0	<LOQ	<LOQ	<LOQ	<LOQ	97.2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	NA
15	<LOQ	<LOQ	<LOQ	<LOQ	97.2	<LOQ	0.7	<LOQ	<LOQ	<LOQ	<LOQ	NA
30	<LOQ	0.6	<LOQ	0.5	93.2	<LOQ	1.8	<LOQ	<LOQ	<LOQ	<LOQ	NA
58	<LOQ	1.3	1.0	<LOQ	78.2	<LOQ	2.4	<LOQ	1.2	<LOQ	<LOQ	<LOQ
86	<LOQ	1.7	<LOQ	<LOQ	80.7	<LOQ	0.8	<LOQ	3.0	<LOQ	<LOQ	NA
120	<LOQ	0.7	<LOQ	<LOQ	74.2	<LOQ	<LOQ	<LOQ	3.1	<LOQ	0.7	NA
177	0.5	<LOQ	<LOQ	<LOQ	80.0	<LOQ	<LOQ	<LOQ	2.1	<LOQ	0.7	NA
259	<LOQ	<LOQ	<LOQ	<LOQ	53.0	<LOQ	<LOQ	<LOQ	5.5	0.5	0.9	NA
365	1.3	<LOQ	<LOQ	<LOQ	51.5	1.4	1.4	0.7	2.1	<LOQ	<LOQ	NA

Table 55 HPLC quantitation of broflanilide and its metabolites from intact TN soil extract (method 1) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP)-OH-8007	UNK1	UNK2	UNK3	UNK4	UNK5	Broflanilide	DM-8007	UNK6	Others
0	<LOQ	<LOQ	1.0	<LOQ	<LOQ	<LOQ	96.4	1.6	<LOQ	NA
15	<LOQ	<LOQ	1.0	0.9	<LOQ	0.8	90.6	1.6	<LOQ	NA
30	<LOQ	<LOQ	0.5	0.8	<LOQ	<LOQ	82.4	2.2	<LOQ	<LOQ
58	<LOQ	<LOQ	0.7	1.8	1.4	0.9	74.2	0.8	<LOQ	NA
86	<LOQ	<LOQ	0.5	<LOQ	1.6	<LOQ	75.4	1.5	<LOQ	NA
120	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	73.1	3.7	<LOQ	NA
177	<LOQ	<LOQ	<LOQ	<LOQ	0.7	<LOQ	75.7	4.9	<LOQ	<LOQ
259	<LOQ	4.3	<LOQ	<LOQ	0.5	<LOQ	67.7	<LOQ	4.3	<LOQ
365	<LOQ	<LOQ	<LOQ	0.5	0.7	<LOQ	59.7	3.0	<LOQ	NA

Table 56 HPLC quantitation of broflanilide and its metabolites from intact TN soil extract (method 2) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP)-OH-8007	UNK1	UNK2	UNK3	UNK4	DC-DM-8007	Broflanilide	DM-8007	UNK8	UMK9
0	1.0	0.5	0.6	0.5	<LOQ	<LOQ	95.7	0.8	<LOQ	<LOQ
15	0.7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	92.6	1.3	<LOQ	<LOQ
30	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	84.7	1.2	<LOQ	0.6
58	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	2.3	74.0	<LOQ	<LOQ	2.6
86	<LOQ	<LOQ	<LOQ	<LOQ	0.7	<LOQ	73.9	1.4	<LOQ	2.8
120	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	74.5	2.1	<LOQ	0.6
177	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	77.1	2.9	<LOQ	1.8
259	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	68.0	2.6	<LOQ	2.4
365	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	60.0	2.5	0.5	1.3

Table 57 HPLC quantitation of broflanilide and its metabolites from processed IL soil extract (method 1) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP)-OH-8007	DC-DM-8007	Broflanilide	DM-8007	UNK 3	UNK 4
0	0.9	<LOQ	95.9	1.4	0.7	<LOQ
15	1.0	<LOQ	93.3	1.3	<LOQ	<LOQ
30	0.7	<LOQ	93.4	0.7	<LOQ	<LOQ
58	0.9	<LOQ	91.6	1.0	<LOQ	<LOQ
86	0.8	<LOQ	91.0	<LOQ	<LOQ	<LOQ
120	0.8	0.7	95.1	<LOQ	<LOQ	<LOQ
177	0.6	0.6	88.6	<LOQ	<LOQ	<LOQ
259	0.5	1.0	86.7	<LOQ	<LOQ	<LOQ
365	0.6	0.7	84.7	<LOQ	<LOQ	0.5

Table 58 HPLC quantitation of broflanilide and its metabolites from processed IL soil extract (method 2) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP)-OH-8007	UNK 1	DC-8007	Broflanilide	DM-8007	UNK 3
0	<LOQ	<LOQ	NA	97.3	1.2	<LOQ
15	<LOQ	<LOQ	NA	95.0	<LOQ	<LOQ
30	<LOQ	<LOQ	NA	94.5	<LOQ	<LOQ
58	<LOQ	<LOQ	NA	93.8	<LOQ	<LOQ
86	<LOQ	<LOQ	NA	92.2	<LOQ	<LOQ
120	<LOQ	<LOQ	NA	96.4	<LOQ	<LOQ
177	<LOQ	<LOQ	NA	90.4	<LOQ	<LOQ
259	<LOQ	<LOQ	<LOQ	87.9	<LOQ	<LOQ
365	<LOQ	0.5	NA	86.1	<LOQ	0.5

Table 59 HPLC quantitation of broflanilide and its metabolites from processed NC soil extract (method 1) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP)-OH-8007	DC-DM-8007	UNK 4	Broflanilide	DM-8007	UNK 5
0	0.8	<LOQ	<LOQ	96.8	0.5	1.2
15	1.0	<LOQ	<LOQ	94.5	1.7	<LOQ
30	0.6	<LOQ	<LOQ	93.0	1.7	<LOQ
58	0.8	0.9	<LOQ	90.0	1.5	<LOQ
86	0.8	<LOQ	<LOQ	89.0	1.5	<LOQ
120	0.8	0.5	<LOQ	86.8	0.9	<LOQ
177	0.9	0.8	<LOQ	85.8	<LOQ	0.7
259	<LOQ	0.9	0.8	82.1	<LOQ	1.1
365	0.5	<LOQ	0.7	79.7	<LOQ	<LOQ

Table 60 HPLC quantitation of broflanilide and its metabolites from processed NC soil extract (method 2) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP)-OH-8007	DC-DM-8007	DC-8007	Broflanilide	DM-8007	UNK 3	Others
0	<LOQ	<LOQ	<LOQ	98.4	1.1	<LOQ	NA
15	<LOQ	<LOQ	<LOQ	96.6	<LOQ	<LOQ	NA
30	<LOQ	<LOQ	<LOQ	94.6	1.0	<LOQ	NA
58	<LOQ	<LOQ	<LOQ	91.6	<LOQ	<LOQ	<LOQ
86	<LOQ	<LOQ	<LOQ	91.2	<LOQ	<LOQ	NA
120	<LOQ	<LOQ	<LOQ	88.8	<LOQ	0.6	NA
177	<LOQ	<LOQ	<LOQ	88.5	<LOQ	0.7	NA
259	<LOQ	<LOQ	<LOQ	82.8	<LOQ	1.0	NA
365	<LOQ	<LOQ	<LOQ	79.6	<LOQ	0.7	<LOQ

Table 61 HPLC quantitation of broflanilide and its metabolites from processed TN soil extract (method 1) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP)-OH-8007	Broflanilide	DM-8007	UNK 2	UNK 3
0	0.8	96.1	0.7	1.9	<LOQ
15	0.6	96.6	<LOQ	<LOQ	<LOQ
30	1.1	94.5	<LOQ	0.5	<LOQ
58	0.9	94.4	<LOQ	0.6	<LOQ
86	1.0	91.4	1.0	<LOQ	<LOQ
120	1.0	92.7	1.1	<LOQ	<LOQ
177	0.9	88.7	<LOQ	<LOQ	0.8
259	0.8	88.9	<LOQ	0.8	0.9
365	0.8	87.4	<LOQ	1.1	<LOQ

Table 62 HPLC quantitation of broflanilide and its metabolites from processed TN soil extract (method 2) in percent of applied radioactivity (mean from duplicate samples)

DAT	S(PFP) OH-8007	UNK 2	Broflanilide	DM-8007	UNK 4	UNK 5
0	<LOQ	<LOQ	98.8	<LOQ	<LOQ	<LOQ
15	<LOQ	<LOQ	97.1	<LOQ	<LOQ	<LOQ
30	<LOQ	<LOQ	95.5	<LOQ	<LOQ	<LOQ
58	<LOQ	<LOQ	94.3	<LOQ	<LOQ	0.6
86	<LOQ	<LOQ	91.9	<LOQ	0.6	<LOQ
120	<LOQ	<LOQ	93.7	<LOQ	<LOQ	<LOQ
177	<LOQ	<LOQ	89.8	<LOQ	<LOQ	<LOQ
259	0.5	<LOQ	89.2	<LOQ	<LOQ	0.8
365	<LOQ	0.6	87.0	<LOQ	<LOQ	1.3

Degradation endpoints were derived from the best-fit kinetic models for broflanilide. The degradation results are given in Table 63.

Table 63 Half-life (DT_{50}) calculation for broflanilide in soil under aerobic conditions

	Full 36- day data set						Truncated 120-day data set					
Soil	Intact soil			Processed soil			Intact soil			Processed soil		
	HPLC Method		Avg.	HPLC Method		Avg.	HPLC Method		Avg.	HPLC Method		Avg.
	1	2		1	2		1	2		1	2	
IL	1000[b]	1000[b]	1000	1000[b]	1000[b]	1000	451	425	438	1000[b]	1000[b]	1000
NC	409	375	392	1000[b]	1000[b]	1000	301	276	288	654	745	699
TN	616	615	616	1000[b]	1000[b]	1000	285	280	282	1000[b]	1000[b]	1000
Intact avg.			669	Processed avg.		1000	Intact avg.		336	Processed avg.		900

Notes:

[a] SFO model was selected as representative model for all evaluations. DT_{50} units are days.

[b] Endpoint exceeds 1000 days. Therefore, 1000 days proposed as a practical, conservative approximation.

In a third study, the soil dissipation/accumulation of broflanilide under field conditions was studied using bare ground plots at five sites in the United States (NC, FL, CA, WA, ND) (Mitchell, 2017, BROFLAN_029). Top soil characteristics are shown in Table 64. A suspension concentrate (SC)

formulation of broflanilide (100 g ai/L) was applied at 2×25 g ai/ha at 7-day intervals for the NC, FL, and CA sites and 1×50 g ai/ha for the WA and ND sites.

Table 64 Top soil (0-6 inches) characteristics (BROFLAN_029).

Soil Name	Texture	Soil series	pH (water)	Cation exchange capacity [meq/100g]	Organic matter [percent]
Soil NC	Loamy sand	Wickham	5.7	3.4	0.66
Soil FL	Sand	Basinger-Smyrna	7.3	4.4	1.4
Soil CA	Loamy sand	Hanford	7.1	4.5	0.53
Soil WA	Sand	Quincy	8.1	8.8	0.26
Soil ND	Clay	Hegne-Fargo	6.9	29.7	3.2

The proposed sampling schedule for soil cores was as follows: prior to and immediately after each test substance application (two applications in NC, FL, and CA; one application in WA and ND) and then 3, 5, 10, 20, 50, 90, 180, 270, 360, 450, and 540 days after last application. Due to the frozen ground, soil samples were not taken at 180 DALT in ND. Due to flooding from Hurricane Matthew, soil samples were not taken at 540 DALT in NC. Soil cores were sectioned into segments of 0–2, 2–4, 4–6, 6–12, 12–18, 18–24, 24–30, 30–36, and 36–42 inches. The top three segments had a length of two inches and all deeper segments had a six-inch length. Core segments were composited by depth within each replicate.

Soil samples were extracted with methanol, followed by methanol/water (7+3). The extracts were combined, diluted 1:5 with methanol/water (1+1) and analysed for broflanilide and its metabolites DM-8007, DC-DM-8007, DC 8007, and S(PFP-OH)-8007 using LC-MS/MS. The limit of quantitation (LOQ) for residues of Broflanilide in soil was 0.001 mg/kg and the limit of detection (LOD) was 0.0002 mg/kg, for each analyte. Mean procedural recoveries from control soil samples fortified with broflanilide and metabolites at 1×, 100× and 600× LOQ were within the acceptable range of 70 to 120 percent.

The measured levels of broflanilide declined from 0.044-0.122 mg/kg at 0 DALT to 0.0002–0.0069 mg/kg at 450–521 DALT, with a few exceptions exclusively found at the 0–5 cm depth. Quantified metabolites were DM-8007 at up to 0.0024 mg/kg and S(PFP)-OH-8007 at up to 0.0008 mg/kg. Concentrations of DC-DM-8007 and DC-8007 were consistently below LOD. The results are shwon in Table 65 to 69.

Table 65 Mean Residues of broflanilide and metabolites (mg/kg) in treated soil samples – North Caroline site

Analyte	Soil Depth (in)	Targeted days after last treatment (actual DALT shown below)												
		T1	-T2	T2	3	5	10	20	50	90	180	270	360	450
		-7 DALT	-1 DALT	0 DALT	3 DALT	5 DALT	10 DALT	20 DALT	49 DALT	94 DALT	177 DALT	267 DALT	365 DALT	455 DALT
Broflanilide	0-2	0.0340	0.0155	0.0600	0.0339	0.0286	0.0236	0.0213	0.0112	0.0029	0.0065	0.0024	0.00096	0.0005
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	0.0340	0.0155	0.0600	0.0339	0.0286	0.0236	0.0213	0.0112	0.0029	0.0065	0.0024	0.00096	0.0005
DM-8007	0-2	<LOD	0.0004	0.0006	0.0008	0.0007	0.0009	0.0007	0.0003	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	0.0004	0.0006	0.0008	0.0007	0.0009	0.0007	0.0003	<LOD	<LOD	<LOD	<LOD	<LOD
S(PFP-OH)-8007	0-2	<LOD	<LOD	<LOD	0.0003	0.0003	0.0003	0.0003	0.0004	0.0002	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

Analyte	Soil Depth (in)	Targeted days after last treatment (actual DALT shown below)												
		T1	-T2	T2	3	5	10	20	50	90	180	270	360	450
		-7 DALT	-1 DALT	0 DALT	3 DALT	5 DALT	10 DALT	20 DALT	49 DALT	94 DALT	177 DALT	267 DALT	365 DALT	455 DALT
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	0.0003	0.0003	0.0003	0.0003	0.0004	0.0002	<LOD	<LOD	<LOD	<LOD
DC-DM-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
DC-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

Table 66 Mean Residues of broflanilide and metabolites (mg/kg) in treated soil samples – Florida site

Analyte	Soil Depth (in)	Targeted days after last treatment (actual DALT shown below)												
		T1	-T2	T2	3	5	10	20	50	90	180	270	360	450
		-7 DALT	-1 DALT	0 DALT	3 DALT	5 DALT	10 DALT	20 DALT	52 DALT	91 DALT	180 DALT	270 DALT	360 DALT	448 DALT
Broflanilide	0-2	0.0406	0.0149	0.0500	0.0325	0.0275	0.0179	0.0142	0.0134	0.0052	0.0044	0.0038	0.0038	0.0018
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	0.0007	0.0003	0.0010
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	0.0406	0.0149	0.0500	0.0325	0.0275	0.0179	0.0142	0.0134	0.0052	0.0044	0.0038	0.0038	0.0018
DM-8007	0-2	<LOD	0.0007	0.0006	0.0013	0.001	0.0007	0.0003	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	0.0007	0.0006	0.0013	0.0010	0.0007	0.0003	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
S(PFP-OH)-8007	0-2	<LOD	0.0004	0.0002	0.0007	0.0007	0.0008	0.0006	0.0005	0.0002	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	0.0003	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	0.0004	<LOD	0.0007	0.0007	0.0008	0.0006	0.0005	0.0002	<LOD	<LOD	<LOD	<LOD
DC-DM-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
DC-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

Table 67 Mean Residues of broflanilide and metabolites (mg/kg) in treated soil samples – California site

Analyte	Soil Depth (in)	Targeted days after last treatment (actual DALT shown below)												
		T1	-T2	T2	3	5	10	20	50	90	180	270	360	450
		-7 DALT	-1 DALT	0 DALT	3 DALT	5 DALT	10 DALT	20 DALT	49 DALT	94 DALT	188 DALT	271 DALT	360 DALT	451 DALT
Broflanilide	0-2	0.0253	0.0163	0.0440	0.0297	0.0278	0.0279	0.025	0.0126	0.0088	0.0047	0.0080	0.0023	0.0033
	2-4	0.0014	<LOD	0.0031	0.0027	0.0026	0.0010	0.0012	0.0012	0.0003	<LOD	<LOD	0.0003	<LOD
	4-6	0.0004	<LOD	0.0014	0.0014	0.0020	0.0006	0.0003	0.0009	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	12-18	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

Analyte	Soil Depth (in)	Targeted days after last treatment (actual DALT shown below)											
		T1	-T2	T2	3	5	10	20	50	90	180	270	360	450
		-7 DALT	-1 DALT	0 DALT	3 DALT	5 DALT	10 DALT	20 DALT	49 DALT	94 DALT	188 DALT	271 DALT	360 DALT	451 DALT
	MAX	0.0253	0.0163	0.0440	0.0297	0.0278	0.0279	0.0250	0.0126	0.0088	0.0047	0.0080	0.0023	0.0033
DM-8007	0-2	<LOD	0.0007	0.0005	0.0011	0.0011	0.0016	0.0022	0.0010	0.0006	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	12-18	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	0.0007	0.0005	0.0011	0.0011	0.0016	0.0022	0.0010	0.0006	<LOD	<LOD	<LOD	<LOD
S(PFP-OH)-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	0.0004	0.0003	0.0003	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	12-18	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	0.0004	0.0003	0.0003	<LOD	<LOD	<LOD
DC-DM-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	12-18	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
DC-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	12-18	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

Table 68 Mean Residues of broflanilide and metabolites (mg/kg) in treated soil samples – Washington site

Analyte	Soil Depth (in)	Targeted days after last treatment (actual DALT shown below)										
		T1	3	5	10	20	50	90	180	270	360	450
		0 DALT	3 DALT	5 DALT	10 DALT	20 DALT	49 DALT	90 DALT	186 DALT	270 DALT	361 DALT	451 DALT
Broflanilide	0-2	0.0775	0.0501	0.0485	0.0204	0.0116	0.0123	0.0065	0.0017	0.0009	0.0004	0.0002
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	0.0775	0.0501	0.0485	0.0204	0.0116	0.0123	0.0065	0.0017	0.0009	0.0004	0.0002
DM-8007	0-2	<LOD	0.0014	0.0024	0.0015	0.0013	0.0004	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	0.0014	0.0024	0.0015	0.0013	0.0004	<LOD	<LOD	<LOD	<LOD	<LOD
S(PFP-OH)-8007	0-2	<LOD	0.0004	0.0004	0.0003	0.0003	0.0005	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	0.0002	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	0.0004	0.0004	0.0003	0.0003	0.0005	0.0002	<LOD	<LOD	<LOD	<LOD
DC-DM-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
DC-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

Analyte	Soil Depth (in)	Targeted days after last treatment (actual DALT shown below)										
		T1	3	5	10	20	50	90	180	270	360	450
		0 DALT	3 DALT	5 DALT	10 DALT	20 DALT	49 DALT	90 DALT	186 DALT	270 DALT	361 DALT	451 DALT
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

Table 69 Mean Residues of broflanilide and metabolites (mg/kg) in treated soil samples – North Dakota site

Analyte	Soil Depth (in)	Targeted days after last treatment (actual DALT shown below)										
		T1	3	5	10	20	50	90	270	360	450	540
		0 DALT	3 DALT	5 DALT	10 DALT	20 DALT	49 DALT	90 DALT	287 DALT	364 DALT	452 DALT	521 DALT
Broflanilide	0-2	0.1219	0.0680	0.0458	0.0502	0.0432	0.0511	0.0431	0.0186	0.0090	0.0045	0.0069
	2-4	0.0006	<LOD	<LOD	<LOD	<LOD	0.0003	0.0013	0.0009	0.0003	0.0005	0.0009
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	12-18	NS	NA	NA	NA	NA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	0.1219	0.0680	0.0458	0.0502	0.0432	0.0511	0.0431	0.0186	0.0090	0.0045	0.0069
DM-8007	0-2	<LOD	0.0007	0.0009	0.0013	0.0013	0.0016	0.0017	0.0004	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	12-18	NS	NA	NA	NA	NA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	0.0007	0.0009	0.0013	0.0013	0.0016	0.0017	0.0004	<LOD	<LOD	<LOD
S(PFP-OH)-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	12-18	NS	NA	NA	NA	NA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
DC-DM-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	12-18	NS	NA	NA	NA	NA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
DC-8007	0-2	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	2-4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	4-6	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	6-12	NS	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	12-18	NS	NA	NA	NA	NA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
	MAX	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

Degradation endpoints were derived from the best-fit kinetic models for broflanilide and metabolite DM-8007. The degradation results are given in Table 70.

Table 70 Dissipation DT_{50} and DT_{90} values for broflanilide and metabolite DM-8007

Site	Soil Profile	Analyte	Model	DT_{50} (d)	DT_{90} (d)
NC soil	0-42 inches	Broflanilide	DFOP	4.3	88.5
		DM-8007	SFO	21.4	71.0
FL soil	0-42 inches	Broflanilide	FOMC	6.0	189.5
		DM-8007	FOMC	7.8	43.5
CA soil	0-42 inches	Broflanilide	FOMC	18.2	390.4
		DM-8007	SFO	37.7	125.1
WA soil	0-42 Inches	Broflanilide	DFOP	5.6	62.3

Site	Soil Profile	Analyte	Model	DT_{50} (d)	DT_{90} (d)
		DM-8007	SFO	16.8	55.7
ND soil	0-42 inches	Broflanilide	DFOP	3.3	392.3
		DM-8007	SFO	91.3	303.3

Soil photolysis

The soil surface photolytic behaviour of broflanilide was investigated in a non-sterile silt loam soil using [A-ring-U-^{14}C]-, [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide at a nominal application rate of 4.7 mg/kg (dry weight), equivalent to 75 g ai/ha. (Ponte, 2017 BROFLAN_030). Soil characteristics are shown in Table 71.

Table 71 Soil characteristics in the United States (BROFLAN_030)

Soil Name	Texture	Soil Origin	Organic matter [percent]	Cation exchange capacity [meq/100g]	pH
IL soil	Silt loam	Carlyle, Illinois,	3.4	11.8	5.9

Soil samples (~2mm thick) were prepared on quartz/Pyrex dishes and subjected to continuous irradiation for 14 days (equivalent to ~38 US solar days or ~32 OECD solar days) at 20 2.6 C using a xenon irradiation source with filters to eliminate infrared light and wavelengths of 290 nm. Dark control samples were prepared in parallel. Volatile organics and CO2 were trapped with ethylene glycol and 10 percent aqueous NaOH solution, respectively. Soil samples were taken at 0, 16h, 24h or 40h, 3, 6, 9 and 14 days.

The soil samples were extracted once with acetonitrile/water (7+3), followed by two times with acetonitrile/0.5 mol/L aqueous HCl (7+3). Extracts were combined and analysed by LSC and HPLC. Selected samples were analysed by TLC to confirm results. The soil remaining after extraction was combusted followed by LSC.

The percentage recovery of the applied radioactivity in moist soil is presented in Tables 72 to 74. Only minor degradation of parent broflanilide was observed from 95–101 percent to 91–95 percent over the irradiation time, while dark controls at 14 days were within the same range at 95 percent. Metabolite DM-8007 occurred only at minor amounts at up to 4.2 percent in C-ring labelled broflanilide, before declining to 2.6 percent at day 14. Unextracted radioactivity increased slightly from 0.8-1.0 percent to 1.5–2.4 percent at day 14.

Table 72 Phototransformation of [A-ring-U-^{14}C]-labelled broflanilide, expressed as percentage (mean of duplicate) of applied radioactivity, on moist irradiated soil samples

Degradate	Incubation period							Dark controls
	0 hours	16 hours	24 hours	3 days	6 days	9 days	14 days	
Broflanilide	100.7	92.2	94.6	97.8	95.5	93.6	90.9	95.0 (14d)
DM-8007	0.0	0.0	0.0	0.0	0.0	0.0	2.3	0.0
Total extracted radioactivity	100.7	92.2	94.6	97.8	95.5	93.6	93.3	95.0-100.7
Volatiles organics	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0
CO_2	NA	0.0	0.0	0.0	0.3	0.5	1.0	0.0
Foam plug	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Unextracted	0.8	0.6	0.9	1.0	1.9	2.1	2.4	0.8-1.3

Degradate	Incubation period							Dark controls
	0 hours	16 hours	24 hours	3 days	6 days	9 days	14 days	
Total	101.5	92.8	95.5	98.8	97.6	96.1	96.6	96.1-101.5

Table 73 Phototransformation of [B-ring-U-^{14}C]-labelled broflanilide, expressed as percentage (mean of duplicate) of applied radioactivity, on moist irradiated soil samples

Degradate	Incubation period							Dark controls
	0 hours	16 hours	24 hours	3 days	6 days	9 days	14 days	
Broflanilide	95.2	102.6	96.2	101.0	95.9	92.6	94.8	95.5 (14d)
Total extracted radioactivity	95.2	102.6	96.2	101.0	95.9	92.6	94.8	92.8-101.7
Volatiles organics	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0
CO_2	NA	0.0	0.0	0.0	0.0	0.1	0.3	0.0
Foam plug	NA	0.0	0.0	0.0	0.0	0.0	0.1	0.0
Unextracted	0.8	0.8	0.9	1.1	1.1	1.8	1.5	0.7-1.0
Total	96.0	103.3	97.0	102.1	97.0	94.4	96.6	93.8-102.7

Table 74 Phototransformation of [C-ring-U-^{14}C]-labelled broflanilide, expressed as percentage (mean of duplicate) of applied radioactivity, on moist irradiated soil samples

Degradate	Incubation period							Dark controls
	0 hours	16 hours	24 hours	3 days	6 days	9 days	14 days	
Broflanilide	95.6	94.4	96.6	94.6	91.1	94.4	90.5	95.6 (14d)
DM-8007	0.0	2.2	2.1	2.4	4.2	2.5	2.6	0.0
Total extracted radioactivity	95.6	96.5	98.7	97.0	95.3	96.8	93.1	95.6-97.9
Volatiles organics	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0
CO_2	NA	0.0	0.0	0.1	0.3	0.6	1.0	0.0-0.5
Foam plug	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Unextracted	1.0	0.9	1.2	1.3	1.5	1.5	2.1	1.0-1.5
Total	96.6	97.4	99.9	98.3	97.1	98.9	96.2	96.6-98.9

Based on the decline rate observed for all three labels, a half-life time (DT_{50}) of 3466 hours under continuous artificial light was estimated (single 1st order kinetics) for broflanilide, equivalent to 389 US solar days, or 347 OECD solar days.

Confined rotational crops

A confined rotational crop study in the United States of America was conducted with [A-ring-U-^{14}C]- and [B-ring-U-^{14}C]-labelled broflanilide, each applied at a rate of 0.15 kg ai/ha to a sandy loam soil (Fleischmann, 2017, BROFLAN_031). After plant-back intervals (PBIs) of 30, 120 and 270 days, the nature and level of radioactive residues were investigated in lettuce (variety Salad Bowl), radish (variety Crimson Giant) and wheat (variety Blanca Royale).

Soil cores were collected from plots on same day of planting crops 30 days, 120 days and 270 days after treatment, as well as at the final harvest and segmented. Crops planted at the 30, 120 and 270 days after treatment intervals were harvested at different stages of maturity. Lettuce was sampled at immature and mature stages of growth. Radish was sampled at maturity dividing the plant into root

(tuber) and foliage. Wheat plants were harvested at growth stages of forage, hay and at maturity where wheat was divided into straw and grain samples.

Liquid samples such as extracts were directly measured by LSC, while samples not amenable to direct radioanalysis were combusted prior to the determination of total radioactivity by LSC. Crop matrices with a TRR ≥ 0.01 mg/kg determined by combustion analysis were extracted two times with acetonitrile/water (8:2), followed by one time with acetonitrile. Representative portions of combined extracts were concentrated to remove acetonitrile, before partitioned against ethyl acetate. Various HPLC methods, TLC and LC-MS were applied for the characterization and identification of the radioactivity. To identify conjugates possibly present in the aqueous extract of B-ring 30 DAT straw samples were hydrolysed by either β-glucosidase, 1 mol/L KOH or 1 mol/L HCl.

At all planting intervals, the majority of the applied radioactivity remained in the 0–6 cm soil horizon. For 30 DAT, radioactive residues in the 0-6 cm soil horizon ranged between 0.043-0.121 mg eq/kg and increased to 0.099–0.139 mg eq/kg at 120 DAT and 0.145–0.158 mg eq/kg at 239 DAT, before declining to 0.052–0.083 mg eq/kg at 270 DAT and 0.049–0.050 mg eq/kg at 338 DAT (Table 75).

Table 75 TRR (mg eq/kg) of Soil Treated with [^{14}C]-Broflanilide

Soil samples	B-label		A-label	
	TRR (mg eq/kg)	TRR (mg eq/kg)	TRR (mg eq/kg)	TRR (mg eq/kg)
30 DAT				
Segment (cm)	Plot 3 (Radish and lettuce)	Plot 4 (Wheat)	Plot 5 (Radish and lettuce)	Plot 6 (Wheat)
0-6	0.043	0.061	0.121	0.073
6-12	0.009	0.009	0.009	0.003
12-18	0.010	0.023	≤0.001	≤0.001
120 DAT				
Segment (cm)	Plot 7 (Radish and lettuce)	Plot 8 (Wheat)	Plot 9 (Radish and lettuce)	Plot 10 (Wheat)
0-6	0.099	0.106	0.133	0.139
6-12	0.005	0.005	0.005	0.008
12-18	≤0.001	0.009	0.003	0.010
239 DAT (Termination of 120 day PBI)				
Segment (cm)	Plot 7/8		Plot 9/10	
0-6	0.145		0.158	
6-12	0.023		0.018	
12-18	0.011		0.005	
270 DAT				
Segment (cm)	Plot 3 (Radish and lettuce)	Plot 4 (Wheat)	Plot 5 (Radish and lettuce)	Plot 6 (Wheat)
0-6	0.070	0.052	0.083	0.059
6-12	0.009	0.011	0.006	0.006
12-18	0.003	0.004	0.002	0.003
338 (Termination of 270 day PBI)				
Segment (cm)	Plot 3/4		Plot 5/6	
0-6	0.049		0.050	
6-12	0.009		0.014	
12-18	0.019		0.002	

Radioactivity for both labels in all matrices was comparable, with consistently higher levels found for the B-ring label. TRR levels were highest in wheat hay and straw for all PBIs, with the tendency to be higher at later PBIs. A summary of all TRRs found is presented in Table 76. Samples containing total radioactive residues >0.01 mg eq/kg were further investigated by solvent extraction and chromatography.

Table 76 Total radioactive residues in rotational crops after application of [A-ring-U-^{14}C]- and [B-ring-U-^{14}C]-labelled broflanilide to bare soil at 0.15 kg ai/ha

Crop parts	A-label	B-label
	TRR (mg eq/kg)	TRR (mg eq/kg)
	30 DAT	
Lettuce (immature)	0.002	0.007
Lettuce (mature)	0.005	0.008
Radish (top)	0.003	0.006
Radish (root)	0.002	0.002
Wheat (forage)	0.003	0.006
Wheat (hay)	0.014	0.030
Wheat (straw)	0.026	0.052
Wheat (grain)	0.004	0.007
	120 DAT	
Lettuce (immature)	0.008	0.013
Lettuce (mature)	0.009	0.020
Radish (top)	0.006	0.008
Radish (root)	0.003	0.006
Wheat (forage)	0.004	0.016
Wheat (hay)	0.016	0.045
Wheat (straw)	0.022	0.038
Wheat (grain)	0.005	0.004
Planting Interval	270 DAT	
Lettuce (immature)	0.012	0.016
Lettuce (mature)	0.002	0.011
Radish (top)	0.015	0.014
Radish (root)	0.003	0.003
Wheat (forage)	0.009	0.016
Wheat (hay)	0.029	0.067
Wheat (straw)	0.028	0.075
Wheat (grain)	0.009	0.013

The radioactivity found in the fractions from the acetonitrile/water extractions, including partitioning behaviour, as well as in the unextracted remainder is presented in Table 77. Extractabilities ranged between 71–92 percent in radish foliage, 77–94 percent TRR in lettuce (immature and mature), 88–92 percent in wheat forage, 36–70 percent TRR in wheat hay, 59–77 percent TRR in straw and 22–85 percent TRR in grain. The radioactivity remaining in the PES was highest at 0.025 mg eq/kg for wheat straw from the treatment with B-labelled broflanilide. No additional characterization of the PES fractions was performed.

Table 77 Extractability of radioactive residues from rotational crops after application of [A-ring-U-^{14}C]- and [B-ring-U-^{14}C]-labelled broflanilide to bare soil at 0.15 kg ai/ha

Crop parts	Distribution of radioactive residues									
	TRR (mg eq.kg)		ACN/H_2O		Organic Partition		Aqueous Partition		PES	
	[1]	[2]	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
A-Label										
Planting Interval: 30 DAT										
Wheat, hay	0.014	0.014	35.7	0.005	32.6	0.005	3.1	< 0.001	64.3	0.009
Wheat, straw	0.026	0.027	70.4	0.019	66.5	0.018	3.9	0.001	29.6	0.008
Planting Interval: 120 DAT										
Wheat, hay	0.016	0.016	37.6	0.006	25.2	0.004	12.4	0.002	62.5	0.010
Wheat, straw	0.022	0.024	66.7	0.016	55.4	0.013	11.3	0.003	33.3	0.008
Planting Interval: 270 DAT										
Radish, foliage	0.015	0.014	71.4	0.010	58.1	0.008	13.3	0.002	28.6	0.004
Lettuce, immature	0.012	0.013	76.9	0.010	60.5	0.008	16.4	0.002	23.1	0.003
Wheat, hay	0.029	0.028	46.4	0.013	39.0	0.011	7.4	0.002	53.6	0.015
Wheat, straw	0.028	0.029	58.6	0.017	47.8	0.014	10.8	0.003	41.4	0.012
Wheat, grain	0.009	0.009	22.2	0.002	15.8	0.001	6.4	0.001	77.8	0.007
B-Label										
Planting Interval: 30 DAT										
Wheat, hay	0.030	0.029	62.1	0.018	46.2	0.013	15.9	0.005	37.9	0.011
Wheat, straw	0.052	0.055	74.5	0.041	70.5	0.039	4.0	0.002	25.5	0.014
Planting Interval: 120 DAT										
Lettuce, immature	0.013	0.011	90.9	0.010	83.7	0.009	7.2	0.001	9.1	0.001
Lettuce, mature	0.020	0.018	94.4	0.017	78.2	0.014	16.2	0.003	5.6	0.001
Wheat, forage	0.016	0.013	92.3	0.012	29.6	0.004	62.6	0.008	7.7	0.001
Wheat, hay	0.045	0.046	69.6	0.032	59.1	0.027	10.5	0.005	30.4	0.014
Wheat, straw	0.038	0.039	76.9	0.030	56.4	0.022	20.5	0.008	23.1	0.009
Planting Interval: 270 DAT										
Radish, foliage	0.014	0.012	91.6	0.011	69.0	0.008	22.6	0.003	8.3	0.001
Lettuce, immature	0.016	0.014	92.9	0.013	63.4	0.009	29.5	0.004	7.1	0.001
Lettuce, mature	0.011	0.012	91.7	0.011	73.0	0.009	18.7	0.002	8.3	0.001
Wheat, forage	0.016	0.017	88.2	0.015	61.6	0.010	26.6	0.005	11.8	0.002
Wheat, hay	0.067	0.067	65.7	0.044	57.4	0.038	8.3	0.006	34.3	0.023
Wheat, straw	0.075	0.079	68.4	0.054	59.2	0.047	9.2	0.007	31.6	0.025
Wheat, grain	0.013	0.013	84.6	0.011	63.7	0.008	20.9	0.003	15.4	0.002

Notes:

[1] TRR determined by combustion/LSC.

[2] TRR determined by calculation (ACN/H_2O (=organic partition + aqueous partition) + PES).

For the 30 day PBI, the results of the identification and characterization of radioactive residues are presented in Table 78. Residues >0.01 mg eq/kg were found in wheat hay and straw only. Parent broflanilide was identified as a minor component ranging between 4.3–10.2 percent TRR (0.001–

0.003 mg eq/kg). Identified metabolites for both labels were DM-8007, ranging between ND–3.7 percent TRR (ND-0.002 mg eq/kg). For the B-label only, additional identified metabolites were B-urea, ranging between 9.7–13.9 percent TRR (0.004–0.005 mg eq/kg) and B-oxam-acid, ranging between 12.5–20 percent TRR (0.004–0.011 mg eq/kg). Other unknown metabolites detected for the B-label were Uk3B-org at up to 12.5 percent TRR (0.007 mg eq/kg) and for the A-label Uk3A-org up to 31.5 percent TRR (0.009 mg eq/kg).

Table 78 Summary of identified/characterized components in wheat hay and straw from rotational crops after application of [A-ring-U-^{14}C]- and [B-ring-U-^{14}C]-labelled broflanilide to bare soil at 0.15 kg ai/ha and a PBI of 30 days

Matrix	Wheat hay		Wheat straw	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg
B-label				
TRR [1]		0.030		0.052
TRR [2]	100.0	0.029	100.0	0.055
Combined extracts	62.1	0.018	74.5	0.041
Organic partition	46.2	0.013	70.5	0.039
Broflanilide	5.5	0.002	4.3	0.002
DM-8007	ND	ND	3.6	0.002
Uk3B-org	8.8	0.002	12.5	0.007
Uk5B	ND	ND	1.8	0.001
Uk6B	ND	ND	2.1	0.001
Uk7B	ND	ND	4.7	0.003
Uk9B	ND	ND	2.1	0.001
Uk10B	ND	ND	3.9	0.002
B-urea	13.9	0.004	9.7	0.005
Uk14B	ND	ND	2.0	0.001
B-oxam-acid	12.5	0.004	20.0	0.011
Uk20B	ND	ND	1.6	0.001
Aqueous partition	15.9	0.005	4.0	0.002
Sum identified	31.9	0.010	37.6	0.020
Sum characterized	8.8	0.002	30.7	0.017
Unextracted	37.9	0.011	25.5	0.014
A-label				
TRR [1]		0.014		0.026
TRR [2]	100	0.014	100	0.027
Combined extracts	35.7	0.005	70.4	0.019
Organic partition	32.6	0.005	66.5	0.018
Broflanilide	9.1	0.001	10.2	0.003
DM-8007	1.5	≤0.001	3.7	0.001
Uk1A	ND	ND	2.3	0.001
Uk3A-org	8.5	0.001	31.5	0.009
Uk6A	1.3	≤0.001	ND	ND
Uk8A	1.3	≤0.001	ND	ND
Uk12A	1.9	≤0.001	ND	ND
Uk14A	6.2	0.001	ND	ND
Uk16A	ND	ND	5.2	0.001
Aqueous partition	3.1	< 0.001	3.9	0.001

Matrix	Wheat hay		Wheat straw	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg
Sum identified	10.6	0.001	13.9	0.004
Sum characterized	19.2	0.002	39.0	0.011
Unextracted	64.3	0.009	29.6	0.008

Notes:

[1)] TRR determined by combustion/LSC.

[2)] Sum of combined extracts + PES.

For the 120 day PBI, the results of the identification and characterization of radioactive residues are presented in Table 79. Residues > 0.01 mg eq/kg were found in lettuce (immature and mature), as well as for wheat forage, hay and straw. Parent broflanilide was identified in varying relative amounts, ranging between ND–45.7 percent TRR, while absolute amounts were consistently < 0.01 mg eq/kg (ND–0.008 mg eq/kg). For the B-label only, additional identified metabolites were B-urea in immature lettuce and wheat matrices, ranging between 11.1–26.6 percent TRR (0.001–0.010 mg eq/kg) and B-oxam-acid in wheat matrices, ranging between 5.4–13.4 percent TRR (0.001–0.006 mg eq/kg). Other unknown metabolites detected for the B-label were Uk3B-org up to 13.8 percent TRR (0.001 mg eq/kg) in immature lettuce and 8.2 percent TRR (0.004 mg eq/kg) in wheat hay as well as for the A-label Uk13A up to 26.8 percent TRR (0.006 mg eq/kg) in wheat straw.

Table 79 Summary of identified/characterized components in lettuce and wheat matrices from rotational crops after application of [A-ring-U-^{14}C]- and [B-ring-U-^{14}C]-labelled broflanilide to bare soil at 0.15 kg ai/ha and a PBI of 120 days

	Lettuce (immature)		Lettuce (mature)		Wheat forage		Wheat hay		Wheat straw	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	% TRR	mg eq/kg	% TRR
B-label										
TRR [1]		0.013		0.020		0.016		0.045		0.038
TRR [2]	100	0.011	100	0.018	100	0.013	100	0.046	100	0.039
Combined extracts	90.9	0.010	94.4	0.017	92.3	0.012	69.6	0.032	76.9	0.030
Organic partition	83.7	0.009	78.2	0.014	29.6	0.004	59.1	0.027	56.4	0.022
Broflanilide	19.6	0.002	45.7	0.008	4.4	0.001	4.7	0.002	ND	ND
DM-8007	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Uk3B-org	13.8	0.001	ND	ND	ND	ND	8.2	0.004	ND	ND
Uk7B	ND	ND	ND	ND	ND	ND	ND	ND	5.7	0.002
B-urea	26.6	0.003	ND	ND	11.1	0.001	22.5	0.010	20.8	0.008
B-oxam-acid	ND	ND	ND	ND	5.4	0.001	13.4	0.006	11.9	0.005
Uk43B	ND	ND	ND	ND	ND	ND	ND	ND	3.7	0.001
Aqueous partition	7.2	0.001	16.2	0.003	62.6	0.008	10.5	0.005	20.5	0.008
Sum identified	46.2	0.005	45.7	0.008	20.9	0.003	40.6	0.018	32.7	0.013
Sum characterized	13.8	0.001	0.0	0.000	0.0	0.000	8.2	0.004	9.4	0.003
Unextracted	9.1	0.001	5.6	0.001	7.7	0.001	30.4	0.014	23.1	0.009
A-label										
TRR [1]	-	-	-	-	-	-		0.016		0.022
TRR [2]	-	-	-	-	-	-	100	0.016	100	0.024

	Lettuce (immature)		Lettuce (mature)		Wheat forage		Wheat hay		Wheat straw	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	% TRR	mg eq/kg	% TRR
Combined extracts	-	-	-	-	-	-	37.6	0.006	66.7	0.016
Organic partition	-	-	-	-	-	-	25.2	0.004	55.4	0.013
Broflanilide	-	-	-	-	-	-	4.1	0.001	16.7	0.004
Uk13A	-	-	-	-	-	-	4.2	0.001	26.8	0.006
Uk17A	-	-	-	-	-	-	15.4	0.002	ND	ND
Aqueous partition	-	-	-	-	-	-	12.4	0.002	11.3	0.003
Sum identified	-	-	-	-	-	-	4.1	0.001	16.7	0.004
Sum characterized	-	-	-	-	-	-	19.6	0.003	26.8	0.006
Unextracted	-	-	-	-	-	-	62.5	0.010	33.3	0.008

Notes:

[1)] TRR determined by combustion/LSC.

[2)] Sum of combined extracts + PES.

For the 270 day PBI, the results of the identification and characterization of radioactive residues are presented in Table 80 and Table 81. Residues > 0.01 mg eq/kg were found in lettuce (immature and mature) and radish leaves, as well as for wheat forage, hay, straw and grain. Parent broflanilide was identified in varying relative amounts, ranging between ND–46.6 percent TRR, while absolute amounts were consistently < 0.01 mg eq/kg (ND–0.006 mg eq/kg). For the B-label only, additional identified metabolites were DM-8007 in radish leaves and wheat straw at 1.8–2.6 percent TRR (< 0.001–0.001 mg eq /kg), B-urea in all matrices (except wheat grain) at 11.8–35.6 percent TRR (0.004–0.010 mg eq/kg) and B-oxam-acid in radish leaves and wheat matrices (except grain), ranging between 11.3–35.6 percent TRR (0.002–0.024 mg eq/kg). Other unknown metabolites detected for the B-label were Uk10B up to 43.9 percent TRR (0.006 mg eq/kg) in wheat grain and for the A-label Uk13A up to 19.4 percent TRR (0.005 mg eq/kg) in wheat hay.

Table 80 Summary of identified/characterized components in lettuce and radish leaves from rotational crops after application of [A-ring-U-^{14}C]- and [B-ring-U-^{14}C]-labelled broflanilide to bare soil at 0.15 kg ai/ha and a PBI of 270 days

	Lettuce (immature)		Lettuce (mature)		Radish leaves	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	% TRR
B-label						
TRR [1]		0.016		0.011		0.014
TRR [2]	100.0	0.014	100.0	0.012	100.0	0.012
Combined extracts	92.9	0.013	91.7	0.011	91.6	0.011
Organic partition	63.4	0.009	73.0	0.009	69.0	0.008
Broflanilide	20.8	0.003	22.5	0.003	2.8	≤0.001
DM-8007	ND	ND	ND	ND	2.6	≤0.001
Uk1B	ND	ND	ND	ND	2.5	≤0.001
Uk3B-org	ND	ND	ND	ND	7.3	0.001
Uk6B	ND	ND	ND	ND	3.6	≤0.001
Uk9B	ND	ND	ND	ND	3.5	≤0.001
B-urea	34.5	0.005	35.6	0.004	31.9	0.004

	Lettuce (immature)		Lettuce (mature)		Radish leaves	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	% TRR
B-oxam-acid	ND	ND	ND	ND	14.9	0.002
Aqueous partition	29.5	0.004	18.7	0.002	22.6	0.003
Sum identified	55.3	0.008	58.1	0.007	52.2	0.006
Sum characterized	0.0	0.000	0.0	0.000	16.9	0.001
Unextracted	7.1	0.001	8.3	0.001	8.3	0.001
A-label						
TRR [1]		0.012	-	-		0.015
TRR [2]	100.0	0.013	-	-	100.0	0.014
Combined extracts	76.9	0.010	-	-	71.4	0.010
Organic partition	60.5	0.008	-	-	58.1	0.008
Broflanilide	46.6	0.006	-	-	18.1	0.002
Uk3A-org	ND	ND	-	-	ND	ND
Uk5A	ND	ND	-	-	ND	ND
Uk9A	ND	ND	-	-	5.3	0.001
Uk13A	ND	ND	-	-	ND	ND
Uk14A	8.3	0.001	-	-	ND	ND
Uk16A	ND	ND	-	-	ND	ND
Uk17A	ND	ND	-	-	6.4	0.001
Uk29A	5.6	0.001	-	-	ND	ND
Uk37A	ND	ND	-	-	5.9	0.001
Uk57A	ND	ND	-	-	ND	ND
Aqueous partition	16.4	0.002	-	-	13.3	0.002
Sum identified	46.6	0.006	-	-	18.1	0.002
Sum characterized	13.9	0.002	-	-	17.6	0.003
Unextracted	23.1	0.003	-	-	28.6	0.004

Notes:

[1)] TRR determined by combustion/LSC.

[2)] Sum of combined extracts + PES.

Table 81 Summary of identified/characterized components in wheat matrices from rotational crops after application of [A-ring-U-^{14}C]- and [B-ring-U-^{14}C]-labelled broflanilide to bare soil at 0.15 kg ai/ha and a PBI of 270 days.

	Wheat forage		Wheat hay		Wheat straw		Wheat grain	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	% TRR
B-label								
TRR [1]		0.016		0.067		0.075		0.013
TRR [2]	100.0	0.017	100.0	0.067	100.0	0.079	100.0	0.013
Combined extracts	88.2	0.015	65.7	0.044	68.4	0.054	84.6	0.011
Organic partition	61.6	0.010	57.4	0.038	59.2	0.047	63.7	0.008
Broflanilide	11.6	0.002	ND	ND	4.7	0.004	ND	ND
DM-8007	ND	ND	ND	ND	1.8	0.001	ND	ND
Uk3B-org	ND	ND	ND	ND	ND	ND	ND	ND
Uk5B	ND	ND	ND	ND	2.4	0.002	ND	ND
Uk6B	ND	ND	ND	ND	2.4	0.002	ND	ND

Component	Wheat forage		Wheat hay		Wheat straw		Wheat grain	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	% TRR
Uk8B	ND	ND	ND	ND	3.0	0.002	ND	ND
Uk9B	ND	ND	ND	ND	3.6	0.003	ND	ND
Uk10B	ND	ND	ND	ND	ND	ND	43.9	0.006
Uk11B	ND	ND	ND	ND	4.7	0.004	ND	ND
Uk12B	ND	ND	ND	ND	ND	ND	12.4	0.002
B-urea	30.2	0.005	15.6	0.010	11.8	0.009	ND	ND
Uk14B	ND	ND	ND	ND	ND	ND	5.2	0.001
B-oxam-acid	11.3	0.002	35.6	0.024	12.4	0.010	ND	ND
Uk20B	ND	ND	ND	ND	2.4	0.002	ND	ND
Uk21B	ND	ND	ND	ND	1.8	0.001	ND	ND
Uk61B	ND	ND	6.3	0.004	ND	ND	ND	ND
Aqueous partition	26.6	0.005	8.3	0.006	9.2	0.007	20.9	0.003
Sum identified	53.1	0.009	51.2	0.034	30.7	0.024	0.0	0.000
Sum characterized	0.0	0.000	6.3	0.004	20.3	0.016	61.5	0.009
Unextracted	11.8	0.002	34.3	0.023	31.6	0.025	15.4	0.002
A-label								
TRR [1]				0.029		0.028		0.009
TRR [2]			100.0	0.028	100.0	0.029	100.0	0.009
Combined extracts			46.4	0.013	58.6	0.017	22.2	0.002
Organic partition			39.0	0.011	47.8	0.014	15.8	0.001
Broflanilide	-	-	ND	ND	8.5	0.002	ND	ND
Uk3A-org	-	-	6.4	0.002	ND	ND	ND	ND
Uk5A	-	-	ND	ND	ND	ND	3.6	< 0.001
Uk9A	-	-	ND	ND	ND	ND	ND	ND
Uk13A	-	-	19.4	0.005	ND	ND	8.7	0.001
Uk14A	-	-	ND	ND	14.5	0.004	ND	ND
Uk16A	-	-	7.0	0.002	ND	ND	ND	ND
Uk17A	-	-	ND	ND	ND	ND	ND	ND
Uk29A	-	-	ND	ND	ND	ND	ND	ND
Uk37A	-	-	ND	ND	ND	ND	ND	ND
Uk57A	-	-	ND	ND	ND	ND	3.6	< 0.001
Aqueous partition	-	-	7.4	0.002	10.8	0.003	6.4	0.001
Sum identified	-	-	0.0	0.000	8.5	0.002	0.0	0.000
Sum characterized	-	-	32.8	0.009	14.5	0.004	15.9	0.001
Unextracted	-	-	53.6	0.015	41.4	0.012	77.8	0.007

Notes:

1) TRR determined by combustion/LSC.

2) Sum of combined extracts + PES.

The proposed metabolic pathway of broflanilide in rotational crops is shown in Figure 6.

Field rotational crop studies

In two field rotational crop trials conducted during the 2016/17 growing seasons in the United States, broflanilide was applied once to bare soil a rate of 0.05 kg ai/ha (1x rate) (Bledsoe, 2019, BROFLAN_032). Wheat, lettuce and radish were planted 30, 60, 90 and 360 DAT and sampled at normal crop maturity.

Parent broflanilide and its metabolites S(PFP-OH)-8007 and DM-8007 were analysed by according to modified method D1417/01, while metabolites B-oxam-acid and B-urea were analysed according to method D1703/01. Homogenized plant samples were extracted after hydration (if necessary), by shaking with acetonitrile. A mixture of salts (magnesium sulfate, sodium chloride, disodium citrate sesquihydrate, and trisodium citrate dihydrate) is then added, followed by mixing with a vortexer and separation by centrifuge. For broflanilide and its metabolites S(PFP-OH)-8007 and DM-8007, an aliquot of the resulting organic layer is diluted in acetonitrile:water (50:50, with 0.1 percent formic acid). Quantification of parent and metabolites was performed by LC-MS/MS with a LOQ of 0.01 mg/kg. Mean procedural recoveries for parent broflanilide and metabolites spiked at 0.01 and 0.1 mg eq/kg to wheat, lettuce and radish matrices were mostly within 70-120 percent and a RSD of <20 percent. Mean recoveries outside this range were found for broflanilide at 0.1 mg/kg in wheat forage (126 percent) and radish top (123 percent), for S(PFP-OH)-8007 at both levels in wheat forage (129 and 125 percent) and for DM-8007 at 0.1 mg eq/kg in wheat forage (131 percent, RSD 27 percent).

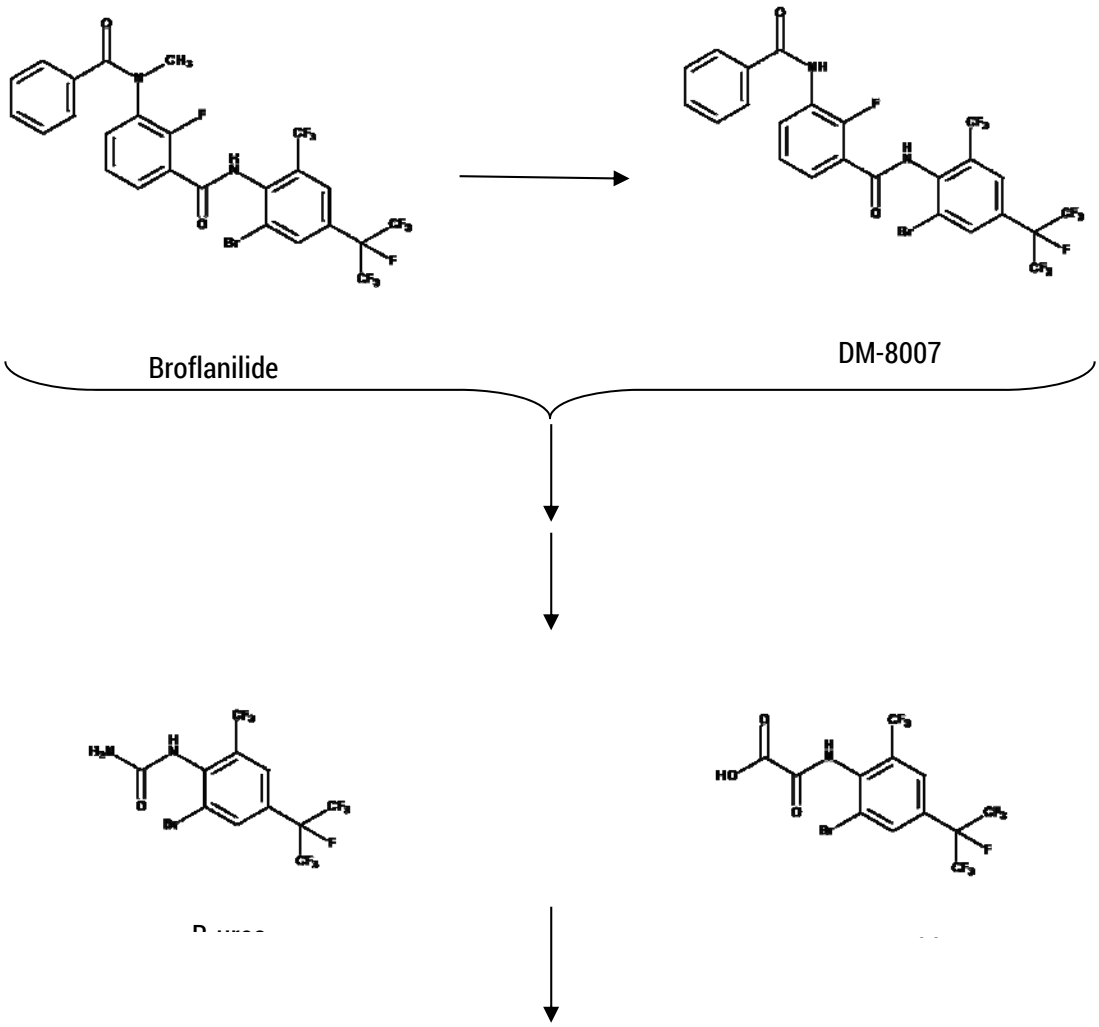


Figure 6 Proposed metabolic pathway of broflanilide in confined rotational crops

The results of the field rotational crop study are provided in Table 82. In general, residues of broflanilide were <LOQ, with the exception of parent broflanilide in lettuce planted at 30 days PBI at 0.013 mg/kg.

Table 82 Residues in rotational crops grown in broflanilide-treated soil in the United States in 2016/2017

Trial ID (Location)	Crop/variety	Commodity	Rate, kg ai./ha	Actual PBI (days)	Residues (mg/kg) [1)]				
					Broflanilide	S(PFP-OH)-8007	DM-8007	B-oxam-acid	B-urea
R160136	Wheat / Forefront HRS	Forage	0.0499	30	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
(Howard, IA)		Hay			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Grain			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Straw			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	Wheat / Expedition HRW	Forage	0.0499	60	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Hay			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Grain			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Straw			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01

Trial ID (Location)	Crop/variety	Commodity	Rate, kg ai./ha	Actual PBI (days)	Residues (mg/kg) [1)]				
					Broflanilide	S(PFP-OH)-8007	DM-8007	B-oxam-acid	B-urea
	Wheat / Expedition HRW	Forage	0.0497	91	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Hay			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Grain			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Straw			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	Wheat / Expedition HRW	Forage	0.0497	361	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Hay			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Grain			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Straw			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
R160137	Lettuce / Black Seeded Simpson	Leaves	0.0477	30	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
(Howard, IA)		Leaves	0.0486	58	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Leaves	0.0502	92	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Leaves	0.0506	361	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
R160138	Radish / Cherry Belle	Tops	0.0495	30	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
(Howard, IA)		Roots			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Tops	0.0483	58	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Roots			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Tops	0.0498	92	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Roots			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Tops	0.0511	361	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Roots			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
R160139	Wheat / AGSTAM 204	Forage	0.0499	30	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
(Yolo, CA)		Hay			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Grain			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Straw			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Forage	0.0519	60	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Hay			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Grain			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Straw			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Forage	0.0519	91	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Hay			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Grain			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Straw			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Forage	0.0499	361	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Hay			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Grain			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Straw			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
R160140	Lettuce / Cimmaron Red Romaine	Leaves	0.0499	30	0.013 [2)]	< 0.01	< 0.01	< 0.01	< 0.01
(Yolo, CA)		Leaves	0.0519	60	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	Lettuce / Buttercrunch	Leaves	0.0519	91	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Leaves	0.0499	361	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01

Trial ID (Location)	Crop/variety	Commodity	Rate, kg ai./ha	Actual PBI (days)	Residues (mg/kg) [1]				
					Broflanilide	S(PFP-OH)-8007	DM-8007	B-oxam-acid	B-urea
R160141	Radish / Crimson Giant	Tops	0.0499	30	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
(Yolo, CA)		Roots			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	Radish / Cherry Belle	Tops	0.0519	60	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Roots			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	Radish / Cherry Belle	Tops	0.0519	91	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Roots			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	Radish / Cherry Belle	Tops	0.0499	361	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		Roots			< 0.01	< 0.01	< 0.01	< 0.01	< 0.01

Notes:

[1] At each location, two treated samples (Rep. A and B) per matrix were collected at each sampling interval from the treated plot. Mean of two replicates is presented.

[2] Mean of multiple analyses of the same sample (range: < 0.01-0.018 mg/kg).

RESIDUE ANALYSIS

Analytical methods

For the analysis of broflanilide and metabolites in various plant and animal matrices, analytical methods suitable for enforcement and data generation purposes were submitted. In the following table an overview of these methods is presented.

Table 83 Overview of analytical methods for broflanilide and metabolites

Method	Matrix	Extraction	Clean-Up	Analyte, Detection, LOQ
Plant materials				
D1417/01 (according to QuEChERS) ILV available	Dry, high starch (wheat grain); dry high protein (bean seed), high water content (tomato), high acid content (citrus), high fat content (soya bean seed), difficult (coffee bean)	Acetonitrile/water (1/1) + buffer salts	Dispersive solid phase extraction with PSA	LC-MS/MS Broflanilide: m/z 663→643, 665→645 S(PFP-OH)-8007: m/z 661→641, 663→643 DM-8007: m/z 649→242, 649→629 LOQ: 0.001 mg/kg
D1703/01 (according to QuEChERS)	Dry, high starch (wheat grain); dry high protein (bean seed), high water content (lettuce), high acid content (orange), high fat content (soya bean seed)	Acetonitrile/water (1/1) + buffer salts	Dispersive solid phase extraction with PSA (optional)	LC-MS/MS B-urea: m/z 451→431, 453→433 B-oxam acid: m/z 478→406, 480→408 LOQ: 0.01 mg/kg
Japanese crop residue method	High water content (radish root and leaves, turnip root and leaves, leek)	Acetonitrile/water (80/20)	Solid phase extraction on C18	LC-MS/MS Broflanilide: m/z 665→645 S(PFP-OH)-8007: m/z 661→641 DM-8007: m/z 649→242 LOQ: 0.01 mg/kg
Korean crop residue method	High water content (cabbage, Chinese cabbage, radish root and leaves, green onion,	Acetonitrile	Partitioned with saturated saline solution, water and dichloromethane;	GC-ECD or HPLC-UV LOQ: 0.02-0.1 mg/kg LC-MS/MS

Method	Matrix	Extraction	Clean-Up	Analyte, Detection, LOQ
	tomato)		solid phase extraction on NH_2	S(PFP-OH)-8007: m/z 661→621, 661→641 LOQ: 0.02 mg/kg
Animal materials				
D1604/01 ILV available	Milk, egg, liver, kidney, muscle, fat	Fat: (1) acetone/hexane (20/80), (2) acetone All others: (1) acetonitrile, (2) acetonitrile/water (80:20)	Liver, kidney, muscle: partitioning with buffer salt solution, dispersive solid phase extraction with PSA	LC-MS/MS Broflanilide: m/z 663→643, 665→645 DC-DM-8007: m/z 545→525, 547→527 DM-8007: m/z 649→242, 651→242 LOQ: 0.001 mg/kg (milk); 0.01 mg/kg (all other matrices)

Plant materials

Method D1417/01 (Jose, 2017, BROFLAN_033 & Jose, 2017, BROFLAN_034)

The method is based on the citrate buffered QuEChERS method. The residues of broflanilide and its metabolites S(PFP-OH)-8007 and DM-8007 are extracted from crop matrices by shaking with acetonitrile and then in the presence of various salts ($MgSO_4$; NaCl; sodium citrate sesquihydrate and sodium citrate dehydrate). For high oil/dry matrices samples, water is previously added before extraction with acetonitrile. An aliquot of the resulting extract is treated with magnesium sulfate and PSA to remove the excess water and fatty acids, respectively. An aliquot is then diluted with 0.1 percent formic acid acetonitrile:water (50:50).

Final determination is accomplished by LC-MS/MS in positive ionization mode, using a BEH C18 column and monitoring ion transitions m/z 663→643, 665→645 for broflanilide, m/z 661→641, 663→643 for S(PFP-OH)-8007 and m/z 649→242, 649→629 for DM-8007. Quantitation was done with external standards in solvent, except dry beans where matrix matched standards were used. The results are shown in Table 84.

Table 84 Recovery data for method D1417/01 measuring broflanilide and its metabolites S(PFP-OH)-8007 and DM-8007 in various plant matrices using LC-MS/MS

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
Wheat, grain	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.001	6	87.1, 101, 103, 94.0, 96.8, 98.1	96.7	5.6	5.8
		0.10	6	100, 89.2, 87.9, 94.1, 93.8, 71.1	89.4	9.9	11
		3.0	6	93.1, 95.3, 90.2, 94.6, 94.9, 97.0	94.2	2.3	2.5
	Overall		18	Range, 71.1-103	93.4	7.0	7.5
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.001	6	99.7, 103, 95.5, 89.1, 98.4, 102	98.0	5.1	5.2
		0.10	6	96.4, 86.5, 82.7, 91.2, 99.1, 90.8	91.1	6.1	6.7
		3.0	6	94.1, 94.4, 92.1, 96.8, 90.6, 96.0	94.0	2.3	2.5
	Overall		18	Range, 82.7-103	94.4	5.3	5.6
Dry beans, seed	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.001	6	88.9, 94.4, 99.2, 92.0, 99.5, 104	96.3	5.6	5.8

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
		0.1	6	112, 108, 115, 117, 113, 108	112	3.7	3.3
	Overall		12	Range, 88.9-117	104	9.4	9.0
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.001	6	104, 93.6, 94.0, 102, 97.2, 98.0	98.1	4.2	4.3
		0.1	6	109, 94.0, 105, 113, 116, 120	110	9.2	8.4
	Overall		12	Range, 93.6-120	104	9.1	8.7
Tomato, whole fruit	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.001	6	105, 98.1, 92.7, 92.2, 113, 103	101	8.0	7.9
		0.1	5	110, 66.6[1], 112, 109, 108, 103	108	3.4	3.1
	Overall		11	Range, 92.2-113	104	7.3	7.0
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.001	6	103, 91.2, 96.8, 102, 119, 99.8	102	9.4	9.2
		0.1	5	113, 71.3[1], 113, 115, 110, 102	111	5.1	4.6
	Overall		11	Range, 80.8-119	106	8.6	8.2
Citrus fruits, whole fruit	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.001	6	113, 105, 98.8, 105, 108, 95.7	104	6.2	6.0
		0.1	6	105, 98.9, 115, 109, 109, 110	108	5.4	5.0
	Overall		12	Range, 95.7-115	106	5.9	5.5
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.001	6	113, 107, 102, 111, 98.6, 107	106	5.4	5.1
		0.1	6	111, 106, 110, 109, 118, 106	110	4.4	4.0
	Overall		12	Range, 98.6-118	108	5.1	4.7
Coffee, bean	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.001	6	88.8, 99.6, 105, 118, 102, 78.0	98.6	14	14
		0.1	6	101, 89.6, 108, 101, 100, 114	102	8.2	8.1
	Overall		12	Range, 78.0-118	100	11	11
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.001	6	99.2, 90.8, 110, 112, 102, 96.8	102	8.1	7.9
		0.1	6	99.2, 96.8, 102, 105, 111, 115	105	7.0	6.7
	Overall		12	Range, 90.8-115	103	7.4	7.1
Soybeans, seed	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.001	6	77.6, 80.4, 79.2, 75.2, 77.6, 88.8	79.8	4.7	5.9
		0.1	6	111, 94.8, 84.0, 100, 104, 93.6	97.9	9.3	9.5
	Overall		12	Range, 75.2-111	88.9	12	13
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.001	6	70.8, 73.6, 70.4, 70.8, 78.8, 82.8	74.5	5.1	6.9
		0.1	6	98.8, 93.2, 100, 106, 112, 102	102	6.4	6.3
	Overall		12	Range, 70.4-120	88.3	15	17
Wheat, grain	Primary Quantitation (*m/z* 661→641)						
	S(PFP-OH)-8007	0.001	6	80.4, 102, 86.4, 86.0, 83.6, 88.8	87.9	7.5	8.5
		0.1	5	83.6, 80.8, 88.0, 88.8, 86.8, 67.6[1]	85.6	3.3	3.9
	Overall		11	Range, 80.4-102	86.6	6.1	7.0
	Confirmatory Quantitation (*m/z* 663→643)						
	S(PFP-OH)-	0.001	6	81.5, 94.6, 93.3, 90.8, 90.2, 85.9	89.4	4.9	5.5

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
	8007	0.1	5	93.8, 84.3, 85.9, 86.6, 91.2, 85.8[1]	88.4	4.0	4.5
	Overall		11	Range, 81.5 – 94.6	89.2	4.4	5.0
Dry beans, seed	Primary Quantitation (*m/z* 661→641)						
	S(PFP-OH)-8007	0.001	6	101, 103, 93.3, 89.8, 103, 101	98.5	5.6	5.7
		0.1	6	107, 96.3, 101, 101, 115, 106	104	6.5	6.2
	Overall		12	Range, 89.8-115	101	6.6	6.5
	Confirmatory Quantitation (*m/z* 663→643)						
	S(PFP-OH)-8007	0.001	6	93.6, 109, 99.1, 99.0, 108, 100	101	5.9	5.8
		0.1	6	108, 99.2, 104, 108, 116, 106	107	5.5	5.2
	Overall		12	Range, 93.6-116	104	6.2	5.9
Tomato, whole fruit	Primary Quantitation (*m/z* 661→641)						
	S(PFP-OH)-8007	0.001	6	86.8, 87.2, 86.0, 88.4, 80.8, 94.0	87.2	4.2	4.9
		0.1	5	119, 52.0[1], 102, 102, 97.6, 87.2	102	11	11
	Overall		11	Range, 80.8-119	93.7	11	12
	Confirmatory Quantitation (*m/z* 663→643)						
	S(PFP-OH)-8007	0.001	6	81.2, 92.0, 80.0, 82.0, 86.8, 88.0	85.0	4.7	5.5
		0.1	5	111, 50.0[1], 102, 89.6, 100, 83.6	97.2	11	11
	Overall		11	Range, 80.0-111	90.6	9.9	11
Citrus fruits, whole fruit	Primary Quantitation (*m/z* 661→641)						
	S(PFP-OH)-8007	0.001	6	106, 109, 103, 102, 95.3, 95.8	102	5.5	5.4
		0.1	6	97.8, 108, 104, 110, 106, 102	105	4.4	4.2
	Overall		12	Range, 95.3-110	103	4.9	4.8
	Confirmatory Quantitation (*m/z* 663→643)						
	S(PFP-OH)-8007	0.001	6	106, 92.8, 96.4, 98.4, 86.8, 101	96.9	6.6	6.9
		0.1	6	99.2, 97.6, 102, 103, 96.8, 98.4	99.5	2.5	2.5
	Overall		12	Range, 86.8-106	98.2	5.0	5.1
Coffee, bean	Primary Quantitation (*m/z* 661→641)						
	S(PFP-OH)-8007	0.001	6	98.0, 95.6, 92.8, 106, 90.8, 96.4	96.6	5.3	5.5
		0.1	6	98.8, 110, 96.4, 87.6, 100, 112	101	9.0	9.0
	Overall		12	Range, 87.6-112	98.7	6.8	6.9
	Confirmatory Quantitation (*m/z* 663→643)						
	S(PFP-OH)-8007	0.001	6	81.2, 98.8, 106, 112, 95.2, 83.6	96.1	12	13
		0.1	6	104, 105, 90.8, 108, 109, 94.8	102	7.4	7.3
	Overall		12	Range, 81.2-112	99.0	10	10
Soybeans, seed	Primary Quantitation (*m/z* 661→641)						
	S(PFP-OH)-8007	0.001	6	71.6, 73.2, 70.4, 87.2, 71.6, 74.4	74.7	6.3	8.4
		0.1	6	120, 97.6, 91.2, 105, 112, 105	105	10	9.7
	Overall		12	Range, 70.4-120	89.9	18	20
	Confirmatory Quantitation (*m/z* 663→643)						
	S(PFP-OH)-8007	0.001	6	76.0, 94.8, 79.6, 93.2, 90.4, 71.2	84.2	9.9	12
		0.1	6	93.6, 112, 99.2, 106, 112, 109	105	7.5	7.1
	Overall		12	Range, 71.2-112	94.8	14	15
Wheat, grain	Primary Quantitation (m/z 649→242)						
	DM-8007	0.001	6	88.6, 92.4, 99.2, 90.2, 100, 99.1	94.9	5.1	5.4

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
		0.1	6	94.3, 91.1, 93.3, 109, 112, 98.3	99.7	8.8	8.8
	Overall		12	Range, 88.6-112	97.3	7.3	7.5
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.001	6	101, 91.1, 115, 104, 104, 97.6	102	8.0	7.8
		0.1	6	110, 99.8, 106, 114, 113, 91.9	106	8.6	8.1
	Overall		12	Range, 91.1-114	104	8.1	7.8
Dry beans, seed	Primary Quantitation (m/z 649→242)						
	DM-8007	0.001	6	94.8, 102, 92.8, 96.4, 72.0, 71.2	88.2	13	15
		0.1	6	100, 92.4, 87.6, 100, 95.2, 98.4	95.6	4.9	5.1
	Overall		12	Range, 71.2-102	91.9	10	11
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.001	6	74.4, 93.2, 91.6, 75.6, 73.6, 52.4[4]	76.8	15	19
		0.1	6	90.8, 101, 95.2, 102, 90.8, 106	97.6	6.3	6.5
	Overall		12	Range, 52.4-106	87.2	15	18
Tomato, whole fruit	Primary Quantitation (m/z 649→242)						
	DM-8007	0.001	6	101, 90.9, 93.2, 107, 107, 101	100	6.8	6.8
		0.1	5	113, 69.3[1], 104, 109, 112, 99.2	107	5.8	5.4
	Overall		11	Range, 90.9-113	103	7.2	6.9
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.001	6	91.2, 94.4, 102, 94.0, 108, 96.4	97.7	6.2	6.4
		0.1	5	115, 68.8[1], 110, 109, 120, 102	111	6.8	6.1
	Overall		11	Range, 91.2-120	104	9.4	9.0
Citrus fruits, whole fruit	Primary Quantitation (m/z 649→242)						
	DM-8007	0.001	5	124[1], 102, 103, 96.4, 104, 102	101	3.0	2.9
		0.1	6	108, 120, 111, 103, 101, 117	110	7.5	6.9
	Overall		11	Range, 96.4-120	106	7.2	6.8
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.001	5	103[1], 96.2, 110, 94.8, 101, 95.7	99.5	6.3	6.4
		0.1	6	98.3, 104, 114, 109, 110, 95.6	105	7.2	6.8
	Overall		11	Range, 94.8-114	103	7.1	6.9
Coffee, bean	Primary Quantitation (m/z 649→242)						
	DM-8007	0.001	5	88.8, 92.0, 102, 165[1], 72.0, 92.0	89.4	11	12
		0.1	6	80.8, 90.4, 88.8, 92.8, 110, 106	94.8	11	12
	Overall		11	Range, 72.0-110	92.3	11	12
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.001	5	81.6, 106, 102, 180[1], 104, 84.8	95.7	12	12
		0.1	6	107, 88.0, 104, 120, 101, 94.0	102	11	11
	Overall		11	Range, 81.6-120	99.3	11	11
Soybeans, seed	Primary Quantitation (m/z 649→242)						
	DM-8007	0.001	6	85.6, 76.5, 70.6, 76.0, 82.6, 76.5	78.0	5.3	6.8
		0.1	6	116, 98.8, 101, 104, 104, 103	104	6.0	5.7
	Overall		12	Range, 70.6-116	91.2	15	16
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.001	6	79.2, 95.9, 71.1, 88.6, 72.0, 79.1	81.0	9.7	12

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
		0.1	6	89.5, 94.4, 77.4, 90.0, 101, 96.2	91.4	8.1	8.8
	Overall		12	Range, 71.1-101	86.2	10	12

Independent laboratory validation of method D1417/01 (Jutson, 2017, BROFLAN_035):

The sample extraction, clean-up and method of determination was mostly identical to D1417/01, with modifications applied to some samples in a second trial (use of a larger amount of buffer salts for some potato, grape and lettuce samples; omitting the clean-up step for grape and lettuce). The results are shown in Table 85.

Table 85 Recovery data for the ILV of method D1417/01, measuring broflanilide and its metabolites S(PFP-OH)-8007 and DM-8007 in various plant matrices

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
Coffee bean, green	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.01	5	108, 112, 135, 112, 119	117	10.6	9.0
		1.0	5	109, 131, 110, 118, 128	119	9.9	8.3
	Overall		10	Range: 108 to 135 percent	118	10.2	8.7
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.01	5	112, 111, 139, 118, 123	121	11.2	9.3
		1.0	5	115, 133, 109, 116, 128	120	10.0	8.3
	Overall		10	Range: 109 to 139 percent	120	10.6	8.8
Kidney bean	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.01	5	122, 118, 114, 116, 119	118	3.1	2.6
		1.0	5	114, 111, 120, 122, 125	119	5.7	4.8
	Overall		10	Range: 111 to 125 percent	118	4.4	3.7
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.01	5	121, 120, 127, 119, 114	120	4.7	3.9
		1.0	5	116, 108, 121, 121, 121	117	5.4	4.6
	Overall		10	Range: 108 to 127 percent	119	5.0	4.3
Soya bean	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.01	5	103, 124, 123, 119, 115	117	8.5	7.3
		1.0	5	93.1, 92.6, 96.6, 84.6, 91.9	91.8	4.4	4.8
	Overall		10	Range: 84.6 to 124 percent	104	6.5	6.0
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.01	5	100, 111, 119, 117, 127	115	10.0	8.7
		1.0	5	91.1, 89.0, 95.0, 83.4, 87.9	89.3	4.3	4.8
	Overall		10	Range: 83.4 to 127 percent	102	7.1	6.7
Grape	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.01	5	92.7, 96.9, 94.9, 104, 104	98.4	5.1	5.2
		1.0	5	89.4, 88.9, 85.7, 88.3, 89.8	88.4	1.7	1.9
	Overall		10	Range: 85.7 to 104 percent	93.4	3.4	3.5
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.01	5	97.1, 92.5, 90.5, 109, 101	98.0	7.3	7.4
		1.0	5	90.6, 89.3, 87.2, 89.9, 90.6	89.5	1.4	1.6

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
	Overall		10	Range: 87.2 to 109 percent	93.8	4.3	4.5
Lettuce	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.01	5	83.8, 117, 98.5, 103, 91.1	98.8	12.7	12.9
		1.0	5	86.0, 84.9, 87.7, 85.2, 83.4	85.4	1.6	1.9
	Overall		10	Range: 83.4 to 117 percent	92.1	7.2	7.4
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.01	5	80.6, 111, 100, 90.0, 92.7	95.0	11.6	12.2
		1.0	5	86.8, 83.3, 86.9, 86.4, 82.6	85.2	2.1	2.5
	Overall		10	Range: 80.6 to 111 percent	90.1	6.8	7.3
Potato	Primary Quantitation (m/z 663→643)						
	Broflanilide	0.01	5	102, 119, 108, 112, 101	108	7.6	7.0
		1.0	5	94.7, 107, 122, 115, 94.7	107	12.2	11.4
	Overall		10	Range: 94.7 to 122 percent	108	9.9	9.2
	Confirmatory Quantitation (m/z 665→645)						
	Broflanilide	0.01	5	95.0, 115, 97.4, 103, 97.4	101	7.9	7.8
		1.0	5	93.5, 101, 120, 115, 93.5	105	12.4	11.8
	Overall		10	Range: 93.5 to 120 percent	103	10.1	9.8
Coffee bean, green	Primary Quantitation (m/z 649→242)						
	DM-8007	0.01	5	105, 103, 124, 113, 123	113	9.8	8.6
		1.0	5	117, 112, 118, 110, 108	113	4.4	3.9
	Overall		10	Range: 103 to 124 percent	113	7.1	6.3
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.01	5	108, 120, 115, 118, 103	113	6.9	6.1
		1.0	5	113, 110, 117, 108, 113	112	3.3	3.0
	Overall		10	Range: 103 to 120 percent	113	5.1	4.6
Kidney bean	Primary Quantitation (m/z 649→242)						
	DM-8007	0.01	5	114, 108, 114, 117, 113	113	3.5	3.0
		1.0	5	105, 100, 111, 115, 115	109	6.4	5.9
	Overall		10	Range: 100 to 117 percent	111	4.9	4.5
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.01	5	102, 108, 100, 113, 92.7	103	7.9	7.7
		1.0	5	117, 101, 110, 112, 115	111	6.2	5.6
	Overall		10	Range: 92.7 to 117 percent	107	7.1	6.6
Soya bean	Primary Quantitation (m/z 649→242)						
	DM-8007	0.01	5	82.8, 91.3, 102, 93.7, 96.8	93.2	7.0	7.5
		1.0	5	82.7, 79.4, 81.5, 72.6, 73.1	77.9	4.7	6.1
	Overall		10	Range: 72.6 to 102 percent	85.6	5.9	6.8
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.01	5	70.8, 72.6, 79.3, 85.0, 78.1	77.1	5.7	7.3
		1.0	5	77.9, 77.0, 76.7, 65.0, 72.7	73.9	5.3	7.2
	Overall		10	Range: 65.0 to 85.0 percent	75.5	5.5	7.3
Grape	Primary Quantitation (m/z 649→242)						
	DM-8007	0.01	5	94.3, 92.5, 86.5, 100, 111	96.9	9.2	9.5
		1.0	5	91.4, 88.5, 85.7, 90.7, 84.3	88.1	3.1	3.5
	Overall		10	Range: 85.7 to 111 percent	92.5	6.2	6.5

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.01	5	95.9, 108, 93.7, 98.7, 113	102	8.2	8.0
		1.0	5	92.6, 87.7, 84.9, 89.1, 86.7	88.2	2.9	3.3
	Overall		10	Range: 84.9 to 113 percent	95.0	5.5	5.7
Lettuce	Primary Quantitation (m/z 649→242)						
	DM-8007	0.01	5	81.0, 118, 99.7, 92.0, 83.5	94.9	15.0	15.8
		1.0	5	84.4, 79.7, 84.9, 82.0, 83.8	83.0	2.1	2.6
	Overall		10	Range: 79.7 to 118 percent	88.9	8.6	9.2
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.01	5	75.8, 113, 90.1, 86.4, 101	93.2	14.2	15.2
		1.0	5	82.8 84.9, 86.9, 84.0, 81.8	84.1	2.0	2.3
	Overall		10	Range: 75.8 to 113 percent	88.7	8.1	8.8
Potato	Primary Quantitation (m/z 649→242)						
	DM-8007	0.01	5	95.4, 127, 98.6, 104, 107	106	12.4	11.6
		1.0	5	95.9, 104, 122, 120, 95.9	108	12.9	12.0
	Overall		10	Range: 95.4 to 127 percent	107	12.6	11.8
	Confirmatory Quantitation (m/z 649→629)						
	DM-8007	0.01	5	112, 111, 109, 128, 103	112	9.3	8.3
		1.0	5	96.3, 105, 121, 114, 91.5	106	12.4	11.7
	Overall		10	Range: 91.5 to 128 percent	109	10.9	10.0
Coffee bean, green	Primary Quantitation (m/z 661→641)						
	S(PFP-OH)-8007	0.01	5	119, 115, 122, 119, 118	118	2.4	2.0
		1.0	5	117, 110, 117, 111, 113	113	3.2	2.8
	Overall		10	Range: 110 to 122 percent	116	2.8	2.4
	Confirmatory Quantitation (m/z 663→643)						
	S(PFP-OH)-8007	0.01	5	122, 118, 113, 116, 124	119	4.7	4.0
		1.0	5	114, 109, 119, 112, 117	114	3.9	3.4
	Overall		10	Range: 109 to 124 percent	116	4.3	3.7
Kidney bean	Primary Quantitation (m/z 661→641)						
	S(PFP-OH)-8007	0.01	5	116, 115, 111, 115, 117	115	2.5	2.1
		1.0	5	107, 100, 109, 107, 110	107	4.0	3.7
	Overall		10	Range: 100 to 117 percent	111	3.2	2.9
	Confirmatory Quantitation (m/z 663→643)						
	S(PFP-OH)-8007	0.01	5	114, 110, 115, 112, 117	114	2.6	2.3
		1.0	5	103, 100, 113, 112, 114	108	6.2	5.7
	Overall		10	Range: 100 to 117 percent	111	4.4	4.0
Soya bean	Primary Quantitation (m/z 661→641)						
	S(PFP-OH)-8007	0.01	5	94.4, 115, 120, 112, 115	111	9.8	8.8
		1.0	5	91.5, 87.4, 93.4, 81.4, 86.7	88.1	4.7	5.3
	Overall		10	Range: 81.4 to 120 percent	100	7.3	7.1
	Confirmatory Quantitation (m/z 663→643)						
	S(PFP-OH)-8007	0.01	5	104, 120, 108, 116, 123	114	8.3	7.3
		1.0	5	87.9, 90.2, 94.2, 80.2, 88.7	88.2	5.1	5.8
	Overall		10	Range: 80.2 to 123 percent	101	6.7	6.5
Grape	Primary Quantitation (m/z 661→641)						

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
	S(PFP-OH)-8007	0.01	5	108, 100, 102, 109, 103	104	3.9	3.7
		1.0	5	91.4, 88.1, 86.8, 91.1, 87.1	88.9	2.2	2.5
	Overall		10	Range: 86.8 to 109 percent	96.6	3.0	3.1
	Confirmatory Quantitation (m/z 663→643)						
	S(PFP-OH)-8007	0.01	5	104, 97.3, 99.3, 104, 104	102	3.1	3.1
		1.0	5	92.2, 90.5, 87.6, 87.5, 88.3	89.2	2.1	2.3
	Overall		10	Range: 87.5 to 104 percent	95.4	2.6	2.7
Lettuce	Primary Quantitation (m/z 661→641)						
	S(PFP-OH)-8007	0.01	5	80.2, 115, 98.1, 88.0, 95.5	95.5	13.2	13.8
		1.0	5	83.2, 81.7, 86.9, 83.2, 82.2	83.4	2.1	2.5
	Overall		10	Range: 80.2 to 115 percent	89.5	7.6	8.1
	Confirmatory Quantitation (m/z 663→643)						
	S(PFP-OH)-8007	0.01	5	79.0, 114, 96.9, 90.0, 103	96.5	13.1	13.6
		1.0	5	85.6, 86.1, 84.1, 86.0, 85.2	85.4	0.8	0.9
	Overall		10	Range: 79.0 to 114 percent	91.0	7.0	7.3
Potato	Primary Quantitation (m/z 661→641)						
	S(PFP-OH)-8007	0.01	5	98.2, 118, 102, 102, 92.2	103	9.6	9.4
		1.0	5	92.3, 102, 119, 115, 93.1	104	12.3	11.8
	Overall		10	Range: 92.2 to 119 percent	103	11.0	10.6
	Confirmatory Quantitation (m/z 663→643)						
	S(PFP-OH)-8007	0.01	5	98.2, 117, 109, 100, 95.8	104	9.0	8.6
		1.0	5	90.3, 102, 117, 115, 93.1	103	12.2	11.8
	Overall		10	Range: 90.3 to 117 percent	96.8	10.6	10.2

Method D1703/01 (Downs, 2017, BROFLAN_036)

The method is based on the citrate buffered QuEChERS method. The residues of the metabolites B-urea and B-oxam acid are extracted from crop matrices by shaking with acetonitrile (for dry matrices addition of water), followed by shaking with a salt solution ($MgSO_4$; NaCl; sodium citrate sesquihydrate and sodium citrate dehydrate). Optional clean-up with PSA. An aliquot of the extract is then diluted with 0.1 percent formic acid in acetonitrile:water (50:50) and analysed.

Final determination is accomplished by LC-MS/MS using a BEH Phenyl column. B-oxam acid was analysed in negative ionization mode and monitoring the ion transitions m/z 478→406, 480→408, while B-urea was quantified in positive ionization mode and monitoring the ion transitions m/z 451→431, 453→433. Quantitation was done with external standards in matrix or solvent. The results are shown in Table 86.

Table 86 Recovery data for method D1703/01 measuring metabolites B-urea and B-oxam acid in various plant matrices by LC-MS/MS

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
Lettuce	Primary Quantitation (m/z 451→431)						
	B-urea	0.01	5	102, 93.2, 90.4, 101, 94.9	96.3	5.0	5.2
		1.0	5	96.5, 92.5, 102, 103, 106	100	5.4	5.4

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
	Overall		10	Range, 90.4-106	98.2	5.3	5.4
	Confirmatory Quantitation (m/z 453→433)						
	B-urea	0.01	5	100, 98.8, 89.9, 98.4, 94.0	96.2	4.2	4.4
		1.0	5	96.0, 104, 91.4, 103, 108	101	6.7	6.6
	Overall		10	Range, 89.9-108	98.4	5.7	5.8
Orange	Primary Quantitation (m/z 451→431)						
	B-urea	0.01	5	98.6, 98.2, 85.7, 92.3, 87.5	92.5	5.9	6.4
		1.0	5	100, 101,88.7, 94.5, 97.9	96.4	5.0	5.2
	Overall		10	Range, 85.7-101	94.4	5.6	5.9
	Confirmatory Quantitation (m/z 453→433)						
	B-urea	0.01	5	99.6, 97.3, 91.9, 91.3, 93.4	94.7	3.6	3.8
		1.0	5	101, 104, 95.0, 97.7, 100	99.5	3.4	3.4
	Overall		10	Range, 91.3-104	97.12	4.2	4.3
Wheat Grain	Primary Quantitation (m/z 451→431)						
	B-urea	0.01	5	97.9, 93.1, 87.8, 95.0, 96.2	94.0	3.9	4.1
		1.0	5	92.2, 95.7, 95.8, 97.4, 99.9	96.2	2.8	2.9
	Overall		10	Range, 87.8-99.9	95.1	3.4	3.6
	Confirmatory Quantitation (m/z 453→433)						
	B-urea	0.01	5	98.8, 100.,96.9, 95.3, 96.9	97.6	1.8	1.9
		1.0	5	90.4, 100, 99.6, 102, 98.5	98.1	4.5	4.6
	Overall		10	Range, 90.4-102	97.8	3.2	3.3
Soybean Seed	Primary Quantitation (m/z 451→431)						
	B-urea	0.01	5	100, 102, 98.4, 97.2, 100	99.5	1.8	1.8
		1.0	5	114, 111, 110, 110, 111	111	1.6	1.5
	Overall		10	Range, 97.2-114	105	6.4	6.0
	Confirmatory Quantitation (m/z 453→433)						
	B-urea	0.01	5	96.5, 102, 97.8, 100, 102	99.7	2.5	2.5
		1.0	5	109, 112, 116, 106, 109	110	3.8	3.4
	Overall		10	Range, 96.5-116	105	6.4	6.1
Kidney Bean	Primary Quantitation (m/z 451→431)						
	B-urea	0.01	5	100, 104, 96.4, 101, 97.0	99.7	3.1	3.1
		1.0	5	111, 101, 113, 111, 108	109	4.7	4.3
	Overall		10	Range, 96.4-113	104	6.1	5.9
	Confirmatory Quantitation (m/z 453→433)						
	B-urea	0.01	5	96.4, 97.9, 104, 95.3, 103	99.3	3.9	4.0
		1.0	5	108, 106, 111, 108, 112	109	2.4	2.2
	Overall		10	Range, 95.3-112	104	6.0	5.7
Lettuce	Primary Quantitation (m/z 478→406)						
	B-oxam-acid	0.01	5	97.3, 94.3, 97.4, 88.4, 94.7	94.4	3.7	3.9
		1.0	5	94.3, 94.2, 102, 107, 95.6	98.6	5.7	5.8
	Overall		10	Range, 88.4-107	96.5	5.0	5.2
	Confirmatory Quantitation (m/z 480→408)						
	B-oxam-acid	0.01	5	95.3, 93.3, 91.5, 99.9, 91.5	94.3	3.5	3.7
		1.0	5	96.1, 100, 104, 109, 94.3	101	6.0	5.9
	Overall		10	Range, 91.5-109	97.5	5.7	5.9

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	SD, %	RSD, %
Orange	Primary Quantitation (m/z 478→406)						
	B-oxam-acid	0.01	5	83.6, 96.7, 90.4, 92.4, 97.8	92.2	5.7	6.2
		1.0	5	101, 104, 111, 102, 101	104	4.2	4.1
	Overall		10	Range, 83.6-111	98.0	7.7	7.9
	Confirmatory Quantitation (m/z 480→408)						
	B-oxam-acid	0.01	5	90.3, 96.0, 92.5, 90.3, 90.0	91.8	2.5	2.8
		1.0	5	107, 109, 110, 105, 102	107	3.2	3.0
	Overall		10	Range, 90.0-110	99.2	8.3	8.3
Wheat Grain	Primary Quantitation (m/z 478→406)						
	B-oxam-acid	0.01	5	94.8, 87.4, 95.1, 93.5, 88.8	91.9	3.6	3.9
		1.0	5	88.4, 99.5, 106, 118, 90.2	100	12.1	12.1
	Overall		10	Range, 87.4-118	96.2	9.6	9.9
	Confirmatory Quantitation (m/z 480→408)						
	B-oxam-acid	0.01	5	90.5, 102, 90.1, 89.8, 79.6	90.4	7.9	8.8
		1.0	5	95.3, 104, 107, 105, 87.8	99.8	8.1	8.1
	Overall		10	Range, 79.6-107.0	95.1	9.0	9.5
Soybean Seed	Primary Quantitation (m/z 478→406)						
	B-oxam-acid	0.01	5	72.9, 80.1, 78.8, 78.9, 78.4	77.8	2.8	3.6
		1.0	5	78.6, 73.9, 86.1, 74.4, 76.9	78.0	4.9	6.3
	Overall		10	Range, 72.9-86.1	77.9	3.6	4.9
	Confirmatory Quantitation (m/z 480→408)						
	B-oxam-acid	0.01	5	74.2, 79.4, 76.2, 77.0, 82.5	77.9	3.2	4.1
		1.0	5	75.6, 74.9, 82.7, 71.2, 65.2	73.9	6.4	8.7
	Overall		10	Range, 65.2-82.7	75.9	5.2	6.9
Kidney Bean	Primary Quantitation (m/z 478→406)						
	B-oxam-acid	0.01	5	71.9, 79.2, 80.9, 72.1, 79.7	76.8	4.4	5.7
		1.0	5	81.4, 84.6, 109, 101, 80.9	91.4	12.8	14.0
	Overall		10	Range, 71.9-109	84.1	11.9	14.1
	Confirmatory Quantitation (m/z 480→408)						
	B-oxam-acid	0.01	5	77.9, 82.8, 74.2, 78.1, 80.0	78.6	3.2	4.0
		1.0	5	76.0, 79.1, 98.3, 96.5, 71.1	84.2	12.4	14.7
	Overall		10	Range, 71.1-98.3	81.4	9.0	11.1

Japanese crop residue method (Kawaguchi, 2020, BROFLAN_037)

The study contains a compilation of validation data from multiple field trials. The homogenized sample materials are extracted with acetonitrile/water (80/20). After filtration an aliquot of the extract is cleaned up by SPE on a C18 cartridge. Analytes are eluted with acetonitrile/water/formic acid (80/20/1/v). If necessary, the eluate is further diluted with acetonitrile/water/formic acid (80/20/1/v).

Final determination is accomplished by LC-MS/MS in positive electrospray ionization using a C18 column and monitoring ion transition m/z 665→645 for broflanilide, m/z 649→242 for DM-8007 and m/z 661→641 for S(PFP-OH)-8007. Quantitation is done with external standards in solvent. The results are shown in Table 87.

Table 87 Recovery data for the method measuring broflanilide and its metabolites S(PFP-OH)-8007 and DM-8007 in high water content plant matrices

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	RSD, %]
Japanese radish (root)	Quantitation (m/z 665→645)					
	Broflanilide	0.01	6	89, 87, 95, 94, 97, 97	93	4.5
		0.5	6	102, 98, 100, 99, 103, 102	101	1.9
	Overall		12	Range, 87-103	97	5.2
Japanese radish (leaf)	Broflanilide	0.01	6	107, 106, 93, 87, 100, 96	98	7.9
		0.5	6	98, 95, 99, 96, 96, 93	96	2.2
		5.0	6	95, 94, 98, 97, 98, 98	97	1.8
	Overall		18	Range, 87-107	97	4.7
Turnip (root)	Broflanilide	0.01	6	89, 87, 88, 85, 87, 81	86	3.3
		0.5	6	93, 91, 93, 91, 91, 91	92	1.1
	Overall		12	Range, 81-93	89	4.0
Turnip (leaf)	Broflanilide	0.01	6	104, 102, 116, 106, 104, 98	105	5.7
		0.5	6	100, 96, 97, 93, 97, 94	96	2.6
		3.0	6	96, 93, 95, 93, 94, 94	94	1.2
	Overall		18	Range, 93-116	98	6.1
Leek	Broflanilide	0.01	6	105, 103, 106, 105, 104, 102	104	1.4
		0.5	6	97, 93, 102, 98, 103, 102	99	3.9
		2.0	6	98, 97, 95, 95, 94, 93	95	2.0
	Overall		18	Range, 93-106	100	4.5
Japanese radish (root)	Quantitation (m/z 649→242)					
	DM-8007	0.01	6	83, 82, 94, 93, 95, 94	90	6.6
		0.5	6	103, 99, 101, 100, 100, 100	101	1.4
	Overall		12	Range, 82-103	95	7.1
Japanese radish (leaf)	DM-8007	0.01	6	98, 96, 98, 97, 103, 93	98	3.3
		0.5	6	101, 94, 102, 98, 102, 102	100	3.3
	Overall		12	Range, 93-102	99	3.4
Turnip (root)	DM-8007	0.01	6	85, 85, 86, 85, 84, 81	84	2.1
		0.5	6	91, 90, 93, 90, 91, 90	91	1.3
	Overall		12	Range, 81-93	88	4.2
Turnip (leaf)	DM-8007	0.01	6	105, 100, 105, 99, 97, 96	100	3.9
		0.5	6	99, 95, 95, 94, 97, 92	95	2.5
	Overall		12	Range, 92-105	98	4.1
Leek	DM-8007	0.01	6	101, 90, 101, 96, 98, 96	97	4.2
		0.5	6	95, 91, 98, 94, 97, 94	95	2.6
	Overall		12	Range, 90-101	96	3.6
Japanese radish (root)	Quantitation (m/z 661→641)					
	S(PFP-OH)-8007	0.01	6	100, 98, 105, 103, 104, 100	101	1.6
		0.5	6	102, 102, 101, 98, 102, 100	102	2.7
	Overall		12	Range, 98-105	101	2.2
Japanese radish (leaf)	S(PFP-OH)-8007	0.01	6	98, 92, 96, 94, 95, 94	95	2.0
		0.5	6	98, 93, 96, 96, 95, 93	95	2.1
	Overall		18	Range, 92-98	95	2.0
Turnip (root)	S(PFP-OH)-8007	0.01	6	90, 89, 92, 84, 88, 83	88	4.0

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean, %	RSD, %]
		0.5	6	94, 92, 95, 92, 94, 91	93	1.7
	Overall		12	Range, 83-95	90	4.2
Turnip (leaf)	S(PFP-OH)-8007	0.01	6	94, 92, 95, 90, 96, 87	92	3.7
		0.5	6	96, 92, 96, 93, 94, 93	94	1.8
	Overall		12	Range, 87-96	93	2.9
Leek	S(PFP-OH)-8007	0.01	6	112, 110, 116, 113, 113, 112	113	1.7
		0.5	6	104, 95, 105, 95, 101, 98	100	4.4
	Overall		12	Range, 95-116	106	7.1

Korean crop residue method (Hiraki, 2020, BROFLAN_038; Hiraki, 2020, BROFLAN_039)

The studies contain compilations of validation data from multiple field trials. The homogenized sample materials are extracted with acetonitrile. After filtration the extract is partitioned with saturated saline solution, water and dichloromethane. The organic phase is dried on anhydrous sodium sulfate and concentrated by evaporation. Clean-up of the extract is performed on a NH_2 SPE cartridge. Analytes are eluted with n-hexane/acetone (80:20).

Final determination in study BROFLAN_038 is accomplished by GC-ECD using a DB-5 column or equivalent (cabbage, radish, tomato) or by HPLC-UV at 226 nm and using a C18 column (Chinese cabbage, green onion, tomato, radish). In study BROFLAN_039, final determination is accomplished by HPLC-UV at 226 nm and using a C18 column (Chinese cabbage, tomato, green onion, radish (broflanilide & DM-8007)) and by LC-MS/MS in positive electrospray ionization mode and monitoring the transitions m/z 661→ 621, 661→ 641 (radish for S(PFP-OH)-8007). Quantitation is done in both studies with external standards in solvent. The results are shown in Tables 88 and 89..

Table 88 Recovery data for study BROFLAN_038 measuring broflanilide and its metabolites S(PFP-OH)-8007 and DM-8007 in high water content plant matrices (n=3)

Matrix	Analyte	Fortification level [mg/kg]	Recovery,%					Reference
			Replicate 1	Replicate 2	Replicate 3	Mean	SD	
Cabbage	Broflanilide	0.1	85.0	80.2	82.3	82.5	2.9	SGR-2017-104 2020/2090104
		0.5	94.5	90.9	97.5	94.3	3.5	
	DM-8007	0.1	99.0	94.3	96.6	96.6	2.4	
		0.5	102.9	99.5	106.2	102.9	3.3	
	S(PFP-OH)-8007	0.1	96.7	91.8	94.3	94.3	2.6	
		0.5	106.0	101.8	108.7	105.5	3.3	
Chinese cabbage	Broflanilide	0.1	99.9	95.7	98.5	98.0	2.2	SGR-2017-103 2020/2090117
		0.5	94.8	95.8	98.8	96.5	2.2	
	DM-8007	0.1	101.2	96.4	96.4	98.0	2.8	
		0.5	94.9	96.4	97.6	96.3	1.4	
	S(PFP-OH)-8007	0.1	105.3	101.7	111.2	106.1	4.5	
		0.5	98.1	98.0	97.6	97.9	0.3	
Radish (root)	Broflanilide	0.1	78.4	74.6	71.4	74.8	4.7	SGR-2017-102 2020/2090125
		0.5	89.1	87.8	86.8	87.9	1.3	
	DM-8007	0.1	100.2	94.3	91.5	95.3	4.7	
		0.5	101.4	100.6	99.1	100.4	1.2	
	S(PFP-OH)-	0.1	109.4	103.0	100.7	104.4	4.3	

Matrix	Analyte	Fortification level [mg/kg]	Recovery,%					Reference
			Replicate 1	Replicate 2	Replicate 3	Mean	SD	
	8007	0.5	113.8	112.5	110.3	112.2	1.6	
Radish (leaves)	Broflanilide	0.1	73.3	83.3	74.3	77.0	7.2	SGR-2017-102 2020/2090125
		0.5	83.0	79.9	78.9	80.6	2.7	
	DM-8007	0.1	101.7	114.7	103.8	106.7	6.5	
		0.5	98.4	95.0	93.7	95.7	2.5	
	S(PFP-OH)-8007	0.1	112.5	114.4	114.0	113.6	0.9	
		0.5	99.1	95.3	93.6	96.0	2.9	
Green onions	Broflanilide	0.1	93.8	97.7	101.5	97.7	3.9	SGR-2017-107 2020/2090145
		0.5	94.0	94.5	89.5	92.7	3.0	
	DM-8007	0.1	87.9	89.3	89.2	88.8	0.9	
		0.5	91.8	88.7	88.5	89.7	2.1	
	S(PFP-OH)-8007	0.1	92.9	89.1	91.6	91.2	2.1	
		0.5	91.5	87.1	84.0	87.5	4.3	
Tomato	Broflanilide	0.1	94.5	95.9	91.1	93.8	2.6	SGR-2017-106 2020/2090154
		0.5	80.3	79.3	84.2	81.3	3.2	
	DM-8007	0.1	90.3	91.0	86.2	89.2	2.9	
		0.5	90.2	91.6	97.5	93.1	4.2	
	S(PFP-OH)-8007	0.1	93.0	93.7	89.1	91.9	2.7	
		0.5	100.3	99.6	107.6	102.5	4.3	

Table 89 Recovery data for study BROFLAN_039 measuring broflanilide and its metabolites S(PFP-OH)-8007 and DM-8007 in high water content plant matrices

Matrix	Analyte	n	Fortification level [mg/kg]	Recovery,%							Reference
				Replicate 1	Replicate 2	Replicate 3	Replicate 4	Replicate 5	Mean	SD	
Chinese cabbage	Broflanilide	3	0.1	94.8	97.4	97.4	-	-	96.5	1.5	DBA-RC-2017-011 2020/2090115
		3	0.5	96.0	95.4	95.0	-	-	95.5	0.5	
	DM-8007	3	0.1	114.8	118.5	117.5	-	-	116.9	1.9	
		3	0.5	113.5	108.9	111.4	-	-	111.3	2.3	
	S(PFP-OH)-8007	3	0.1	102.9	101.2	103.0	-	-	102.4	1.0	
		3	0.5	94.4	96.0	96.9	-	-	95.8	1.3	
Radish (root)	Broflanilide	5	0.02	101.2	107.8	106.8	107.1	108.4	106.3	2.9	R1941 2020/2090129
		5	0.2	97.1	95.7	95.9	94.1	93.0	95.2	1.6	
	DM-8007	5	0.02	100.1	100.6	99.8	103.7	100.7	101.0	1.6	
		5	0.2	92.0	90.7	91.8	900.0	88.2	90.5	1.5	
	S(PFP-OH)-8007	5	0.02	92.2	81.7	90.7	99.3	91.7	91.1	6.3	
		5	0.2	93.8	98.6	95.8	94.7	95.3	95.6	1.8	
Radish (leaves)	Broflanilide	5	0.04	93.2	105.0	104.5	98.8	100.9	100.5	4.8	R1941 2020/2090129
		5	0.4	84.6	83.8	82.6	84.9	84.3	84.0	0.9	
	DM-8007	5	0.04	98.1	106.1	105.9	103.4	103.7	103.4	3.2	
		5	0.4	85.9	84.2	82.7	85.6	83.7	84.4	1.3	
	S(PFP-OH)-8007	5	0.04	81.3	86.3	85.5	101.1	79.8	86.8	8.5	
		5	0.4	77.7	79.2	75.4	72.0	77.5	76.8	2.1	
Green onions	Broflanilide	3	0.1	111.9	113.1	112.5	-	-	112.5	0.6	DBA-RC-2017-

Matrix	Analyte	n	Fortifi-cation level [mg/kg]	Recovery,%							Reference
				Repli-cate 1	Repli-cate 2	Repli-cate 3	Repli-cate 4	Repli-cate 5	Mean	SD	
		3	0.5	97.4	98.1	100.4	-	-	98.6	1.6	017
	DM-8007	3	0.1	103.4	102.6	102.8	-	-	102.9	0.4	2020/2090142
		3	0.5	92.5	93.7	92.9	-	-	93.0	0.6	
	S(PFP-OH)-8007	3	0.1	98.0	85.1	95.9	-	-	93.0	6.9	
		3	0.5	83.3	84.2	86.9	-	-	84.8	1.9	
Tomato	Broflanilide	3	0.02	104.9	104.8	103.2	-	-	104.3	1.0	R1943
		3	0.2	96.0	97.2	102.1	-	-	98.4	3.2	2020/2090156
	DM-8007	3	0.02	98.0	94.6	90.1	-	-	94.2	4.0	
		3	0.2	96.6	95.4	101.6	-	-	97.9	3.3	
	S(PFP-OH)-8007	3	0.02	99.1	99.4	84.0	-	-	94.2	8.8	
		3	0.2	76.5	92.9	85.3	-	-	84.9	8.2	

Animal materials

Method D1604/01 (Malinsky, 2017, BROFLAN_040)

Residues of broflanilide in the livestock commodity samples of milk, egg, liver, kidney and muscle are extracted with acetonitrile followed by acetonitrile/water (80:20). Liver, kidney and muscle are further partitioned with a salt solution ($MgSO_4$; NaCl; sodium citrate sesquihydrate and sodium citrate dehydrate), followed by clean-up with PSA. Samples of fat are extracted with acetone/hexane (20/80), followed by acetone. An aliquot of the extracts from all commodities are then diluted with 0.1 percent formic acid in acetonitrile/water (1:1).

Final determination is accomplished by LC-MS/MS in positive ionization mode, using a BEH C18 column and monitoring ion transitions m/z 663→643, 665→645 for broflanilide, m/z 545→525, 547→527 for DC-DM-8007 and m/z 649→242, 651→242 for DM-8007. Quantitation was done with external standards in solvent. The results are in Table 90.

Table 90 Recovery data for method D1604/01 measuring broflanilide and its metabolites DC-DM-8007 and DM-8007 in various animal matrices using LC-MS/MS

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean,%	SD,%	RSD, %
Milk		Primary Quantitation (*m/z* 663→643)					
	Broflanilide	0.001	5	105, 110, 120, 113, 120	114	6.4	5.6
		0.01	5	97.6, 111, 101, 102, 107	104	5.4	5.3
		Overall	10	Range: 97.6-120	109	7.7	7.1
	Confirmatory Quantitation (*m/z* 665→645)						
	Broflanilide	0.001	5	121, 112, 108, 105, 110	111	6.1	5.5
		0.01	5	105, 100, 104, 97.6, 88.8	99	6.5	6.6
		Overall	10	Range: 88.8-121	105	8.7	8.3
Egg	Primary Quantitation (*m/z* 663→643)						
	Broflanilide	0.01	5	107, 72.8, 94.8, 92.8, 92.8	92.1	12.3	13.4
		0.1	5	80.4, 86.8, 83.2, 81.2, 79.6	82.2	2.9	3.5
		Overall	10	Range: 72.8-107	87.2	9.9	11.4
	Confirmatory Quantitation (*m/z* 665→645)						

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean,%	SD,%	RSD, %
	Broflanilide	0.01	5	96.8, 91.2, 104, 94.8, 89.2	95.2	5.8	6.0
		0.1	5	85.6, 80.0, 80.4, 88.0, 91.6	85.1	5.0	5.8
		Overall	10	Range: 80.0-104	90.2	7.3	8.1
Fat	Primary Quantitation (*m/z* 663→643)						
	Broflanilide	0.01	5	93.2, 100, 87.2, 98.4, 84.8	92.6	6.6	7.1
		0.1	5	83.6, 83.2, 84.8, 92.0[1)], 85.2[1)]	85.8	3.6	4.2
		Overall	10	Range, 83.2-100	89.2	6.2	6.9
	Confirmatory Quantitation (*m/z* 665→645)						
	Broflanilide	0.01	5	84.4, 94.8, 94.0, 108, 92.0	94.7	8.7	9.2
		0.1	5	91.6, 79.2, 84.8, 97.6[1)], 90.9[1)]	88.8	7.0	7.9
		Overall	10	Range, 79.2-108	91.8	8.1	8.8
Liver	Primary Quantitation (*m/z* 663→643)						
	Broflanilide	0.01	5	112, 128, 128, 116, 119	120	7.0	5.8
		0.1	5	108, 106, 141, 117, 122	119	14.0	11.8
		Overall	10	Range, 106-141	119	10.5	8.8
	Confirmatory Quantitation (*m/z* 665→645)						
	Broflanilide	0.01	5	113, 124, 105, 101, 111	111	9.0	8.1
		0.1	5	117, 115, 125, 100, 116	115	8.9	7.8
		Overall	10	Range, 100-125	112	8.7	7.7
Kidney	Primary Quantitation (*m/z* 663→643)						
	Broflanilide	0.01	5	109, 117, 114, 127, 114	116	6.7	5.7
		0.1	5	108, 122, 119, 119, 103	114	8.3	7.3
		Overall	10	Range, 103-127	115	7.1	6.2
	Confirmatory Quantitation (*m/z* 665→645)						
	Broflanilide	0.01	5	117, 120, 111, 100, 138	117	13.8	11.8
		0.1	5	97.5, 116, 127, 125, 119	117	11.7	10.0
		Overall	10	Range, 97.5-138	117	12.1	10.3
Muscle	Primary Quantitation (*m/z* 663→643)						
	Broflanilide	0.01	5	98.0, 107, 102, 110, 99.0	103	5.0	4.9
		0.1	5	90.0, 94.0, 102, 113, 97.0	99.1	8.8	8.9
		Overall	10	Range, 90.0-113	101	7.1	7.0
	Confirmatory Quantitation (*m/z* 665→645)						
	Broflanilide	0.01	5	113, 104, 113, 104, 98.5	106	6.4	6.0
		0.1	5	117, 105, 95.5, 103, 112	106	8.2	7.7
		Overall	10	Range, 95.5-117	106	6.9	6.5
Milk	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.001	5	90.8, 97.2, 80.4, 81.2, 81.6	86.2	7.5	8.6
		0.01	5	99.2, 78.8, 88.8, 91.2, 104	92.4	9.8	11
		Overall	10	Range, 78.8-104	89.3	8.8	9.9
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.001	5	81.6, 100, 97.6, 105, 88.8	94.6	9.3	9.9
		0.01	5	102, 91.6, 91.2, 92.0, 92.4	93.8	4.4	4.7
		Overall	10	Range, 81.6-105	94.2	6.9	7.3
Egg	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.01	5	96.4, 75.2, 91.6, 103, 102	93.7	11.3	12.1
		0.1	5	87.6, 85.6, 73.6, 98.8, 85.6	86.2	9.0	10.4

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean,%	SD,%	RSD, %
		Overall	10	Range, 73.6-103	90.0	10.4	11.6
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.01	5	100, 79.6, 81.6, 80.4, 86.4	85.7	8.6	10.1
		0.1	5	78.4, 84.0, 75.6, 76.8, 74.4	77.8	3.8	4.8
		Overall	10	Range, 74.4-100	81.8	7.5	9.2
Fat	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.01	10	86.4, 75.6, 80.4, 91.6, 84.4 84.8, 78.0, 84.4, 93.6, 82.4	84.2	5.6	6.6
		0.1	10	78.0, 75.6, 72.8, 76.4, 91.2 95.2, 91.2, 87.6, 89.9[1)], 88.1[1)]	84.6	8.0	9.5
		Overall	20	Range: 64.4-86.5	72.8	6.5	8.9
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.01	10	79.6, 91.2, 84.8, 86.4, 87.6 97.6, 89.6, 78.8, 93.2, 98.0	88.7	6.6	7.5
		0.1	10	93.6, 88.4, 82.4, 86.4, 92.8 88.8, 90.8, 94.8, 90.5[1)], 92.4[1)]	90.1	3.7	4.1
		Overall	20	Range: 68.5-84.7	74.7	5.5	7.4
Liver	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.01	5	105, 112, 97.5, 94.0, 114	104	8.6	8.3
		0.1	5	96.5, 109, 85.5, 103, 98.5	98.5	8.7	8.8
		Overall	10	Range, 85.5-114	102	8.7	8.6
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.01	5	78.5, 110, 100, 89.5, 87.0	92.9	12.0	13.0
		0.1	5	99.0, 102, 82.5, 96.5, 105	96.9	8.6	8.9
		Overall	10	Range, 78.5-110	94.9	10.1	10.6
Kidney	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.01	5	116, 93.5, 97.0, 104, 105	103	8.7	8.4
		0.1	5	106, 122, 109, 109, 106	110	6.6	6.0
		Overall	10	Range, 93.5-122	107	8.1	7.6
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.01	5	102, 101, 99.5, 114, 116	106	7.7	7.2
		0.1	5	96.5, 99.0, 103, 123, 112	107	10.9	10.2
		Overall	10	Range, 96.5-123	107	8.9	8.4
Muscle	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.01	5	91.5, 114, 107, 99.0, 109	104	8.8	8.5
		0.1	5	92.5, 119, 98.0, 101, 93.5	101	10.5	10.5
		Overall	10	Range, 91.5-119	102	9.3	9.1
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.01	5	109, 100, 106, 94.5, 97.0	101	6.1	6.0
		0.1	5	83.5, 78.5, 92.5, 84.0, 86.5	85.0	5.1	6.0
		Overall	10	Range, 78.5-109	93.2	10.1	10.8
Milk	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.001	5	106, 113, 112, 112, 101	109	5.1	4.7
		0.01	5	106, 94.4, 98.8, 89.6, 106	99	7.3	7.4
		Overall	10	Range, 89.6-113	104	7.8	7.6
	Confirmatory Quantitation (*m/z* 547→527)						

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean,%	SD,%	RSD, %
	DC-DM-8007	0.001	5	113, 111, 110, 97.2, 110	108	6.4	5.9
		0.01	5	104, 122, 102, 128, 107	112	11.5	10.2
		Overall	10	Range, 97.2-128	110	9.0	8.1
Egg	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.01	5	108, 88.8, 120, 103, 131	110	16.2	14.7
		0.1	5	91.2, 101, 94.0, 94.8, 98.4	95.9	3.9	4.1
		Overall	10	Range 88.8-131	103	13.4	13.0
	Confirmatory Quantitation (*m/z* 547→527)						
	DC-DM-8007	0.01	5	95.2, 93.6, 92.8, 91.2, 77.6	90.1	7.1	7.9
		0.1	5	84.4, 90.8, 89.2, 82.4, 90.0	87.4	3.7	4.3
		Overall	10	Range, 77.6-95.2	88.7	5.6	6.2
Fat	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.01	10	91.6, 87.6, 89.2, 90.4, 94.4, 95.6, 94.8, 87.2, 103, 94.4	92.8	4.7	5.1
		0.1	10	93.2, 98.0, 81.2, 87.6, 114, 92.4, 80.4, 83.2, 92.7[1)], 89.1[1)]	91.2	9.8	10.8
		Overall	20	Range, 80.4-114	92.0	7.6	8.2
	Confirmatory Quantitation (*m/z* 547→527)						
	DC-DM-8007	0.01	10	86.0, 89.2, 85.6, 84.8, 93.2 102, 92.0, 88.0, 97.6, 92.4	91.1	5.5	6.1
		0.01	10	98.0, 77.6, 82.4, 85.2, 100 112, 110, 116, 106[1)], 97.6[1)]	98.5	13.1	13.3
		Overall	20	Range, 77.6-116	94.8	10.5	11.1
Liver	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.01	10	70.5, 78.0, 67.5, 92.0, 72.0 75.0, 103, 97.0, 79.0, 96.0	83.0	12.8	15.4
		0.1	5	98.5, 95.5, 117, 101, 98.5	102	8.6	8.4
		Overall	15	Range, 67.5-117	89.4	14.6	16.3
	Confirmatory Quantitation (*m/z* 547→527)						
	DC-DM-8007	0.01	10	76.0, 106, 71.5, 68.0, 81.0 82.0, 88.0, 75.0, 92.0, 93.5	83.3	11.6	14.0
		0.1	5	104, 116, 110, 116, 102	110	6.5	6.0
		Overall	15	Range, 68.0-116	92.1	16.2	17.7
Kidney	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.01	5	98.0, 115, 71.5, 79.0, 90.0	90.7	17.0	18.7
		0.1	5	82.5, 103, 90.0, 89.5, 72.0	87.3	11.2	12.8
		Overall	10	Range, 71.5-115	89	13.7	15.4
	Confirmatory Quantitation (*m/z* 547→527)						
	DC-DM-8007	0.01	5	82.5, 83.5, 87.5, 94.5, 72.0	84.0	8.2	9.8
		0.1	5	92.5, 100.5, 96.5, 95.0, 91.0	95.1	3.7	3.9
		Overall	10	Range, 72.0-100.5	89.6	8.4	9.4
Muscle	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.01	5	117, 121, 115, 118, 120	118	2.4	2.0
		0.1	5	109, 130, 117, 98.0, 114	114	11.7	10.3
		Overall	10	Range, 98-130	116	8.3	7.2
	Confirmatory Quantitation (*m/z* 547→527)						

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean,%	SD,%	RSD, %
	DC-DM-8007	0.01	5	99.0, 103, 114, 113, 125	111	10.2	9.2
		0.1	5	107, 139, 107, 126, 89.0	114	19.4	17.1
		Overall	10	Range, 89-139	112	14.7	13.1

Notes:

[1)] Average of more than one value.

Independent laboratory validation of method D1604/01 (Sheng & Wrigley, 2017, BROFLAN_041):

The sample extraction, clean-up and method of determination were identical to D1604/01. Recovery data are in Table 91.

Table 91 Recovery data for the ILV of method D1604/01, measuring broflanilide and its metabolites DC-DM-8007 and DM-8007 in various animal matrices

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean,%	SD,%	RSD, %]
Milk		Primary Quantitation (*m/z* 663→643)					
	Broflanilide	0.001	5	99.9, 87.2, 86.2, 92.4, 95.0	92.2	5.64	6.12
		0.01	5	92.4, 93.2, 91.7, 93.1, 92.7	92.6	0.601	0.65
		Overall	10	Range: 86.2-99.9	92.4	3.79	4.10
	Confirmatory Quantitation (*m/z* 665→645)						
	Broflanilide	0.001	5	110, 98.6, 97.0, 111,106	104	6.26	6.00
		0.01	5	92.4, 93.6, 93.0, 92.5, 93.9	93.1	0.664	0.71
		Overall	10	Range: 92.4-111	98.7	7.25	7.35
Egg	Primary Quantitation (*m/z* 663→643)						
	Broflanilide	0.01	5	90.4, 86.2, 83.8, 85.7, 77.4	84.7	4.74	5.60
		0.1	5	86.6, 84.0, 85.3, 87.3, 85.9	85.8	1.23	1.44
		Overall	10	Range: 77.4-90.4	85.3	3.32	3.89
	Confirmatory Quantitation (*m/z* 665→645)						
	Broflanilide	0.01	5	88.0, 85.7, 82.0, 82.3, 89.9	85.6	3.47	4.06
		0.1	5	86.1, 83.5, 85.6, 86.7, 84.6	85.3	1.27	1.49
		Overall	10	Range: 82.0-89.9	85.4	2.47	2.89
Fat	Primary Quantitation (*m/z* 663→643)						
	Broflanilide	0.01	5	89.1, 89.4, 75.0, 78.5, 76.9	81.8	6.94	8.49
		0.1	5	88.5, 76.9, 76.4, 81.2, 74.2	79.4	5.65	7.12
		Overall	10	Range: 74.2-89.4	80.6	6.09	7.56
	Confirmatory Quantitation (*m/z* 665→645)						
	Broflanilide	0.01	5	76.3, 84.7, 68.6, 63.7, 73.8	73.4	7.98	10.9
		0.1	5	87.4, 76.3, 76.3, 80.6, 73.1	78.8	5.53	7.02
		Overall	10	Range: 63.7-87.4	76.1	7.06	9.28
Liver	Primary Quantitation (*m/z* 663→643)						
	Broflanilide	0.01	5	111, 108, 108, 103, 113	109	3.98	3.66
		0.1	5	108, 99.4, 105, 105, 110	105	3.93	3.73
		Overall	10	Range: 99.4-111	107	4.06	3.80
	Confirmatory Quantitation (*m/z* 665→645)						
	Broflanilide	0.01	5	109, 111, 118, 109, 112	112	3.92	3.51

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean,%	SD,%	RSD, %]
		0.1	5	106, 98.7, 106, 103, 111	105	4.58	4.37
		Overall	10	Range: 98.7-118	108	5.43	5.01
Muscle	Primary Quantitation (*m/z* 663→643)						
	Broflanilide	0.01	5	65.0, 70.1, 78.3, 95.4, 97.4	81.3	14.7	18.0
		0.1	5	107, 94.9, 101, 88.0, 108	99.8	8.51	8.52
		Overall	10	Range: 65.0-108	90.5	15.0	16.5
	Confirmatory Quantitation (*m/z* 665→645)						
	Broflanilide	0.01	5	71.9, 71.8, 76.9, 95.0, 93.2	81.8	11.5	14.0
		0.1	5	105, 93.8, 103, 90.3, 105	99.4	6.87	6.91
		Overall	10	Range: 71.8-105	90.6	12.9	14.2
Milk	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.001	5	89.3, 77.6, 81.5, 84.9, 89.4	84.5	5.09	6.02
		0.01	5	87.3, 90.5, 90.3, 88.1, 89.1	89.1	1.39	1.56
		Overall	10	Range: 77.6-90.5	86.8	4.25	4.90
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.001	5	83.7, 83.5, 79.7, 79.1, 88.2	82.8	3.65	4.41
		0.01	5	86.6, 88.8, 88.1, 89.3, 89.8	88.5	1.27	1.43
		Overall	10	Range: 79.1-89.8	85.7	3.94	4.60
Egg	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.01	5	86.7, 83.9, 78.6, 78.3, 87.1	82.9	4.28	5.16
		0.1	5	85.3, 78.0, 84.1, 83.2, 85.2	83.1	3.00	3.61
		Overall	10	Range: 78.3-87.1	83.0	3.48	4.20
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.01	5	91.0, 84.3, 80.1, 80.1, 75.4	82.2	5.88	7.15
		0.1	5	85.5, 79.2, 82.9, 82.4, 83.9	82.8	2.32	2.80
		Overall	10	Range: 75.4-91.0	82.5	4.22	5.12
Fat	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.01	5	74.1, 77.2, 64.9, 64.4, 71.0	70.3	5.61	7.98
		0.1	5	86.5, 70.8, 72.4, 77.2, 69.4	75.3	6.94	9.22
		Overall	10	Range: 64.4-86.5	72.8	6.49	8.91
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.01	5	77.9, 80.2, 69.2, 68.8, 76.5	74.5	5.20	6.98
		0.1	5	84.7, 70.6, 73.2, 77.1, 68.5	74.8	6.38	8.53
		Overall	10	Range: 68.5-84.7	74.7	5.49	7.35
Liver	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.01	5	103, 105, 95.4, 104, 102	102	3.87	3.79
		0.1	5	104, 96.6, 107, 103, 109	104	4.68	4.50
		Overall	10	Range: 95.4-109	103	4.17	4.05
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.01	5	111, 113, 113, 103, 100	108	6.20	5.74
		0.1	5	107, 97.8, 105, 105, 109	105	4.11	3.93
		Overall	10	Range: 97.8-113	106	5.28	4.96
Muscle	Primary Quantitation (*m/z* 649→242)						
	DM-8007	0.01	5	73.4, 72.2, 82.0, 92.0, 104	84.7	13.3	15.7
		0.1	5	108, 95.5, 101, 88.2, 107	99.8	8.16	8.18
		Overall	10	Range: 72.2-108	92.2	13.1	14.2

Matrix	Analyte	Fortification Level [mg/kg]	n	Recoveries,%	Mean,%	SD,%	RSD, %]
	Confirmatory Quantitation (*m/z* 651→242)						
	DM-8007	0.01	5	72.5, 74.3, 84.3, 99.1, 101	86.3	13.5	15.7
		0.1	5	106, 94.4, 101, 87.2, 106	98.8	7.91	8.01
		Overall	10	Range: 72.5-106	92.6	12.3	13.3
Milk	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.001	5	97.1, 91.2, 95.0, 93.2, 99.7	95.2	3.31	3.48
		0.01	5	94.4, 94.7, 94.3, 93.9, 95.6	94.6	0.65	0.69
		Overall	10	Range: 91.2-99.7	94.9	2.28	2.40
	Confirmatory Quantitation (*m/z* 547→527)						
	DC-DM-8007	0.001	5	91.1, 92.0, 90.5, 92.7, 94.9	92.2	1.72	1.86
		0.01	5	94.4, 95.9, 94.9, 93.2, 95.4	94.7	1.02	1.08
		Overall	10	Range: 91.1-95.9	93.5	1.88	2.01
Egg	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.01	5	89.1, 84.4, 83.5, 82.3, 84.7	84.8	2.58	3.04
		0.1	5	90.7, 89.7, 88.6, 88.1, 86.8	88.8	1.51	1.70
		Overall	10	Range: 82.3-90.7	86.8	2.89	3.33
	Confirmatory Quantitation (*m/z* 547→527)						
	DC-DM-8007	0.01	5	87.7, 88.2, 83.1, 83.6, 83.5	85.2	2.53	2.96
		0.1	5	89.3, 87.1, 89.6, 87.8, 87.8	88.3	1.07	1.21
		Overall	10	Range: 83.1-89.6	86.8	2.45	2.83
Fat	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.01	5	82.2, 83.0, 72.1, 67.4, 75.3	76.0	6.66	8.76
		0.1	5	87.1, 74.5, 76.0, 77.3, 72.4	77.5	5.68	7.33
		Overall	10	Range: 67.4-87.1	76.7	5.88	7.67
	Confirmatory Quantitation (*m/z* 547→527)						
	DC-DM-8007	0.01	5	82.9, 83.0, 73.5, 74.0, 73.7	77.4	5.05	6.52
		0.01	5	87.2, 77.6, 76.2, 77.6, 74.3	78.6	5.01	6.37
		Overall	10	Range: 73.5-87.2	78.0	4.78	6.13
Liver	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.01	5	102, 101, 103, 101, 104	102	1.44	1.41
		0.1	5	106, 97.5, 107, 106, 113	106	5.41	5.12
		Overall	10	Range: 97.5-113			
	Confirmatory Quantitation (*m/z* 547→527)						
	DC-DM-8007	0.01	5	104, 104, 106, 105, 107	105	1.46	1.39
		0.1	5	107, 98.1, 108, 104, 111	106	5.06	4.78
		Overall	10	Range: 98.1-111	106	3.52	3.33
Muscle	Primary Quantitation (*m/z* 545→525)						
	DC-DM-8007	0.01	5	77.6, 82.1, 87.5, 99.6, 107	90.7	12.2	13.4
		0.1	5	109, 98.9, 105, 94.5, 111	104	6.85	6.61
		Overall	10	Range: 77.6-111	97.2	11.6	11.9
	Confirmatory Quantitation (*m/z* 547→527)						
	DC-DM-8007	0.01	5	77.6, 81.8, 89.1, 100, 105	90.7	11.6	12.8
		0.1	5	110, 98.5, 106, 94.1, 111	104	7.41	7.13
		Overall	10	Range: 77.6-111	97.3	11.6	11.9

Stability of pesticides in stored analytical samples

Plant matrices

The storage stability of parent broflanilide and its metabolites S(PFPOH)-8007 and DM-8007 under frozen conditions in lettuce, kidney bean, soya bean, potato and grape was determined over a period up to 24 month. (Delinsky, 2020, BROFLAN_042).

Each analyte was added to homogenized samples at 0.01 mg/kg and stored deep frozen at approximately -20 °C. Storage durations of approximately 0, 3, 5, 9, 12, 15, 18, and 24 months were tested for broflanilide and DM-8007. Metabolite S(PFP-OH)-8007 in potato was tested at storage intervals of 0, 1, 2, 6, 9, 12, 15, 21, and 24 months, while in lettuce, kidney bean, soybean, and grape S(PFP-OH)-8007 has been tested at 0, 1, 2, 6, 7, 9, 15, 21, and 25 months (except for lettuce and kidney bean where the final timepoint was 28 months instead of 25). Due to a fortification error at the first fortification event with S(PFP-OH)-8007, the fortification had to be repeated. All samples were analysed in triplicates according to method D1417/01. The results are in Table 92 to 94.

Table 92 Storage stability of broflanilide and metabolites in plant matrices fortified at 0.01 mg/kg

Crop	A: mean% remaining; B: mean% concurrent recovery									
	Grapes (fruits)		Kidney bean (dried seeds)		Lettuce (leaves)		Potato (tubers)		Soybean (dried seeds)	
Day (Months)	A	B	A	B	A	B	A	B	A	B
Broflanilide										
0 (0)	98	86	99	94[1]	116	108	111	96	101	103
92 – 103 (3)	89	86	99	94[1]	103	108	109	96	103	103
153 – 160 (5)	92	105	88	105	91	107	100	108	89	111
280 – 281 (9)	88	93	86	91	92	96	82	81	89	86
370 – 379 (12)	92	96	97	102	78	81	92	105	95	99
462 – 467 (15)	102	121	94	111	95	107	94	100	86	85
566 – 568 (18)	107	104	96	107	96	104	94	78	91	118
729 – 730 (24)	111	116	112	123	116	126	104	112	110	118
S(PFPOH)-8007										

Crop	A: mean% remaining; B: mean% concurrent recovery									
	Grapes (fruits)		Kidney bean (dried seeds)		Lettuce (leaves)		Potato (tubers)		Soybean (dried seeds)	
Day (Months)	A	B	A	B	A	B	A	B	A	B
0 (0)	77	79	100	100[1]	98	105	96	97	104	109
0 (0)[2)]	112	124	112	113	104	119	na	na	116	114
28 – 30 (1)	98	106	108	105	107	106	106	102	105	100
36 (1)[2)]	105	116	148	123	117	105	na	na	107	104
48 – 56 (2)	112	104	107	109	106	107	121	123	108	107
56 (2)	na	na	na	na	na	na	99	104	na	na
50 (2)[2)]	na	na	111	123	na	na	na	na	na	na
175 – 177 (6)	96	93	92	109	93	90	87	80	91	93
205 – 206 (7)[2)]	110	98	118	114	112	99	na	na	112	108
265 – 274 (9)	106	110	125	116	106	92	106	108	126	111
358 (12)	na	na	na	na	na	na	99	106	na	na
448 – 449 (15)[2)]	123	110	123	112	123	106	na	na	116	110
462 (15)	na	na	na	na	na	na	88	81	na	na
631-632 (21)[2)]	108	99	112	107	112	98	na	na	103	92
625 (21)	na	na	na	na	na	na	110	112	na	na
774 -775 (25)[2)]	113	101	na	na	na	na	na	na	115	110
736 (24)	na	na	na	na	na	na	89	92	na	na
849 – 850 (28)[2)]	na	na	117	102	119	97	na	na	na	na
DM-8007										
0 (0)	83	86	72	80[1]	106	107	96	98	72	78
92 – 103 (3)	91	86	72	80[1]	107	107	108	98	79	78
153 – 153 (5)	109	109	88	109	110	110	124	128	108	107
160 (5)	na	na	na	na	na	na	104	106	na	na
280 – 281 (9)	98	103	79	91	92	100	88	88	114	118
370 – 378 (12)	99	102	104	122	103	94	109	120	97	102
462 – 478 (15)	95	115	108	82	111	103	110	105	93	106
566 – 572 (18)	89	95	106	111	91	103	83	72	83	115
729 – 730 (24)	127	101	99	123	125	127	114	113	133	117

Notes:

na Not applicable

[1)] The duplicate concurrent recovery sample was spiked at the incorrect level and the data were rejected.

[2)] Data from the third fortification event of storage stability samples. All other data are from the second fortification event.

The storage stability of metabolites B-oxam-acid and B-urea under frozen conditions in lettuce, grape, wheat grain, soya bean dried seed and kidney bean dried seed was determined over a period up to 16 month (Delinsky, 2019, BROFLAN_043). In addition, the storage stability of broflanilide and metabolites S(PFP-OH)-8007, DM-8007, B-oxam-acid and B-urea in bee related matrices (honey and pollen) was investigated. The results are shown in Table 93

Each analyte was added separately to homogenized samples at 0.1 mg/kg (honey and pollen: 0.01 mg/kg), stored deep frozen at approximately -20 °C and analysed after storage durations of approximately 0. 2, 5, 9, 12-13, and 16 months. The plant commodity samples were analysed for B-oxam-

acid and B-urea residues using BASF Method D1703/01, while the bee related matrices were analysed according to method L0334/02 (validation not available).

Table 93 Storage stability of broflanilide and metabolites in honey and pollen fortified at 0.01 mg/kg

	A: mean in stored samples, percent of nominal (uncorrected) B: mean in procedural, freshly-spiked samples (concurrent recovery)											
Crop	Honey						Pollen					
Compound	Broflanilide		S(PFP-OH)-8007		DM-8007		Broflanilide		S(PFP-OH)-8007		DM-8007	
Months	A	B	A	B	A	B	A	B	A	B	A	B
0	97	94	100	99	101	92	104	107	122	122	105	113
2	101	94	86	99	87	92	108	108	105	122	95	113
5	95	99	99	99	88	102	96	131	124	114	116	118
9	93	98	86	93	84	96	95	115	106	116	107	118
16	92	93	99	101	87	97	98	99	101	98	87	110

Table 94 Storage stability of B-oxam-acid and B-urea metabolites in honey and pollen, fortified at 0.01 mg/kg, and plant matrices, fortified at 0.1 mg/kg

	A: mean in stored samples, percent of nominal (uncorrected) B: mean in procedural, freshly-spiked samples (concurrent recovery)																					
	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
Crop	Honey			Pollen			Lettuce (leaves)			Grape (fruit)			Wheat (grain)			Soybean (dried seed)			Kidney bean (dry seed)		
Months	A	B		A	B		A	B		A	B		A	B		A	B		A	B	
	B-oxam-acid																					
0	86	85		82	81		89	89		98	96		82	86		84	83		92	92	
2	82	85		79	81		90	89		88	96		80	86		80	83		88	92	
5	95	106		81	85		87	93		90	97		83	88		79	84		79	88	
9	75	81		81	83		89	96		82	101		79	91		87	96		84	98	
12-13	na	na		na	na		82	85		86	92		73	74		76	78		79	80	
16	81	88		75	82		85	87		83	91		78	88		79	75		79	91	
	B-urea																					
0	99	98		99	102		98	95		100	99		97	102		99	98		111	105	
2	102	98		86	102		86	95		100	99		89	102		90	98		87	105	
5	110	117		95	78		87	95		91	94		91	94		91	96		97	95	
9	85	88		85	92		85	94		89	100		94	96		78	91		86	96	
12-13	na	na		na	na		82	92		88	95		92	83		86	92		99	88	
16	113	107		104	112		90	99		87	100		98	106		97	100		93	111	

The storage stability of parent broflanilide and its metabolites S(PFPOH)-8007 and DM-8007 under frozen conditions in Chinese cabbage, cabbage, tomato, radish (root & leaves) and green onion was determined over a period up to 6 month (Hiraki, 2020, BROFLAN_044, Hiraki, 2020, BROFLAN_045).

Each analyte was added to homogenized samples at a rate of 0.1, 0.2, 0.4 or 0.5 mg/kg, stored deep frozen at -20°C and analysed after 20, 104, 119 and 178 days (BROFLAN_044) and 7, 17, 23, 33, 34 and 35 days (BROFLAN_045). All samples were analysed according to the Korean residue method. The results are shown in Table 95

Table 95 Storage stability of broflanilide and metabolites S(PFPOH)-8007 and DM-8007 in plant matrices (Studies BROFLAN_044 and BROFLAN_045)

Analyte	Level [mg/kg]	Days	% remaining			
			Rep 1	Rep 2	Rep 3	Mean ± SD
Chinese cabbage (BROFLAN_044)						
Broflanilide	0.5	104	103.6	99.8	112.7	105.4 ± 6.6
DM-8007	0.5	104	101.8	101.6	103.6	102.3 ± 1.1
S(PFP-OH)-8007	0.5	104	102.7	94.6	108.7	102.0 ± 7.1
Tomato (BROFLAN_044)						
Broflanilide	0.2	20	109.7	110.3	109.6	109.9 ± 0.4
DM-8007	0.2	20	96.6	105.7	104.5	102.3 ± 4.9
S(PFP-OH)-8007	0.2	20	72.2	82.2	87.4	80.6 ± 7.7
Radish (root) (BROFLAN_044)						
Broflanilide	0.2	178	92.3	90.9	91.7	91.6 ± 0.7
DM-8007	0.2	178	95.7	94.5	93.5	94.6 ± 1.1
S(PFP-OH)-8007	0.2	178	92.3	92.4	102.3	95.7 ± 5.7
Radish (leaves) (BROFLAN_044)						
Broflanilide	0.4	179	88.8	91.7	88.4	89.6 ± 1.8
DM-8007	0.4	179	85.8	89.4	91.9	89.0 ± 3.1
S(PFP-OH)-8007	0.4	179	76.8	85.0	80.0	80.6 ± 4.1
Green onion (BROFLAN_044)						
Broflanilide	0.5	119	103.0	107.0	110.0	106.7 ± 3.5
DM-8007	0.5	119	103.4	100.6	100.8	101.6 ± 1.6
S(PFP-OH)-8007	0.5	119	98.8	96.7	98.2	97.9 ± 1.1
Cabbage (BROFLAN_045)						
Broflanilide	0.1	35	81.3	81.4	81.6	81.4 ± 0.2
DM-8007	0.1	35	94.9	95.4	96.0	95.4 ± 0.6
S(PFP-OH)-8007	0.1	35	92.4	92.9	93.6	93.0 ± 0.6
Chinese cabbage (BROFLAN_045)						
Broflanilide	0.1	23	91.3	92.4	85.3	89.7 ± 4.3
DM-8007	0.1	23	91.3	95.2	83.7	90.1 ± 6.5
S(PFP-OH)-8007	0.1	23	99.8	102.4	88.8	97.0 ± 7.4
Radish (root) (BROFLAN_045)						
Broflanilide	0.1	33	74.3	74.8	74.6	74.6 ± 0.3
DM-8007	0.1	33	94.0	94.5	94.0	94.2 ± 0.3
S(PFP-OH)-8007	0.1	33	102.1	102.4	102.3	102.3 ± 0.1
Radish (leaves) (BROFLAN_045)						
Broflanilide	0.1	34	74.6	72.8	73.4	73.6 ± 1.2
DM-8007	0.1	34	104.8	102.3	103.0	103.4 ± 1.2
S(PFP-OH)-8007	0.1	34	115.1	111.9	112.8	113.3 ± 1.5
Green onion (BROFLAN_045)						
Broflanilide	0.1	7	97.0	92.1	94.4	94.5 ± 2.6
DM-8007	0.1	7	86.9	90.3	85.8	87.7 ± 2.7
S(PFP-OH)-8007	0.1	7	89.0	85.3	86.1	86.8 ± 2.2
Tomato (BROFLAN_045)						
Broflanilide	0.1	17	83.1	85.2	80.0	82.8 ± 3.2
DM-8007	0.1	17	79.9	81.5	81.0	80.8 ± 1.0

Analyte	Level [mg/kg]	Days	% remaining			
			Rep 1	Rep 2	Rep 3	Mean ± SD
S(PFP-OH)-8007	0.1	17	83.1	84.7	85.0	84.3 ± 1.2

The storage stability of parent broflanilide and its metabolites S(PFPOH)-8007 and DM-8007 under frozen conditions in radish (root & leaves), turnil (root and leaves) and leek was determined over a period up to 7 month (Kawaguchi, 2020, BROFLAN_046).

Each analyte was added to homogenized samples at a rate of 0.5 mg/kg, stored frozen at -20 °C and analysed after 19 to 206 days of storage. All samples were analysed according to the Japanese residue method. The results are shown in Table 96

Table 96 Storage stability of broflanilide and metabolites S(PFPOH)-8007 and DM-8007 in plant matrices (BROFLAN_046)

Matrix	Days	% remaining	Days	% remaining	Days	% remaining
Broflanilide			DM-8007		S(PFPOH)-8007	
Leek	21	92	19	95	19	99
	19	94	21	92	21	95
	32	93	32	92	32	96
	161	98	161	102	141	104
	162	99	162	98	141	102
	204	89	169	96	141	102
Radish (leaf)	39	96	39	96	39	98
	40	96	40	96	40	96
	166	97	166	93	160	96
	176	97	176	98	160	93
	194	95	188	96	160	98
	206	104	188	91	176	92
Radish (root)	39	94	39	98	39	97
	40	95	40	88	40	96
	166	103	166	104	160	103
	176	96	176	98	160	103
	194	109	188	98	160	102
	206	102	188	102	176	93
Turnip (leaf)	33	98	33	94	33	98
	84	96	84	94	84	96
	99	96	99	90	99	93
Turnip (root)	33	90	33	92	33	88
	84	90	84	93	84	88
	99	96	99	88	99	88

Animal matrices

The storage stability of parent broflanilide and its metabolites DM-8007 and DC-DM-8007 under frozen conditions in muscle, liver, kidney, milk and fat was determined over a period up to 2 month with a dairy cow feeding study. (Xu, 2019, BROFLAN_082).

For each freezer storage interval, duplicate samples were fortified at 0.01 mg/kg (milk, fat) or 0.1 mg/kg (muscle, liver, kidney), placed in the freezer at -20 ºC and analysed after 0, 1 and 2 months. All samples were analysed in duplicates according to method D1604/01. The results are shown in Table 97.

Table 97 Storage stability of broflanilide in animal matrices

	A: mean in stored samples, percent of nominal (uncorrected) B: mean in procedural, freshly-spiked samples (concurrent recovery)									
	Muscle		Liver		Kidney		Milk		Fat	
Months	A	B	A	B	A	B	A	B	A	B
	Broflanilide									
0	120	94.1	92.9	93.1	92.6	91.0	97.3	96.5	120	94.1
1	107	115	88.3	94.0	81.3	90.9	86.0	86.3	107	115
2	78.5	88.3	93.5	102	93.5	102	94.6	104	78.5	88.3
	DM-8007									
0	93.3	90.5	91.2	94.2	88.7	93.1	93.3	89.1	121	88.3
1	90.4	95.2	86.9	97.9	83.4	91.0	82.6	78.9	94.0	98.3
2	112	119	110	118	111	121	106	122	98.1	106
	DC-DM-8007									
0	92.7	93.2	90.7	95.7	90.5	90.0	92.9	93.7	115	93.1
1	67.3	96.5	76.5	101	49.1	94.7	80.3	83.1	106	116
2	59.4	101	51.1	97.9	29.4	91.9	89.9	99.3	74.4	87.6

USE PATTERN

GAP information taken from the submitted labels, for all crops supported with residue data is summarized in Table 98.

Table 98 List of uses of broflanilide

Crop/ Commodity	Country	Formulation		Application				PHI (days)
		Active substance content	Type	Method	Rate	Water volume (L/ha)	No or Seasonal max. (interval)	
Welsh onion (including spring onion)	Republic of Korea	5 percent (w/w)	SC	Foliar spray	2.5 g ai/hL		3 (10 days)	7
Welsh onion (including spring onion)	Republic of Korea	5 percent (w/w)	EC	Foliar spray	2.5 g ai/hL		3 (10 days)	7
Subgroup of green onion	Japan	5 percent (w/w)	SC	Foliar spray	1.25-2.5 g ai/hL	1000-3000	3 (not stated)	1
Cabbage	Republic of Korea	5 percent (w/w)	EC	Foliar spray	2.5 g ai/hL		2 (not stated)	14
Cabbage	China	100 g/L	SC	Foliar spray	10.5-24 g ai/ha		1	5
Cabbage	Japan	5 percent (w/w)	SC	Foliar spray	1.25-2.5 g ai/hL	1000-3000	3 (not stated)	1

Crop/ Commodity	Country	Formulation		Application				PHI (days)
		Active substance content	Type	Method	Rate	Water volume (L/ha)	No or Seasonal max. (interval)	
Cabbage	Japan	20 percent (w/w)	SC	Foliar spray	1.25-2.5 g ai/hL	1000-3000	3 (not stated)	1
Chinese cabbage	Republic of Korea	5 percent (w/w)	SC	Foliar spray	2.5 g ai/hL		2 (not stated)	14
Chinese cabbage	Republic of Korea	5 percent (w/w)	EC	Foliar spray	2.5 g ai/hL	1500	2 (not stated)	14
Chinese cabbage	China	100 g/L	SC	Foliar spray	10.5-24 g ai/ha		1	5
Chinese cabbage	Japan	5 percent (w/w)	SC	Foliar spray	1.25-2.5 g ai/hL	1000-3000	3 (not stated)	1
Tomato (including cherry tomato)	Republic of Korea	5 percent (w/w)	EC	Foliar spray	2.5 g ai/hL		2 (7 days)	2
Japanese radish	Republic of Korea	5 percent (w/w)	EC	Foliar spray	2.5 g ai/hL		3 (not stated)	14
Radish	Japan	5 percent (w/w)	SC	Foliar spray	1.25-2.5 g ai/hL	1000-3000	3 (not stated)	1
Subgroup of tuberous and corm vegetables	United States	100 g/L	SC	Soil application (in furrow)	22-50 g ai/ha	min. 46.8	1	F
Subgroup of Tuberous and corm vegetables	United States	300 g/L	SC	Soil application (in furrow)	22-50 g ai/ha	min. 46.8	1	F
Potato	Canada	100 g/L	SC	Soil application (in furrow)	25 g ai/ha	min. 46.8	1	F
Sweet potato	Japan	5 percent (w/w)	SC	Foliar spray	1.25-2.5 g ai/hL	1000-3000	3 (not stated)	1
Cereals (barley, oat, wheat, triticale, rye, millet, sorghum, amaranth, buckwheat, cañihua, chia, cram-cram, huauzontle, quinoa, spelt)	United States	300 g/L	FS	Seed treatment	50 g ai/t		1	F
Cereals (barley, oat, wheat, triticale, rye, millet, sorghum)	United States	16.7 g/L	FS	Seed treatment	50 g ai/t		1	F
Cereals (barley, oat, wheat, triticale, rye, millet, sorghum, canary seed, buckwheat)	Canada	300 g/L	FS	Seed treatment	50 g ai/t		1	F
Cereals (barley, oat, wheat, triticale, rye, canary seed)	Canada	16.7 g/L	FS	Seed treatment	50 g ai/t		1	F
Maize, including sweet corn	United States	100 g/L	SC	Soil application (in furrow)	16-50 g ai/ha	min. 46.8	1	F

Crop/ Commodity	Country	Formulation		Application				PHI (days)
		Active substance content	Type	Method	Rate	Water volume (L/ha)	No or Seasonal max. (interval)	
Maize, including sweet corn	United States	300 g/L	SC	Soil application (in furrow)	15-50 g ai/ha	min. 46.8	1	F
Maize, including sweet corn	Canada	100 g/L	SC	Soil application (in furrow)	25 g ai/ha	min. 50	1	F
Coffee	Colombia	100 g/L	SC	Foliar spray	18 g ai/ha		2 (30 days)	45

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as broflanilide equivalents. When residues were not detected they are shown as below the LOQ, e.g., < 0.01 mg/kg. Application rates, spray concentrations and mean residue results have generally been rounded to two significant figures. Values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are underlined.

Laboratory reports included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for percent recovery. A summary of the residue trials is shown in Table 98.

Table 99 Broflanilide – supervised residue trials

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Green onion (Welsh onion)	Indoor	Foliar spray	Republic of Korea	Table 100
Leek	Outdoor	Foliar spray	Japan	Table 101
Cabbage	Indoor & outdoor	Foliar spray	United States, Canada, South Korea, China	Table 102
Chinese cabbage	Indoor & outdoor	Foliar spray	Republic of Korea, China	Table 103
Tomatoes	Indoor & outdoor	Foliar spray	United States,	Table 104
Radish	Indoor	Foliar spray	Republic of Korea	Table 105
Japanese radish	Outdoor	Foliar spray	Japan	Table 106
Turnip	Indoor	Foliar spray	Japan	Table 107
Potato	Outdoor	Foliar spray & in-furrow	United States, Canada	Table 108
Wheat	Outdoor	Seed treatment	United States, Canada	Table 109
Barley	Outdoor	Seed treatment	United States, Canada	Table 110
Maize	Outdoor	In-furrow	United States, Canada	Table 111
Coffee	Outdoor	Foliar spray	Brazil, Colombia	Table 112
Turnip tops	Indoor	Foliar spray	Japan	Table 113

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Wheat forage, hay and straw	Outdoor	Seed treatment	United States, Canada	Table 114
Maize forage and stover	Outdoor	In-furrow	United States, Canada	Table 115
Barley hay and straw	Outdoor	Seed treatment	United States, Canada	Table 116

Bulb vegetables

Green onion (Welsh onion)

Two greenhouse trials were conducted with green onion in the Republic of Korea in the 2017 growing season (BROFLAN_047, BROFLAN_048). Plants received 3 spray applications of broflanilide at nominal rates of 2.5 g ai/hL (37.5-50 g ai/ha) with a 7 day interval between applications. Green onions were collected at 0, 7 and 14 DALA (in one trial samples were also taken additionally at 21 DALA). Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using the Korean residue method with a validated limit of quantification of 0.1 mg/kg and a limit of detection of 0.01 mg/kg.

Additionally, a total of three field trials were conducted with green onion in Japan in the 2013 and 2014 growing seasons (BROFLAN_049, BROFLAN_050). Plants received 3 spray applications of broflanilide at nominal rates of 2.5 g ai/hL with a 7±1 day interval between applications. Leeks were collected at 1, 3 and 7 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using the Japanese residue method with a LOQ of 0.01 mg/kg. The results are shown in Table 100.

Table 100 Residues of broflanilide in green onion following foliar treatment

Location, Year, Trial No., (variety)	Application			DALA	Residues found [mg/kg]			Report, Reference, Storage period
	N (RTI)	g ai/hL	GS at final appl.		Broflanilide[1]	DM-8007	S(PFP-OH)-8007	
Japan, Kochi, 2013, JP2013C277B (Kujofuto)	3 (7)	2.5	Harvest	1 3 7	0.31 <u>0.38</u> 0.13	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	2020/2090147 Nakamura, 2015, BROFLAN_049 Storage time: max. 132 days; Procedural recoveries: 90.0-113% at 0.01 mg/kg (n=6) & 0.5 mg/kg (n=6)
Japan, Miyazaki, 2013, JP2013C277C (Kujofuto)	3 (7)	2.5	Early harvest	1 3 7	0.40 <u>0.46</u> 0.19	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	
Japan, Kagoshima, 2014, JP2014C129C (Asagi-kei Kujo)	3 (6-7)	2.5	Mid of growing stage	1 3 7	<u>1.32</u> 0.77 0.28	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	2020/2090150 Nakamura, 2015, BROFLAN_050 Storage time: max. 25 days; Procedural recoveries: 93-98% at 2 mg/kg (n=6)
Republic of Korea, 41-6,Sinryewon-ro, Yesan-eup, Yesan-gun, Chungcheongnam-do, 2017, DBA-RC-2017-017 (not stated)	3 (7)	2.5	Not stated	0 7 14	1.39, 1.39, 1.38 (1.39) 0.82, 0.80, 0.80 (0.81) 0.32, 0.31, 0.33 (0.32)	< 0.01 (3) < 0.01 (3) < 0.01 (3)	< 0.01 (3) < 0.01 (3) < 0.01 (3)	2020/2090142 Yoon, 2018, BROFLAN_047 Storage time: 119 days; Procedural recoveries: 83.3-113.1% at 0.1 and 0.5 mg/kg (n=3)

Location, Year, Trial No., (variety)	Application			DALA	Residues found [mg/kg]			Report, Reference, Storage period
	N (RTI)	g ai/hL	GS at final appl.		Broflanilide[1]	DM-8007	S(PFP-OH)-8007	
Republic of Korea, 527 Changsori, Yesan-eup, Yesan-gun, Chungcheongnam-do, 2017, SGR-2017-107 (not stated)	3 (7)	2.5	Not stated	0	0.48, 0.47, 0.46 (0.47)	< 0.01 (3)	< 0.01 (3)	2020/2090145 Park, 2018, BROFLAN_048 Storage time: 7 days Procedural recoveries: 84.0-101.5% at 0.1 and 0.5 mg/kg (n=3)
				7	0.41, 0.41, 0.42 (0.41)	< 0.01 (3)	< 0.01 (3)	
				14	0.14, 0.14, 0.14 (0.14)	< 0.01 (3)	< 0.01 (3)	
				21	0.07, 0.07, 0.07 (0.07)	< 0.01 (3)	< 0.01 (3)	

Notes:

GS= growth stage; RTI= repeated treatment interval, days

[1] values in parentheses represent mean values.

Leek

A total of three field trials were conducted with leek in Japan in the 2013 and 2014 growing seasons (BROFLAN_049, BROFLAN_050). Plants received 3 spray applications of broflanilide at nominal rates of 2.5 g ai/hL with a 7±1 day interval between applications. Leeks were collected at 1, 3 and 7 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using the Japanese residue method with a LOQ of 0.01 mg/kg. The results are shown in Table 101.

Table 101 Residues of broflanilide in leek following foliar treatment

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/hL	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
Japan, Ibaraki, 2013, JP2013C277A (Shuitsu)	3 (6-8)	2.5	Early harvest	Leek	1	0.20	< 0.02	< 0.01	2020/2090147 Nakamura, 2015, BROFLAN_049 Storage time: max. 132 days; procedural recoveries: 90.0-113% at 0.01 and 0.5 mg/kg
					3	0.16	< 0.02	< 0.01	
					7	0.14	< 0.02	< 0.01	
Japan, Aomori, 2014, JP2014C129A (Natsu Ohgi Power)	3 (7-8)	2.5	Late stage of growth	Leek	1	0.22	< 0.02	< 0.01	2020/2090150 Nakamura, 2015, BROFLAN_050 Storage time: max. 25 days Procedural recoveries: 93-98% at 2 mg/kg (n=6)
					3	0.14	< 0.02	< 0.01	
					7	0.07	< 0.02	< 0.01	
Japan, Ishikawa, 2014, JP2014C129B (White star)	3 (7)	2.5	Growing stage	Leek	1	0.10	< 0.02	< 0.01	
					3	0.07	< 0.02	< 0.01	
					7	0.04	< 0.02	< 0.01	

Notes:

GS= growth stage; RTI= repeated treatment interval, days

[1] Mean from 2 analytical replicate samples. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

Brassica vegetables

Cabbage

A total of 10 field trials were conducted with cabbage in Canada and the United States during the 2015 and 2016 growing seasons (BROFLAN_051). Plants received 2 spray applications of broflanilide at nominal rates of 25 g ai/ha (water volume ranged from 283–446 L/ha) with a 6–8 day interval between applications. Samples were collected at 1 day after the last application. Additionally, in one decline trial, plants were also collected at 0, 1, 3, 7 and 14 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg.

One greenhouse trial was conducted with cabbage in Republic of Korea in the 2018 growing season (BROFLAN_052). Plants received 3 spray applications of broflanilide at nominal rates of 37.5–50 g ai/ha (water volume: 2000 L/ha) with a 7 day interval between applications. Plants were collected at 7, 14, 21, and 30 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using the Korean residue method with a validated limit of quantification of 0.1 mg/kg and a limit of detection of 0.01 mg/kg.

A total of 24 field trials were conducted with cabbage in China during the 2015–2018 growing seasons (BROFLAN_053, BROFLAN_054, BROFLAN_055, BROFLAN_056). Plants received 2 or 3 spray applications of broflanilide at nominal rates of 22.5–45 g ai/ha (water volume: 750–900 L/ha), with a 7 day interval between applications. Plants were collected at (0, 1, 2), 3, 5 and 7 DALA. Additionally, some decline trials were performed with 1 application at 33.8 or 45 g ai/ha, were samples were collected at various time points, up to 21 days. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using a method similar to D1417/01 with a LOQ of 0.01 mg/kg for broflanilide and 0.001 mg/kg or 0.01 mg/kg for metabolites DM-8007 and S(PFP-OH)-8007. The results are in Table 102.

Table 102 Residues of broflanilide in cabbage following foliar treatment in China the Republic of Korea and the United States

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
China, Liaoning Province, 2018 (Lvyuan)	2 (7)	25	45	Cabbage head	0	0.02	< 0.01	< 0.01	2020/2090096 Min, 2019, BROFLAN_053 Storage time: <8 months Procedural recoveries: 72-118% at 0.01-0.5 mg/kg
					1	< 0.01	< 0.01	< 0.01	
					3	0.07	< 0.01	< 0.01	
					5	0.04	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
China, Shanxi Province, 2018 (Zhonggan 56)	2 (7)	25	45	Cabbage head	0	< 0.01	< 0.01	< 0.01	
					1	< 0.01	< 0.01	< 0.01	
					3	< 0.01	< 0.01	< 0.01	
					5	< 0.01	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
China Anhui Province, 2018 (Xiaguang)	2 (7)	25	45	Cabbage head	0	0.58	< 0.01	< 0.01	
					1	0.49	< 0.01	< 0.01	
					3	0.43	< 0.01	< 0.01	
					5	0.17	< 0.01	< 0.01	
					7	0.10	< 0.01	< 0.01	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
China Guizhou Province, 2018 (Jingfend Yihao)	2 (7)	25	45	Cabbage head	0 1 3 5 7	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	
China Ningxia Hui Autonomous Region, 2018 (Lvbao)	2 (7)	25	45	Cabbage head	3 5 7	0.04 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	
China Jiangsu Province, 2018 (Chunfeng)	2 (7)	25	45	Cabbage head	3 5 7	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	
China Zhuliu Town, Changle County, Weifang City, Shandong Province 2015 (Zhonggan 21)	2 (7)	30	Not stated	Cabbage head	3 5 7	0.284 0.155 0.149	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2020/2090099 Yongquan, 2017, BROFLAN_054 Storage time: <20 months Procedural recoveries: 75.5-109.2% at 0.01-1.0 mg/kg
	3 (7)	30	Not stated	Cabbage head	3 5 7	0.480 0.353 0.278	0.022 < 0.01 < 0.01	0.021 < 0.01 < 0.01	
	2 (7)	45	Not stated	Cabbage head	3 5 7	0.642 0.467 0.239	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	
	3 (7)	45	Not stated	Cabbage head	3 5 7	1.081 1.334 0.843	0.029 < 0.01 < 0.01	0.016 0.014 < 0.01	
	1	45	Not stated	Cabbage head	0 1 2 3 5 7 14 21	1.198 1.119 0.788 0.713 <u>0.483</u> 0.450 0.385 0.167	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	
China Zhuliu Town, Changle County, Weifang City, Shandong Province 2016 (Zhonggan 21)[2]	2 (7)	30	Not stated	Cabbage head	3 5 7	0.910 0.615 0.248	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	
	3 (7)	30	Not stated	Cabbage head	3 5 7	1.343 0.968 0.557	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	
	2 (7)	45	Not stated	Cabbage head	3 5 7	2.277 1.130 0.714	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	
	3 (7)	45	Not stated	Cabbage head	3 5 7	2.910 1.743 0.891	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
	1	45	Not stated	Cabbage head	0	4.466	< 0.01	< 0.01	
					1	4.498	< 0.01	< 0.01	
					2	3.915	< 0.01	< 0.01	
					3	1.670	< 0.01	< 0.01	
					5	1.590	< 0.01	< 0.01	
					7	0.406	< 0.01	< 0.01	
					14	0.359	< 0.01	< 0.01	
					21	0.255	< 0.01	< 0.01	
China Songzhuang Village, Qinyang City, Henan Province, 2015 (Moyu 50)	2 (7)	30	Not stated	Cabbage head	3	0.927	< 0.01	< 0.01	
					5	0.517	< 0.01	0.012	
					7	0.244	< 0.01	< 0.01	
	3 (7)	30	Not stated	Cabbage head	3	1.242	< 0.01	0.016	
					5	0.851	< 0.01	< 0.01	
					7	0.376	< 0.01	< 0.01	
	2 (7)	45	Not stated	Cabbage head	3	1.109	< 0.01	< 0.01	
					5	0.795	0.028	0.018	
					7	0.344	< 0.01	0.016	
	3 (7)	45	Not stated	Cabbage head	3	1.589	0.051	0.025	
					5	1.328	0.031	0.022	
					7	0.827	< 0.01	0.017	
	1	45	Not stated	Cabbage head	0	1.256	< 0.01	< 0.01	
					1	1.042	< 0.01	< 0.01	
					2	0.442	< 0.01	< 0.01	
					3	0.362	< 0.01	< 0.01	
					5	0.329	< 0.01	< 0.01	
					7	0.279	< 0.01	< 0.01	
					14	0.244	< 0.01	< 0.01	
					21	0.127	< 0.01	< 0.01	
China Songzhuang Village, Qinyang City, Henan Province, 2016 (Moyu 50)	2 (7)	30	Not stated	Cabbage head	3	0.602	< 0.01	< 0.01	
					5	0.273	< 0.01	< 0.01	
					7	0.464	< 0.01	< 0.01	
	3 (7)	30	Not stated	Cabbage head	3	0.772	< 0.01	< 0.01	
					5	0.479	< 0.01	< 0.01	
					7	0.386	< 0.01	< 0.01	
	2 (7)	45	Not stated	Cabbage head	3	1.001	< 0.01	< 0.01	
					5	0.813	< 0.01	< 0.01	
					7	0.646	< 0.01	< 0.01	
	3 (7)	45	Not stated	Cabbage head	3	1.407	0.023	0.014	
					5	0.866	< 0.01	< 0.01	
					7	0.648	< 0.01	< 0.01	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
	1	45	Not stated	Cabbage head	0	4.055	< 0.01	< 0.01	
					1	4.192	< 0.01	< 0.01	
					2	3.750	< 0.01	< 0.01	
					3	3.368	< 0.01	< 0.01	
					5	1.581	< 0.01	< 0.01	
					7	0.753	< 0.01	< 0.01	
					14	0.429	< 0.01	< 0.01	
					21	0.315	< 0.01	< 0.01	
China Xinqiao Town, Zhangjiajie City, Hunan Province, 2015 (Jingfeng No.1)	2 (7)	30	Not stated	Cabbage head	3	1.105	< 0.01	< 0.01	
					5	0.545	< 0.01	< 0.01	
					7	0.218	< 0.01	< 0.01	
	3 (7)	30	Not stated	Cabbage head	3	1.180	0.032	0.017	
					5	0.641	< 0.01	< 0.01	
					7	0.323	< 0.01	< 0.01	
	2 (7)	45	Not stated	Cabbage head	3	1.190	< 0.01	< 0.01	
					5	0.872	< 0.01	< 0.01	
					7	0.398	< 0.01	0.015	
	3 (7)	45	Not stated	Cabbage head	3	1.711	0.056	0.033	
					5	1.413	0.040	0.023	
					7	0.847	0.022	< 0.01	
	1	45	Not stated	Cabbage head	0	1.232	< 0.001	< 0.01	
					1	0.987	< 0.01	< 0.01	
					2	0.683	< 0.01	< 0.01	
					3	0.352	< 0.01	< 0.01	
					5	0.314	< 0.01	< 0.01	
					7	0.269	< 0.01	< 0.01	
					14	0.245	< 0.01	< 0.01	
					21	0.131	< 0.01	< 0.01	
China Xinqiao Town, Zhangjiajie City, Hunan Province, 2016 (Jingfeng No.1)[2]	2 (7)	30	Not stated	Cabbage head	3	0.293	< 0.01	< 0.01	
					5	0.102	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
	3 (7)	30	Not stated	Cabbage head	3	0.320	< 0.01	< 0.01	
					5	0.208	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
	2 (7)	45	Not stated	Cabbage head	3	0.444	< 0.01	< 0.01	
					5	0.279	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
	3 (7)	45	Not stated	Cabbage head	3	0.680	< 0.01	< 0.01	
					5	0.392	< 0.01	< 0.01	
					7	0.089	< 0.01	< 0.01	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
	1	45	Not stated	Cabbage head	0	1.132	< 0.01	< 0.01	
					1	1.063	< 0.01	< 0.01	
					2	0.621	< 0.01	< 0.01	
					3	0.224	< 0.01	< 0.01	
					5	0.122	< 0.01	< 0.01	
					7	0.114	< 0.01	< 0.01	
					14	0.095	< 0.01	< 0.01	
					21	0.041	< 0.01	< 0.01	
China Zhuliu Town, Changle County, Weifang City, Shandong Province 2016 (Zhonggan 21)[2]	2 (7)	22.5	Not stated	Cabbage head	3	1.012	< 0.001	0.007	2020/2090106 Yongquan, 2017, BROFLAN_055 Storage time: <1 months Procedural recoveries: 74.1-111.6% at 0.001-1.0 mg/kg
					5	0.820	< 0.001	0.005	
					7	0.245	< 0.001	< 0.001	
	3 (7)	22.5	Not stated	Cabbage head	3	1.180	0.001	0.008	
					5	0.941	< 0.001	0.002	
					7	0.337	< 0.001	0.003	
	2 (7)	33.8	Not stated	Cabbage head	3	1.694	0.001	0.010	
					5	0.864	0.001	0.002	
					7	0.550	< 0.001	0.003	
	3 (7)	33.8	Not stated	Cabbage head	3	1.928	0.002	0.011	
					5	0.951	0.001	0.004	
					7	0.723	< 0.001	0.004	
	1	33.8	Not stated	Cabbage head	0	0.494	< 0.001	< 0.001	
					1	0.527	< 0.001	0.004	
					3	0.206	< 0.001	0.001	
					5	0.117	< 0.001	< 0.001	
					7	0.086	< 0.001	< 0.001	
					10	0.123	< 0.001	< 0.001	
					14	0.050	< 0.001	< 0.001	
					21	0.046	< 0.001	< 0.001	
China Zhuliu Town, Changle County, Weifang City, Shandong Province 2017 (Zhonggan 21)	2 (7)	22.5	Not stated	Cabbage head	3	0.319	0.001	0.001	
					5	0.342	0.001	< 0.001	
					7	0.144	< 0.001	< 0.001	
	3 (7)	22.5	Not stated	Cabbage head	3	0.349	0.001	0.004	
					5	0.341	0.001	0.003	
					7	0.187	< 0.001	0.002	
	2 (7)	33.8	Not stated	Cabbage head	3	0.616	0.001	0.004	
					5	0.581	0.001	0.003	
					7	0.517	< 0.001	0.003	
	3 (7)	33.8	Not stated	Cabbage head	3	0.703	0.001	0.006	
					5	0.628	0.002	0.005	
					7	0.615	< 0.001	0.004	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
	1	33.8	Not stated	Cabbage head	0	0.513	< 0.001	0.001	
					1	0.516	< 0.001	0.003	
					3	0.322	< 0.001	0.002	
					5	0.160	< 0.001	< 0.001	
					7	0.124	< 0.001	< 0.001	
					10	0.110	< 0.001	< 0.001	
					14	0.023	< 0.001	< 0.001	
					21	0.012	< 0.001	< 0.001	
China Fucheng Town, Fengyang County, Anhui Province 2016 (Huamei)	2 (7)	22.5	Not stated	Cabbage head	3	0.909	0.001	0.004	
					5	0.709	< 0.001	0.003	
					7	0.381	< 0.001	< 0.001	
	3 (7)	22.5	Not stated	Cabbage head	3	1.056	0.001	0.008	
					5	0.826	< 0.001	0.006	
					7	0.414	< 0.001	0.004	
	2 (7)	33.8	Not stated	Cabbage head	3	1.670	0.001	0.009	
					5	0.782	< 0.001	0.005	
					7	0.571	< 0.001	0.004	
	3 (7)	33.8	Not stated	Cabbage head	3	1.836	0.001	0.010	
					5	0.840	< 0.001	0.005	
					7	0.652	< 0.001	0.003	
	1	33.8	Not stated	Cabbage head	0	0.452	< 0.001	< 0.001	
					1	0.485	< 0.001	0.004	
					3	0.189	< 0.001	0.001	
					5	0.109	< 0.001	< 0.001	
					7	0.080	< 0.001	< 0.001	
					10	0.111	< 0.001	< 0.001	
					14	0.049	< 0.001	< 0.001	
					21	0.045	< 0.001	< 0.001	
China Fucheng Town, Fengyang County, Anhui Province 2017 (Chunfeng)	2 (7)	22.5	Not stated	Cabbage head	3	0.146	< 0.001	0.001	
					5	0.113	< 0.001	< 0.001	
					7	0.079	< 0.001	< 0.001	
	3 (7)	22.5	Not stated	Cabbage head	3	0.175	0.001	0.001	
					5	0.138	< 0.001	< 0.001	
					7	0.106	< 0.001	< 0.001	
	2 (7)	33.8	Not stated	Cabbage head	3	0.180	0.001	0.001	
					5	0.130	< 0.001	< 0.001	
					7	0.080	< 0.001	< 0.001	
	3 (7)	33.8	Not stated	Cabbage head	3	0.207	0.001	0.002	
					5	0.181	< 0.001	0.001	
					7	0.123	< 0.001	< 0.001	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
	1	33.8	Not stated	Cabbage head	0	0.933	< 0.001	0.002	
					1	1.008	< 0.001	0.003	
					3	0.564	< 0.001	0.002	
					5	0.420	< 0.001	0.001	
					7	0.348	< 0.001	< 0.001	
					10	0.134	< 0.001	< 0.001	
					14	< 0.01	< 0.001	< 0.001	
					21	< 0.01	< 0.001	< 0.001	
China Xinqiao Town, Zhangjiajie City, Hunan Province, 2016 (Jingfeng No.1)[2]	2 (7)	22.5	Not stated	Cabbage head	3	< 0.01	< 0.001	< 0.001	
					5	< 0.01	< 0.001	< 0.001	
					7	< 0.01	< 0.001	< 0.001	
	3 (7)	22.5	Not stated	Cabbage head	3	< 0.01	< 0.001	< 0.001	
					5	< 0.01	< 0.001	< 0.001	
					7	< 0.01	< 0.001	< 0.001	
	2 (7)	33.8	Not stated	Cabbage head	3	< 0.01	< 0.001	< 0.001	
					5	< 0.01	< 0.001	< 0.001	
					7	< 0.01	< 0.001	< 0.001	
	3 (7)	33.8	Not stated	Cabbage head	3	< 0.01	< 0.001	< 0.001	
					5	< 0.01	< 0.001	< 0.001	
					7	< 0.01	< 0.001	< 0.001	
	1	33.8	Not stated	Cabbage head	0	0.045	< 0.001	< 0.001	
					1	0.043	< 0.001	< 0.001	
					3	0.030	< 0.001	< 0.001	
					5	0.029	< 0.001	< 0.001	
					7	0.012	< 0.001	< 0.001	
					10	< 0.01	< 0.001	< 0.001	
					14	< 0.01	< 0.001	< 0.001	
					21	< 0.01	< 0.001	< 0.001	
China Xinqiao Town, Zhangjiajie City, Hunan Province, 2017 (Jingfeng No.1)	2 (7)	22.5	Not stated	Cabbage head	3	< 0.01	< 0.001	< 0.001	
					5	< 0.01	< 0.001	< 0.001	
					7	< 0.01	< 0.001	< 0.001	
	3 (7)	22.5	Not stated	Cabbage head	3	< 0.01	< 0.001	< 0.001	
					5	< 0.01	< 0.001	< 0.001	
					7	< 0.01	< 0.001	< 0.001	
	2 (7)	33.8	Not stated	Cabbage head	3	< 0.01	< 0.001	< 0.001	
					5	< 0.01	< 0.001	< 0.001	
					7	< 0.01	< 0.001	< 0.001	
	3 (7)	33.8	Not stated	Cabbage head	3	< 0.01	< 0.001	< 0.001	
					5	< 0.01	< 0.001	< 0.001	
					7	< 0.01	< 0.001	< 0.001	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
	1	33.8	Not stated	Cabbage head	0	0.035	< 0.001	< 0.001	
					1	0.020	< 0.001	< 0.001	
					3	0.010	< 0.001	< 0.001	
					5	< 0.01	< 0.001	< 0.001	
					7	< 0.01	< 0.001	< 0.001	
					10	< 0.01	< 0.001	< 0.001	
					14	< 0.01	< 0.001	< 0.001	
					21	< 0.01	< 0.001	< 0.001	
China Zhaoqing City, Guangdong Province 2018 (Sijipingtou)	2 (7)	22.5	Not stated	Cabbage head	2h	0.158	< 0.01	< 0.01	2020/2090110 Yongquan, 2019, BROFLAN_056 Storage time: 7 months Procedural recoveries: 72-105% at 0.01-1 mg/kg
					3	0.054	< 0.01	< 0.01	
					5	0.042	< 0.01	< 0.01	
					7	0.019	< 0.01	< 0.01	
					10	0.013	< 0.01	< 0.01	
China Jinzhong City, Shanxi Province 2018 (Xingshu 608)	2 (7)	22.5	Not stated	Cabbage head	2h	0.124	< 0.01	< 0.01	
					3	0.052	< 0.01	< 0.01	
					5	0.037	< 0.01	< 0.01	
					7	0.018	< 0.01	< 0.01	
					10	0.014	< 0.01	< 0.01	
China, Guoxian, Tongzhou District, Beijing 2018 (Qiuyulvli)	2 (7)	22.5	Not stated	Cabbage head	2h	0.137	< 0.01	< 0.01	
					3	0.048	< 0.01	< 0.01	
					5	0.037	< 0.01	< 0.01	
					7	0.019	< 0.01	< 0.01	
					10	0.014	< 0.01	< 0.01	
China, Guiyang City, Guizhou Province 2018 (Qiangan No.1)	2 (7)	22.5	Not stated	Cabbage head	2h	0.151	< 0.01	< 0.01	
					3	0.053	< 0.01	< 0.01	
					5	0.038	< 0.01	< 0.01	
					7	0.020	< 0.01	< 0.01	
					10	0.014	< 0.01	< 0.01	
China Changchun, Jilin Province, 2018 (Jingke)	2 (7)	22.5	Not stated	Cabbage head	3	0.058	< 0.01	< 0.01	
					5	0.049	< 0.01	< 0.01	
					7	0.014	< 0.01	< 0.01	
					10	0.013	< 0.01	< 0.01	
China Kunming City, Yunnan Province 2018 (Niuxin)	2 (7)	22.5	Not stated	Cabbage head	3	0.048	< 0.01	< 0.01	
					5	0.039	< 0.01	< 0.01	
					7	0.014	< 0.01	< 0.01	
					10	0.013	< 0.01	< 0.01	
Canada, QC, St-Marc-sur-Richelieu, 2015 R150120 (Bronco)	2 (7)	25 25	47-49	With wrapper leaves	1	0.0082	< 0.001	< 0.001	2016/7009963 Schreier, 2017, BROFLAN_051 Storage time: max. 479 days (~16 months) Procedural recoveries: 98.0-116% at 0.001-0.4 mg/kg
				Without wrapper leaves	1	0.0014	< 0.001	< 0.001	
United States, NY, Alton, 2015 R150114 (Farao)	2 (7)	25 25	49	With wrapper leaves	0	0.097	< 0.001	0.0011	
					1	0.170	0.021	0.0016	
					3	0.106	0.0018	0.0014	
					7	0.100	0.0023	0.0016	
					14	0.056	< 0.001	0.0011	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
				Without wrapper leaves	0 1 3 7 14	0.013 0.0089 0.0062 0.0035 0.0024	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	
United States, GA, Weston, 2015 R150115 (Blue Vintage)	2 (7)	25 27	49	With wrapper leaves	1	0.0042	< 0.001	< 0.001	
				Without wrapper leaves	1	0.0089	< 0.001	< 0.001	
United States, FL, Bradeton, 2015 R150116 (Bravo)	2 (7)	28 25	Headed cabbage	With wrapper leaves	1	0.047	< 0.001	< 0.001	
				Without wrapper leaves	1	0.0020	< 0.001	< 0.001	
United States, IA, Richland, 2015 R150117 (Early Flat Dutch)	2 (7)	25 26	79	With wrapper leaves	1	0.120	0.0010	< 0.001	
				Without wrapper leaves	1	0.0023	< 0.001	< 0.001	
United States, MN, Paynesville, 2015 R150118 (Pennant)	2 (8)	25 25	79	With wrapper leaves	1	0.015	< 0.001	< 0.001	
				Without wrapper leaves	1	0.0016	< 0.001	< 0.001	
United States, IA, Lime Spring, 2015 R150119 (Thunderhead)	2 (7)	26 25	Heads almost formed/ Mature	With wrapper leaves	1	0.250	< 0.001	< 0.001	
				Without wrapper leaves	1	0.0041	< 0.001	< 0.001	
United States, IA, Bagley, 2015 R150121 (not stated)	2 (6)	26 26	49	With wrapper leaves	1	0.015	< 0.001	< 0.001	
				Without wrapper leaves	1	0.011	< 0.001	< 0.001	
United States, OK, Lebanon, 2015 R150122 (Late Flat Dutch)	2 (7)	26 26	84	With wrapper leaves	1	0.030	< 0.001	< 0.001	
				Without wrapper leaves	1	0.0029	< 0.001	< 0.001	
United States, CA, Fresno, 2015	2 (7)	25 25	49	With wrapper leaves	1	0.015	0.0014	< 0.001	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
R150123 (Supreme Vantage)				Without wrapper leaves	1	0.0113	< 0.001	< 0.001	
Republic of Korea, Wanju-gun, Jeollabuk-do, 2018 SGR--2017-104 (Capaitata)	3 (7-9)	37.5-50	Not stated	Whole cabbage	7 14 21 30	0.02 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	2020/2090104 Park, 2018, BROFLAN_052 Storage time: 35 days Procedural recoveries: 80.2-108.7% at 0.1 & 0.5 mg/kg

Notes:

GS= growth stage; RTI= repeated treatment interval, days

[1] Mean values from duplicate or triplicate field samples. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

[2] Same location and year, but different formulation and therefore considered independent.

Chinese cabbage

Two greenhouse trials were conducted with Chinese cabbage in the Republic of Korea in the 2017 and 2018 growing seasons (BROFLAN_057, BROFLAN_058). Plants received 2 spray applications of broflanilide at nominal rates of 37.5–50 g ai/ha (water volume: 2000 L/ha) with a 7 or 9 day interval between applications. Plants were collected at 7, 14 and 21 DALA (one trial additionally at 30 DALA). Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using the Korean residue method using HPLC-UV with a LOQ of 0.1 mg/kg and a limit of detection of 0.01 mg/kg.

A total of 10 field trials were conducted with Chinese cabbage in China during the 2015-2016 and 2018 growing seasons (BROFLAN_059, BROFLAN_060). Plants received 2 or 3 spray applications of broflanilide at nominal rates of 25, 30 or 45 g ai/ha (water volume: 600-900 L/ha), with a 7 day interval between applications. Plants were collected at 5 and 7 DALA, and some trials additionally at 0, 3 and 10 DALA. Additional decline trials were performed with 1 application at 45 g ai/ha and samples were collected at 0, 1, 2, 3, 7, 14, 21, 28 and 42 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were extracted with acetonitrile, followed by addition of sodium chloride. The supernatant was diluted with methanol-water (1:1) and determined by LC-MS/MS with a LOQ of 0.001 mg/kg. The results are shown in Table 103.

Table 103 Residues of broflanilide in Chinese cabbage following foliar treatment in South Korea and China

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
Republic of Korea, Buyeo-gun, Chungcheongnam-do, 2017 DBA-RC-2017-011 (Ttugsim Cross)	2 (7)	50	Not stated	Cabbage head	7 14 21	1.05 0.53 0.34	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2020/2090115 Yoon, 2018, BROFLAN_057 Storage time: 104 days Procedural recoveries: 94.4-118.5% at 0.1 & 0.5 mg/kg
Republic of Korea, Iksan-si, Jeollabuk-do 2018 SGR-2017-103 (Cheonggwang Cross)	2 (7)	46-50	Not stated	Cabbage head	7 14 21 30	0.70 0.21 0.02 < 0.01	0.03 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	2020/2090117 Park, 2018, BROFLAN_058 Storage time: 23 days Procedural recoveries: 94.8-111.2% at 0.1 & 0.5 mg/kg
China Huliuzi Village, Chagu Port Town, Wuqing District, Tianjin 2015 (Xinlv)	2 (7)	30	46	Cabbage head	5 7	0.388 0.322	0.002 0.002	0.001 < 0.001	2020/2090098 Yuting, 2017, BROFLAN_059 Storage time: <24 month Procedural recoveries: 77-111% at 0.001-1 mg/kg
	3 (7)	30	47	Cabbage head	5 7	0.435 0.177	0.002 0.002	0.001 < 0.001	
	2 (7)	45	46	Cabbage head	5 7	1.065 0.488	0.003 0.002	0.001 < 0.001	
	3 (7)	45	47	Cabbage head	5 7	1.005 0.393	0.002 0.002	0.001 < 0.001	
	1	45	45	Cabbage head	0 1 2 3 7 14 21 28 42	2.67 0.367 0.374 <u>0.386</u> 0.150 0.032 0.007 0.003 0.002	< 0.001 0.001 0.002 0.002 0.002 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001	
China Huliuzi Village, Chagu Port Town, Wuqing District, Tianjin 2016 (Jinlv 60)	2 (7)	30	45	Cabbage head	5 7	0.671 0.549	0.002 0.002	0.001 0.002	
	3 (7)	30	46	Cabbage head	5 7	0.596 0.622	0.003 0.012	0.001 0.006	
	2 (7)	45	45	Cabbage head	5 7	0.927 0.857	0.003 0.003	0.002 0.002	
	3 (7)	45	46	Cabbage head	5 7	2.133 1.783	0.008 0.007	0.005 0.005	
	1	45	44	Cabbage head	0 1 2 3 7 14 21 28 42	1.21 0.396 0.212 <u>0.407</u> 0.213 0.259 0.163 0.017 0.001	< 0.001 < 0.001 < 0.001 < 0.001 0.001 0.003 0.002 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001	
China Hongqi Village, Nangang District, Harbin City,	2 (7)	30	47	Cabbage head	5 7	0.740 0.551	0.003 0.002	0.003 0.002	
	3 (7)	30	48	Cabbage head	5 7	1.617 0.284	0.006 0.001	0.001 0.001	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
Heilongjiang Province 2015 (586-4)	2 (7)	45	47	Cabbage head	5	0.475	0.001	< 0.001	
					7	0.716	0.003	< 0.001	
	3 (7)	45	48	Cabbage head	5	2.440	0.005	< 0.001	
					7	0.227	0.001	0.001	
	1	45	46	Cabbage head	0	1.91	0.002	0.003	
					1	3.16	0.002	0.005	
					2	0.948	0.001	0.002	
					3	0.608	0.001	0.001	
					7	<u>1.83</u>	0.002	0.004	
					14	0.413	< 0.001	0.001	
					21	0.057	< 0.001	< 0.001	
					28	0.056	< 0.001	< 0.001	
					42	0.005	< 0.001	< 0.001	
China Hongqi Village, Nangang District, Harbin City, Heilongjiang Province 2016 (586-4)	2 (7)	30	47	Cabbage head	5	0.33	0.001	< 0.001	
					7	0.234	0.001	< 0.001	
	3 (7)	30	48	Cabbage head	5	0.886	0.004	< 0.001	
					7	0.634	0.003	0.001	
	2 (7)	45	47	Cabbage head	5	1.054	0.004	< 0.001	
					7	0.499	0.002	0.001	
	3 (7)	45	48	Cabbage head	5	0.770	0.004	< 0.001	
					7	0.752	0.003	< 0.001	
	1	45	46	Cabbage head	0	1.11	< 0.001	< 0.001	
					1	1.40	< 0.001	< 0.001	
					2	0.995	< 0.001	< 0.001	
					3	<u>0.990</u>	< 0.001	< 0.001	
					7	0.476	< 0.001	< 0.001	
					14	0.089	< 0.001	< 0.001	
					21	0.094	< 0.001	< 0.001	
					28	0.022	< 0.001	< 0.001	
					42	< 0.001	< 0.001	< 0.001	
China Xianyang City, Shaanxi Province 2018 (Beijing New No.3)	2 (7)	25	48	Cabbage head	0	1.325	< 0.001	0.002	2020/2090097 Na, 2019, BROFLAN_060
					3	0.886	< 0.001	< 0.001	
					5	0.647	0.002	< 0.001	
					7	0.608	< 0.001	< 0.001	
					10	0.579	0.002	< 0.001	
China Penglai City, Shandong Province 2018, (Jiaozhou Chinese cabbage)	2 (7)	25	48	Cabbage head	0	1.045	< 0.001	0.002	Storage time: max. 9 month Procedural recoveries: 100-110% at 0.05 mg/kg
					3	1.120	< 0.001	0.002	
					5	0.675	< 0.001	0.002	
					7	1.900	0.002	0.003	
					10	1.365	0.002	0.003	
China Jiyuan City, Henan Province 2018, (Damaobian cabbage)	2 (7)	25	48	Cabbage head	5	0.004	< 0.001	< 0.001	
					7	0.002	< 0.001	< 0.001	
China Suzhou City, Anhui Province 2018 (Fengkang 70)	2 (7)	25	48	Cabbage head	0	0.739	< 0.001	< 0.001	
					3	0.382	< 0.001	< 0.001	
					5	0.406	< 0.001	< 0.001	
					7	0.153	< 0.001	< 0.001	
					10	0.002	< 0.001	< 0.001	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
China Zhangjiajie City, Hunan Province 2018 (Jindian Hualiang Early Maturing cabbage No.5)	2 (7)	25	48	Cabbage head	5 7	0.220 0.199	< 0.001 < 0.001	< 0.001 < 0.001	
China Jiulongpo District, Chongqing City 2018 (Shandong Chinese cabbage)	2 (7)	25	48	Cabbage head	0 3 5 7 10	1.745 1.510 1.395 0.878 0.934	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	

Notes:

GS= growth stage; RTI= repeated treatment interval, days

[1] Mean values from duplicate or triplicate field samples. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

Fruiting vegetables, other than cucurbits

Tomatoes

A total of 20 field trials were conducted with tomatoes (3 with cherry tomatoes) in the United States during the 2015 growing season (BROFLAN_061). Plants received 2 foliar spray applications of broflanilide at nominal rates of 25 g ai/ha with a 7±1 day interval between applications. Samples were collected at 1 days after the last application. Additionally, in two decline trial, plants were also collected at 0, 1, 3, 7 and 10 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 (according to QuEChERS) with a LOQ of 0.001 mg/kg.

Additionally, two greenhouse trials were conducted with tomatoes in the Republic of Korea in the 2018 and 2019 growing seasons (BROFLAN_062, BROFLAN_063). Plants received 2 or 3 spray applications of broflanilide at 2.5 g ai/hL with a 7 day interval between applications. Plants were collected at 0, 1, 3, 5 and 7 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using the Korean residue method with a validated limit of quantification of 0.1 mg/kg and a LOQ of 0.01 mg/kg. The results are shown in Table 104.

Table 104 Residues of broflanilide in tomatoes following foliar treatment in Canada, the Republic of Korea and the United States

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg]			Report, Reference, Storage period
	N (RTI)	g ai/ha (g ai/hL)	GS at final appl.			Broflanilide[1]	DM-8007	S(PFP-OH)-8007	
Republic of Korea, Buyeo-gun, Chungcheongnam-do 2018 SGR-2017-106 (Bravo)	2 (7)	52.5 (2.5)	Not stated	Fruit	0 1 3 5 7	0.13 0.11 (0.11, 0.11, 0.12) 0.10 0.08 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	2020/2090154 Park, 2018, BROFLAN_062 Storage time: 17 days Procedural recoveries: 79.3-107.6% at 0.1 & 0.5 mg/kg

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg]			Report, Reference, Storage period
	N (RTI)	g ai/ha (g ai/hL)	GS at final appl.			Broflanilide[1]	DM-8007	S(PFP-OH)-8007	
Republic of Korea, Iksan-si, Jeollabuk-do 2019 R1943 (High-Q)	3 (7)	66.9 66.9 67.5 (2.5)	Not stated	Fruit	0 1 3 5 7	0.32 0.29 0.28 0.24 0.22	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	2020/2090156 Yoon, 2019, BROFLAN_063 Storage time: 20 days Procedural recoveries: 76.5-104.9% at 0.02 & 0.2 mg/kg
Canada, St-Marc-sur-Richelieu, PQ 2015, R150139 (Celebrity)	2 (6 days)	24.7 25.1 (10)	87-88	Fruit	1	0.040 (0.043, 0.036)	< 0.001	< 0.001	2020/2081627 Reeves, 2020, BROFLAN_061 Storage time: max. 510 days (17 months) Procedural recoveries: 86.0-113% at 0.001 & 0.1 mg/kg
Canada Branchton, ON 2015, R150144 (TSH 18)	2 (7)	26.7 25.1 (13)	84-84	Fruit	1	0.031 (0.026, 0.036)	< 0.001	< 0.001	
United States, Fresno, CA 2015, R150132 Cherry Tomato (Naomi)	2 (7)	25.0 24.7 (8.9)	84	Fruit	1	0.028 (0.031, 0.025)	< 0.001	< 0.001	
United States, Paso Robles, CA 2015, R150133 Cherry Tomato (Sungold)	2 (7)	25.0 25.8 (8.9)	88	Fruit	1	0.076 (0.077, 0.075)	< 0.001	< 0.001	
United States, Porterville, CA 2015, R150134 Cherry Tomato (Big red cherry)	2 (7)	25.4 25.9 (8.9)	89	Fruit	0 1 3 7 10	0.040 0.026 (0.022, 0.029) 0.025 0.020 0.019	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	
United States, Alton, NY 2015, R150135 (POLBIG F1)	2 (7)	25.6 25.6 (9.1)	85-86	Fruit	1	0.018 (0.021, 0.015)	< 0.001	< 0.001	
United States, Jeffersonville, GA 2015, R150136 (Amelia)	2 (8)	25.5 26.4 (9.1)	71	Fruit	1	0.015 (0.015, 0.014)	< 0.001	< 0.001	
United States, Sneads, FL 2015, R150137 (FL 137)	2 (7)	24.9 24.9 (12)	87	Fruit	1	0.0022 (0.0019, 0.0024)	< 0.001	< 0.001	
United States, Clermont, FL 2015, R150138 (Better Boy)	2 (7)	24.7 25.7 (9.0)	85	Fruit	1	0.0049 (0.0045, 0.0052)	< 0.001	< 0.001	
United States, Leonard, MO 2015, R150140 (Celebrity)	2 (7)	25.6 25.3 (9.4)	75	Fruit	1	0.019 (0.016, 0.022)	< 0.001	< 0.001	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg]			Report, Reference, Storage period
	N (RTI)	g ai/ha (g ai/hL)	GS at final appl.			Broflanilide[1]	DM-8007	S(PFP-OH)-8007	
United States, York, NE, 2015, R150141 (Better Bush)	2 (6 days)	25.4 25.6 (13)	83	Fruit	1	0.0017 (0.0017, 0.0017)	< 0.001	< 0.001	
United States, Richland, IA 2015, R150142 (Delicious)	2 (7)	25.9 25.6 (12)	88	Fruit	1	0.023 (0.019, 0.026)	< 0.001	< 0.001	
United States, Paynesville, MN 2015, R150143 (Mountain Merit)	2 (7)	25.1 24.9 (13)	89	Fruit	1	0.014 (0.016, 0.012)	< 0.001	< 0.001	
United States, Fitchburg, WI 2015, R150145 (Mountain Fresh)	2 (7)	25.2 25.3 (13)	69-84	Fruit	1	0.078 (0.0089, 0.0066)	< 0.001	< 0.001	
United States, Stafford, KS 2015, R150146 (Husky Red)	2 (7)	25.6 24.5 (12)	88	Fruit	1	0.014 (0.013, 0.014)	< 0.001	< 0.001	
United States, Lafayette, IN 2015, R150147 (Roma/Heinz 3406)	2 (7)	24.5 25.1 (8.8)	81	Fruit	1	0.0097 (0.0095, 0.0099)	< 0.001	< 0.001	
United States, Yuba City, CA 2015, R150148 (Heinz H 4107)	2 (7)	25.5 25.4 (9.1)	88	Fruit	1	0.024 (0.028, 0.019)	< 0.001	< 0.001	
United States, Sanger, CA 2015, R150149 (San Marzano)	2 (6)	24.4 24.7 (8.9)	87	Fruit	1	0.012 (0.014, 0.010)	< 0.001	< 0.001	
United States, Yuma, AZ 2015, R150150 (Mountain Fresh)	2 (7)	25.8 26.0 (9.0)	86	Fruit	1	0.011 (0.014, 0.0078)	< 0.001	< 0.001	
United States, Woodland, CA 2015, R150151 (Heinz H 4107)	2 (6)	26.2 25.7 (8.9)	85	Fruit	0 1 3 7 10	0.015 0.015 (0.013, 0.016) 0.012 0.014 0.015	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	

Notes:

GS= growth stage; RTI= repeated treatment interval, days

[1] Mean values from duplicate or triplicate field samples, individual values in parentheses. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

Root and tuber vegetables

Radish

Two greenhouse trials were conducted with radishes in the Republic of Korea in the 2018 and 2019 growing seasons (BROFLAN_064, BROFLAN_065). Plants received 3 spray applications of broflanilide at 2.5 g ai/hL with a 7–9 day interval between applications. Plants were collected at 7, 14, 21 and 30 DALA or 0, 7, 14 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using the Korean residue method with a LOQ of 0.1 mg/kg and a LOD of 0.01 mg/kg. The results are shown in Table 105.

Table 105 Residues of broflanilide in radish following foliar treatment in the Republic of Korea

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/hL	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
Republic of Korea, Iksan-si, Jeollabuk-do 2018 SGR-2017-102 (Sativus)	3 (7-9)	2.5	Not stated	Roots	7	< 0.01	< 0.01	< 0.01	2020/2090125 Park, 2018, BROFLAN_064 storage time: max. 34 days Procedural recoveries: 71.4-114.7% at 0.1 & 0.5 mg/kg
					14	< 0.01	< 0.01	< 0.01	
					21	< 0.01	< 0.01	< 0.01	
					30	< 0.01	< 0.01	< 0.01	
				Tops	7	1.76	0.25	0.15	
					14	0.58	0.08	< 0.01	
					21	0.18	< 0.01	< 0.01	
					30	0.12	< 0.01	< 0.01	
Republic of Korea, Buyeo-gun, Chungcheongnam-do, 2019 R1941 (Early Spring Altari)	3 (7)	2.5	Not stated	Roots	0	< 0.02	< 0.02	< 0.02	2020/2090129 Jeong, 2020, BROFLAN_065 Storage time: max. 179 days (6 months) Procedural recoveries: 74.0-108.4% at 0.02 & 0.2 mg/kg
					7	< 0.02	< 0.02	< 0.02	
					14	< 0.02	< 0.02	< 0.02	
				Tops	0	2.05	< 0.04	< 0.04	
					7	1.52	< 0.04	< 0.04	
					14	0.14	< 0.04	< 0.04	

Notes:

GS= growth stage; RTI= repeated treatment interval, days

[1] Mean values from duplicate or triplicate field samples, individual values in parentheses. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

Japanese radish

A total of six field trials were conducted with Japanese radish in Japan in the 2013 and 2014 growing seasons (BROFLAN_066, BROFLAN_067). Plants received 3 foliar spray applications of broflanilide at nominal rates of 2.5 g ai/hL (37.5–75 g ai/ha) with a 7±1 day interval between applications. Radish roots and tops were collected at 1, 3 and 7 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using the Japanese residue method with a LOQ of 0.01 mg/kg (Table 106)

Table 106 Residues of broflanilide in Japanese radish following foliar treatment in Japan

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/hL	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	

Location, Year, Trial No., (variety)	Application			Sample	DALA	Residues [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/hL	GS at final appl.			Broflanilide	DM-8007	S(PFP-OH)-8007	
Fukui, 2013 JP2013C275A (Taibyo Sobuton)	3 (7)	2.5	Not stated	Roots	1	< 0.01	< 0.01	< 0.01	2020/2090131 Nakamura, 2014, BROFLAN_066 Storage time: max. 117 days (~4 month) Procedural recoveries: 83-96% at 0.1 mg/kg
					3	< 0.01	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
				Tops	1	1.53	< 0.01	< 0.01	
					3	0.68	< 0.01	< 0.01	
					7	0.46	< 0.01	< 0.01	
Nara, 2013 JP2013C275B (Taibyo Sobuton)	3 (7)	2.5	Not stated	Roots	1	< 0.01	< 0.01	< 0.01	
					3	< 0.01	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
				Tops	1	3.46	< 0.01	0.01	
					3	2.94	< 0.01	< 0.01	
					7	1.75	< 0.01	< 0.01	
Wakayama, 2013 JP2013C275C (Taibyo Sobuton)	3 (6-8)	2.5	Not stated	Roots	1	< 0.01	< 0.01	< 0.01	
					3	< 0.01	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
				Tops	1	3.94	< 0.01	< 0.01	
					3	3.64	< 0.01	< 0.01	
					7	3.26	0.02	< 0.01	
Fukui, 2014 JP2014C124A (Taibyo Sobuton)	3 (7)	2.5	Not stated	Roots	1	< 0.01	< 0.01	< 0.01	2020/2090133 Nakamura, 2015, BROFLAN_067 Storage time: max. 169 days (5.6 month) Procedural recoveries: 84-101% at 0.1 mg/kg
					3	< 0.01	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
				Tops	1	0.69	< 0.01	< 0.01	
					3	0.80	< 0.01	< 0.01	
					7	0.40	< 0.01	< 0.01	
Nara, 2014 JP2014C124B (Taibyo Sobuton)	3 (7)	2.5	Not stated	Roots	1	< 0.01	< 0.01	< 0.01	
					3	< 0.01	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
				Tops	1	1.92	< 0.01	< 0.01	
					3	1.75	< 0.01	< 0.01	
					7	1.44	< 0.01	< 0.01	
Kochi, 2014 JP2014C124C (Daishi)	3 (7)	2.5	Not stated	Roots	1	< 0.01	< 0.01	< 0.01	
					3	< 0.01	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
				Tops	1	4.40	< 0.01	< 0.01	
					3	3.98	< 0.01	< 0.01	
					7	2.92	< 0.01	< 0.01	

Notes:

GS= growth stage; RTI= repeated treatment interval, days

[1] Mean values from analytical duplicate. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

Turnip

A total of three greenhouse trials were conducted with turnip in Japan in the 2014/15 growing season. Plants received 3 foliar spray applications of broflanilide at 2.5 g ai/hL with a 7±1 day interval between applications. Turnip roots were collected at 1, 3 and 7 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using the Japanese residue method with a LOQ of 0.01 mg/kg (Table 107)

Table 107 Residues of broflanilide in turnip roots following foliar treatment in Japan

Location, Year, Trial No., (variety)	Application			DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/hL	GS at final appl.		Broflanilide	DM-8007	S(PFP-OH)-8007	
Ibaraki, 2014 JP2014C125A (Taibyo Hikari)	3 (6)	2.5	Not stated	1 3 7	0.01 0.01 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2020/2090138 Nakamura, 2015, BROFLAN_068
Mie, 2015 JP2014C125B (Taibyo Hikari)	3 (7)	2.5	Not stated	1 3 7	0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	Storage time: max. 91 days (3 month) Procedural recoveries: 96-119% at 0.1 mg/kg
Miyazaki, 2014 JP2014C125C (Taibyo Hikari)	3 (6-8)	2.5	Not stated	1 3 7	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	

Notes:

GS= growth stage; RTI= repeated treatment interval, days

[1] Mean values from analytical duplicate. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

Potato

A total of 21 field trials were conducted with cabbage in Canada and the United States during the 2015–16 growing seasons (BROFLAN_069). Two treatments were applied: 1) Plants received 2 spray applications of broflanilide at nominal rates of 25 g ai/ha with a 6–8 day interval between applications. Samples were collected at 13-14 days after the last application. Additionally, in one decline trial, plants were also collected at 0, 7, 14, 18 and 21 DALA. 2) One in-furrow application, at planting at 50 g ai/ha. Samples were collected at 80–146 days after the application. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg (Table 108). The procedural recoveries ranged from 70.3-118 percent at 0.001 & 0.1 mg/kg and the samples were stored at a maximum of 450 days before analysis.

Table 108 Residues of broflanilide in potatoes tuber following foliar and in-furrow treatment in Canada and the United States (Study 2016/7009341, Crawford, 2017, BROFLAN_069)

Location, Year, Trial No., (variety)	Application			DALA	Residues [mg/kg][1]		
	N (RTI)	g ai/ha	GS at final appl.		Broflanilide	DM-8007	S(PFP-OH)-8007
Canada, Outlook, SK, 2015, R150263 (E3 Wisconsin Norland)	2 (7)	25.2 25.4	BBCH 79-82	14*	< 0.001	< 0.001	< 0.001
	1	50.0	At planting	98*	<u>< 0.001</u> (< 0.001, < 0.001)	< 0.001	< 0.001
Canada, Saskatchewan, AB, 2015 R150264, (Russet Burbank)	2 (7)	25.9 25.4	BBCH 71	14*	< 0.001	< 0.001	< 0.001
United States, North Rose, NY, 2015 R150065 (Red Norland)	2 (7)	28.2 25.7	BBCH 47	0 7 14 18 21	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001
	1	52	At planting	89*	<u>0.0049</u> (0.0061, 0.0037)	< 0.001	< 0.001
United States, Waterloo, NY,	2 (7)	25.1 23.1	BBCH 49	14*	< 0.001	< 0.001	< 0.001

Location, Year, Trial No., (variety)	Application			DALA	Residues [mg/kg][1]		
	N (RTI)	g ai/ha	GS at final appl.		Broflanilide	DM-8007	S(PFP-OH)-8007
2015, R150066 (Red Norland)	1	50.0	At planting	88*	0.0029 (0.0037, 0.0020)	< 0.001	< 0.001
United States, Weedsport, NY, 2015, R150067 (Chieftrain)	2 (7)	25.5 25.4	47	14*	< 0.001	< 0.001	< 0.001
	1	50.2	At planting	88*	0.0018 (0.0021, 0.0014)	< 0.001	< 0.001
United States, Germansville, PA, 2015, R150069 (Dark Red Norland)	2 (6)	25.0 24.9	75-81	14*	< 0.001	< 0.001	< 0.001
	1	50.2	At planting	80*	0.0015 (0.0015, 0.0015)	< 0.001	< 0.001
United States, Chula, GA, 2016, R150070 (Red Pontiac)	2 (7)	25.3 24.7	48	14*	< 0.001	< 0.001	< 0.001
	1	49.9	At planting	83*	0.0023 (0.0025, 0.002)	< 0.001	< 0.001
United States, Hobe Sound, FL, 2015, R150071 (Red Lasoda)	2 (7)	24.8 24.9	47	14*	0.0030	< 0.001	< 0.001
	1	50.2	At planting	102*	0.034 (0.039, 0.028)	0.0017 (0.0018, 0.0016)	< 0.001
United States, Delavan, WI, 2015, R150072 (Atlantic)	2 (7)	25.1 25.1	BBCH 46	14*	0.0063	< 0.001	< 0.001
	1	50.4	At planting	102*	0.015 (0.027, 0.0031)	< 0.001	< 0.001
United States, Gardner, ND, 2015, R150073 (Russet Norkotah)	2 (7)	25.9 26.1	87	14*	< 0.001	< 0.001	< 0.001
	1	51.6	At planting	127*	< 0.001 (< 0.001, 0.001)	< 0.001	< 0.001
United States, York, NE, 2015, R150075 (Yukon gold)	2 (7)	24.9 24.8	BBCH 75	14*	0.0022	< 0.001	< 0.001
	1	50.2	At planting	99*	0.0017 (0.0015, 0.0019)	< 0.001	< 0.001
United States, St. John, KS, 2016, R150076 (Red Pontiac)	2 (7)	24.9 24.9	BBCH 71	14*	< 0.001	< 0.001	< 0.001
	1	50.3	At planting	92*	< 0.001 (< 0.001, < 0.001)	< 0.001	< 0.001
United States, Lewiston, UT, 2015, R150078 (Agata)	1	47.7	At planting	92*	0.0021 (0.0025, 0.0016)	< 0.001	< 0.001
United States, Porterville, CA, 2015, R150079 (Red Pontiac)	2 (7)	25.4 25.5	BBCH 48	14*	< 0.001	< 0.001	< 0.001
	1	50.2	At planting	146	< 0.001 (< 0.001, 0.001)	< 0.001	< 0.001
United States, Yakima, WA, 2015, R150080 (Organic Desiree)	2 (7)	25.6 25.7	Late bloom	14*	< 0.001	< 0.001	< 0.001
	1	49.5	At planting	118*	0.0026 (0.0030, 0.0021)	< 0.001	< 0.001
United States, American Falls, ID, 2015, R150081 (Russet Burbank)	2 (7)	26.1 26.1	47-48	0 7 14 18 21	0.0012 < 0.001 < 0.001 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001
	1	49.8	At planting	112*	0.0049 (0.0061, 0.0037)	< 0.001	< 0.001
United States, Ashton, ID, 2015	2 (8)	23.9 23.9	BBCH 47-48	14*	< 0.001	< 0.001	< 0.001

Location, Year, Trial No., (variety)	Application			DALA	Residues [mg/kg][1]		
	N (RTI)	g ai/ha	GS at final appl.		Broflanilide	DM-8007	S(PFP-OH)-8007
R150082 (Russet Burbank)	1	53.8	At planting	98*	0.0046 (0.0053, 0.0038)	< 0.001	< 0.001
United States, Tulelake, CA, 2015, R150083 (Standard Russet Norkotah)	2 (7)	26.4 25.9	88	14*	0.0011	< 0.001	< 0.001
	1	50.5	At planting	112*	0.0012 (0.0013, 0.0010)	< 0.001	< 0.001
	1	50.9	At planting	96*	0.0012 (0.0014, < 0.001)	< 0.001	< 0.001
United States, Dana, IA, 2015, R150267 (Carola)	2 (7)	25.2 25.0	85	14*	0.0068	< 0.001	< 0.001
	1	49.7	At planting	108*	< 0.001 (< 0.001, < 0.001)	< 0.001	< 0.001
United States, Frenchtown, NJ, 2015, R150269 (Waneta)	2 (7)	25.8 25.9	BBCH 46	14*	< 0.001	< 0.001	< 0.001
	1	52.0	At planting	91*	0.0015 (0.0013, 0.0016)	< 0.001	< 0.001
United States, Lewiston, UT, 2015, R150273 (Regina)	2 (7)	24.2 23.9	BBCH 47	14*	< 0.001	< 0.001	< 0.001

Notes:

GS= growth stage; RTI= repeated treatment interval, days

[1] Mean values from duplicate field samples, individual values in parentheses. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

* Crop maturity.

Cereal grains

Wheat

A total of 25 field trials were conducted on winter or spring wheat in Canada and the United States during the 2015/16 growing season (BROFLAN_070). The seeds were treated with broflanilide at a nominal concentration of 100 g ai/ton of seed. The treated seeds were planted at a rate of 84.0–134.4 kg/ha, (target rate 100 kg/ha), resulting in an actual test substance application rate of 8.14–13.3 g ai/ha. Samples were collected at 83–274 days after planting. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg. Procedural recoveries: range from 66-137 percent at 0.001 and 0.1 mg/kg and the samples were stored up to 444 days. The results are shown in Table 109.

Table 109 Residues of broflanilide in wheat grain following seed treatment in Canada and the United States (Study 2016/7006466; Wyatt, 2017, BROFLAN_070)

Location, Year, Trial No., (variety)	Application	Days after planting	Residues [mg/kg][1]		
	g ai/t seed (g ai/ha)		Broflanilide	DM-8007	S(PFP-OH)-8007
Canada, Minto, MB, 2015, R150019 (CDC Utmost)	104.3 (10.6)	97	< 0.001	< 0.001	< 0.001
Canada, Glenboro, MB, 2015, R150020 CDC Utmost)	104.3 (10.4)	89	< 0.001	< 0.001	< 0.001
Canada, Brandon, MB, 2015, R150021 (Glenn)	107.0 (10.7)	93	< 0.001	< 0.001	< 0.001

Location, Year, Trial No., (variety)	Application g ai/t seed (g ai/ha)	Days after planting	Residues [mg/kg][1]		
			Broflanilide	DM-8007	S(PFP-OH)-8007
Canada, Alvena, SK, 2015, R150022 (CDC Go)	104.3 (10.4)	113	< 0.001	< 0.001	< 0.001
Canada, Wakaw, SK, 2015, R150023 (CDC Go)	104.3 (10.4)	109	< 0.001	< 0.001	< 0.001
Canada, Hepburn, SK, 2015, R150024 (Glenn)	107.0 (10.7)	112	< 0.001	< 0.001	< 0.001
Canada, Fort Saskatchewan, AB, 2015, R150025 (CDC Go)	104.3 (10.4)	123	< 0.001[2]	< 0.001	< 0.001
United States, Seven Springs, NC, 2015, R150001 (Progeny 357)	95.2 (12.7)	226	< 0.001	< 0.001	< 0.001
United States, Proctor, AR, 2015 R150002 (Progeny 357)	95.2 (9.53)	238	< 0.001	< 0.001	< 0.001
United States, Richland, IA, 2015, R150003 (Branson)	99.6 (9.78)	274	< 0.001	< 0.001	< 0.001
United States, Carlyle, IL, 2015 R150004 (Branson)	99.6 (9.64)	259	< 0.001	< 0.001	< 0.001
United States, York, NE, 2015 R150005 (Prosper)	105.3 (10.8)	83	< 0.001	< 0.001	< 0.001
United States, Stafford, KS, 2015, R150006 (TAM 111)	98.6 (13.3)	232	< 0.001	< 0.001	< 0.001
United States, Uvalde, TX, 2015, R150007 (TAM 113)	96.9 (9.50)	175	< 0.001	< 0.001	< 0.001
United States, Carrington, ND, 2015, R150008 (Glenn)	107.0 (10.7)	97	< 0.001	< 0.001	< 0.001
United States, Sykeston, ND, 2015, R150009 (Glenn)	107.0 (10.7)	102	< 0.001	< 0.001	< 0.001
United States, Grand Island, NE, 2015, R150010 (Prosper)	105.3 (11.5)	85	< 0.001	< 0.001	< 0.001
United States, Hastings, NE, 2015, R150011 (Prosper)	105.3 (10.9)	104	< 0.001	< 0.001	< 0.001
United States, Jamestown, ND, 2015, R150012 (Prosper)	105.3 (10.8)	89	< 0.001	< 0.001	< 0.001
Canada, Taber, AB, 2015 R150013 (CDC Utmost)	104.3 (10.0)	96	< 0.001	< 0.001	< 0.001
United States, Plainview, TX, 2015, R150014 (TAM 111)	98.6 (10.1)	203	< 0.001	< 0.001	< 0.001
United States, Wall, TX, 2015 R150015 (TAM 113)	96.9 (8.14)	180	< 0.001	< 0.001	< 0.001
United States, Larned, KS, 2015 R150016 (TAM 111)	98.6 (13.3)	233	< 0.001	< 0.001	< 0.001
United States, Hinton, OK, 2015, R150017(TAM 113)	96.9 (9.91)	253	< 0.001	< 0.001	< 0.001
United States, Payette, ID, 2015 R150018 (Alpowa)	101.2 (11.7)	99	< 0.001	< 0.001	< 0.001

Notes:

[1] Mean values from duplicate field samples, individual values in parentheses. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

[2] Average of several re-analyses.

Barley

A total of 16 field trials were conducted on barley in Canada and the United States during the 2015 and 2016 growing seasons (BROFLAN_071). The seeds were treated with broflanilide at a nominal concentration of 100 g ai/ton of seed. The treated seeds were planted at a rate of 95.2 to 107 kg/ha, (target rate 100 kg/ha) resulting in an actual test substance application rate of 9.52–10.7 g ai/ha. Barley grain was harvested at maturity (BBCH 87-90). Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg. Procedural recoveries ranged from 88- to 133 percent at 0.001 and 0.1 mg/kg, and samples were stored up to 585 days before analysis. The results are shown in Table 110

Table 110 Residues of broflanilide in barley grain following seed treatment in Canada and the United States (Study 2016/7009340, Greenland, 2017, BROFLAN_071

Location, Year, Trial No., (variety)	Application	BBCH stage at harvest	Residues [mg/kg][1]		
	g ai/t (g ai/ha)		Broflanilide	DM-8007	S(PFP-OH)-8007
Canada, Taber, AB, 2015, R150029 (AC Metcalfe)	101.1 (10.0)	89	< 0.001	< 0.001	< 0.001
Canada, Minto, MB, 2015, R150036 (AC Metcalfe)	101.1 (10.1)	89	< 0.001	< 0.001	< 0.001
Canada, Blaine Lake, SK, 2015, R150037 (AC Metcalfe)	101.1 (9.88)	89	< 0.001	< 0.001	< 0.001
Canada, Hague, SK, 2015, R150038 (AC Metcalfe)	101.1 (10.0)	89	< 0.001	< 0.001	< 0.001
Canada, Neepawa, MB, 2016, R150039 (AC Metcalfe)	104.4 (9.84)	89	< 0.001	< 0.001	< 0.001
Canada, Alvena, SK, 2015, R150040 (AC Metcalfe)	101.1 (10.0)	89	< 0.001	< 0.001	< 0.001
Canada, Fort Saskatchewan, AB, 2015, R150041 (AC Metcalfe)	101.1 (9.52)	87	< 0.001	< 0.001	< 0.001
United States, North Rose, NY, 2015 R150026 (Quest)	97.8 (9.97)	89	< 0.001	< 0.001	< 0.001
United States, Gardner, ND, 2015 R150027 (Quest)	97.8 (10.5)	89	< 0.001	< 0.001	< 0.001
United States, Fitchburg, WI, 2015, R150028 (Quest)	97.8 (10.7)	89	< 0.001	< 0.001	< 0.001
United States, Grand Island, NE, 2015, R150030 (Lacey)	94.8 (9.96)	89	< 0.001	< 0.001	< 0.001
United States, Prosser, NE, 2016 R150031 (Lacey)	103.8 (10.4)	89	< 0.001	< 0.001	< 0.001
United States, Jamestown, ND, 2015, R150032 (Quest)	97.8 (10.6)	90	< 0.001	< 0.001	< 0.001
United States, Trenton, UT, 2015, R150033 (Lenetah)	99.1 (10.3)	89	< 0.001	< 0.001	< 0.001
United States, Madera, CA, 2015, R150034 (Strider)	100.6 (10.4)	89	< 0.001	< 0.001	< 0.001
United States, Aberdeen, ID, 2015, R150035 (Lenetah)	99.1 (10.1)	89	< 0.001	< 0.001	< 0.001

Notes:

[1] Mean values from duplicate field samples, individual values in parentheses. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

Maize

A total of 25 field trials were conducted on maize (20) and sweet corn (5) in Canada and the United States during the 2015 and 2016 growing seasons (BROFLAN_072). Plants received one in-furrow application, at planting at 50 g ai/ha. Sweet corn kernel + cob with husk removed (K+CWHR) were collected 62–94 DAA, while maize gain samples were collected 112–164 days after application (DAA). Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg. A reduced validation set (n=3) for corn grain at two levels (0.001 and 0.1 mg/kg) had acceptable mean recoveries within the range of 70–120 percent with an RSD <20 percent for all analytes, while the reduced validation set for K+CWHR had somewhat higher mean recoveries at up to 129 percent. Procedural recoveries: 83–163 percent at 0.001 & 0.1 mg/kg. Samples were stored up to 267 days for grain and up to 357 days for K+CWHR. The results are showing in Table 111

Table 111 Residues of broflanilide in sweet corn kernel + cob with husk removed (K+CWHR) and maize grain following in-furrow treatment at planting in Canada and the United States (Study 2016/7006467, Wyatt, 2017, BROFLAN_072)

Location, Year, Trial No., (variety)	Application	Sample	DAA	Residues [mg/kg][1]		
	g ai/ha			Broflanilide	DM-8007	S(PFP-OH)-8007
Canada, Taber, AB, 2016 R150222 (Sweet corn-Trinity)	50.6	K+CWHR	91	< 0.001	< 0.001	< 0.001
United States, Alton, NY, 2015 R150201 (A1027687)	50.0	K+CWHR Grain	81 112	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Baptistown, NJ 2015 R150202 (TA545-33EZ)	51.2	K+CWHR Grain	83 139	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Newberry, FL, 2015 R150203 (Sweet corn-Passion II)	49.9	K+CWHR	62	< 0.001	< 0.001	< 0.001
United States, Conklin, MI, 2015 R150204 (DKC 46-20 RIB, A1031276)	49.7	K+CWHR Grain	92 150	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Delavan, WI, 2016 R150205 (DKC 49-94 RIB)	50.6	K+CWHR Grain	89 158	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Fitchburg, WI, 2015 R150206 (G95D32-3110)	49.9	K+CWHR Grain	80 147	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Richland, IA, 2015, R150207 (Pioneer P1023AM)	49.9	K+CWHR Grain	78 125	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Lime Springs, IA, 2016, R150208 (AI031275 DKC46-37 RIB)	50.4	K+CWHR Grain	94 154	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Geneva, MN, 2016 R150209 (NuTech 5D-196AMX)	50.0	Grain	164	< 0.001	< 0.001	< 0.001
United States, Manilla, IN, 2015 R150210 (Pioneer P0942AMX)	48.8	Grain	140	< 0.001	< 0.001	< 0.001
United States, Highland, IL, 2015 R150211 (FS 63SV1 RIB)	49.9	Grain	131	< 0.001	< 0.001	< 0.001
United States, Carlyle, IL, 2015 R150212 (N78N-3111)	51.0	Grain	140	< 0.001	< 0.001	< 0.001
United States, Mason, IL, 2015 R150213 (N49W-3000GT)	50.8	Grain	125	< 0.001	< 0.001	< 0.001
United States, Hedrick, IA, 2015 R150214 (Pioneer P1023AM)	50.0	Grain	128	< 0.001	< 0.001	< 0.001
United States, Grandview, IA, 2015 R150215 (Pioneer P0506AM)	49.8	Grain	126	< 0.001	< 0.001	< 0.001
United States, Kirksville, MO, 2015 R150216 (Pioneer P0506AM)	50.1	Grain	125	< 0.001	< 0.001	< 0.001
United States, York, NE, 2015 R150217 (Pioneer P0876HR)	49.7	Grain	142	< 0.001	< 0.001	< 0.001

Location, Year, Trial No., (variety)	Application	Sample	DAA	Residues [mg/kg][1]		
	g ai/ha			Broflanilide	DM-8007	S(PFP-OH)-8007
United States, Osceola, NE, 2015 R150218 (Pioneer P0876HR)	50.1	Grain	138	< 0.001	< 0.001	< 0.001
United States, Brunswick, NE, 2015 R150219 (DKC 54-40RIB)	50.7	Grain	148	< 0.001	< 0.001	< 0.001
United States, Stafford, KS, 2016 R150220 (P1151AM)	49.8	Grain	151	< 0.001	< 0.001	< 0.001
United States, Hinton, OK, 2015 R150221 (DKC 65-19)	50.0	Grain	116	< 0.001	< 0.001	< 0.001
United States, Porterville, CA, 2015 R150223 (Sweet corn-Bodacious)	50.1	K+CWHR	77	< 0.001	< 0.001	< 0.001
United States, Ephrata, WA, 2015 R150224 (Sweet corn-Basin)	50.9	K+CWHR	84	< 0.001	< 0.001	< 0.001
United States, Canby, OR, AB, 2015, R150225 (Sweet corn– Honey N Pearl-L)	51.2	K+CWHR	85	< 0.001	< 0.001	< 0.001

Notes:

[1] Mean values from duplicate field samples, individual values in parentheses. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

Seeds for beverages and sweets

Coffee

A total of nine field trials were conducted on coffee in Brazil and Colombia during the 2016 and 2018/19 growing seasons, respectively (BROFLAN_073, BROFLAN_074). Plants received 2 foliar applications of broflanilide at nominal rates of 18 g ai/ha with an RTI of up to 30 days. Coffee cherries were harvested at 45, 60, 75 and 90 DALA. Coffee cherries sampled in Colombia were de-pulped, dried and threshed. For the coffee cherries sampled in Brazil only threshing is mentioned for all trials, while drying and de-pulping is mentioned for some trials only. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg (Table 112).

Table 112 Residues of broflanilide in coffee beans following foliar treatment in Brazil and Colombia

Location, Year, Trial No., (variety)	Application			DALA	Residues [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.		Broflanilide	DM-8007	S(PFP-OH)-8007	
Brazil, Ibiporã, PR 2016, PR G150327 (Catuai)	2 (30)	18.0	75-83	45	< 0.001[2]	< 0.001	< 0.001	2020/2090092 de Matos, 2018, BROFLAN_073
				60	0.0015	< 0.001	< 0.001	
				75	0.0013	< 0.001	< 0.001	
				90	0.0012	< 0.001	< 0.001	
Brazil, Rolândia, PR, 2016 PR G150328 (Mundo Novo)	2 (30)	18.0	75-83	45	0.0018	< 0.001	< 0.001	Storage time: max. 139 days (~4.5 months)
				60	< 0.001[2]	< 0.001	< 0.001	
				75	0.0034[2]	< 0.001	< 0.001	
				90	< 0.001	< 0.001	< 0.001	
Brazil, Cambé, PR 2016, PR G150329 (Catuai)	2 (30)	18.0	75-83	45	0.0014[2]	< 0.001	< 0.001	Procedural recoveries: 92.3-114% at 0.001 & 0.1 mg/kg
				60	0.0037[2]	< 0.001	< 0.001	
				75	< 0.001	< 0.001	< 0.001	
				90	< 0.001	< 0.001	< 0.001	
Brazil, Antônio do Jardim, SP 2016, PR G150330 (Catucai Multiline)	2 (30)	18.0	79-85	45	0.0016	< 0.001	< 0.001	
				60	0.0012	< 0.001	< 0.001	
				75	< 0.001	< 0.001	< 0.001	
				90	< 0.001	< 0.001	< 0.001	

Location, Year, Trial No., (variety)	Application			DALA	Residues [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/ha	GS at final appl.		Broflanilide	DM-8007	S(PFP-OH)-8007	
Brazil, Indianópolis, MG 201, PR G150331 (Catuai)	2 (30)	18.0	73-81	45	0.0039[2]	< 0.001	< 0.001	
				60	0.0022	< 0.001	< 0.001	
				75	0.0027	< 0.001	< 0.001	
				90	< 0.001	< 0.001	< 0.001	
Colombia, Ciudad Bolivar, Antioquia, 2018/19, G180132 (Castillo)	2 (30)	18.0	77-81	45	< 0.001	< 0.001	< 0.001	2019/2076212 Jose, 2020, BROFLAN_074
				60	< 0.001	< 0.001	< 0.001	
				75	< 0.001	< 0.001	< 0.001	
				90	< 0.001	< 0.001	< 0.001	
Colombia, Concordia, Antioquia, 2018/19 G180133 (Castillo)	2 (30)	18.0	79-81	45	0.0050	< 0.001	< 0.001	Storage time: max. 163 days (~5.5 months)
				60	0.0020	< 0.001	< 0.001	
				75	0.0025	< 0.001	< 0.001	
				90	0.0017	< 0.001	< 0.001	
Colombia, Jericó, Antioquia, 2018/19, G180134 (Catimore)	2 (30)	18.0	77-81	45	< 0.001	< 0.001	< 0.001	Procedural recoveries: 91.3-110% at 0.001 & 0.1 mg/kg
				60	< 0.001	< 0.001	< 0.001	
				75	< 0.001	< 0.001	< 0.001	
				90	< 0.001	< 0.001	< 0.001	
Colombia, Palestina, Caldas, 2018/19, G180135 (Caturra)	2 (30)	18.0	77-81	45	0.0023	< 0.001	< 0.001	
				60	0.0011	< 0.001	< 0.001	
				75	< 0.001	< 0.001	< 0.001	
				90	< 0.001	< 0.001	< 0.001	

Notes:

GS= Growth stage.

RTI= repeated treatment interval, days.

[1] The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

[2] Mean of several measurements.

Feed commodities

Turnip tops

A total of 3 greenhouse trials were conducted with turnip in Japan in the 2014/15 growing season. Plants received 3 foliar spray applications of broflanilide at nominal rates of 2.5 g ai/hL with a 7±1 day interval between applications. Turnip tops were collected at 1, 3 and 7 DALA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using the Japanese residue method with a LOQ of 0.01 mg/kg (Table 113).

Table 113 Residues of broflanilide in turnip tops following foliar treatment in Japan

Location, Year, Trial No., (variety)	Application			DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/hL	GS at final appl.		Broflanilide	DM-8007	S(PFP-OH)-8007	
Ibaraki, 2014 JP2014C125A (Taibyo Hikari)	3 (6 days)	2.5	Not stated	1	2.58	< 0.01	< 0.01	2020/2090138 Nakamura, 2015, BROFLAN_068 Storage time: max. 91 days (3 month) Procedural recoveries: 92-
				3	2.29	< 0.01	< 0.01	
				7	2.04	< 0.01	< 0.01	
Mie, 2015 JP2014C125B (Taibyo Hikari)	3 (7 days)	2.5	Not stated	1	1.95	< 0.01	< 0.01	
				3	1.62	< 0.01	< 0.01	
				7	1.49	< 0.01	< 0.01	

Location, Year, Trial No., (variety)	Application			DALA	Residues found [mg/kg][1]			Report, Reference, Storage period
	N (RTI)	g ai/hL	GS at final appl.		Broflanilide	DM-8007	S(PFP-OH)-8007	
Miyazaki, 2014 JP2014C125C (Taibyo Hikari)	3 (6-8 days)	2.5	Not stated	1 3 7	1.42 0.99 0.90	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	102% at 0.1 mg/kg

Notes:

[1] Mean values from analytical duplicate. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

Wheat forage, hay and straw

A total of 25 field trials were conducted on winter or spring wheat in Canada and the United States during the 2015–16 growing seasons (BROFLAN_070). The seeds were treated with broflanilide at a nominal concentration of 100 g ai/t of seed (8.14–13.3 g ai/ha). Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 (according to QuEChERS) with a validated limit of quantification of 0.001 mg/kg. A reduced validation set (n=3) for wheat forage at two levels (0.001 and 0.1 mg/kg) had acceptable mean recoveries within the range of 70–120 percent with an RSD < 20 percent for broflanilide, while for metabolites DM-8007 and S(PFP-OH)-8007 mean recoveries at the 0.1 mg/kg level were outside of this range at 125 percent and 144 percent, respectively. Procedural recoveries for all analytes were consistently > 120 percent at the 0.1 mg/kg level. Samples were stored up to 44 days before analysed. The results are shown in Table 114.

Table 114 Residues of broflanilide in wheat forage, hay and straw following seed treatment in Canada and the United States (Study 2016/7006466; Wyatt, 2017; BROFLAN_070)

Location, Year, Trial No., (variety)	Application	Sample	Days after planting	Residues found [mg/kg][1]		
	g ai/t seed (g ai/ha)			Broflanilide	DM-8007	S(PFP-OH)-8007
Canada, Minto, MB, 2015 R150019 (CDC Utmost)	104.3 (10.6)	Forage	31	< 0.001	< 0.001	< 0.001
		Hay	60	< 0.001	< 0.001	< 0.001
		Straw	97	< 0.001	< 0.001	< 0.001
Canada, Glenboro, MB, 2015 R150020 CDC Utmost)	104.3 (10.4)	Forage	26	< 0.001	< 0.001	< 0.001
		Hay	48	< 0.001	< 0.001	< 0.001
		Straw	89	< 0.001	< 0.001	< 0.001
Canada, Brandon, MB, 2015 R150021 (Glenn)	107.0 (10.7)	Forage	26	< 0.001	< 0.001	< 0.001
		Hay	54	< 0.001	< 0.001	< 0.001
		Straw	93	0.0011, <0.001 (0.001)	< 0.001	< 0.001
Canada, Alvena, SK, 2015 R150022 (CDC Go)	104.3 (10.4)	Forage	35	< 0.001	< 0.001	< 0.001
		Hay	68	<0.001, 0.0014 (0.012)[2]	< 0.001	< 0.001
		Straw	113	< 0.001	< 0.001	< 0.001
Canada, Wakaw, SK, 2015 R150023 (CDC Go)	104.3 (10.4)	Forage	34	< 0.001	< 0.001	< 0.001
		Hay	74	< 0.001	< 0.001	< 0.001
		Straw	109	< 0.001	< 0.001	< 0.001
Canada, Hepburn, SK, 2015 R150024 (Glenn)	107.0 (10.7)	Forage	36	< 0.001	< 0.001	< 0.001
		Hay	74	< 0.001	< 0.001	< 0.001
		Straw	112	< 0.001	< 0.001	< 0.001
Canada, Fort Saskatchewan, AB, 2015 R150025 (CDC Go)	104.3 (10.4)	Forage	36	< 0.001	< 0.001	< 0.001
		Hay	55	< 0.001	< 0.001	< 0.001
		Straw	123	< 0.001	< 0.001	< 0.001
United States, Seven Springs, NC, 2015, R150001 (Progeny 357)	95.2 (12.7)	Forage	158	< 0.001	< 0.001	< 0.001
		Hay	193	< 0.001	< 0.001	< 0.001
		straw	226	< 0.001	< 0.001	< 0.001

Location, Year, Trial No., (variety)	Application g ai/t seed (g ai/ha)	Sample	Days after planting	Residues found [mg/kg][1]		
				Broflanilide	DM-8007	S(PFP-OH)-8007
United States, Proctor, AR, 2015, R150002 (Progeny 357)	95.2 (9.53)	Forage	169	< 0.001	< 0.001	< 0.001
		Hay	196	< 0.001	< 0.001	< 0.001
		Straw	238	< 0.001	< 0.001	< 0.001
United States, Richland, IA, 2015, R150003 (Branson)	99.6 (9.78)	Forage	190	< 0.001	< 0.001	< 0.001
		Hay	220	< 0.001	< 0.001	< 0.001
		Straw	274	< 0.001	< 0.001	< 0.001
United States, Carlyle, IL, 2015 R150004 (Branson)	99.6 (9.64)	Forage	183	< 0.001	< 0.001	< 0.001
		Hay	212	< 0.001	< 0.001	< 0.001
		Straw	274	< 0.001	< 0.001	< 0.001
United States, York, NE, 2015, R150005 (Prosper)	105.3 (10.8)	Forage	29	< 0.001	< 0.001	< 0.001
		Hay	56	< 0.001	< 0.001	< 0.001
		Straw	83	< 0.001	< 0.001	< 0.001
United States, Stafford, KS, 2015, R150006 (TAM 111)	98.6 (13.3)	Forage	147	< 0.001	< 0.001	< 0.001
		Hay	194	< 0.001	< 0.001	< 0.001
		Straw	232	< 0.001	< 0.001	< 0.001
United States, Uvalde, TX, 2015, R150007 (TAM 113)	96.9 (9.50)	Forage	84	< 0.001	< 0.001	< 0.001
		Hay	145	< 0.001	< 0.001	< 0.001
		Straw	172	< 0.001	< 0.001	< 0.001
United States, Carrington, ND, 2015, R150008 (Glenn)	107.0 (10.7)	Forage	35	< 0.001	< 0.001	< 0.001
		Hay	57	< 0.001	< 0.001	< 0.001
		Straw	97	< 0.001	< 0.001	< 0.001
United States, Sykeston, ND, 2015 R150009 (Glenn)	107.0 (10.7)	Forage	35	< 0.001	< 0.001	< 0.001
		Hay	57	< 0.001	< 0.001	< 0.001
		Straw	102	< 0.001	< 0.001	< 0.001
United States, Grand Island, NE, 2015 R150010 (Prosper)	105.3 (11.5)	Forage	26	< 0.001	< 0.001	< 0.001
		Hay	57	< 0.001	< 0.001	< 0.001
		Straw	85	< 0.001	< 0.001	< 0.001
United States, Hastings, NE, 2015 R150011 (Prosper)	105.3 (10.9)	Forage	34	< 0.001	< 0.001	< 0.001
		Hay	57	< 0.001	< 0.001	< 0.001
		Straw	104	0.0011, <0.001 (0.001)[2]	< 0.001	< 0.001
United States, Jamestown, ND, 2015, R150012 (Prosper)	105.3 (10.8)	Forage	38	< 0.001	< 0.001	< 0.001
		Hay	57	< 0.001	< 0.001	< 0.001
		Straw	89	< 0.001	< 0.001	< 0.001
Canada, Taber, AB, 2015 R150013 (CDC Utmost)	104.3 (10.0)	Forage	34	< 0.001	< 0.001	< 0.001
		Hay	46	< 0.001	< 0.001	< 0.001
		Straw	96	< 0.001	< 0.001	< 0.001
United States, Plainview, TX, 2015 R150014 (TAM 111)	98.6 (10.1)	Forage	117	< 0.001	< 0.001	< 0.001
		Hay	152	< 0.001	< 0.001	< 0.001
		Straw	203	< 0.001	< 0.001	< 0.001
United States, Wall, TX, 2015 R150015 (TAM 113)	96.9 (8.14)	Forage	81	< 0.001	< 0.001	< 0.001
		Hay	133	< 0.001	< 0.001	< 0.001
		Straw	180	< 0.001	< 0.001	< 0.001
United States, Larned, KS, 2015 R150016 (TAM 111)	98.6 (13.3)	Forage	145	< 0.001, 0.0012 (0.0011)[2]	< 0.001	< 0.001
		Hay	197	< 0.001	< 0.001	< 0.001
		Straw	233	< 0.001	< 0.001	< 0.001
United States, Hinton, OK, 2015 R150017 (TAM 113)	96.9 (9.91)	Forage	161	< 0.001	< 0.001	< 0.001
		Hay	203	< 0.001	< 0.001	< 0.001
		Straw	253	< 0.001	< 0.001	< 0.001
United States, Payette, ID, 2015 R150018 (Alpowa)	101.2 (11.7)	Forage	25	< 0.001	< 0.001	< 0.001
		Hay	57	< 0.001	< 0.001	< 0.001
		Straw	99	< 0.001	< 0.001	< 0.001

Notes:

[1] Mean values in parentheses. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

[2] Average of several re-analyses.

Maize forage and stover

A total of 25 field trials were conducted on maize in Canada and the United States during the 2015 and 2016 growing seasons (BROFLAN_072). Plants received one in-furrow application, at planting at 50 g ai/ha. Maize forage was collected at 62–110 DAA and maize stover from 99-164 DAA. Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 (according to QuEChERS) with a LOQ of 0.001 mg/kg. Procedural recoveries ranged from 66 -186 percent at 0.001 and 0.1 mg/kg> Samples were stored for up to 315 days (forage) and for up to 672 days (stover) before analysis. The results are shown in Table 115.

Table 115 Residues of broflanilide in maize forage and stover following in-furrow treatment at planting in Canada and the United States (Study 2016/7006467; Wyatt, 2017; BROFLAN_072)

Location, Year, Trial No., (variety)	Application (g ai/ha)	Sample	DAA	Residues [mg/kg][1]		
				Broflanilide	DM-8007	S(PFP-OH)-8007
Canada, Taber, AB, 2016, R150222 (Sweet corn-Trinity)	50.6	Forage	91	< 0.001	< 0.001	< 0.001
		Stover	146	< 0.001	< 0.001	< 0.001
United States, Alton, NY, 2015, R150201 (A1027687)	50.0	Forage	81	< 0.001	< 0.001	< 0.001
		Stover	112	< 0.001	< 0.001	< 0.001
United States, Baptistown, NJ, 2015 R150202 (TA545-33EZ)	51.2	Forage	83	< 0.001	< 0.001	< 0.001
		Stover	139	< 0.001	< 0.001	< 0.001
United States, Newberry, FL, 2015 R150203 (Sweet corn-Passion II)	49.9	Forage	62	< 0.001	< 0.001	< 0.001
		Stover	112	< 0.001	< 0.001	< 0.001
United States, Conklin, MI, 2015 R150204 (DKC 46-20 RIB, A1031276)	49.7	Forage	92	< 0.001	< 0.001	< 0.001
		Stover	150	< 0.001	< 0.001	< 0.001
United States, Delavan, WI, 2016 R150205 (DKC 49-94 RIB)	50.6	Forage	89	< 0.001	< 0.001	< 0.001
		Stover	158	< 0.001	< 0.001	< 0.001
United States, Fitchburg, WI, 2015 R150206 (G95D32-3110)	49.9	Forage	80	< 0.001	< 0.001	< 0.001
		Stover	147	< 0.001	< 0.001	< 0.001
United States, Richland, IA, 2015 R150207 (Pioneer P1023AM)	49.9	Forage	78	< 0.001	< 0.001	< 0.001
		Stover	125	< 0.001	< 0.001	< 0.001
United States, Lime Springs, IA, 2016 R150208 (AI031275 DKC46-37 RIB)	50.4	Forage	94	< 0.001	< 0.001	< 0.001
		Stover	154	< 0.001	< 0.001	< 0.001
United States, Geneva, MN, 2016 R150209 (NuTech 5D-196AMX)	50.0	Forage	110	< 0.001	< 0.001	< 0.001
		Stover	164	< 0.001	< 0.001	< 0.001
United States, Manilla, IN, 2015 R150210 (Pioneer P0942AMX)	48.8	Forage	91	< 0.001	< 0.001	< 0.001
		Stover	140	< 0.001	< 0.001	< 0.001
United States, Highland, IL, 2015 R150211 (FS 63SV1 RIB)	49.9	Forage	99	< 0.001	< 0.001	< 0.001
		Stover	131	< 0.001	< 0.001	< 0.001
United States, Carlyle, IL, 2015 R150212 (N78N-3111)	51.0	Forage	97	< 0.001	< 0.001	< 0.001
		Stover	140	< 0.001	< 0.001	< 0.001
United States, Mason, IL, 2015 R150213 (N49W-3000GT)	50.8	Forage	92	< 0.001	< 0.001	< 0.001
		Stover	125	< 0.001	< 0.001	< 0.001
United States, Hedrick, IA, 2015 R150214 (Pioneer P1023AM)	50.0	Forage	93	< 0.001	< 0.001	< 0.001
		Stover	128	< 0.001	< 0.001	< 0.001
United States, Grandview, IA, 2015 R150215 (Pioneer P0506AM)	49.8	Forage	91	< 0.001	< 0.001	< 0.001
		Stover	126	< 0.001	< 0.001	< 0.001
United States, Kirksville, MO, 2015 R150216 (Pioneer P0506AM)	50.1	Forage	82	< 0.001	< 0.001	< 0.001
		Stover	125	< 0.001	< 0.001	< 0.001
United States, York, NE, 2015 R150217 (Pioneer P0876HR)	49.7	Forage	92	< 0.001	< 0.001	< 0.001
		Stover	142	< 0.001	< 0.001	< 0.001

Location, Year, Trial No., (variety)	Application (g ai/ha)	Sample	DAA	Residues [mg/kg][1]		
				Broflanilide	DM-8007	S(PFP-OH)-8007
United States, Osceola, NE, 2015 R150218 (Pioneer P0876HR)	50.1	Forage Stover	96 138	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Brunswick, NE, 2015 R150219 (DKC 54-40RIB)	50.7	Forage Stover	88 148	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Stafford, KS, 2016 R150220 (P1151AM)	49.8	Forage Stover	108 151	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Hinton, OK, 2015 R150221 (DKC 65-19)	50.0	Forage Stover	73 116	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Porterville, CA, 2015 R150223 (Sweet corn-Bodacious)	50.1	Forage Stover	77 109	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Ephrata, WA, 2015 R150224 (Sweet corn-Basin)	50.9	Forage Stover	84 105	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001
United States, Canby, OR, AB, 2015 R150225 (Sweet corn – Honey N Pearl-L)	51.2	Forage Stover	85 99	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001

Notes:

[1] Mean values in parentheses. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

Barley hay and straw

A total of 16 field trials were conducted on barley in Canada and the United States during the 2015 and 2016 growing seasons (BROFLAN_071). The seeds were treated with broflanilide at a nominal concentration of 100 g ai/ton of seed (9.52–10.7 g ai/ha). Barley hay was harvested at early milk to soft dough stage (BBCH 73 to 85) and allowed to field dry to 10 to 20 percent moisture content before being collected. Barley straw was harvested at grain maturity (BBCH 97–90) Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 (according to QuEChERS) with a LOQ of 0.001 mg/kg. Procedural recoveries ranged from 96–133 percent at 0.001 and 0.1 mg/kg. Samples were stored for up to 604 days (hay) or 561 days (straw) before analysed. The results are shown in Table 116.

Table 116 Residues of broflanilide in barley hay following seed treatment in Canada and the United States (Study 2016/7009340; Greenland, 2017; BROFLAN_07)

Location, Year, Trial No., (variety)	Application g ai/t seed (g ai/ha)	Sample	BBCH at harvest	Residues found [mg/kg][1]		
				Broflanilide	DM-8007	S(PFP-OH)-8007
Canada, Minto, MB, 2015 R150036 (AC Metcalfe)	101.1 (10.1)	Hay Straw	73-77 89	< 0.001	< 0.001	< 0.001
Canada, Blaine Lake, SK, 2015 R150037 (AC Metcalfe)	101.1 (9.88)	Hay Straw	77-83 89	< 0.001	< 0.001	< 0.001
Canada, Hague, SK, 2015 R150038 (AC Metcalfe)	101.1 (10.0)	Hay Straw	75-77 89	< 0.001	< 0.001	< 0.001
Canada, Neepawa, MB, 2016, R150039 (AC Metcalfe)	104.4 (9.84)	Hay Straw	77 89	< 0.001	< 0.001	< 0.001
Canada, Alvena, SK, 2015 R150040 (AC Metcalfe)	101.1 (10.0)	Hay Straw	75-77 89	< 0.001	< 0.001	< 0.001
Canada, Fort Saskatchewan, AB, 2015, R150041 (AC Metcalfe)	101.1 (9.52)	Hay Straw	83 87	< 0.001	< 0.001	< 0.001

Location, Year, Trial No., (variety)	Application g ai/t seed (g ai/ha)	Sample	BBCH at harvest	Residues found [mg/kg][1]		
				Broflanilide	DM-8007	S(PFP-OH)-8007
Canada, Taber, AB, 2015 R150029 (AC Metcalfe)	101.1 (10.0)	Hay Straw	73-83 89	< 0.001	< 0.001	< 0.001
United States, North Rose, NY, 2015, R150026 (Quest)	97.8 (9.97)	Hay Straw	73 89	< 0.001	< 0.001	< 0.001
United States, Gardner, ND, 2015, R150027 (Quest)	97.8 (10.5)	Hay Straw	85 89	0.0047, 0.0016 (0.0032)[2]	< 0.001	< 0.001
United States, Fitchburg, WI, 2015, R150028 (Quest)	97.8 (10.7)	Hay Straw	75 89	< 0.001	< 0.001	< 0.001
United States, Grand Island, NE, 2015, R150030 (Lacey)	94.8 (9.96)	Hay Straw	85 89	< 0.001	< 0.001	< 0.001
United States, Prosser, NE, 2016, R150031 (Lacey)	103.8 (10.4)	Hay Straw	75 89	< 0.001, 0.0026 (0.0018)	< 0.001	< 0.001
United States, Jamestown, ND, 2015, R150032 (Quest)	97.8 (10.6)	Hay Straw	75 90	< 0.001	< 0.001	< 0.001
United States, (Trenton, UT, 2015, R150033 (Lenetah)	99.1 (10.3)	Hay Straw	85 89	< 0.001	< 0.001	< 0.001
United States, Madera, CA, 2015, R150034 (Strider)	100.6 (10.4)	Hay Straw	75-85 89	< 0.001	< 0.001	< 0.001
United States, Aberdeen, ID, 2015, R150035 (Lenetah)	99.1 (10.1)	Hay Straw	85 89	< 0.001	< 0.001	< 0.001

Notes:

[1] Mean values in parentheses. The values of each metabolite were converted to the value of MCI-8007 (Conversion factor; DM-8007: 1.02, S(PFP-OH)-8007: 1.00).

[2] Replicate analyses for Broflanilide: replicate 1: 0.0027, 0.0062, and 0.0051 with an avg. of 0.0047 mg/kg; replicate 2: 0.0021, 0.0014, 0.0011, and 0.0019 with an avg. of 0.0016 mg/kg.

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of residue during processing

[B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide were incubated in aqueous citrate buffer solutions over the concentration range 0.1 to 1.5 mg/L under three sets of conditions, each designed to simulate an appropriate process: 90 °C (pH 4, 20 minutes) to simulate pasteurization, 100 °C (pH 5, 60 minutes), to simulate boiling, baking and brewing, and 120 °C (pH 6, 20 minutes) to simulate sterilization (Strathdee, 2017, BROFLAN_075; Amendment 1: BROFLAN_076).

Total recovered radioactivity was measured for each test solution by LSC. Radioactive components were characterized by fractionation and co-chromatography with authenticated reference compounds using HPLC-UV and TLC. Selected samples were also analysed by LC-MS to identify components.

Preliminary solubility testing was performed in aqueous media buffered to pH 4, 5 and 6 using ^{14}C-radiolabeled forms of Broflanilide over the concentration range 0.1 to 1.5 mg/L. The recovery of radioactivity indicated that Broflanilide in aqueous media adsorbed to glassware over these concentration ranges with 51.3 to 66.5 percent of the radioactivity recovered from the aqueous media. Subsequent adsorption to glassware testing indicated that the majority of radioactivity could be recovered by soaking

vessels with acetonitrile following removal of the [^{14}C]-buffered media with a total of ≥ 85.0 percent of the applied radioactivity recovered from the buffered media and acetonitrile washes (Table 117).

Table 117 Hydrolysis of broflanilide under simulated processing conditions, in percent of total applied radioactivity (Percent TAR)

Test Material	[C-ring-U-^{14}C]-Broflanilide			[B-ring-U-^{14}C]-Broflanilide		
	pH 4, 90 °C (20 min)	pH 5, 100 °C (60 min)	pH 6, 120 °C (20 min)	pH 4, 90 °C (20 min)	pH 5, 100 °C (60 min)	pH 6, 120 °C (20 min)
Total (prior to treatment) [1)]	80.4	75.7	72.0	110.3	109.4	91.7
Total (post treatment) [2)]	79.7	86.6	91.6	98.2	94.9	92.0
Broflanilide [3)]	78.6	85.6	89.9	97.2	93.8[4)]	86.6
Unknown [2) 3)]	1.1	1.0	1.7	1.0	1.1[5)]	5.4

Notes:

[1)] Mean of two replicates.
[2)] Mean of three replicates.
[3)] Sum of unknown peaks, each < 3% TAR.
[4)] Mean of two replicates.

Residues after processing

The fate of broflanilide during processing of raw agricultural commodity (RAC) was investigated in tomato, potato, maize, wheat and coffee. As a measure of the transfer of residues into processed products, a processing factor was used, which is defined as:

Processing factor = Residue in processed product (mg/kg) ÷ Residue in raw agricultural commodity (mg/kg)

If residues in the RAC were below the LOQ, no processing factor could be derived. In case of residues below the LOQ in the processed product, the numeric value of the LOQ was used for the calculation and the PF was expressed as “less than” (e.g. < 0.5).

Tomato

The transfer of residues of broflanilide into processed commodities was investigated in tomato from three supervised field trials conducted in the United States during the 2015 growing season (Reeves, 2020, BROFLAN_077). The trials were performed with two foliar applications at an exaggerated rate of 125 g ai/ha + spray adjuvant (non-ionic surfactant) at BBCH 86–89) with a RTI of 6-7 days and harvest at 1 DALA. Tomatoes were processed into the commodities of blanched tomatoes, blanching water, canned tomatoes, ketchup after pasteurization, paste, peeled tomatoes, puree, raw juice, sundried tomatoes, tomato peel, vegetable stock, wash water, washed tomatoes and wet pomace, using common commercial practices.

Tomato juice, paste and puree: Tomatoes were chopped, passed through an initial screen, heated to 91–97 °C and passed through another screen. The pulp material not passing was collected as wet pomace. The resultant tomato juice was further processed to puree and paste by evaporating water under heat.

Sun-dried tomatoes were obtained by slicing tomatoes and drying them in a dehydrator at 49–54 °C for 24–26 hours.

Canned tomatoes were produced by blanching tomatoes with steam for 1–3 minutes, followed submerging into water for removal of the skin. Tomatoes were placed in cans with heated tomato juice, sealed and pressure cooked for 50–60 minutes.

Blanched tomatoes were placed into boiling water for 30–60 seconds.

Peeled tomatoes were obtained by peeling by hand. The remaining peeled tomatoes were blended and sieved. The material passing through the sieve was collected as raw juice. Alternatively, raw juice was produced by using a juicer.

Ketchup was produced by evaporation of tomato juice to a solid content of 30–35 percent, followed by adding vinegar, sugar and salt. After stirring, the mixture was passed through a sieve. The material passing through was ketchup.

Vegetable stock was produced by cooking diced and cored tomatoes with water for 45–60 minutes, followed by sieving. The material passing through was vegetable stock.

Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg. Procedural recoveries for all analytes in the RAC and all processed commodities ranged between 71.2–130 percent.

Residue of broflanilide in the RAC and processed commodities are presented in Table 130. Residues of metabolites S(PFP-OH)-8007 and DM-8007 in tomato RAC samples were below LOQ (< 0.001 mg/kg). In processed fractions, residues of metabolite S(PFP-OH)-8007 were below LOQ (< 0.001 mg/kg) in all samples except for sun-dried tomatoes (up to 0.0045 mg/kg), tomato peel (up to 0.0015 mg/kg) and wet pomace (up to 0.0024 mg/kg). Residues of metabolite DM-8007 were below LOQ (< 0.001 mg/kg) in all processed fraction samples except for sun-dried tomatoes (up to 0.0054 mg/kg), tomato peel (up to 0.0013 mg/kg) and wet pomace (up to 0.0016 mg/kg) (Table 118).

Table 118 Summary of broflanilide residues in processed tomato commodities

Trial Identification Location (variety)	Commodity or Matrix	Broflanilide (mg/kg)	Processing Factor
R150260 Clermont, FL, United States, 2015 251 g ai/ha DALA= 1 day (Better Boy)	Fruit RAC	0.033*	-
	Tomato Processor RAC	0.050	-
	Blanched tomatoes	0.0019	0.038
	Blanching water	< 0.001	< 0.02
	Canned tomatoes	0.015	0.3
	Ketchup after pasteurization	0.100	2.0
	Paste	0.110	2.2
	Raw juice	0.033	0.66
	Tomato peel	0.140	2.8
	Sun-dried tomatoes	0.340	6.8
	Wash water	< 0.001	< 0.02
	Washed tomatoes	0.028	0.56
	Vegetable stock	0.0015	0.03
	Wet pomace	0.024	0.48
	Peeled tomatoes	0.0033	0.066
	Puree	0.034	0.68
R150261 Sanger, CA, United States, 2015 245 g ai/ha DALA=1 day (UG 19406)	Fruit RAC	0.0715*	-
	Tomato Processor RAC	0.069	-
	Blanched tomatoes	< 0.001	< 0.014
	Blanching water	< 0.001	< 0.014
	Canned tomatoes	0.0053	0.077
	Ketchup after pasteurization	0.026	0.38
	Paste	0.024	0.35

Trial Identification Location (variety)	Commodity or Matrix	Broflanilide (mg/kg)	Processing Factor
	Raw juice	0.0059	0.086
	Tomato peel	0.300	4.35
	Sun-dried tomatoes	0.740	10.7
	Wash water	0.016	0.23
	Washed tomatoes	0.066	0.96
	Vegetable stock	0.0032	0.046
	Wet pomace	0.270	3.9
	Peeled tomatoes	0.016	0.23
	Puree	0.0096	0.14
R150262 Stratford, CA United States, 2015 252 g ai/ha DALA= 1 day (Roma 5608)	Fruit RAC	0.0375*	-
	Tomato Processor RAC	0.036	-
	Blanched tomatoes	< 0.001	< 0.028
	Blanching water	< 0.001	< 0.028
	Canned tomatoes	0.0042	0.12
	Ketchup after pasteurization	0.082	2.3
	Paste	0.079	2.2
	Raw juice	0.0088	0.24
	Tomato peel	0.150	4.17
	Sun-dried tomatoes	0.170	4.7
	Wash water	0.014	0.39
	Washed tomatoes	0.022	0.61
	Vegetable stock	0.0021	0.058
	Wet pomace	0.099	2.8
	Peeled tomatoes	0.0037	0.10
	Puree	0.032	0.89

Notes:

RAC: Raw agricultural commodity.

* Mean of two replicate samples.

Potato

The transfer of residues of broflanilide into processed commodities was investigated in potato from three supervised field trials conducted in the United States during the 2015 growing season (Crawford, 2017, BROFLAN_078). The trials were performed with one in-furrow application at planting, followed by one foliar application 14 days prior to harvest (BBCH 45–48) at exaggerated rates of 126–128 g ai/ha for the first application and 125–149 g ai/ha for the second application + adjuvant. Potato samples were collected at 14 DALA and for trial R150085 at 23 DALA. Potatoes were processed into peeled potatoes, wet peel, boiled potatoes, microwave boiled potatoes, baked potatoes, fried potatoes, crisps, chips, granules/flakes, process waste, ensiled, starch, dried pulp and protein using simulated commercial processing procedures. Samples were stored frozen for a maximum of 344 days (11.3 months).

Potato flakes were produced by removing the peel by using a steam peeler. Peels were collected and pressed. The peeled potatoes were cut into slabs, washed to remove starch and steam cooked for 35–45 minutes. The wash water was filtered and centrifuged to obtain the starch sample. The cooked slices were mashed, followed by mixing with additives and drying the potato mash in a single drum dryer. The resultant sheet is broken up and fed into a hammer mill for a uniform sizing of the finished flakes.

Potato crisp and fried potatoes were produced by peeling washed potatoes with Hobart peeler, and cut to slices. After washing to remove free starch, the slices were fried in oil at 163–191 °C for 90–100 seconds (crisps) or 3 minutes (fried potatoes).

Boiled potatoes (hand peeled) and microwave boiled potatoes (unpeeled) were obtained by boiling in water until an internal temperature of 88–92 °C was reached. Baked potatoes were placed in an oven at 210 °C till the same internal temperature was reached.

Fresh fries were produced by slicing washed unpeeled potatoes into strips, followed by deep frying for 2.5–3.0 minutes at 177–191 °C

Potato pulp and protein were produced by milling washed potatoes, followed by pressing. The pressed wet pulp was placed in a dryer for dehydration to obtain the dried pulp. The potato fruit water collected in pressing was centrifuged and filtered, followed by thermal processing at ~90 °C while adjusting the pH to ~4 with sulphuric acid to precipitate the protein. To obtain ensiled potatoes, an aliquot of the milled potatoes was placed in a bag silo, sealed and kept at room temperature for 3 weeks.

Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg. Procedural recoveries for all analytes in the RAC and all processed commodities ranged between 55.6–129 percent.

Residue of broflanilide in the RAC and processed commodities are presented in Table 131. Residues of metabolites S(PFP-OH)-8007 and DM-8007 in potato field RAC and processor RAC samples were below LOQ (< 0.001 mg/kg), except for trial R150085, where residues of DM-8007 were at 0.001 mg/kg. In processed fractions, residues of metabolite S(PFP-OH)-8007 were below LOQ (< 0.001 mg/kg) in all samples. Residues of metabolite DM-8007 were below LOQ (< 0.001 mg/kg) in all processed fraction samples except for wet peel (up to 0.005 mg/kg), dried pulp (up to 0.003 mg/kg) and potato protein (up to 0.0014 mg/kg) (Table 119)

Table 119 Summary of broflanilide residues in processed potato commodities

Trial Identification Location (variety)	Commodity or Matrix	Broflanilide (mg/kg)	Processing Factor
R150085 North Rose, NY, United States, 2015 275 g ai/ha DALA= 23 (Red Norland)	Tuber Field RAC	0.008	-
	Tuber Processor RAC	0.010	-
	Peeled potato	< 0.001	< 0.10
	Peel, wet	0.035	3.5
	Boiled potatoes	< 0.001	< 0.10
	Microwave/boiled potatoes (unpeeled)	0.0059	0.59
	Baked potato	0.0018	0.18
	Fried potato	< 0.001	< 0.10
	Crisps (fries)	< 0.001	< 0.10
	Chips	0.0016	0.16
	Granules/Flakes	< 0.001	< 0.10
	Process waste	0.0069	0.69
	Ensiled	0.0043	0.43
	Starch	0.0027	0.27
	Dried pulp	0.023	2.3
	Potato protein	0.018	1.8
R150086 Weedsport, NY United States, 2015 253 g ai/ha DALA=14 (Chieftain)	Tuber Field RAC	0.0074	-
	Tuber Processor RAC	0.0054	-
	Peeled potato	< 0.001	< 0.19
	Peel, wet	0.013	2.4
	Boiled potatoes	< 0.001	< 0.19
	Microwave/boiled potatoes (unpeeled)	0.0012	0.22

Trial Identification Location (variety)	Commodity or Matrix	Broflanilide (mg/kg)	Processing Factor
	Baked potato	0.0011	0.20
	Fried potato	< 0.001	< 0.19
	Crisps (fries)	< 0.001	< 0.19
	Chips	< 0.001	< 0.19
	Granules/Flakes	< 0.001	< 0.19
	Process waste	< 0.001	< 0.19
	Ensiled	0.0019	0.35
	Starch	< 0.001	< 0.19
	Dried pulp	0.0064	1.2
	Potato protein	0.0077	1.4
R150087 American Falls, ID, United States, 2015 252 g ai/ha DALA= 14 (Russet Burbank)	Tuber Field RAC	0.010	-
	Tuber Processor RAC	0.017	-
	Peeled potato	< 0.001	< 0.06
	Peel, wet	0.015	0.88
	Boiled potatoes	< 0.001	< 0.06
	Microwave/boiled potatoes (unpeeled)	0.0026	0.15
	Baked potato	0.0031	0.18
	Fried potato	< 0.001	< 0.06
	Crisps (fries)	< 0.001	< 0.06
	Chips	< 0.001	< 0.06
	Granules/Flakes	< 0.001	< 0.06
	Process waste	0.002	0.12
	Ensiled	0.0026	0.15
	Starch	< 0.001	< 0.06
	Dried pulp	0.0093	0.55
	Potato protein	0.0190	1.1

Notes:

RAC: Raw agricultural commodity.

Maize

The transfer of residues of broflanilide into processed commodities was investigated in maize from 3 supervised field trial conducted in Brazil during the 2015 growing season (Jordan, 2017, BROFLAN_079). The trials were performed with two foliar applications at an exaggerated rate of 90 g ai/ha + oil adjuvant at BBCH 85 and 87 with a RTI of 10 days and harvest at maturity, 14 DALA. At one site, due to a calculation error by the field investigator, the corn was treated at 9 g ai/ha (Trial R150325), one-tenth the targeted rate due to an oversight by the field investigator. Maize was processed to bran, dry milling grits, dry milling meal, dry milling flour, flour-wet process, germ, germ-wet milling, gluten, gluten feed meal, milled by-products, wet milling RBD oil, dry milling RBD oil, wet milling starch using common commercial practices. Samples were stored frozen for a maximum of 205 days (7 months).

Before processing, whole corn samples were dried (if necessary), followed by cleaning by means of aspiration and screening. Screening and light impurities were combined to produce milled by-products.

Dry milling process: cleaned whole corn was moisture conditioned to 20–22 percent, followed by milling. The corn stock from the mill was dried and screened using sieves of various sizes to obtain bran,

germ, and large grits from grits, meal and flour. The germ was dried and flaked, followed by extraction with hexane in a batch extractor. Resulting factions from the solvent extraction were miscella and solvent extracted germs. Miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. The crude oil was alkali refined, resulting in soapstock and alkali refined oil. While the soapstock was discarded, the alkali refined oil was beached, filtered and steam bathed at 220–230 °C to obtain refined bleached-deodorized oil (RBD oil).

Wet milling process: cleaned whole corn was steeped in 49–54 °C water containing 0.1–0.2 percent sulfur dioxide (sulphurous acid), followed by milling and removal of germ and hull using a hydroclone. Germ and hull were dried and separated by screening. The germ was further processed as described for dry milling. The corn stock was screened. The material not passing through was discarded, while the process water (with starch and gluten) passing through the screen was batch centrifuged to separate the starch and gluten. Both fractions were dried subsequently.

Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg. Procedural recoveries for all analytes in the RAC and all processed commodities ranged between 68–132 percent.

Residue of broflanilide in the RAC and processed commodities are presented in Table 132. Residues of metabolites S(PFP-OH)-8007 and DM-8007 in maize grain RAC and processed commodities were below LOQ (< 0.001 mg/kg) (Table 120).

Table 120 Summary of broflanilide residues in processed maize commodities

Trial Identification Location (variety)	Commodity or Matrix	Broflanilide (mg/kg)	Processing Factor
R150324 Ponta Grossa, Paraná, Brazil, 2015 201 g ai/ha DALA=14 (LG6030)	Grain RAC	0.0062	-
	Bran	0.0089	1.44
	Dry Milling Grits	0.0014	0.23
	Dry Milling Meal	0.0082	1.32
	Dry Milling Flour	0.013	2.10
	Flour-Wet Process	< 0.001	< 0.16
	Germ	0.0046	0.74
	Germ (Wet Milling)	0.0076	1.23
	Gluten	0.010	1.61
	Gluten Feed Meal	0.042	6.77
	Milled By-products	0.039	6.29
	Wet Milling RBD Oil	0.0022	0.35
	Dry Milling RBD Oil	0.005	0.81
	Wet Milling Starch	< 0.001	< 0.16
R150325 Santo Antonio de Posse, São Paulo Brazil, 2015 20 g ai/ha DALA=14 (30F53YH)	Grain RAC	< 0.001	-
	Bran	< 0.001	N/A
	Dry Milling Grits	< 0.001	N/A
	Dry Milling Meal	< 0.001	N/A
	Dry Milling Flour	< 0.001	N/A
	Flour-Wet Process	< 0.001	N/A
	Germ	< 0.001	N/A
	Germ (Wet Milling)	< 0.001	N/A
	Gluten	< 0.001	N/A
	Gluten Feed Meal	< 0.001	N/A
	Milled By-products	0.002	N/A
	Wet Milling RBD Oil	< 0.001	N/A
	Dry Milling RBD Oil	< 0.001	N/A
	Wet Milling Starch	< 0.001	N/A

Trial Identification Location (variety)	Commodity or Matrix	Broflanilide (mg/kg)	Processing Factor
R150326 Estrela do Sul, Minas Gerais Brazil, 2015 183 g ai/ha DALA=14 (NS90PRO2)	Grain RAC	< 0.001	-
	Bran	0.002	N/A
	Dry Milling Grits	< 0.001	N/A
	Dry Milling Meal	0.0015	N/A
	Dry Milling Flour	0.002	N/A
	Flour-Wet Process	< 0.001	N/A
	Germ	0.001	N/A
	Germ (Wet Milling)	< 0.001	N/A
	Gluten	0.0013	N/A
	Gluten Feed Meal	0.0036	N/A
	Milled By-products	0.016	N/A
	Wet Milling RBD Oil	< 0.001	N/A
	Dry Milling RBD Oil	< 0.001	N/A
	Wet Milling Starch	< 0.001	N/A

Notes:

RAC: Raw agricultural commodity.

Wheat

The transfer of residues of broflanilide into processed commodities was investigated in wheat from 3 supervised field trial conducted in Brazil during the 2015 growing season (Jordan, 2017, BROFLAN_080). The trials were performed with two foliar applications at an exaggerated rate of 90 g ai/ha + non-ionic adjuvant at BBCH 73 and 77–83 with a RTI of 10 days. Wheat grain was harvested at maturity, 14 DALA. Wheat grains were processed into wheat meal, bran, flour, middlings, shorts and germ and various other wheat processed fractions, according to simulated commercial procedures. Samples were stored frozen for a maximum of 351 days (~12 months).

Before processing, whole wheat grain samples were dried (if necessary), followed by cleaning by means of aspiration and screening. Screening and light impurities were combined to produce milled by-products.

For germ production a sample of the cleaned wheat grain was moisture adjusted and passed through a disc mill, followed by sieving. The material on top of the sieve was aspirated to remove the bran from the germ fraction. The germ and reduced endosperm was separated by sieving with multiple screens The germ material was aspirated once more to remove additional bran and endosperm.

For flour production, breaking of a sample of cleaned, moisture adjusted wheat grain was accomplished by three break rolls. The material passing through the break rolls was fed onto a break sifter screen. The material passing through the 120 screen is Break flour, while the material not passing was passed through the 25 screen. The material passing through the 25 screen is middlings, while the material exiting is bran. The middlings were fed into a reduction system, using two reductions rolls. After passing through the reduction rolls, the material was fed onto a reduction sifter screen. The material passing through is reduction flour, while the material not passing through is shorts. Representative amounts of break and reduction flours were combined to receive white flour.

For production of whole meal flour and whole meal bread, cleaned wheat grains were milled in a pin mill. The resultant whole meal flour was used to bake bread using a bread machine.

For production of gluten, gluten feed meal and starch, break flour was mixed with water. The resultant dough was kneaded as water washed away the starch, leaving the gluten behind. The starch was

separated from the water using centrifugation and dried in a dehydrator. For gluten feed meal, reduction flour is used instead of break flour, but the same procedure is utilized.

Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg. Procedural recoveries for all analytes in the RAC and all processed commodities ranged between 66–137 percent.

Residue of broflanilide in the RAC and processed commodities are presented in Table 133. Residues of metabolites S(PFP-OH)-8007 and DM-8007 in wheat grain RAC and processed fractions, were below LOQ (< 0.001 mg/kg) in all samples, except for milled by-products (up to 0.0079 mg/kg for S(PFP-OH)-8007; 0.0049 mg/kg for DM-8007), germ (up to 0.0013 mg/kg for S(PFP-OH)-8007; 0.0015 mg/kg for DM-8007) and gluten (up to 0.0015 mg/kg for S(PFP-OH)-8007; 0.0023 mg/kg for DM-8007) (Table 121)

Table 121 Summary of broflanilide residues in processed wheat commodities

Trial Identification Location (variety)	Commodity or Matrix	Broflanilide (mg/kg)	Processing Factor
R150270 Castro, Paraná, Brazil, 2015 180 g ai/ha DALA=14 (TBIO Sinuelo)	Grain RAC	0.12	-
	Bran	0.15	1.24
	Flour	0.038	0.30
	Middlings	0.047	0.38
	Shorts	0.09	0.72
	Gluten	0.10	0.80
	Gluten Feed Meal	0.082	0.66
	Milled By-products	1.04	8.3
	Starch	0.0052	0.04
	Germ	0.35	2.77
	Whole Meal Flour	0.12	0.98
	Whole Grain Bread	0.0079	0.63
R150271 Ponta Grossa, Paraná, Brazil, 2015 180 g ai/ha DALA=14 (TBIO Sinuelo)	Grain RAC	0.14	-
	Bran	0.13	0.92
	Flour	0.074	0.54
	Middlings	0.058	0.42
	Shorts	0.064	0.47
	Gluten	0.68	4.97
	Gluten Feed Meal	0.28	2.07
	Milled By-products	1.67	12.2
	Starch	0.0033	0.02
	Germ	0.24	1.75
	Whole Meal Flour	0.086	0.63
	Whole Grain Bread	0.11	0.78
R150272 Tibagi, Paraná, Brazil, 2015 180 g ai/ha DALA=14 (TBIO Sinuelo)	Grain RAC	0.18	-
	Bran	0.16	0.90
	Flour	0.081	0.44
	Middlings	0.061	0.33
	Shorts	0.065	0.36
	Gluten	0.75	4.14
	Gluten Feed Meal	0.33	1.83
	Milled By-products	1.23	6.8
	Starch	0.0038	0.02
	Germ	0.21	1.14
	Whole Meal Flour	0.093	0.51
	Whole Grain Bread	0.083	0.45

Notes:

RAC: Raw agricultural commodity.

Coffee

The transfer of residues of broflanilide into processed commodities was investigated in coffee beans from three supervised field trial conducted in Brazil during the 2018 growing season (José, 2019, BROFLAN_081). The trials were performed with two foliar applications at an exaggerated rate of 90 g ai/ha + non-ionic adjuvant at BBCH 75 and 81 with a RTI of 30 days. Sampling was done at 45 DALA. Coffee beans were dried and processed into concentrated liquor, instant coffee and roasted and ground beans. Samples were stored frozen for a maximum of 263 days (~9 months).

To produce roasted coffee, the green beans were placed in a roster and roasted for 20 minutes at 200 °C. After equilibration for > 18 hours, the roasted beans were grinded in a cone mill.

For liquor extract coffee, beans were roasted for 80–90 minutes at 180 °C. After equilibration for > 18 hours, the bean were broken up in a cone mill and sieved for fines removal. The ground coffee was extracted in columns with 92±4 °C hot water for 15±2 minutes. The extract was dried using a spray dryer for the production of instant coffee.

Residues of broflanilide and metabolites DM-8007 and S(PFP-OH)-8007 were determined using method D1417/01 with a LOQ of 0.001 mg/kg. Procedural recoveries for all analytes in the RAC and all processed commodities ranged between 70.5–134 percent.

Residue of broflanilide in the RAC and processed commodities are presented in Table 134. Residues of metabolites S(PFP-OH)-8007 and DM-8007 in coffee beans (RAC) and processed fractions, were below LOQ (< 0.001 mg/kg) in all samples, except for dried coffee cherry (up to 0.0091 mg/kg for S(PFP-OH)-8007; 0.0058 mg/kg for DM-8007). The results are shown in Table 122.

Table 122 Summary of broflanilide residues in processed coffee commodities

Trial Identification Location (variety)	Commodity or Matrix	Broflanilide (mg/kg)	Processing Factor
G172053 186 g ai/ha DALA=45 Rio Claro, SP, Brazil, 2018 (Catuai amarelo)	Coffee beans (RAC)	0.0072	-
	Dried coffee cherry	0.28	39
	Concentrated liquor	< 0.001	< 0.14
	Instant coffee	< 0.001	< 0.14
	Roasted and ground coffee beans	0.0059	0.82
G172054 188 g ai/ha DALA=45 Campinas, SP, Brazil, 2018 (Catuai amarelo)	Coffee beans (RAC)	0.011	-
	Dried coffee cherry	0.47	43
	Concentrated liquor	< 0.001	< 0.09
	Instant coffee	< 0.001	< 0.09
	Roasted and ground coffee beans	0.0042	0.38
G172055 186 g ai/ha DALA=45 Leme, SP, Brazil, 2018 (Mundo Novo)	Coffee beans (RAC)	0.0039	-
	Dried coffee cherry	0.33	85
	Concentrated liquor	< 0.001	< 0.26
	Instant coffee	< 0.001	< 0.26
	Roasted and ground coffee beans	0.0092	2.36

Notes:

RAC: Raw agricultural commodity.

Table 123 Overview of processing factors

Raw commodity	Processed commodity	Individual processing factors	Median or best estimate processing factor
Tomato	Blanched tomatoes	< 0.014, < 0.028, 0.038,	0.038
	Blanching water	< 0.014, < 0.02, < 0.028	< 0.028
	Canned tomatoes	0.077, 0.12, 0.3	0.12
	Ketchup after pasteurization	0.38, 2.0, 2.3	2.0
	Paste	0.35, 2.2, 2.2	2.2
	Raw juice	0.086, 0.24, 0.66	0.24
	Tomato peel	2.8, 4.17, 4.35	4.17
	Sun-dried tomatoes	4.7, 6.8, 10.7	6.8
	Wash water	< 0.02, 0.23, 0.39	0.23
	Washed tomatoes	0.56, 0.61, 0.96	0.61
	Vegetable stock	0.03, 0.046, 0.058	0.046
	Wet pomace	0.48, 2.8, 3.9	2.8
	Peeled tomatoes	0.066, 0.10, 0.23	0.10
	Puree	0.14, 0.68, 0.89	0.68
Potato	Peeled potato	< 0.06, < 0.10, < 0.19	< 0.19
	Peel, wet	0.88, 2.4, 3.5	2.4
	Boiled potatoes	< 0.06, < 0.10, < 0.19	< 0.19
	Microwave/boiled potatoes (unpeeled)	0.15, 0.22, 0.59	0.22
	Baked potato	0.18, 0.18, 0.20	0.18
	Fried potato	< 0.06, < 0.10, < 0.19	< 0.10
	Crisps (fries)	< 0.06, < 0.10, < 0.19	< 0.10
	Chips	< 0.06, 0.16, < 0.19	0.16
	Granules/Flakes	< 0.06, < 0.10, < 0.19	< 0.10
	Process waste	0.12, < 0.19, 0.69	< 0.19
	Ensiled	0.15, 0.35, 0.43	0.35
	Starch	< 0.06, < 0.19, 0.27	0.27
	Dried pulp	0.55, 1.2, 2.3	1.2
	Potato protein	1.1, 1.4, 1.8	1.4
Maize	Bran	1.44	1.44
	Dry Milling Grits	0.23	0.23
	Dry Milling Meal	1.32	1.32
	Dry Milling Flour	2.10	2.10
	Flour-Wet Process	< 0.16	< 0.16
	Germ	0.74	0.74
	Germ (Wet Milling)	1.23	1.23
	Gluten	1.61	1.61
	Gluten Feed Meal	6.77	6.77
	Milled By-products	6.29	6.29
	Wet Milling RBD Oil	0.35	0.35
	Dry Milling RBD Oil	0.81	0.81
	Wet Milling Starch	< 0.16	< 0.16
Wheat	Bran	0.90, 0.92, 1.24	0.92
	Flour	0.30, 0.44, 0.54	0.44
	Middlings	0.33, 0.38, 0.42	0.38
	Shorts	0.36, 0.47, 0.72	0.47
	Gluten	0.80, 4.14, 4.97	4.14
	Gluten Feed Meal	0.66, 1.83, 2.07	1.83
	Milled By-products	6.8, 8.3, 12.2	8.3
	Starch	0.02, 0.02, 0.04	0.02
	Germ	1.14, 1.75, 2.77	1.75
	Whole Meal Flour	0.51, 0.63, 0.98	0.63
	Whole Grain Bread	0.45, 0.63, 0.78	0.63

Raw commodity	Processed commodity	Individual processing factors	Median or best estimate processing factor
Coffee	Dried coffee cherry	39, 43, 85	43
	Concentrated liquor	< 0.09, < 0.14, < 0.26	< 0.14
	Instant coffee	< 0.09, < 0.14, < 0.26	< 0.14
	Roasted and ground coffee beans	0.38, 0.82, 2.36	0.82

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Lactating cows

The transfer of residues of broflanilide into animal matrices was investigated in a study with dairy cows for 43 consecutive days in order for milk residues to reach plateau (Xu, 2019, BROFLAN_082). The experimental design is presented in Table 124.

Table 124 Dosing regimen for broflanilide in lactating cows

Treatment group	Actual dose [ppm feed (DM basis)] [1)]	Dose amount (mg/day)[2)]	Average dose mg ai/kg bw/ day[3)]
Control	0	0	0
I	0.015	0.33	0.00062
II	0.152	3.7	0.00672
III	1.50	34.7	0.06468
IV	10.1	239.2	0.43586

Notes:

[1)] Based on average mg Broflanilide received per day and the body weight of each animal during the dosing period.

[2)] Dose was calculated as mean over all 6 weeks of dose amount mg/kg day (actual dose mg/kg feed DM multiplied by dry feed intake in kg/day).

[3)] Average dose (mg ai/kg bw) was calculated by dose amount (mg/kg day) divided by mean body weight of animals per group. The body weights were calculated as mean per dose group from page 352 (Control: 514.5 kg, Group I: 534.5 kg, Group II: 551 kg, Group III: 536.5 kg, Group IV: 548.8 kg).

Three cows per treatment group (+ three cows for depuration at 10 ppm) were dosed via oral bolus with broflanilide in gelatin capsules once daily. Milk samples were collected twice daily and pooled on study days (-1), 1, 4, 7, 10, 13, 16, 20, 22, 25, 27, 30, 34, 37, and 41. Additional milk samples were taken on the depuration cows on study days 44, 48, and 55. Cream and whey samples were taken from all the study animals on study day 22 and additionally from the depuration cows on their necropsy days on study days 41, 44, 48, and 55. All cows were sacrificed within 24 hours of administration of the last dose, except the three depuration cows, which were necropsied at 3, 7, and 14 days following the last dose. Samples of liver, kidney, muscle, perirenal fat, mesenterial fat and subcutaneous fat were collected and taken for analysis.

Samples were analysed for parent broflanilide as well as metabolites DM-8007 and DC-DM-8007 using method D1604/01 with a LOQ of 0.001 mg/kg for milk and 0.01 mg/kg for tissues. Procedural recoveries for all matrices and analytes ranged between 75.9–118 percent. LOD for residues in livestock samples was set at 20 percent of the LOQ, equivalent to 0.0002 mg/kg for milk per analyte and 0.002 mg/kg for fat, liver, kidney and muscle. Maximum storage time for milk was 43 days, muscle 28 days, liver and kidney 36 days and fat 42 days.

Residues of parent broflanilide above LOQ were detected in milk only in the highest treatment group (10 ppm) at up to 0.0018 mg/kg (mean over 41 days at 0.001 mg/kg). A plateau in milk was reached after 7 days. In cream, broflanilide was detected >LOQ in the 1.5 and 10 ppm treatment groups, while in skim milk, broflanilide was consistently below LOD (Table 125).

Residues of metabolite DM-8007 were detected at levels >LOQ in milk samples from cows dosed at 0.15 ppm, 1.5 ppm and 10 ppm. A plateau in milk was reached after 7 days in cows dosed at 10 ppm. In cream, DM-8007 was detected at levels >LOQ in all dosing groups (Table 126). Residues of DC-DM-8007 were only sporadically found above LOQ in milk from the 10 ppm dose group. In cream, DC-DM-8007 was detected >LOQ in the 1.5 and 10 ppm treatment groups, while in skim milk, it was consistently below LOD (Table 127).

Table 125 Residues of broflanilide in milk (including skim milk and cream)

Study Day	Broflanilide: group mean residue (*highest individual*) in mg/kg				
	Control Group *(0 ppm)*	Group I *(0.015 ppm)*	Group II *(0.15 ppm)*	Group III *(1.5 ppm)*	Group IV *(10 ppm)*
-1	<LOD	<LOD	<LOD	<LOD	<LOD
1	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00024) *0.00035*
4	<LOD	<LOQ (0.00039) *0.00077*	<LOD	<LOD	<LOD (0.00072) *0.0009*
7	<LOD	<LOD	<LOD	<LOQ (0.00021) *0.00022*	<LOQ (0.00098) *0.0017*
10	<LOD	<LOD	<LOD	<LOQ (0.00021) 0.00024	<LOQ (0.0010) *0.0012*
13	<LOD	<LOD	<LOD	<LOQ (0.0002) *0.00021*	<LOQ (0.00097) *0.0014*
16	<LOD	<LOD	<LOD	<LOD	0.00122 *0.0018*
20	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00095) *0.0015*
22	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00062) *0.0011*
25	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00088) *0.0014*
27	<LOD	<LOQ (0.00027) *0.00041*	<LOQ (0.00027) *0.00041*	<LOQ (0.00027) *0.00035*	0.00137 *0.0018*
30	<LOD	<LOQ (0.00037) *0.0007*	<LOD	<LOQ (0.00028) *0.00035*	0.00118 *0.0015*
34	<LOD	<LOD	<LOD	<LOQ (0.00022) *0.00025*	0.00109 *0.0013*
37	<LOD	<LOQ (0.0004) *0.00081*	<LOQ (0.00035) *0.00065*	<LOD	0.00091 *0.0012*
41	<LOD	<LOD	<LOD	<LOQ (0.00021) *0.00022*	0.00091 *0.0013*
44	-	-	-	-	<LOD
48	-	-	-	-	<LOD
55	-	-	-	-	<LOD

Study Day	Broflanilide: group mean residue (*highest individual*) in mg/kg				
	Control Group *(0 ppm)*	Group I *(0.015 ppm)*	Group II *(0.15 ppm)*	Group III *(1.5 ppm)*	Group IV *(10 ppm)*
Mean (Days 27-41)	<LOD	<LOQ (0.00029)	<LOQ (0.00024)	<LOQ (0.00023)	<LOQ(0.00099)
Cream (Day22)	<LOQ (0.0005) *0.0011*	<LOQ (0.00021) *0.00023*	<LOQ (0.00036) *0.00048*	0.0034 *0.0051*	0.0143 *0.016*
Cream (Day41)	-	-	-	-	0.0117 *0.015*
Cream (Day44)	-	-	-	-	0.0017 *0.0017*
Cream (Day48)	-	-	-	-	<LOD
Cream (Day55)	-	-	-	-	<LOD
Skim milk (Day22)	<LOD	<LOD	<LOD	<LOD	<LOD
Skim milk (Day41)	-	-	-	-	<LOD
Skim milk (Day44)	-	-	-	-	<LOD
Skim milk (Day48)	-	-	-	-	<LOD
Skim milk (Day55)	-	-	-	-	<LOD

Table 126 Residues of metabolite DM-8007 in milk (including skim milk and cream)

Study Day	DM-8007: group mean residue (*highest individual*) in mg/kg				
	Control Group *(0 ppm)*	Group I *(0.015 ppm)*	Group II *(0.15 ppm)*	Group III *(1.5 ppm)*	Group IV *(10 ppm)*
-1	<LOD	<LOD	<LOD	<LOD	<LOD
1	<LOD	<LOD	<LOQ (0.00025) *0.00034*	0.0018 *0.0024*	0.0155 *0.022*
4	<LOD	<LOD	<LOQ (0.00085) *0.0012*	0.0057 *0.0070*	0.047 *0.059*
7	<LOD	<LOD	0.00114 *0.0013*	0.0093 *0.01*	0.067 *0.083*
10	<LOD	<LOD	0.0017 *0.0024*	0.0103 *0.011*	0.082 *0.094*
13	<LOD	<LOQ (0.00021) *0.00023*	0.00158 *0.0025*	0.0096 *0.0120*	0.078 *0.12*
16	<LOD	<LOQ (0.00021) *0.00022*	0.00137 *0.0018*	0.0084 *0.0092*	0.073 *0.089*
20	<LOD	<LOD	0.00117 *0.0014*	0.0079 *0.01*	0.073 *0.085*
22	<LOD	<LOQ (0.00023) *0.00025*	0.00101 *0.0011*	0.0082 *0.0096*	0.048 *0.084*
25	<LOD	<LOQ (0.00024) *0.00031*	<LOQ (0.00089) *0.0015*	0.0056 *0.0081*	0.070 *0.096*

Study Day	DM-8007: group mean residue (*highest individual*) in mg/kg				
	Control Group *(0 ppm)*	Group I *(0.015 ppm)*	Group II *(0.15 ppm)*	Group III *(1.5 ppm)*	Group IV *(10 ppm)*
27	<LOD	<LOQ (0.00025) *0.00029*	0.0016 *0.0018*	0.0117 *0.014*	0.086 *0.10*
30	<LOD	<LOQ (0.00032) *0.00042*	0.00147 *0.0017*	0.011 *0.014*	0.078 *0.10*
34	<LOD	0.00040 *0.00072*	0.0017 *0.0023*	0.013 *0.014*	0.089 *0.095*
37	<LOD	<LOQ (0.00026) *0.00035*	0.00137 *0.0014*	0.0114 *0.013*	0.086 *0.097*
41	<LOD	<LOQ (0.00028) *0.00031*	0.0015 *0.0021*	0.0135 *0.016*	0.081 *0.097*
44	-	-	-	-	0.045 *0.054*
48	-	-	-	-	0.029 *0.051*
55	-	-	-	-	0.0016 *0.0016*
Mean (Days 27-41)	<LOD	<LOQ (0.0003)	0.00153	0.0121	0.0841
Cream (Day22)	<LOD	0.0027 *0.0044*	0.0197 *0.022*	0.15 *0.18*	1.047 *1.3*
Cream (Day41)	-	-	-	-	1.067 *1.10*
Cream (Day44)	-	-	-	-	0.46 *0.46*
Cream (Day48)	-	-	-	-	0.42 *0.42*
Cream (Day55)	-	-	-	-	0.027 *0.027*
Skim milk (Day22)	<LOD	<LOD	<LOD	0.001 *0.0016*	0.0067 *0.014*
Skim milk (Day41)	-	-	-	-	0.0025 *0.0028*
Skim milk (Day44)	-	-	-	-	0.0029 *0.0029*
Skim milk (Day48)	-	-	-	-	0.0022 *0.0022*
Skim milk (Day55)					0.00039 *0.00039*

Table 127 Residues of metabolite DC-DM-8007 in milk (including skim milk and cream)

Study Day	DC-DM-8007: group mean residue (*highest individual*) in mg/kg				
	Control Group *(0 ppm)*	Group I *(0.015 ppm)*	Group II *(0.15 ppm)*	Group III *(1.5 ppm)*	Group IV *(10 ppm)*
-1	<LOD	<LOD	<LOD	<LOD	<LOD

Study Day	DC-DM-8007: group mean residue (*highest individual*) in mg/kg				
	Control Group *(0 ppm)*	Group I *(0.015 ppm)*	Group II *(0.15 ppm)*	Group III *(1.5 ppm)*	Group IV *(10 ppm)*
1	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00021) *0.00024*
4	<LOD	<LOD	<LOD	<LOD	<LOQ (0.0005) *0.0008*
7	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00073) *0.00084*
10	<LOD	<LOD	<LOD	<LOD	0.00102 *0.0013*
13	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00085) *0.0013*
16	<LOD	<LOD	<LOD	<LOD	0.00109 *0.0015*
20	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00092) *0.0011*
22	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00059) *0.00099*
25	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00087) *0.0012*
27	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00097) *0.0012*
30	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00083) *0.0011*
34	<LOD	<LOD	<LOD	<LOD	0.00106 *0.0011*
37	<LOD	<LOD	<LOD	<LOD	0.00107 *0.0013*
41	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00091) *0.0011*
44	-	-	-	-	<LOQ (0.00051) *0.0006*
48	-	-	-	-	<LOQ (0.00027) *0.00054*
55	-	-	-	-	<LOD
Mean (Days 27-41)	<LOD	<LOD	<LOD	<LOD	<LOQ (0.00097)
Cream (Day22)	<LOD	<LOD	<LOQ (0.0002) *0.00021*	0.0019 *0.0023*	0.0137 *0.015*
Cream (Day41)	-	-	-	-	0.012 *0.013*
Cream (Day44)	-	-	-	-	0.0078 *0.0078*
Cream (Day48)	-	-	-	-	0.0053 *0.0053*
Cream (Day55)	-	-	-	-	<LOQ (0.00024) *0.00024*

Study Day	DC-DM-8007: group mean residue (*highest individual*) in mg/kg				
	Control Group *(0 ppm)*	Group I *(0.015 ppm)*	Group II *(0.15 ppm)*	Group III *(1.5 ppm)*	Group IV *(10 ppm)*
Skim milk (Day22)	<LOD	<LOD	<LOD	<LOD	<LOD
Skim milk (Day41)	-	-	-	-	<LOD
Skim milk (Day44)	-	-	-	-	<LOD
Skim milk (Day48)	-	-	-	-	<LOD
Skim milk (Day55)	-	-	-	-	<LOD

In tissues, mean levels of broflanilide and of DC-DM-8007 were < LOQ in all treatment groups (Table 128). Metabolite DM-8007 was detected above LOQ in all tissues from the 10 ppm dosing group with a maximum of 0.79 mg/kg in mesenterial fat, while in the lower dose groups DM-8007 was not consistently found in all tissues (Table 128).

Table 128 Residues of broflanilide and metabolites in cow tissues

Tissue	Mean Residues (*maximum individual*), mg/kg							
	Control Group *(0 ppm)*	Group I *(0.015 ppm)*	Group II *(0.15 ppm)*	Group III *(1.5 ppm)*	Group IV *(10 ppm)*	Group IV Dep 1 [1] *(10 ppm)*	Group IV Dep 2 [2] *(10 ppm)*	Group IV Dep 3 [3] *(10 ppm)*
Broflanilide								
Muscle	<LOD	<LOD	<LOD	<LOD	<LOQ (0.0042) *0.012*	<LOD	<LOD	<LOD
Liver	<LOD	<LOD	<LOD	<LOD	<LOQ (0.0031) *0.0035*	<LOD	<LOD	<LOD
Kidney	<LOD	<LOD	<LOD	<LOD	<LOQ (0.0021) *0.0024*	<LOD	<LOD	<LOD
Fat Perirenal	<LOD	<LOD	<LOD	<LOD	<LOQ (0.0070) *0.0086)*	<LOD	<LOD	<LOD
Fat Mesenterial	<LOD	<LOD	<LOD	<LOQ (0.0020) *0.0021*	<LOQ (0.0094) *0.0110*	<LOQ (0.0039) *0.0039*	<LOQ (0.0039) *0.0039*	<LOD
Fat Subcutaneous	<LOD	<LOD	<LOD	<LOQ (0.0024) *0.0027*	<LOQ (0.0077) *0.0085*	<LOQ (0.0087) *0.0087*	<LOQ (0.0021) *0.0021*	<LOD
DM-8007								
Muscle	<LOD	<LOD	<LOD	<LOQ (0.0050) *0.0067*	0.026 *0.038*	0.026 *0.026*	0.026 *0.026*	<LOD
Liver	<LOD	<LOD	<LOD	0.0109 *0.013*	0.074 *0.078*	0.034 *0.034*	0.033 *0.033*	<LOQ (0.0026) *0.0026*

Tissue	Mean Residues (*maximum individual*), mg/kg							
	Control Group *(0 ppm)*	Group I *(0.015 ppm)*	Group II *(0.15 ppm)*	Group III *(1.5 ppm)*	Group IV *(10 ppm)*	Group IV Dep 1 [1] *(10 ppm)*	Group IV Dep 2 [2] *(10 ppm)*	Group IV Dep 3 [3] *(10 ppm)*
Kidney	<LOD	<LOD	<LOD	<LOQ (0.0088) *0.010*	0.0663 *0.08*	0.035 *0.035*	0.069 *0.069*	<LOD
Fat Perirenal	<LOD	<LOQ (0.0030) *0.0041*	<LOQ (0.0095) *0.0130*	0.1023 *0.11*	0.4867 *0.61*	0.37 *0.37*	0.32 *0.32*	0.018 *0.018*
Fat Mesenterial	<LOD	<LOQ (0.0036) *0.0045*	0.014 *0.016*	0.1243 *0.16*	0.72 *0.79*	0.56 *0.56*	0.67 *0.67*	0.02 *0.02*
Fat Subcutaneous	<LOD	<LOQ (0.0025) *0.003*	<LOQ (0.0078) *0.01*	0.088 *0.11*	0.51 *0.55*	0.46 *0.46*	0.40 *0.40*	0.022 *0.022*
DC-DM-8007								
Muscle	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
Liver	<LOD	<LOD	<LOD	<LOD	<LOQ (0.0023) *0.0025*	<LOD	<LOD	<LOD
Kidney	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
Fat Perirenal	<LOD	<LOD	<LOD	<LOQ (0.002) *0.002*	<LOQ (0.0073) *0.0088*	<LOQ (0.0064) *0.0064*	<LOQ (0.0058) *0.0058*	<LOD
Fat Mesenterial	<LOD	<LOD	<LOD	<LOQ (0.0021) *0.0024*	<LOQ (0.0091) *0.01*	<LOQ (0.0085) *0.0085*	<LOQ (0.0074) *0.0074*	<LOD
Fat Subcutaneous	<LOD	<LOD	<LOD	<LOD	<LOQ (0.0063) *0.0069*	<LOQ (0.0063) *0.0063*	<LOQ (0.0049) *0.0049*	<LOD

Notes:

[1] 2 days withdrawal.

[2] 6 days withdrawal.

[3] 13 days withdrawal.

Laying hens

The transfer of residues of broflanilide into animal matrices was investigated in a study with laying hens (Ray, 2017, BROFLAN_083). The experimental design is presented in Table 128.

Table 129: Dosing regimen for broflanilide in laying hens

Treatment group	Dose (ppm)	Average mg ai/kg dry feed	Average mg as/kg bw
1× Group	0.02	0.021	0.00117
5× Group	0.10	0.102	0.00551
25× Group	0.50	0.509	0.02734

The hens in the treatment groups (12 animals per group + 12 hens for depuration at 0.5 ppm) were treated orally with broflanilide in a gelatin capsule once daily. Eggs were collected for sampling at selected intervals in the evening after dosing and on the following morning before the next dose and combined. Hens were sacrificed on the day after the last dose (control and 1× group on day 29, 5× group on day 36 and 25× group on day 50). Additionally, the hens from the depuration subgroups were sacrificed at day 53, 57 and 64. Samples of liver, muscle (thigh, breast) and fat (abdominal, subcutaneous) were collected and taken for analysis.

Samples were analysed for parent broflanilide as well as metabolites DM-8007 and DC-DM-8007 using method D1604/01 with a LOQ of 0.01 mg/kg for egg and tissues. Procedural recoveries for all matrices and analytes ranged between 75–113 percent. The method limitation of detection (LOD) for residues in egg and tissue samples was set at 20 percent of the LOQ, equivalent to 0.002 mg/kg. Maximum storage time of egg and tissues was 12 and 15 days, respectively.

In the egg samples, residues of broflanilide and metabolite DC-DM-8007 were < 0.002 mg/kg (<LOD) over the entire dosing period at the 0.02, 0.10 and 0.50 ppm dose levels. Residues of metabolite DM-8007 were < 0.002 mg/kg (<LOD) in the 0.02 and 0.10 mg/kg dose levels as well, but were found at up to 0.023 mg/kg in the 0.5 ppm dose level (Table 130). A plateau in eggs was not reached (Figure 7).

Table 130 Residues of metabolite DM-8007 in eggs

Study day	DM-8007: group mean residue (*highest individual*) in mg/kg		
	25× Group (0.50 ppm)		
	Broflanilide	DC-DM-8007	DM-8007
-1	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (< 0.002)
1	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (< 0.002)
4	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (0.002)
7	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (0.006)
10	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0100 *0.0103*
13	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0112 *0.0124*
16	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0138 *0.0167*
19	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0145 *0.0192*
22	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0145 *0.0164*
25	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0146 *0.0156*
28	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0156 *0.0209*
31	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0169 *0.0226*
34	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0178 *0.0224*
37	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0164 *0.0214*
40	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0161 *0.0198*

Study day	DM-8007: group mean residue (*highest individual*) in mg/kg		
	25× Group (0.50 ppm)		
	Broflanilide	DC-DM-8007	DM-8007
43	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0159 *0.0177*
46	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0179 *0.0206*
49	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0182 *0.0189*
52	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0195 *0.0218*
56	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0116 *0.0132*
59	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (0.0057)
63	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (0.0039)

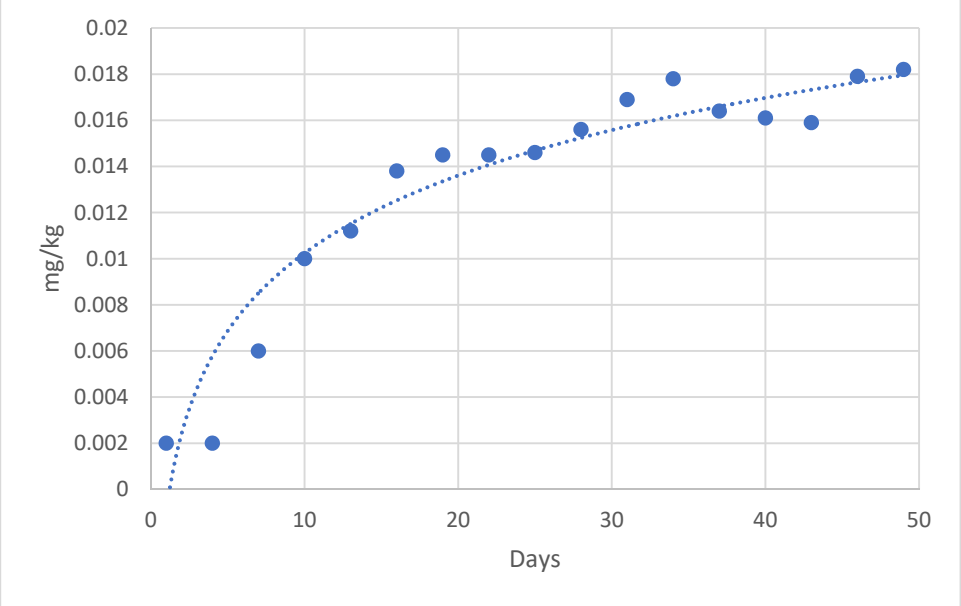


Figure 7 Time course of the concentrations of metabolite DM-8007 in eggs

In the tissue samples, residues of broflanilide and metabolite DC-DM-8007 were <LOQ over the entire dosing period at the 0.02, 0.10 and 0.50 ppm dose levels. Residues of metabolite DM-8007 were found in liver >LOQ only at the highest dose at up to 0.021 mg/kg, while in muscle levels never reached LOQ level. On the other hand, levels of DM-8007 in fat increased from 0.01 mg/kg at the lowest dose level to up to 0.15 mg/kg at the highest dose level (Table 131).

Table 131 Residues of broflanilide and metabolites DM-8007 and DC-DM-8007 in poultry tissues

Matrix	Study day	Feeding level (ppm)	Mean (highest individual), mg/kg		
			Broflanilide	DC-DM-8007	DM-8007
Liver	29	0	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (< 0.002)
	29	0.02	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (< 0.002)
	36	0.10	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01

Matrix	Study day	Feeding level (ppm)	Mean (highest individual), mg/kg		
			Broflanilide	DC-DM-8007	DM-8007
	50	0.50	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0185 (0.0211)
Muscle (thigh and breast)	29	0	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (< 0.002)
	29	0.02	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (< 0.002)
	36	0.10	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01
	50	0.50	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01
Fat (abdominal and subcutaneous)	29	0	< 0.01 (< 0.002)	< 0.01 (< 0.002)	< 0.01 (< 0.002)
	29	0.02	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0103 (0.0108)
	36	0.10	< 0.01 (< 0.002)	< 0.01 (< 0.002)	0.0338 (0.0392)
	50	0.50	< 0.01 (< 0.002)	< 0.01 (0.0025)	0.1372 (0.1522)

APPRAISAL

Broflanilide is a meta-diamide insecticide for the control of chewing-insect pests. It is the precursor to its active form desmethyl broflanilide, which acts by binding to the GABA receptor, resulting in a block of inhibitory neurotransmission and death of target insects. At the Fifty-first Session of the CCPR, broflanilide was scheduled for evaluation as a new compound in 2020 and rescheduled to the 2022 JMPR.

The Meeting received information on identity, physicochemical properties, metabolism (plant, confined rotational crops and animals), environmental fate, field rotational crops, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials, fate of residues in processing, and livestock feeding studies.

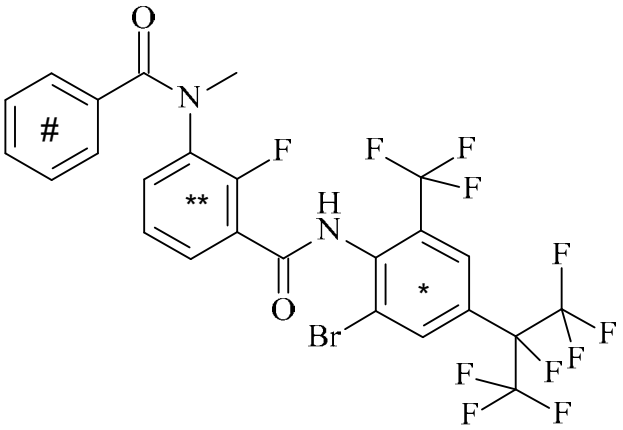
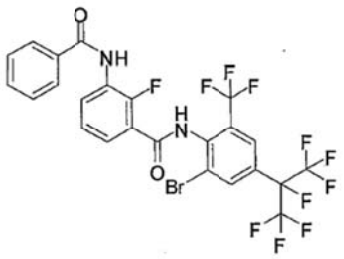
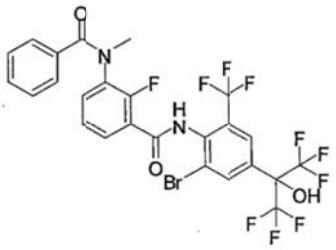
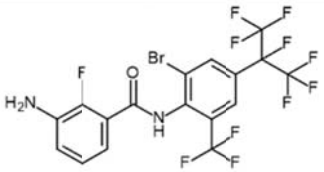
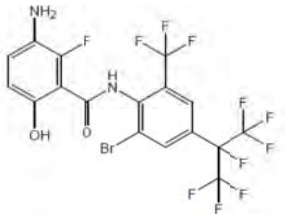
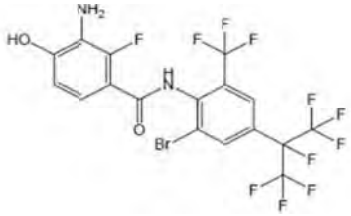
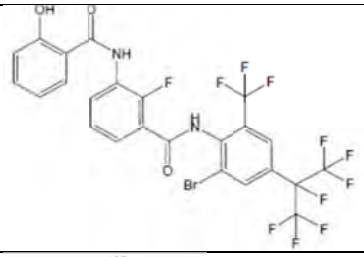
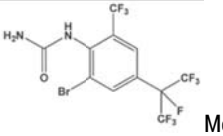
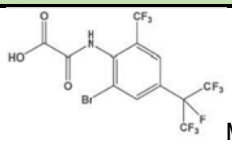
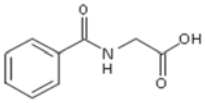
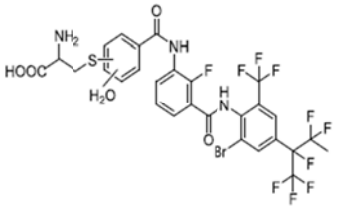


Figure 8 Chemical structure of broflanilide (IUPAC name: *N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-3-(*N*-methylbenzamido) benzamide). Molecular weight of 663.3 g/mol. * B-ring; # C-ring; ** A-ring

Table 132 Overview of metabolites and codes referred to in the appraisal

Code Names	Chemical Names (IUPAC)	Structure
DM-8007, MLP-8473 (Reg. No 5856361)	3-benzamido-*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluorobenzamide	 Molar mass: 649.3 g/mol
S(PFP-OH)-8007 (Reg. No 5959598)	*N*-[2-bromo-4-(1,1,1,3,3,3-hexafluoro-2-hydroxypropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-3-(*N*-methylbenzamido)benzamide	 Molar mass: 661.3 g/mol
DC-DM-8007 Reg.No. 5936906	3-amino-*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluorobenzamide	 Molecular mass: 545.1 g/mol
DC-DM-(A4-OH)-8007 KAK-1606-146	3-amino-*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-6-hydoxybenzamide	
DC-DM-(A6-OH)-8007 267-022-015-2	3-amino-*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-4-hydoxybenzamide	
DM-(C2-OH)-8007 267-014-033-4	*N*-2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluoro-3-(2-hydoxybenamido)benzamide	
B-urea (Reg. No 6065386)	[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]urea	 Molar mass: 451.1 g/mol

Code Names	Chemical Names (IUPAC)	Structure
B-oxam-acid (Reg. No 6066332)	*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]oxamic acid	 Molar mass: 480.1 g/mol
Hippuric acid	N-benzoylglycine	
H-U27B (and related but not fully elucidated H-U27C)	Hydroxyl cysteine conjugate of DM-8007	

Physical and chemical properties

Parent broflanilide and metabolites DM-8007, S(PFP-OH)-8007 and DC-DM-8007 have low solubility in water, but at least broflanilide has a good solubility in organic solvent. The logP_{OW} at 20 or 25 °C and at pH of 4, 7 and 10 ranges between 3.8–5.9 for these compounds, suggesting that the parent and metabolites have the potential to partition into fat. Parent broflanilide was shown to be hydrolytically stable at pH 4, 7 and 9. Broflanilide was not prone to photodegradation in water pH 7 (DT_{50}: 69–89 days), but showed slow degradation at pH 5 (DT_{50}: 14–20 days) and moderately fast degradation at pH 9 (DT_{50}: 3–6 days). The vapour pressure indicates that broflanilide is not volatile.

Plant metabolism

The metabolic fate in plants was investigated following application of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide to cabbage, tomato, Japanese radish, soya bean, rice, wheat and tea.

Cabbage

Cabbages grown outdoors received two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide at nominal rates of 0.025 kg ai/ha each at immature stage (BBCH 45) and 7 days later (BBCH 46). Immature cabbage (BBCH 46) was harvested 6 days after application 1, while mature cabbage (BBCH 49) was harvested 21 days after application 2.

TRRs were highest in cabbage (inner and outer leaves) taken at 6 DAT1 ranging between 0.304–0.352 mg eq/kg. In cabbage (inner and outer leaves) taken at 21 DAT2, about half of the previous TRR for the B-ring was found, while levels for the C-ring remained the same.

The outer leaves received a surface rinse with acetonitrile before homogenization, which released 55–66 percent of the TRR. Homogenized samples of the rinsed outer leaves and the inner leaves were subjected to extraction with acetonitrile (twice) and acetonitrile:water (1+1) (once). Extracted radioactivity was similar for both labels ranging between 92–95 percent TRR, while the PES accounted for 6–8 percent TRR.

Parent broflanilide was the major identified residue in immature and mature cabbage (inner and outer leaves) accounting for 66–84 percent TRR (0.10–0.25 mg/kg). Additionally, two minor metabolites were identified, namely S(PFP-OH)-8007 and DM-8007 accounting for 3.8–7.6 percent TRR (0.01–

0.012 mg eq/kg) and 2.9–7.9 percent TRR (0.009–0.021 mg eq/kg), respectively. The unextracted residue was not further characterized

Tomato

Tomato grown outdoors received two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide at nominal rates of 0.025 kg ai/ha each. The first application occurred at the pre-bud stage (approx. BBCH 49–50) and the second application 83 days later at the beginning ripening stage (approximately BBCH 79–81). Immature tomatoes and leaves (BBCH 75) were harvested 71 days after application 1, while mature tomatoes and leaves (approximately BBCH 88) were harvested 10 days after application 2.

TRR was very low or non-detected in tomato leaves and immature fruits form harvest 1. In samples from harvest 2, radioactivity was highest in leaves, ranging between 0.904–1.596 mg eq/kg while in tomato fruits, levels were significantly lower at 0.01 mg eq/kg. Samples of tomato leaves and fruits received a surface rinse with acetonitrile before homogenization, which released 70–80 percent TRR.

Portions of the tomato leaves from harvest 2 were subjected to extraction with acetonitrile (twice) and acetonitrile:water (1+1) (once). Extracted radioactivity was similar for both labels ranging between 96–99 percent TRR in tomato leaves and 70–80 percent TRR in tomato fruit. The PES in tomato fruit accounted for 20–30 percent TRR, but was < 0.003 mg eq/kg in absolute concentration and not further analysed.

Parent broflanilide was the major identified residue, accounting for 87–89 percent TRR (0.76–1.3 mg/kg) in tomato leaves and 60–68 percent TRR (0.006–0.007 mg eq/kg) in tomato fruit. Additionally, metabolites S(PFP-OH)-8007 and DM-8007 were identified in tomato leaves and fruit at minor levels accounting for 3.0–3.4 percent TRR (0.0003–0.051 mg eq/kg) and 3.4–4.0 percent TRR (0.0004–0.060 mg eq/kg), respectively.

Japanese radish

Japanese radish grown indoors received two applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide. The first treatment was applied to the soil at a rate of 0.4 kg ai/ha immediately after seeding, and a second treatment was applied foliar 41 days later at a rate of 0.225 kg ai/ha, 29 days before the final harvest. Plants (leaves and root) were collected at three sampling points: 40DAT1 (intermediate harvest-1), 14DAT2 (intermediate harvest-2) and 29DAT2 (final harvest).

TRRs were highest in radish leaves ranging between 3.6–4.4 mg eq/kg for the 14DAT2 and 29 DAT2 sampling time points. In radish roots, the TRR was at least two orders in magnitude lower, ranging between 0.0036–0.0119 mg eq/kg.

Radish leaves were surface-rinsed with acetonitrile (except the intermediate harvest-1) and the root was further separated into peel and flesh. All samples were homogenized by blending with dry ice. Portions of the samples were subjected to extraction with acetonitrile:water (8+2) (twice) and acetonitrile:0.1M HCl (8+2) (once).

The extracted radioactivity in radish leaves ranged between 95–99 percent TRR, except for leaves from intermediate harvest 1 for the C-ring label (70 percent TRR). In these samples the radioactivity in the PES accounted for 30 percent TRR, but was < 0.0021 mg eq/kg in absolute concentration. In radish roots, the sum of the radioactivity found in peel and flesh extracts accounted for 54–96 percent TRR, while 4.3–47 percent TRR remained in the PES. However, absolute concentrations in the PES were throughout < 0.0056 mg eq/kg.

Parent broflanilide was the major identified residue in radish leaves, accounting for 77–82 percent TRR (2.8–3.6 mg/kg). Additionally, metabolites S(PFP-OH)-8007 and DM-8007 were identified at minor levels, accounting for 1.7–2.9 percent TRR (0.067–0.12 mg eq/kg) and 2.5–3.3 percent TRR (0.11–0.13 mg eq/kg), respectively. Residues in roots were not further investigated.

Soya bean

Soya bean grown outdoors received two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide at nominal rates of 0.025 kg ai/ha each. The first application occurred at bud formation (approx. BBCH 49–51) and the second application 77 days later at the beginning of the pod and seed ripening stage (approx. BBCH 79–81). Soya bean forage and hay samples were harvested at 21DAT1 (BBCH 69) and 35DAT1 (BBCH 74), respectively. The mature soya bean seeds were harvested at 12DAT2.

Similar TRR levels were found for both labels, with the highest levels in soya bean forage ranging between 0.460–0.433 mg eq/kg. In soya bean seeds, the detected radioactivity was < 0.01 mg eq/kg for both labels and was not further analysed.

Portions of soya bean forage and hay were subjected to extraction with acetonitrile (twice) and acetonitrile:water (1+1) (once). The extracted radioactivity was similar for both labels ranging between 92–93 percent TRR in soya bean forage and 89–91 percent TRR in soya bean hay.

Parent broflanilide was the major identified residue, accounting for 75–76 percent TRR (0.32–0.34 mg/kg) in soya bean forage and 67–71 percent TRR (0.19 mg eq/kg) in soya bean hay. Additionally, metabolites S(PFP-OH)-8007 and DM-8007 were identified in soya bean forage and hay at minor levels accounting for 3.8–5.6 percent TRR (0.010–0.021 mg eq/kg) and 5.1–8.3 percent TRR (0.022–0.023 mg eq/kg), respectively.

Rice

Rice grown indoors received two applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide The first treatment was applied to the flooding water at a rate of 0.3 kg ai/ha immediately after seed transplantation, followed by a foliar application at a rate of 0.15 kg ai/ha 73 days later. The rice plants were collected at 13DAT2 (intermediate harvest: foliage) and at 32DAT2 (the final harvest: husked rice, hulls, straw and root).

The TRR (sum of foliage surface rinse, extracts and PES) was generally similar for both labels, with the highest levels found in rice hulls and straw at 5.5–6.8 mg eq/kg and 4.2–4.9 mg eq/kg, respectively. In husked rice, levels were at least one order in magnitude lower at 0.021 mg eq/kg for the B-ring label and 0.11 mg eq/kg for the C-ring label.

Only the foliage from the intermediate harvest was surface-rinsed with acetonitrile. Portions of the foliage, husked rice, straw and hulls were subjected to extraction with acetonitrile:water (8+2) (twice), followed by SPE fractionation of the extracts.

The extracted radioactivity from rice forage, husked rice, hulls and straw ranged between 85–98 percent TRR, except for husked rice for the C-ring label (18 percent TRR). In this sample the radioactivity in the PES accounted for 82 percent TRR Further characterization of the PES using acid/enzyme treatments showed that 21 percent TRR accounted for the starch fraction and 7.2 percent TRR for the protein fraction. Since the absolute measured radioactivity in the extracts was similar for both labels, it was assumed that the higher radioactivity in the PES from C-ring label was due to incorporation of $^{14}CO_2$, into the plant matrix.

Parent broflanilide was the major identified residue, accounting for 84–87 percent TRR (1.0–1.6 mg/kg) in rice foliage, 83–90 percent TRR (4.6–8.1 mg/kg) in hulls and 85–87 percent TRR (3.6–4.1 mg/kg) in straw. In husked rice, although the relative amounts of parent broflanilide differed between the two labels, accounting for 64 percent TRR using the B-ring label and 13 percent TRR using the C-ring label, the radioactive residue levels were similar (0.013–0.014 mg eq/kg). Additionally, metabolites S(PFP-OH)-8007 and DM-8007 were identified in all matrices accounting for 1.0–8.5 percent TRR (0.002–0.28 mg eq/kg) and 0.8–5.4 percent TRR (0.001–0.26 mg eq/kg), respectively.

Wheat

Wheat grown indoors received seed treatment of [B-ring-U-^{14}C]-labelled broflanilide applied at 10 g ai/100 kg seeds, corresponding to actual application rate of 0.022 kg ai/ha. Immature wheat plants (wheat forage) were collected at growth stage BBCH 39 (77 DAT), and half of the forage was allowed to dry for 8 days at room temperature to produce wheat hay. Mature wheat plants were harvested at growth stage BBCH 89 (154 DAT) and were separated into straw and grains.

TRR levels in wheat matrices were generally low, with the highest level measured in wheat straw at 0.029 mg eq/kg, while the TRR in wheat grain was up to 0.011 mg eq/kg.

Portions of wheat straw and grains were subjected to extraction with acetonitrile:water (1+1) (twice), followed by acetonitrile (once). The straw extracts were further partitioned with ethyl acetate, followed by fractionation using SPE. The PES of wheat straw and grains were characterized by enzyme solubilization using macerozyme, tyrosinase and amylase.

Extractability with solvents was higher in straw (79 percent TRR) compared to grains (29 percent TRR). Further characterization of the PES released additionally 6.7 percent TRR from wheat straw and 24 percent TRR from wheat grain. No individual components could be identified in either matrix. In wheat straw, one unknown component accounted for 14 percent TRR, but the level was < 0.01 mg eq/kg.

Tea grown outdoors received two foliar applications of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide at nominal rates of 0.1 kg ai/ha each with a RTI of 14 days. Tea leaves were harvested at 7days after the second application (7DAT2) and 14DAT2.

The TRR was generally similar for both labels and harvest times, ranging between 15–20 mg eq/kg. Tea leaves were surface-rinsed with acetonitrile followed by extraction with acetonitrile (twice) and acetonitrile:water (1+1) (once). The extracted radioactivity from tea leaves ranged between 99.3–99.6 percent TRR, with over 97 percent radioactive being removed by the acetonitrile rinse.

Parent broflanilide was the major identified residue, accounting for 96–97 percent TRR (14–19 mg/kg). Additionally, metabolites S(PFP-OH)-8007 and DM-8007 were identified at minor levels of 1.0–1.4 percent TRR (0.14–0.27 mg eq/kg) and up to 1.0 percent TRR (0.20 mg eq/kg), respectively.

Summary of plant metabolism

In all plant metabolism studies, broflanilide was degraded into DM-8007 via demethylation or into S(PFP-OH)-8007 via oxidative defluorination (substitution of fluorine with hydroxy group). Parent broflanilide was the major identified component in all matrices, while both metabolites were detected at minor levels.

Animal metabolism

The Meeting received studies on the metabolism of broflanilide in laboratory animals, lactating goats and laying hens. The evaluation of the metabolism studies in laboratory animals was carried out by the WHO Core Assessment Group.

Lactating goats

In lactating goats, the metabolic fate of broflanilide was investigated using [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide. The compound was administered orally once daily (after morning milking) for 10 consecutive days at 19 ppm (0.62 mg/kg bw day) and 20 ppm (0.73 mg/kg bw) for the B-ring and C-ring label, respectively.

The majority of the radioactivity was found in faeces, at 51–75 percent of the applied radioactivity (AR). In urine, 24 percent AR was found in the C-ring treatment, while only 0.7 percent AR was found in the B-ring treatment. In edible tissues, the highest TRRs were found in fats (omental, subcutaneous and renal), ranging from 2.6–3.4 mg eq/kg for both labels and in liver, ranging from 0.46 mg eq/kg (C-ring) to 2.2 mg eq/kg (B-ring). In muscle and kidney, TRRs were lower for both labels, ranging between 0.22–0.37 mg eq/kg and 0.25–0.27 mg eq/kg, respectively.

In whole milk, the radioactive residues ranged between 0.12–0.43 mg eq/kg for both labels. Residue levels reached a plateau after approximately 6 days and 2 days for the B-ring and C-ring labels, respectively. In milk fat, residues were ~2 orders in magnitude higher compared to skim milk for both labels, reaching up to 4.1 mg eq/kg.

Samples of flank and loin muscles, liver and kidney were extracted twice with acetonitrile:water (1:1) and once with acetonitrile. Skim milk was extracted twice with acetone:water (1:1) and once with acetone. Fats and milk fat were extracted twice with acetone/hexane (1+4) and once with acetone. Solvent extraction released at least 88 percent TRR from most matrices, except for liver (42–68 percent TRR, both labels) and kidney (76 percent TRR, B-ring label).

The PES from liver (B and C-ring labels) and kidney (B-ring label only) were further characterized by enzyme solubilization using protease and lipase, followed by incubations with 1 mol/L HCl and 1 mol/L NaOH, which released additionally 16–59 percent TRR. The extracts and solubilizates were also treated with *β*-glucuronidase in order to cleave conjugates to their respective aglycones.

Parent broflanilide was only detected as a minor residue in muscle, kidney and liver, accounting for 0.5–6.7 percent TRR (0.005–0.022 mg/kg). A major identified metabolite using both labels was DM-8007 in muscle, milk, fats, liver (only C-label) and kidney, accounting for 21.3–99.9 percent TRR (0.01–3.4 mg eq/kg). In the B-label treated goat only, metabolite DC-DM-8007 was detected at major proportions in muscle, milk, fats, liver and kidney, ranging from 29–67 percent TRR (0.017–2.3 mg eq/kg), while in the C-label only, hippuric acid was detected in skim milk, liver and kidney at 19–69 percent TRR (0.018–0.13 mg/kg). Also, hydroxylated and conjugated DC-DM-(A4-OH)-8007, DC-DM-(A6-OH)-8007 and DM-(C2-OH)-8007 were identified in liver, accounting for up to 15 percent TRR (0.32 mg eq/kg), 11 percent TRR (0.24 mg eq/kg) and 17 percent TRR (0.078 mg eq/kg), respectively, and in kidney (B-label only), accounting for less than 10 percent TRR (0.007 to 0.019 mg eq/kg).

Laying hens

In laying hens, the metabolic fate of broflanilide was investigated using [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide. The compound was administered orally once daily for 14 consecutive days to 10 laying hens per label, at 14 ppm (0.86 mg/kg bw day) and 15 ppm (0.84 mg/kg bw) for the B- and C-label, respectively. Eggs and excreta samples were collected twice daily, at approximately 12 hour intervals. Samples of breast muscle, thigh (leg) muscle, abdominal and subcutaneous fat, liver and the entire gastrointestinal tract were collected after sacrifice, which occurred 6 hr after the last dose.

The majority of the radioactivity was found in excreta at 56–65 percent AR. In edible tissues radioactivity was highest in fat at 15–19 mg eq/kg, followed by egg yolk at 3.4–3.6 mg eq/kg and liver at

1.8–2.6 mg eq/kg. Incorporation of radioactivity into egg whites reached steady state within 3–4 days, while no plateau was reached in egg yolks.

Egg white, egg yolk, and muscle samples were extracted twice with acetonitrile:water (1:1) and then once with acetonitrile. Fat samples were extracted twice with acetone:hexane (1:4) then once with acetone. Liver samples were initially extracted three times with acetonitrile:water (1:1) and then once with acetonitrile. Extraction with solvents released at least 89 percent TRR from all matrices, except for liver where 65–72 percent TRR were released. The PES of hen liver from both radiolabels were further characterized by enzyme solubilization using protease and lipase, followed by incubations with 1 mol/L HCl and 1 mol/L NaOH, which released additionally 29–36 percent TRR.

Parent broflanilide was only tentatively identified (TLC analysis) in egg white from the B-label at 2.1 percent TRR (0.0004 mg/kg). The predominant identified residue for both labels was metabolite DM-8007 in all matrices, accounting for 57100 percent TRR (0.013–19 mg eq/kg). As a minor metabolite only occurring with the B-label, DC-DM-8007 was detected in all matrices, accounting for up to 3 percent TRR (0.55 mg eq/kg) in subcutaneous fat. In egg white, DC-DM-8007 accounted for 16 percent TRR, but residues were low (0.003 mg eq/kg). H-U27B (B-label), a hydroxyl cysteine conjugate of DM-8007 and the similar, but structurally not fully elucidated compound H-U27C (C-label) were identified in liver only, accounting for 5.3 percent TRR (0.131 mg eq/kg) and 3.3 percent TRR (0.061 mg eq/kg), respectively.

Summary of livestock metabolism

Generally, the transfer of radioactivity into animal food and feed matrices was low. The metabolism in lactating goats and laying hens is similar, starting with N-demethylation of parent broflanilide to form the main metabolite DM-8007, which is either hydroxylated and conjugated, or cleaved to DC-DM-8007 (identified using the B-label) and hippuric acid (identified using the C-label), via the intermediate benzoic acid. DC-DM-8007 is subsequently hydroxylated, followed by conjugation.

Environmental fate in soil

The Meeting received studies on aerobic soil degradation, soil photolysis, confined rotational crop metabolism and field rotational crops.

In two aerobic soil degradation studies performed with fresh soil under laboratory conditions, ^{14}C-labelled broflanilide (A-ring, B-ring, or C-ring), was very persistent with estimated half-lives in various soils ranging between 288 to 1000 days. Identified metabolites were DM-8007 (up to 4.9 percent AR), S(PFP-OH)-8007 (up to 1.2 percent AR) and DC-DM-8007 (up to 2.3 percent AR). On the contrary, in a soil dissipation study under field conditions, broflanilide and metabolite DM-8007 were not, or only moderately persistent with estimated half-lives, ranging from 3.3 to 18 days and from 8 to 91 days, respectively. Therefore, the Meeting concluded that broflanilide does not have the potential to accumulate in soil.

Half-life of ^{14}C-labelled broflanilide for soil photolysis was estimated (single 1st order kinetics) to 389 US solar days, or 347 OECD solar days. The only identified metabolite was DM-8007 at up to 4.2 percent AR. The Meeting concluded that photolysis does not represent a significant degradation pathway for broflanilide.

A confined rotational crop metabolism study was conducted with [A-ring-U-^{14}C]- and [B-ring-U-^{14}C]-labelled broflanilide, each applied at a rate of 0.15 kg ai/ha to a sandy loam soil. After plant-back intervals (PBIs) of 30, 120 and 270 days, the nature and level of radioactive residues were investigated in lettuce, radish and wheat.

Radioactivity for both labels in all matrices was comparable, with consistently higher levels found for the B-ring label. TRR levels were highest in wheat hay and straw for all PBIs, ranging between 0.014–0.067 mg/eq kg and 0.022–0.075 mg eq/kg, respectively, with the tendency to be higher at later PBIs.

Crop matrices with TRRs ≥0.01 mg/kg were extracted two times with acetonitrile/water (8:2), followed by one time with acetonitrile. Extractabilities ranged between 71–92 percent in radish foliage, 77–94 percent TRR in lettuce (immature and mature), 88–92 percent in wheat forage, 36–70 percent TRR in wheat hay, 58–77 percent TRR in straw and 22–84 percent TRR in grain. The highest radioactivity remaining in the PES was found in wheat straw from the treatment with B-labelled broflanilide (0.025 mg eq/kg), but no further characterization was performed.

In food matrices, parent broflanilide was identified in lettuce (immature and mature) at 19.6–46.6 percent TRR (0.002–0.008 mg/kg) at 120 and 270 day PBI and in radish leaves at 2.8–18.1 percent TRR (< 0.001–0.002 mg/kg) for the 270 day PBI only. In feed matrices, parent broflanilide was identified as a minor component in wheat forage, hay and straw, accounting for 4.3–16.7 percent TRR (0.001–0.004 mg/kg) in all PBIs.

Metabolite DM-8007 was a minor residue in wheat hay and straw and in radish leaves ranging between 1.5–3.7 percent TRR (< 0.001–0.002 mg eq/kg) at 30 and 270 day PBIs.

For the B-label only, an additional identified metabolite was B-urea, accounting for 9.7–30.2 percent TRR (0.004–0.010 mg eq/kg) in wheat forage, hay and straw in all PBIs, for 31.9 percent TRR (0.004 mg eq/kg) for the 270 day PBI only in radish leaves, for 26.6–34.5 percent TRR (0.003–0.005 mg eq/kg) at 120 and 270 day PBIs in immature lettuce and for 35.6 percent TRR (0.004 mg eq/kg) for the 270 day PBI only in mature lettuce. Once again for the B-label only, metabolite B-oxam-acid was identified in wheat forage, hay and straw, accounting for 5.4–35.6 percent TRR (0.001–0.024 mg eq/kg) and in radish leaves, accounting for 14.9 percent TRR (0.002 mg eq/kg) for the 270 day PBI only

In two field rotational crop trials, conducted during the 2016/17 growing seasons in the United States, broflanilide was applied once to bare soil at 0.05 kg ai/ha. Wheat, lettuce and radish were planted 30, 60, 90 and 360 DAT and sampled at normal crop maturity.

Residues of broflanilide as well as metabolites S(PFP-OH)-8007, DM-8007, B-oxam-acid and B-urea were < LOQ (0.01 mg eq/kg), with the exception of parent broflanilide in lettuce planted at 30 days PBI (0.013 mg/kg).

Summary of environmental fate in soil

The Meeting concluded that residues of broflanilide are not very persistent under field conditions and does not have the potential to accumulate in soil. From rotational crop studies, the Meeting concluded that significant carry-over of broflanilide residues in succeeding crops is unlikely.

Methods of analysis

The Meeting received analytical methods for the determination of broflanilide and metabolites S(PFP-OH)-8007, DM-8007, B-urea and B-oxamic acid in plant matrices and for broflanilide and metabolites DC-DM-8007 and DM-8007 in animal matrices.

For matrices of plant origin, a method for all analytes based on QuEChERS employed extraction with acetonitrile/water + buffer salts, followed by clean-up using dispersive solid phase extraction (dSPE) with PSA (optional for B-urea and B-oxamic acid). All analytes were determined by LC-MS/MS with an LOQ of 0.001 for broflanilide and metabolites S(PFP-OH)-8007 and DM-8007, and 0.01 mg/kg for B-urea and B-oxamic acid. Two additional methods employed extraction with acetonitrile or acetonitrile/water, followed

by clean-up using liquid-liquid extraction and/or SPE. Final determination was done by LC-MS/MS, GC-ECD or HPLC-UV with LOQs ranging between 0.01–0.1 mg/kg.

For animal matrices, the method employed extraction with acetonitrile, followed by acetonitrile/water for milk, egg, liver, kidney and muscle. Samples of fat were extracted with acetone/hexane, followed by acetone. Liver, kidney and muscle were further partitioned with a salt solution (MgSO4; NaCl; sodium citrate sesquihydrate and sodium citrate dehydrate), followed by clean-up with PSA. Final determination for all analytes was accomplished by LC-MS/MS with an LOQ of 0.001 mg/kg for milk and 0.01 mg/kg for all other matrices.

The Meeting concluded that suitable methods are available to measure residues of broflanilide and metabolites S(PFP-OH)-8007, DM-8007, B-urea and B-oxamic acid in plant matrices as well as broflanilide and metabolites DC-DM-8007 and DM-8007 in animal matrices.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the storage stability of broflanilide and its metabolites S(PFPOH)-8007, DM-8007, B-oxam-acid and B-urea in a variety of plant matrices stored under frozen conditions. Samples were fortified at levels ranging from 0.01 to 0.5 mg/kg.

Residues of broflanilide and metabolite DM-8007 were stable in high acid matrices (grapes), high protein matrices (kidney beans), high water matrices (lettuce), high starch matrices (potato) and high oil matrices (soya been seed) for at least 24 months.

Residues of metabolite S(PFP-OH)-8007 were stable for at least 25 months in high acid matrices (grapes) and high oil matrices (soya been seed), for at least 28 months in high protein matrices (kidney beans) and high water matrices (lettuce), and for at least 24 months in high starch matrices (potato).

Residues of B-oxam-acid and B-urea were stable in high acid matrices (grapes), high protein matrices (kidney beans), high water matrices (lettuce), high starch matrices (wheat grain) and high oil matrices (soya been seed) for at least 16 months.

All samples from field trials were analysed within the tested storage stability time.

For animal matrices, the Meeting received information on the storage stability of broflanilide and its metabolites DM-8007 and DC-DM-8007 in muscle, liver, kidney, milk and fat stored at -20 °C. Samples were fortified at 0.01 mg/kg.

Residues of broflanilide and metabolite DM-8007 were stable in all tested matrices for at least 2 months. Metabolite DC-DM-8007 was not stable in muscle and kidney, but stable up to one month in liver and at least 2 months in milk and fat. Samples were analysed within this time frame, except for the analysis of DC-DM-8007 in muscle (maximum storage 28 days) as well as in kidney and liver (maximum storage 36 days).

Definition of the residue

In food commodities from plant metabolism studies conducted on cabbage, Japanese radish, tomato, soya bean, rice and tea, the predominant residue was parent broflanilide, accounting for 66–84 percent TRR in cabbage, 77–82 percent TRR in Japanese radish leaves, 60–68 percent TRR in tomato fruit, 13–64 percent TRR in husked rice and 96–97 percent TRR in tea. In studies with wheat (seed treatment) and in Japanese radish root, TRR levels were too low for identification. In feed matrices, residues of broflanilide accounted for 75–76 percent TRR in soya bean forage, 67–71 percent TRR in soya bean hay, 83–90 percent TRR in rice hulls and 85–87 percent TRR in rice straw.

The Meeting concluded that parent broflanilide is a major residue in plants and is a suitable marker compound for compliance with MRLs.

Analytical methods are available for monitoring broflanilide in all plant matrices.

On deciding which compounds should be included in the residue definition for risk assessment, the Meeting considered the likely occurrence of the compounds and the toxicological properties of the metabolites S(PFP-OH)-8007 and DM-8007.

The Meeting concluded that S(PFP-OH)-8007 is covered by the health based guidance value of broflanilide, but is toxicologically 3 times more potent. In plant metabolism studies, S(PFP-OH)-8007 was identified as a minor metabolite (< 10 percent TRR), with levels between 3 and 33 times lower than parent. The metabolite was analysed in various food and feed commodities from supervised field trials and residues above LOQ were detected occasionally, but always at least one order in magnitude lower compared to parent. Taking into account its higher potency, its contribution to the overall dietary exposure was still insignificant compared to parent residues (+0.1 percent relative), due to the low concentrations found in treated commodities.

Metabolite DM-8007 was identified in plant metabolism studies as a minor metabolite (< 10 percent TRR), with levels between 8 to 2500 times lower than the parent. Additionally, the metabolite was analysed in various food and feed commodities from supervised field trials and residues above the LOQ were only detected occasionally, but always at least one order in magnitude lower, compared to parent. The Meeting concluded that metabolite DM-8007 is of no greater toxicity than parent broflanilide and is covered by the toxicological reference values of the parent.

The Meeting noted that broflanilide represents the major part of the residues in plant commodities, sufficiently addressing the overall potential dietary exposure from plant commodities and agreed to set the definition of the residue for dietary risk assessment for plant commodities as parent broflanilide.

In animal metabolism studies performed with lactating goats and laying hens, the predominant metabolic pathway is N-demethylation of parent broflanilide to form DM-8007. Its subsequent cleavage results in DC-DM-8007 (identified using the B-label) and hippuric acid (identified using the C-label), via the intermediate benzoic acid. Depending on the label and tissue, some percentages may appear higher/lower than truly present due to the selective radiodetection.

Parent broflanilide was only detected as a minor residue in muscle, kidney and liver from lactating goats (0.5–6.7 percent TRR) and tentatively in egg white (2.1 percent TRR). In a cow feeding study, residues of broflanilide were detected in milk from the 10 ppm feeding level (approximately 7 times higher than the maximum dietary burden) at up to 0.0018 mg/kg and in cream from the 1.5 ppm (approximatley maximum dietary burden) and the 10 ppm feeding levels at up to 0.016 mg/kg. In all other tissues broflanilide was not detected. The Meeting noted that broflanilide is not a suitable marker for the residue definition for compliance with the MRL for animal commodities alone.

The predominant identified residue in metabolism studies was metabolite DM-8007, accounting for 21–100 percent TRR (0.01–3.4 mg eq/kg) in lactating goat matrices and for 57–100 percent TRR (0.013–19 mg eq/kg) in laying hen matrices. In a cow feeding study, residues of DM-8007 were detected in milk (up to 0.12 mg/kg), cream (up to 1.3 mg/kg), fats (up to 0.79 mg/kg), liver (up to 0.078 mg/kg) and in muscle and kidney (up to 0.08 mg/kg). In matrices from laying hens, residues of DM-8007 were found in eggs, liver and fat at up to 0.023 mg/kg, 0.021 mg/kg and 0.15 mg/kg, respectively.

Hence, the Meeting decided to include parent broflanilide and metabolite DM-8007 into the residue definition for compliance with MRLs.

Analytical methods are available for measuring broflanilide and DM-8007 in animal matrices.

In muscle and fat tissues of all animals investigated, residue concentrations of the sum of broflanilide and DM-8007 were 8–60 times higher in fat compared to muscle. Similarly, levels were approximately 200–300 times higher in milk fat compared to skim milk and approximately 400 times higher in egg yolk compared to egg white. The log P_{ow} of DM-8007 is 5.8 in pH 7 buffer solution. The Meeting concluded that residues according to the residue definition are fat-soluble.

On deciding which compounds should be additionally included in the residue definition for risk assessment, the Meeting considered the likely occurrence and toxicological properties for the candidates DC-DM-8007 and hippuric acid as well as the hydroxylated and conjugated metabolites DC-DM-(A4-OH)-8007, DC-DM-(A6-OH)-8007 and DM-(C2-OH)-8007.

In goat metabolism study (20 ppm dose level), DC-DM-8007 was detected in muscle, milk, fats and kidney at similar proportions as DM-8007 (13–67 percent TRR), but higher proportion in liver. In laying hens (15 ppm dose level), DC-DM-8007 occurred mostly at minor proportions in all matrices (0.8–3.0 percent TRR), with the exception of egg white (16 percent TRR). In the cow feeding study, DC-DM-8007 residues above LOQ were only occasionally found at the 1.5 and 10 ppm feeding levels in milk (up to 0.0015 mg/kg) and in cream (up to 0.015 mg/kg), but levels were at least one order in magnitude lower compared to DM-8007. In all cow tissues, as well as in any matrices from a laying hen feeding study, DC-DM-8007 was < LOQ. The Meeting concluded that DC-DM-8007 does not significantly contributes to the dietary exposure and is covered by the toxicological reference value of the parent.

In goat metabolism study, hippuric acid accounted for 19–69 percent TRR in skim milk, liver and kidney. Compared to DM-8007, the residue levels were similar (kidney, liver) or higher (~3 fold in skim milk), but the metabolite was not analysed in the livestock feeding studies. In rat metabolism studies hippuric acid was found in urine at levels of 11 percent AD and is commonly found in mg or g/l concentrations in human urine. Hippuric acid is of no toxicological concern. Hence, the Meeting decided to not include hippuric acid into the residue definition for dietary risk assessment for animal commodities.

In addition, the hydroxylated and conjugated metabolites DC-DM-(A4-OH)-8007, DC-DM-(A6-OH)-8007 and DM-(C2-OH)-8007 were identified in liver and kidney from goats (15, 11 and 17 percent TRR, respectively), at lower or similar levels or higher relative to DM-8007. However, they were not detected in muscle, milk and fat. The Meeting concluded that these metabolites do not significantly contribute to dietary exposure and are covered by the toxicological reference values of the parent.

The Meeting decided to include broflanilide and metabolite DM-8007 in the residue definition for dietary exposure purposes for animal commodities

Definition of the residue for compliance with the MRL and for dietary risk assessment for plant commodities: *Broflanilide*

Definition of the residue for compliance with the MRL and for dietary risk assessment for animal commodities: *Sum of broflanilide plus 3-benzamido-N-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluorobenzamide (DM-8007), expressed as broflanilide.*

The residue is fat-soluble.

Results of supervised residue trials on crops

Supervised trials were available for the use of broflanilide on green onion (Welsh onion), leek, cabbage, Chinese cabbage, tomatoes, radish, Japanese radish, turnip, potato, wheat, barley, maize and coffee. No trials according to GAP were provided on turnip.

Green onions, Subgroup of

The critical GAP for green onions in Japan allows three foliar applications of broflanilide at 2.5 g ai /hL with a PHI of 1 day.

Field trials with green onion in Japan were performed according to the GAP (±25 percent). The ranked order of broflanilide residues was (n=3): 0.38, 0.46, 1.32 mg/kg.

Field trials with leek in Japan were performed according to the GAP (±25 percent). The ranked order of residues was (n=3): 0.10, 0.20, 0.22 mg/kg.

The Meeting noted that residues in green onion and leek belong to different populations and could not be combined.

The Meeting concluded that the number of trials is insufficient to estimate maximum residue levels for broflanilide in green onions and leek.

Cabbage and Chinese cabbage

The critical GAP for cabbage and Chinese cabbage in Japan allows three foliar applications of broflanilide at 2.5 g ai /hL, and PHI of 14 days. No trials were provided matching this GAP.

A GAP for cabbage and Chinese cabbage in China allows a maximum of one foliar application of broflanilide at 24 g ai/ha and PHI of 5 days.

A total of 12 field trials conducted with cabbage in China were provided. In trials conducted at 33.8 g ai/ha, broflanilide residues were (n=6): < 0.01, 0.03, 0.11, 0.12, 0.16 and 0.42 mg/kg. In trials conducted at 45 g ai/ha, residues were (n=6): 0.12, 0.31, 0.33, 0.48 and 1.6 (2) mg/kg.

The proportionality approach was used in both datasets, and scaling factors of 0.71 or 0.53 were applied, giving residues in ranked order (n=12): < 0.01, 0.02, 0.06, 0.08, 0.09, 0.11, 0.17 (2), 0.26, 0.30 and 0.84 (2) mg/kg.

In field trials conducted with Chinese cabbage (n=4) in China at 45 g ai/hg, the ranked order of residues was (n=4): 0.39, 0.41, 0.99, 1.8 mg/kg. By applying the scaling factor of 0.53, residues were: 0.21, 0.22, 0.53 and 0.95 mg/kg.

The Meeting recognized that the residue population from trials on cabbage and Chinese cabbage were not significantly different according to the Kruskal-Wallis H-test and decided to combine the data sets. The ranked order of residues for estimating maximum residue levels and dietary risk assessment was (n=16): < 0.01, 0.02, 0.06, 0.08, 0.09, 0.11, 0.17 (2), 0.21, 0.22, 0.26, 0.30, 0.53, 0.84(2) and 0.95 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg and an STMR of 0.19 mg/kg for broflanilide in cabbage and Chinese cabbage.

For animal feed, the Meeting estimated a highest residue of 0.95 mg/kg and median residue of 0.19 mg/kg for broflanilide in cabbage.

Tomato (including cherry tomato)

The critical GAP for tomato in the Republic of Korea allows two foliar applications of broflanilide at 2.5 g ai/hL with a RTI of 7 days and a PHI of 2 days. Information on the spray volume was not provided.

A total of 20 field trials conducted with tomato in the United States were performed with 2× 25 g ai/ha (translating into a spray concentration of 8.8–13 g ai/hL) with a RTI of 7 days and harvest after 1 DALA.

The Meeting noted that the proportionality principle could not be applied to trials as the resultant scaling factors are outside the acceptable range (not lower than 0.3).

Hence, the Meeting concluded that no maximum residue level could be estimated for broflanilide in tomato.

Radish, Japanese

The critical GAP for Japanese radish in Japan allows three foliar applications of broflanilide at 2.5 g ai/hL with a PHI of 1 day.

Field trials with Japanese radish in Japan were performed according to the GAP (±25 percent). The ranked order of broflanilide residues in radish roots were (n=6): < 0.01(6) mg/kg.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and an STMR of 0.01 mg/kg for broflanilide in radish, Japanese.

Tuberous and corm vegetables, Subgroup of

The critical GAP for subgroup of tuberous and corm vegetables in the United States allows one in-furrow soil application of broflanilide at 50 g ai/ha and the PHI covered by conditions of use.

Field trials conducted with potato from Canada and the United States were performed according to GAP (±25 percent). The ranked order of broflanilide residues was (n=20): < 0.001(5), 0.0012(2), 0.0015(2), 0.0017, 0.0018, 0.0021, 0.0023, 0.0026, 0.0029, 0.0046. 0.0049(2), 0.015, 0.034 mg/kg.

The Meeting estimated a maximum residue level of 0.04 mg/kg and an STMR of 0.00175 mg/kg for broflanilide in the subgroup of tuberous and corm vegetables.

For animal feed, the Meeting estimated a highest residue of 0.034 mg/kg and median residue of 0.00175 mg/kg for broflanilide in potato culls.

Cereal grains, Group of (except rice)

The critical GAP for cereals (barley, oat, wheat, triticale, rye, millet, sorghum, amaranth, buckwheat, cañihua, chia, cram-cram, huauzontle, quinoa, spelt) in the United States allows for seed treatment with broflanilide at a concentration of 50 g ai/t seeds. The critical GAP for maize, including sweet corn in the United States allows one in-furrow soil application of broflanilide at 50 g ai/ha and the PHI covered by conditions of use.

The Meeting considered that both treatments are similar, as they are both soil treatments. In addition, the results of a seed treatment metabolism study performed with wheat at 100 g ai/ton seeds demonstrated that uptake of radioactivity through the roots into the plant is very limited (TRR maximum of 0.011 mg ai/kg). Therefore, the Meeting decided to consider all data sets for wheat, barley and maize to explore a potential group recommendation.

Field trials with wheat were conducted in Canada and the United States at an exaggerated rate of 100 g ai/ton of seed, giving broflanilide residues of < 0.001 (25) mg/kg.

Field trials with barley were conducted in Canada and the United States at an exaggerated rate of 100 g ai/ton of seed, giving broflanilide residues of < 0.001 (16) mg/kg.

In field trials with maize conducted in Canada and the United States according to the GAP, broflanilide residues were < 0.001 (20) mg/kg.

In field trials with sweet corn conducted in the United States following GAP treatment (±25 percent), broflanilide residues were < 0.001 (12), mg/kg.

The Meeting noted that for the overdosed seed treatment trials in wheat and barley, as well as for the in-furrow treatment of maize all residues were <LOQ.

Hence, the Meeting estimated a maximum residue level of 0.001(*) mg/kg for broflanilide for the group of cereals grains, except rice.

The Meeting also estimated an STMR of 0 mg/kg for the group of cereals grains, except rice and sweet corns, and an STMR of 0.001 mg/kg for sweet corns.

Coffee beans, green

The critical GAP for coffee in Colombia allows two foliar applications of broflanilide at 18 g ai/ha with a RTI of 30 days and a PHI of 45 days.

In field trials conducted with coffee in Brazil and Colombia following GAP (±25 percent), the ranked order of broflanilide residues was (n=9): < 0.001(2), 0.0015, 0.0016, 0.0023, 0.0034, 0.0037, 0.0039, 0.005 mg/kg.

The Meeting estimated a maximum residue level of 0.01 mg/kg, and a STMR of 0.0023 mg/kg for broflanilide in coffee beans, green.

Residues in animal feeds

Wheat forage

The critical GAP for wheat in Canada and the United States allows for seed treatment with broflanilide at a concentration of 50 g ai/t seeds with no livestock feeding restrictions.

Field trials were conducted with wheat in Canada and the United States at an exaggerated rate of 100 g ai/ton of seed. The ranked order of residues in wheat forage was (n=25): < 0.001(24), 0.0011 mg/kg.

The Meeting noted that trials were overdosed and decided to set 0.001 mg/kg (as received) as highest and median residue for broflanilide in wheat forage.

Maize forage

The critical GAP for maize in the United States allows one in-furrow soil application of broflanilide at 50 g ai/ha and the PHI covered by conditions of use.

In field trials conducted with maize in Canada and the United States, broflanilide residues in maize forage following GAP (± 25) were < 0.001(25) mg/kg, as received.

The Meeting estimated a highest and median residue of 0.001 mg/kg (as received) for broflanilide in maize forage.

Cereal grains (including pseudocereals) feed products with low water (<20 percent) content (hay, straw), Subgroup of (except rice)

The critical GAP for wheat and barley in the United States allows for seed treatment with broflanilide at 50 g ai/t seeds with no livestock feeding restrictions. The critical GAP for maize in the United States allows one in-furrow soil application of broflanilide at 50 g ai/ha and the PHI covered by conditions of use. As discussed previously, the Meeting considered that both treatments are similar.

Field trials were conducted with wheat and barley in Canada and the United States at an exaggerated rate of 100 g ai/ton of seed. The ranked order of residues for wheat hay was (n=25): < 0.001(24), 0.0012 mg/kg and for barley hay was (n=16): < 0.001(14), 0.0018, 0.0032 mg/kg, as received.

The Meeting decided to combine the data sets for wheat and barley hay as they were considered similar and apply proportionality principle to the residues from the overdosed trials. Therefore, a scaling factor of 0.5 was applied to residues >LOQ, resulting in a total residue population of (n=41): 0.0006, 0.0009, < 0.001(38), 0.0016 mg/kg, as received.

The ranked order of residues for wheat straw was (n=25): < 0.001(23), 0.001(2) mg/kg and for barley straw was (n=16): < 0.001(16) mg/kg, as received.

The Meeting decided to combine the data sets for wheat and barley straw and apply the scaling factor of 0.5, resulting in a total residue population of (n=41): 0.0005(2), < 0.001(39), mg/kg, as received.

The ranked order of residues in maize stover following GAP treatment (±25) was (n=25): < 0.001(25) mg/kg as received.

Based on the more critical hay data, the Meeting estimated a highest residue of 0.0016 mg/kg (as received), a median residue of 0.001 mg/kg (as received) and a maximum residue level of 0.01 mg/kg (dw, based on 88 percent DM content) for the subgroup of cereal grains (including pseudocereals) feed products with low water (<20 percent) content (hay, straw), except rice feed products.

Fate of residues during processing

The Meeting received information on the hydrolysis of [B-ring-U-^{14}C]- and [C-ring-U-^{14}C]-labelled broflanilide, simulating typical processing conditions (90 °C, pH 4, 20 minutes to simulate pasteurization, 100 °C, pH 5, 60 minutes to simulate boiling, baking and brewing and 120 °C, pH 6, 20 minutes to simulate sterilization). No significant hydrolysis of broflanilide was observed at the conditions studied.

The Meeting concluded that broflanilide is stable under the conditions of pasteurization, boiling, baking and brewing, as well as sterilization.

The fate of broflanilide residues has been examined simulating household and commercial processing of potato, maize, wheat and coffee, and the results are shown in Table 133.

Table 133 Estimated processing factors for maximum residue and dietary exposure of processed commodities according to the residue definition broflanilide

Crop	Residue (mg/kg) in RAC		Processed commodity	Individual PF	Median or best estimate PF	Residue (mg/kg) in processed commodity	
	MRL	STMR				MRL-P	STMR-P
Potato	0.04	0.0018	Starch	< 0.06, < 0.19, 0.27	< 0.19	-	0.0003
			Process waste	0.12, < 0.19, 0.69	< 0.19	-	0.0003
			Dried pulp	0.55, 1.2, 2.3	1.2	-	0.0022
Maize	0.001	0	Bran	1.4	1.4	0.002	0
			Dry milling flour	2.1	2.1	0.002	0
			Germ	0.74, 1.2	0.99	-	0
			Gluten	1.6	1.6	-	0
			Gluten feed meal	6.77	6.77	-	0
			Milled by-products	6.29	6.29	-	0
			RBD oil	0.35, 0.81	0.28	-	0
			Starch	< 0.16	< 0.16	-	0
Wheat	0.001	0	Flour	0.30, 0.44, 0.54	0.44	-	0

Crop	Residue (mg/kg) in RAC		Processed commodity	Individual PF	Median or best estimate PF	Residue (mg/kg) in processed commodity	
	MRL	STMR				MRL-P	STMR-P
			Gluten	0.80, 4.14, 4.97	4.1	-	0
			Milled by-products	6.8, 8.3, 12.2	8.3	-	0
			Starch	0.02, 0.02, 0.04	0.02	-	0
			Germ	1.14, 1.75, 2.77	1.75	0.002	0
			Whole grain bread	0.45, 0.63, 0.78	0.63	-	0
Coffee	0.01	0.0023	Instant coffee	< 0.09, < 0.14, < 0.26	< 0.09	-	0.0002
			Roasted and ground coffee beans	0.38, 0.82, 2.36	0.82	-	0.0019

Residues in animal commodities

Farm animal feeding studies

The Meeting received feeding studies with broflanilide on lactating cows and laying hens.

The study with lactating cows was conducted at treatment rates of 0.015, 0.15, 1.5 and 10 ppm. Residues of parent broflanilide were only detected in milk from the 10 ppm group at up to 0.0018 mg/kg and in cream from the 1.5 and 10 ppm groups at up to 0.016 mg/kg.

Residues of metabolite DM-8007 above LOQ were detected in milk from the 0.15 ppm, 1.5 ppm and 10 ppm groups at up to 0.12 mg/kg and in cream from in all groups at up to 1.3 mg/kg. Metabolite DM-8007 was also detected above LOQ in fats from all groups at up to 0.79 mg/kg, in liver from the 1.5 and 10 ppm groups at up to 0.078 mg/kg and in muscle and kidney from the 10 ppm group at up to 0.08 mg/kg.

The study with laying hens was conducted at treatment rates of 0.02, 0.10 and 0.50 ppm. In eggs and tissues, residues of broflanilide and metabolite DC-DM-8007 were consistently below LOQ for all dose levels.

Residues of metabolite DM-8007 above LOQ were only found in eggs and liver from the 0.5 ppm group at up to 0.023 and 0.021 mg/kg, respectively, as well as in fat from all groups at up to 0.15 mg/kg.

Estimated maximum and mean dietary burdens of livestock and animal commodities maximum residue levels

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are presented in Annex 6. The calculations were made according to the livestock diets from the United States-Canada, European Union, Australia and Japan in the OECD Table (Annex 6 of the 2006 JMPR Report). The summary results are shown in Table 134.

Table 134 Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden for broflanilide, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	Max.	Mean	Max.	Mean	Max.	Mean	Max.	Mean
Beef cattle	0.052	0.004	1.3	0.26	0.021	0.004	-	-
Dairy cattle	0.019	0.0026	1.3❶	0.26❷	0.02	0.004	0.001	0.001

Animal dietary burden for broflanilide, ppm of dry matter diet								
	United States-Canada		European Union		Australia		Japan	
	Max.	Mean	Max.	Mean	Max.	Mean	Max.	Mean
Poultry – broiler	-	-	0.018	0.001	-	-	-	-
Poultry – layer	-	-	0.33❸	0.065❹	-	-	-	-

Notes:

❶ Highest maximum beef or dairy cattle burden suitable for MRL estimates for mammalian meat and milk.

❷ Highest mean beef or dairy cattle burden suitable for STMR estimates for mammalian meat and milk.

❸ Highest maximum broiler or laying hen burden suitable for MRL estimates for poultry products and eggs.

❹ Highest mean broiler or laying hen burden suitable for STMR estimates for poultry products and eggs.

Animal commodities maximum residue levels

For beef and dairy cattle, a maximum and mean dietary burden of 1.3 ppm and 0.26 ppm were estimated, respectively.

For maximum residue level estimation, the maximum dietary burden of 1.3 ppm was evaluated by interpolating between the 0.15 and 1.5 ppm dosing levels of the lactating cow feeding study (Table 135).

Table 135 Maximum residue level estimation of broflanilide in cattle commodities

Maximum residue level beef or dairy cattle	Feed level (ppm)	Sum of broflanilide + DM-8007 in milk (mg/kg)	Sum of broflanilide + DM-8007 in cream (mg/kg)	Sum of broflanilide + DM-8007 (mg/kg)			
				Liver	Kidney	Muscle	Fat
Feeding study	0.15	0.003[a]	0.021[a]	< 0.02	< 0.02	< 0.02	0.026
	1.5	0.012[a]	0.153[a]	0.023	0.02	< 0.02	0.17
Dietary burden and highest residue	1.3	0.011	0.13	0.023	0.02	< 0.02	0.15

Notes:

[a] Mean at plateau level.

For the STMR estimation, the mean dietary burden of 0.26 ppm was evaluated by interpolating between the 0.15 and 1.5 ppm dosing levels of the lactating cow feeding study (Table 136).

Table 136 STMR estimation of broflanilide in cattle commodities

STMR beef or dairy cattle	Feed level (ppm)	Sum of broflanilide + DM-8007 in milk (mg/kg)	Sum of broflanilide + DM-8007 in cream (mg/kg)	Sum of broflanilide + DM-8007 (mg/kg)			
				Liver	Kidney	Muscle	Fat
Feeding study	0.15	0.003 [a]	0.021 [a]	< 0.02	< 0.02	< 0.02	0.024
	1.5	0.012 [a]	0.153 [a]	0.02	< 0.02	< 0.02	0.13
Dietary burden and mean residue	0.26	0.004	0.032	0.02	< 0.02	< 0.02	0.033

Notes:

[a] Mean at plateau level.

The Meeting estimated a maximum residue level for milks at 0.015 mg/kg, milk (fat) at 0.4 mg/kg (assuming 40 percent fat content in cream), edible offal (mammalian) at 0.03 mg/kg and 0.15 mg/kg for meat from mammals (fat) and mammalian fats.

The Meeting estimated STMR values of 0.004 mg/kg in milks, 0.08 mg/kg in milk (fat) (assuming 40 percent fat content in cream), 0.02 mg/kg in edible offal (mammalian), 0.02 mg/kg in muscle from mammals and 0.033 mg/kg in mammalian fats.

For broiler and laying poultry, a maximum and mean dietary burden of 0.33 ppm and 0.065 ppm were estimated, respectively. For maximum residue level estimation, the maximum dietary burden of 0.33 ppm was evaluated by interpolating between the 0.1 and 0.5 ppm dosing levels of the laying hen feeding study (Table 137).

Table 137 Maximum residue level estimation of broflanilide in poultry commodities

Maximum residue level broiler or layer poultry	Feed level (ppm)	Sum of broflanilide + DM-8007 in eggs (mg/kg)	Sum of broflanilide + DM-8007 (mg/kg)		
			Liver	Muscle	Fat
Feeding study	0.1	< 0.02	< 0.02	< 0.02	0.049
	0.5	0.033	0.031	< 0.02	0.16
Dietary burden and highest residues	0.33	0.027	0.026	< 0.02	0.113

For the STMR estimation, the mean dietary burden of 0.065 ppm was evaluated by interpolating between the 0.02 and 0.1 ppm dosing levels of the laying hen feeding study (Table 138).

Table 138 STMR estimation of broflanilide in poultry commodities

STMR broiler or layer poultry	Feed level (ppm)	Sum of broflanilide + DM-8007 in eggs (mg/kg)	Sum of broflanilide + DM-8007 (mg/kg)		
			Liver	Muscle	Fat
Feeding study	0.02	< 0.02	< 0.02	< 0.02	0.020
	0.1	< 0.02	< 0.02	< 0.02	0.044
Dietary burden and mean residues	0.065	< 0.02	< 0.02	< 0.02	0.034

The Meeting recommended a maximum residue level of 0.03 mg/kg for eggs, 0.03 mg/kg for poultry edible offal, 0.02(*) mg/kg for poultry meat and 0.15 mg/kg for poultry fats.

The Meeting estimated an STMR value of 0.02 mg/kg in eggs, poultry edible offal and poultry muscle as well as 0.034 mg/kg for poultry fats.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI.

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *Broflanilide*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *Sum of broflanilide plus 3-benzamido-N-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl)phenyl]-2-fluorobenzamide (DM-8007), expressed as broflanilide*

The residue is fat-soluble.

Table 139 Recommendations for residues of broflanilide from the 2022 JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)	STMR or STMR-P mg/kg	HR or HR-P mg/kg

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VB 0041	Cabbages, Head	2	-	0.19	
VB 0467	Chinese cabbage, (type Pe-tsai)	2	-	0.19	
SB 0716	Coffee bean, green	0.01	-	0.0023	-
MO 0105	Edible offal (mammalian)	0.03	-	0.02	-
PE 0112	Eggs	0.03	-	0.02	-
GC 0080	Cereal grains, Group of (except rice)	0.001*	-	0 (cereal grains) 0.001 (sweet corns)	-
AS 3569	Maize, bran	0.002	-	0	-
CF 1255	Maize, flour	0.002	-	0	-
MF 0100	Mammalian fats	0.15	-	0.033	-
MM 0095	Meat (from mammals other than marine mammals)	0.15 (fat)	-	0.02 (muscle) 0.033 (fat)	-
FM 0183	Milk fats	0.4	-	0.08	-
ML 0106	Milks	0.015	-	0.004	-
VR 0591	Radish, Japanese	0.01*	-	0.01	-
PO 0111	Poultry edible offal	0.03	-	0.02	-
PM 0110	Poultry meat	0.02*	-	0.02 (muscle) 0.034 (fat)	-
PF 0111	Poultry fats	0.15	-	0.034	-
VR 2071	Subgroup of tuberous and corm vegetables	0.04	-	0.00175	
AS 3304	Subgroup of cereal grains (including pseudocereals) feed products with low water (<20 percent) content (hay, straw), except rice feed products	0.01 (dw)	-	Median 0.001 (ar)	Highest 0.0016 (ar)
CF 1210	Wheat, germ	0.002	-	0	-
For dietary risk assessment and/or dietary burden calculations					
	Coffee bean, instant coffee	-	-	0.0002	
SM 0716	Coffee bean, roasted	-	-	0.0019	
OR 0645	Maize oil, edible	-	-	0	
	Maize starch	-	-	0	
	Maize germ	-	-	0	
	Potato, starch	-	-	0.0005	
CF 1211	Wheat, flour	-	-	0	
CF 3522	Wheat, gluten meal	-	-	0	
	Wheat starch	-	-	0	
CP 1212	Wheat, wholemeal bread	-	-	0	

Notes:

ar As received.

dw Dry weight.

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for broflanilide is 0–0.02 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for broflanilide were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2019 JMPR Report.

The IEDIs ranged from 0–1 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of broflanilide from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The Meeting determined that establishment of an acute reference dose is unnecessary for broflanilide and concluded that the acute exposure of residues of broflanilide from uses considered by the Meeting is unlikely to present a public health concern.

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BROFLAN_043	Delinsky, D.	2019	Freezer storage stability of BAS 450 I (Reg. No GLP. 5672774) and metabolites S(PFP-OH)-8007 (Reg. No GLP. 5959598), DM-8007 (Reg. No GLP. 5856361), B-oxam-acid (Reg. No GLP. 6066332) and B-urea (Reg. No GLP. 6065386) in selected plant and bee matrices. BASF Agricultural Solutions, Research Triangle Park NC, United States of America 2018/7003926. GLP. Unpublished
BROFLAN_044	Hiraki, M.	2020	Broflanilide 5 percent SC-Summary report of the storage stability in Korean crop residue study. Dongbang Agro Corp. Research Institute, Yanghwa-myeon ,Buyeo-gun, Chungcheongnam-do Korea Republic of 2020/2107560. no GLP. Unpublished
BROFLAN_045	Hiraki, M.	2020	Broflanilide 5 percent EC-Summary report of the storage stability in Korean crop

Code	Author	Year	Title, Institute, Report reference
			residue study. HanKook SamKong Co. Ltd., Korea Republic of. 2020/2107559. no GLP. Unpublished
BROFLAN_046	Kawaguchi, T.	2020	MCI-8007 (Broflanilide) flowable-Summary report of the storage stability of Japanese radish, turnip and leek Japan Plant Protection Association, Nakazato 2-28-10, Kitazu, Tokyo 114-0015, Japan. 2020/2091594. no GLP. Unpublished
BROFLAN_047	Yoon, T.	2018	Magnitude of Broflanilide residues in green onion following applications of Broflanilide 5 percent SC. Dongbang Agro Corp. Research Institute, Yanghwa-myeon ,Buyeo-gun, Chungcheongnam-do Korea Republic of, Korea Republic of. 2020/2090142, DBA-RC-2017-017. GLP. Unpublished
BROFLAN_048	Park, C.	2018	Magnitude of Broflanilide residues green onion following applications of Broflanilide 5 percent EC. HanKook SamKong Co. Ltd., Korea Republic of. 2020/2090144, SGR-2017-107. GLP. Unpublished
BROFLAN_049	Nakamura, K.	2015	Crop residue study of MCI-8007 in leek treated with MCI-8007 (MIE-1209) flowable. Japan Plant Protection Association, Nakazato 2-28-10, Kita-ku, Tokyo 114-0015, Japan. 2020/2090147, JP2013C277. GLP. Unpublished
BROFLAN_050	Nakamura, K.	2015	Crop residue study of MCI-8007 in leek treated with MCI-8007 (MIE-1209) flowable. Japan Plant Protection Association, Nakazato 2-28-10, Kita-ku, Tokyo 114-0015, Japan. 2020/2090150, JP2014C129. GLP. Unpublished
BROFLAN_051	Schreier, T.	2017	Magnitude of the residue of Broflanilide in brassica vegetables following applications of BAS 450 I. Precision Study Management LLC, Amarillo TX, United States of America. 2016/7009963. GLP. Unpublished
BROFLAN_052	Park, C.	2018	Magnitude of Broflanilide residues in cabbage following applications of Broflanilide 5 percent EC. Hankook Samgong Co. Ltd., Seoul, Korea Republic of. 2020/2090104, SGR-2017-104. GLP. Unpublished
BROFLAN_053	Min, H.	2019	Pesticide residue testing report of Broflanilide 9.5 percent suspension concentrate in cabbages. IPP-CAAS-The Institute of Plant Protection-Chinese Academy of Agricultural Sciences, Beijing, China. 2020/2090096. GLP. Unpublished
BROFLAN_054	Yongquan, Z.	2017	Trial report on the residue of Broflanilide in cabbage IPP-CAAS-The Institute of Plant Protection-Chinese Academy of Agricultural Sciences, Beijing, China. 2020/2090099. no GLP. Unpublished
BROFLAN_055	Yongquan, Z.	2017	Residue test report of Broflanilide in cabbages. IPP-CAAS-The Institute of Plant Protection-Chinese Academy of Agricultural Sciences, Beijing, China. 2020/2090106, 001-15P286-1724. GLP. Unpublished
BROFLAN_056	Yongquan, Z.	2019	Residue test report of Broflanilide in cabbages IPP-CAAS-The Institute of Plant Protection-Chinese Academy of Agricultural Sciences, Beijing, China. 2020/2090110, 001-15P286-1865. GLP. Unpublished
BROFLAN_057	Yoon, T.	2018	Magnitude of Broflanilide residues in chinese cabbage following applications of Broflanilide 5 percent SC. Dongbang Agro Corp. Research Institute, Yanghwa-myeon ,Buyeo-gun, Chungcheongnam-do Korea Republic of, Korea Republic of. 2020/2090115, DBA-RC-2017-011. GLP. Unpublished
BROFLAN_058	Park, C.	2018	Magnitude of Broflanilide residues in chinese cabbage following applications of Broflanilide 5 percent EC. HanKook SamKong Co. Ltd., Korea Republic of. 2020/2090117, SGR-2017-103. GLP. Unpublished
BROFLAN_059	Yuting, Z.	2017	Trial report on the residue of Broflanilide in chinese cabbage Tianjin Institute of Agriculture Quality Standards and Testing Techno GLPlogy, Tianjin, China. 2020/2090098. no GLP. Unpublished
BROFLAN_060	Na, L.	2019	BAS 450 00 I residue in Chinese cabbage 2018 in China Tianjin Institute of Agriculture Quality Standards and Testing Techno GLPlogy, Tianjin, China. 2020/2090097. GLP. Unpublished
BROFLAN_061	Reeves, L.	2020	Magnitude and decline of Broflanilide residues following applications of BAS 450 I to fruiting vegetables (crop group 8) Eurofins Agroscience Services Inc., Lancaster PA, United States of America. 2020/2081627. GLP. Unpublished

Code	Author	Year	Title, Institute, Report reference
BROFLAN_062	Park, C.	2018	Magnitude of Broflanilide residues in tomato following application of Broflanilide 5 percent EC. HanKook SamKong Co. Ltd., Korea Republic of. 2020/2090154, SGR-2017-106. GLP. Unpublished
BROFLAN_063	Yoon, T.	2019	Magnitude of Broflanilide residues in tomato following applications of Broflanilide 5 percent SC. Dongbang Agro Corp. Research Institute, Yanghwa-myeon ,Buyeo-gun, Chungcheongnam-do Korea Republic of, Korea Republic of. 2020/2090156, R1943. GLP. Unpublished
BROFLAN_064	Park, C.	2018	Magnitude of Broflanilide residues in radish following applications of Broflanilide 5 percent EC. HanKook SamKong Co. Ltd., Korea Republic of. 2020/2090125, SGR-2017-102. GLP. Unpublished
BROFLAN_065	Jeong, H.	2020	Magnitude of Broflanilide residues in radish following applications of Broflanilide 5 percent SC. Dongbang Agro Corp. Research Institute, Yanghwa-myeon ,Buyeo-gun, Chungcheongnam-do Korea Republic of, Korea Republic of. 2020/2090129, R1941. GLP. Unpublished
BROFLAN_066	Nakamura, K.	2014	Crop residue study of MCI-8007 in Japanese radish treated with MCI-8007 (MIE-1209) flowable. Japan Plant Protection Association, Nakazato 2-28-10, Kita-ku, Tokyo 114-0015, Japan. 2020/2090131, JP2013C275. GLP. Unpublished
BROFLAN_067	Nakamura, K.	2015	Crop residue study of MCI-8007 in Japanese radish treated with MCI-8007 (MIE-1209) flowable. Japan Plant Protection Association, Nakazato 2-28-10, Kita-ku, Tokyo 114-0015, Japan. 2020/2090133, JP2014C124. GLP. Unpublished
BROFLAN_068	Nakamura, K.	2015	Crop residue study of MCI-8007 in turnip treated with MCI-8007 (MIE-1209) flowable. Japan Plant Protection Association, Nakazato 2-28-10, Kita-ku, Tokyo 114-0015, Japan. 2020/2090138, JP2014C125. GLP. Unpublished
BROFLAN_069	Crawford, L.	2017	Magnitude of the residue of Broflanilide, (BAS 450 I) in potatoes following foliar or in-furrow applications of BAS 450 00 I.Landis International Inc., Valdosta GA, United States of America. 2016/7009341. GLP. Unpublished
BROFLAN_070	Wyatt, D.	2017	Magnitude of the residues of Broflanilide in or on wheat raw agricultural commodities following seed treatment with BAS 450 01 I.The Carringers Inc., Apex NC, United States of America. 2016/7006466. GLP. Unpublished
BROFLAN_071	Greenland, R.	2017	Magnitude of the residues of BAS 450I in barley following seed treatment with BAS 450 01 I. Stewart Agricultural Research Services Inc., Clarence MO, United States of America 2016/7009340. GLP. Unpublished
BROFLAN_072	Wyatt, D.	2017	Magnitude of the residues of Broflanilide in or on field corn and sweet corn raw agricultural commodities following one in-furrow application of BAS 450 00 I. The Carringers Inc., Apex NC, United States of America. 2016/7006467. GLP. Unpublished
BROFLAN_073	de Matos, C.	2018	Residue study of broflanilide in coffee (beans), after treatment with BAS 450 00 I, under field conditions in Brazil. BASF SA, Guaratingueta, Brazil. 2020/2090092. GLP. Unpublished
BROFLAN_074	Jose, W	2020	Residue study of broflanilide in coffee (grain) after treatment with BAS 450 00 I under field conditions in Colombia. BASF S.A.-Global Environmental and Consumer Safety Laboratory-GENCS, Sao Paulo, Brazil. 2019/2076212. GLP. Unpublished
BROFLAN_075	Strathdee, A.	2017	14C-BAS 450 I: Simulated processing-hydrolysis at 90°C, 100°C and 120°C. Charles River Laboratories, Tranent East Lothian EH33 2NE, United Kingdom. 2017/1077933. GLP. Unpublished
BROFLAN_076	Strathdee, A.	2017	Amendment 1: 14C-BAS 450 I: Simulated processing-hydrolysis at 90°C, 100°C and 120°C. Charles River Laboratories, Tranent East Lothian EH33 2NE, United Kingdom. 2017/1205294. GLP. Unpublished
BROFLAN_077	Reeves, L.	2020	Magnitude of BAS 450 I residues in tomato processed fractions Eurofins Agroscience Services Inc., Forsyth GA, United States of America. 2020/2036100. GLP. Unpublished
BROFLAN_078	Crawford, L.	2017	Magnitude and concentration of the residue of Broflanilide, (BAS 450 I) in potato processed commodities following in-furrow and foliar applications of BAS 450 00 I. Landis International Inc., Valdosta GA, United States of America.

Code	Author	Year	Title, Institute, Report reference
			2017/7000465. GLP. Unpublished
BROFLAN_079	Jordan, J.	2017	Magnitude of the residues of BAS 450 I in corn processed fractions following applications of BAS 450 00 I. BASF Corp., Research Triangle Park NC, United States of America. 2016/7009342. GLP. Unpublished
BROFLAN_080	Jordan, J.	2017	Magnitude of the residues of BAS 450 I in wheat processed fractions following applications of BAS 450 00 I. BASF Crop Protection., Research Triangle Park NC, United States of America. 2017/7016659. GLP. Unpublished
BROFLAN_081	José, W	2019	Residue study of broflanilide in coffee (dried bean, grains and processed fractions), after treatment with BAS 450 00 I under field conditions in Brazil. BASF S.A.-Global Environmental and Consumer Safety Laboratory-GENCS, Sao Paulo, Brazil. 2019/2050329. GLP. Unpublished
BROFLAN_082	Xu, A.	2017	A meat and milk magnitude of the residue study with BAS 450 I in lactating dairy cows. PASC-Primera Analytical Solutions Corp., Princeton NJ, United States of America. 2017/7016675 GLP. Unpublished
BROFLAN_083	Ray, W.	2017	Magnitude of the residues in eggs and tissues of laying hens following oral administration of BAS 450 I. Symbiotic Research LLC, Mount Olive NJ, United States of America. 2017/7016766. GLP. Unpublished

CHLORANTRANILIPROLE (230)

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EXPLANATION

Chlorantraniliprole was first evaluated for residues and toxicological aspects by the 2008 JMPR. The 2008 JMPR established an ADI for chlorantraniliprole of 0–2 mg/kg bw and concluded that an ARfD was not necessary.

Definition of the residue for both compliance with MRL and estimation of dietary intake (for plant and animal commodities): chlorantraniliprole.

The residue is fat soluble.

It was evaluated for additional maximum residue levels in 2010, 2013, 2014, 2016, and 2019 (extra). At the Fifty-second CCPR (2021), chlorantraniliprole was listed for consideration of further additional maximum residue levels by the 2022 JMPR.

The current Meeting received new information on use patterns, supervised residue trials and storage studies on avocado and tea.

RESIDUE ANALYSIS

Analytical methods

The Meeting received a new method of analysis for tea (Woodward and Tsuchizawa (2010)).

For tea leaves, powdered dried tea leaves (5 g) was soaked in 20 mL of water for 2 hours, and then added to acetonitrile-water (8:2, v/v) and shaken for 30 minutes. After filtration, the extract was loaded onto an SPE cartridge (conditioned with acetonitrile (5 mL) and water (5 mL)), washed with 5 mL of acetonitrile-water (2:8, v/v) and eluted with 10 mL of acetonitrile-water (8:2, v/v).

For tea infusion, 9 g of dried tea leaves was extracted with 540 mL of boiling water for 5 minutes. The filtered extract was cleaned up by SPE cartridge with the same procedure as that for tea leaves.

Both samples after clean-up were introduced to LC-MS/MS using ESI (C18 column, 5 mmol/L ammonium acetate (aq) – 5 mmol ammonium acetate in methanol, 50:50->5:95, v/v). Ion monitored was 484.0 -> 285.8 for chlorantraniliprole. Calibration curve was linear (R^2 > 0.999) between 0.001 and 0.04 mg/kg. LOD and LOQ for chlorantraniliprole were 0.003 and 0.01 mg/kg, respectively.

The validation data were shown in Table 1.

Table 1 Method validation data for tea

Analyte	Matrix	Fortification mg/kg	n	Recovery range % (mean)	RSD percent
Chlorantraniliprole	Tea leaves	0.01	5	100-114 (107)	4.8
		0.5	5	96-101 (99)	1.9
		50	5	96-102 (99)	2.7
	Tea infusion	0.01	5	84-101 (95)	7.4
		0.5	5	94-103 (98)	3.8
		20	5	100-104 (102)	1.8

Avocados were analysed using Method 13294, evaluated by the 2008 JMPR.

In summary, 5 g of avocado with 10 mL of water was mixed with 40 mL of acetonitrile, blended (approximately 2 minutes) and centrifuged. Acetonitrile (50 mL) was added to the supernatant and centrifuged again. An aliquot (5 mL) of supernatant was added to 20 mL of water and cleaned up by SPE (SAX, HLB) cartridge column eluted with acetonitrile-ethyl acetate (4:1, v/v). After filtration, the sample was introduced to LC-MS/MS using ESI (C18 column, 0.01 mol/L formic acid (aq) – 0.01 mol/L formic acid in methanol). Ions monitored were 484 -> 453 and 484 -> 286 for chlorantraniliprole. Calibration curve was linear (R^2 > 0.99) between 0.004 and 0.2 mg/kg. LOD and LOQ for chlorantraniliprole were 0.003 and 0.01 mg/kg, respectively.

Stability of pesticide residues in stored analytical samples

The Meeting received storage stability studies for tea (Woodward and Tsuchizawa (2010), Kawano (2021a) and Kawano (2021b)). Tea leaves fortified with chlorantraniliprole at 0.5 or 0.1 mg/kg were stored frozen at approximately -20 °C for 39–131 days and analysed in duplicate (Table 2). Samples at day 0 was not analysed; however, concurrent recovery in the analysis of field trial samples were between 79–111 percent at the fortification levels of 0.01–30 mg/kg (Table 3).

Table 2 Storage stability of chlorantraniliprole in tea leaves

Analyte	Matrix	Fortification mg/kg	Storage period (days)	Mean recovery (%)
Chlorantraniliprole	Tea leaves [a]	0.5	66	102
	Tea leaves [a]	0.5	39	99
	Tea leaves [b]	0.1	131	95
	Tea leaves [c]	0.1	53	93

Notes:

[a] Woodward and Tsuchizawa (2010).

[b] Kawano (2021a).

[c] Kawano (2021b).

Table 3 Concurrent recovery of chlorantraniliprole in tea leaves

Analyte	Matrix	Fortification mg/kg	n	Recovery range % (mean)	RSD percent
Chlorantraniliprole	Tea leaves [a]	0.01	3	102-116 (111)	7.0
	Tea leaves [a]	0.01	3	73-83 (79)	6.9
	Tea leaves [a]	30	3	90-91 (90)	0.64
	Tea leaves [a]	30	3	88-94 (92)	3.5
	Tea leaves [b]	0.1	4	84-93 (89)	3.9
	Tea leaves [c]	0.1	2	87-113 (100)	NA

Notes:

[a] Woodward and Tsuchizawa (2010).

[b] Kawano (2021a).

[c] Kawano (2021b).

USE PATTERN

The Meeting received the GAP for avocado and tea as shown in Table 4. The labels provided cover broader spectrum of uses.

Table 4 Use pattern of chlorantraniliprole

Crop	Country	Concentration g ai/kg Formulation	Application							
			Type	kg ai/ha	g ai/L	Application volume (L/ha)	Growth stage	No	minimum RTI (days)	PHI (days)
Avocado	United States	350 g ai/kg, WG	Foliar spray	0.112/a	-	-	NA	2 [a]	10	1
Tea	Japan	100 g ai/kg, FL	Foliar spray	-	0.05	4000 [b]	NA	1 [c]	NA	3

[a] According to the US label, the application rate for each application (0.074–0.112 kg ai/ha) and the maximum seasonal rate (0.224 kg ai/ha) indicates that cGAP can be 3 × 0.074 kg ai/ha or 2 × 0.112 kg ai/ha. As the PHI is the same for these GAPs (1 day), the latter is considered more critical.

[b] Recommended maximum application volume.

[c] Maximum 1 application before first pick and maximum 1 application between picks (e.g. maximum 1 application before first pick, up to a total of 2 (1+1) before second pick, 3 (1+1+1) before third pick etc.).

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue levels were reported as measured. When residue concentrations were less than LOQ, they are shown as below the LOQ, e.g., < 0.01 mg/kg. Residue values from the trials conducted according to the maximum GAP were used for the estimation of maximum residue levels, STMR and HR. These results are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. The results of field trials are shown in tables indicated in Table 5.

Table 5 Results of supervised field trials for chlorantraniliprole

Commodity	Result
Avocado	Table 6
Tea	Table 7

Avocado (Shepard (2021))

The Meeting received five supervised trials conducted in 2020 and 2021 on avocado in the United States of America. In these trials, avocados received two foliar applications of chlorantraniliprole (350 g/kg WG) at 0.111–0.114 kg ai/ha with intervals of 9–10 days. In two trials, plants were harvested at 1 day after the last application (DALA) and other three trials were decline study (harvested at 0–7 DALA). Two trials were conducted at the same venue in the same year.

Samples harvested were stored at -20 °C for up to 6.5 months before analysis. Since the storage stability data from the 2008 JMPR had covered a diverse range of crops and demonstrated stability of chlorantraniliprole for at least 24 months, it was considered that the data should be sufficient to cover the storage stability of chlorantraniliprole in avocados in this submission.

The residues of chlorantraniliprole in avocado, with stone removed, were analysed by Method 13294 (LOQ=0.01 mg/kg). Two samples for each trial were duplicatedly analysed and the averages were shown as residue data. Procedural recoveries of chlorantraniliprole in avocado were 92–100 percent.

The residues of chlorantraniliprole in avocado when harvested around 1 DALA were 0.063–0.16 mg/kg (Table 6).

Table 6 Residues of chlorantraniliprole in avocado after foliar application of chlorantraniliprole 350 g/kg WG

Location, year (variety)	No	RTI (day)	kg ai/ha	DALA	Residue (mg/kg) Chlorantraniliprole	Note
GAP (United States)	2	10	2 × 0.112	1		
Homestead, FL, United States 2020 (Monroe)	2	10	0.112 0.111	-0 0 1 3 7	0.048 0.033 0.040 0.063/[a] 0.047	
Lemon Cove, CA, United States 2021 (Lamb hass)	2	10	0.112 0.112	-0 0 1 3 7	0.058 0.14 0.081 0.066 0.083/[b]	
Porterville, CA, United States 2021 (Hass)	2	10	0.112 0.114	-0 0 1 3 7	0.026 0.063 0.081 0.075 0.094/[c]	
Santa Paula, CA, United States 2021 (Hass)	2	10	0.113 0.112	1	0.16/[d]	[e]
Santa Paula, CA, United States 2021 (Hass)	2	9	0.112 0.112	1	0.043	[e]

Notes:

[a] The higher individual analytical value in duplicate samples: 0.064 mg/kg.

[b] The higher individual analytical value in duplicate samples: 0.12 mg/kg.

[c] The higher individual analytical value in duplicate samples: 0.11 mg/kg.

[d] The higher individual analytical value in duplicate samples: 0.20 mg/kg.

[e] Trials were conducted in the same year and the same place. The difference of application dates was 39 days.

Tea (Woodward and Tsuchizawa (2010), Kawano (2021a) and Kawano (2021b))

The Meeting received 10 supervised trials conducted in 2006-2021 on tea in Japan. In these trials, tea received one foliar application of chlorantraniliprole (0.05 g ai/L, dilution of formulation at 100 g/kg FL) and harvested 3–21 days after treatment (DAT). Samples after harvest were stored at <-20 °C for up to 3.5 months before analysis.

Dried tea leaves were prepared from fresh tea leaves harvested to be steamed twice at 100 °C for 1 minute, and then dried three times in an oven at 80 °C for 120 minutes with intervals of 30 minutes.

The residues of chlorantraniliprole in tea leaves were analysed by LC-MS/MS method (LOQ=0.01 mg/kg). Procedural recoveries of chlorantraniliprole in dried tea leaves were 73–116 percent.

The residues of chlorantraniliprole in dried tea leaves when harvested 3 DAT were 19–32 mg/kg (Table 7).

Table 7 Residues of chlorantraniliprole in tea, green after foliar application of chlorantraniliprole 100g/kg FL

Location, year (variety)	Application No	g ai/L	Application volume L/ha	DAT	Residue in tea leaves (mg/kg) Chlorantraniliprole	Reference
GAP (Japan)	1	0.05		3		
Shizuoka, Japan 2006 (Yabukita)	1	0.05	4000	3 7 14 21	25 21 4 0.35	Woodward and Tsuchizawa (2010)
Nara, Japan 2006 (Yabukita)	1	0.05	4000	3 7 14 21	29 14 4.5 0.88	Woodward and Tsuchizawa (2010)
Ibaraki, Japan 2020 (Yabukita)	1	0.05	3000	3 7 14 21	19 7.6 2.2 0.14	Kawano (2021a).
Saitama, Japan 2020 (Hokumei)	1	0.05	4000	3 7 14 21	28 13 5.1 0.83	Kawano (2021a).
Shizuoka, Japan 2020 (Yabukita)	1	0.05	4000	3 7 14 21	28 16 6.9 0.69	Kawano (2021a).
Nara, Japan 2020 (Yabukita)	1	0.05	3400	3 7 14 21	19 14 7.1 1.8	Kawano (2021a).
Kochi, Japan 2020 (Yabukita)	1	0.05	3800	3 7 14 21	32 7.2 1.8 0.5	Kawano (2021a).
Miyazaki, Japan 2020 (Yabukita)	1	0.05	3200	3 7 14 21	22 6.5 1.2 0.06	Kawano (2021a).
Shizuoka, Japan 2021 (Yabukita)	1	0.05	4000	3 7 14 21	24 21 7.0 5.2	Kawano (2021b)
Miyazaki, Japan 2021 (Minamisayaka)	1	0.05	4000	3 7 14 21	23 16 6.5 1.1	Kawano (2021b)

FATE OF RESIDUES IN STORAGE AND PROCESSING

Tea (Kawano (2021a) and Kawano (2021b))

Tea infusion was prepared from dried tea leaves (stored at -20 °C for up to 3.5 months) as follows: 9 g of dried tea leaves were soaked in 540 mL of boiling water for 5 minutes and filtered. Tea infusion was analysed with LC-MS/MS method with LOQ of 0.01 mg/kg. The procedural recoveries for tea infusion were 88–101 percent.

The processing factors from 32 trials ranged from 0.0053 to 0.011 with a best estimate (median) of 0.0081 (Table 8, Table 9).

Table 8 Processing factor of chlorantraniliprole from dried tea leaves to infusion

Location, year (variety)	Application No	g ai/L	Application L/ha	DAT	Residue in tea leaves (mg/kg) Chlorantraniliprole	Residue in tea infusion (mg/L) Chlorantraniliprole	Processing factor	Reference
GAP (Japan)	1	0.05		3				
Ibaraki, Japan 2020 (Yabukita)	1	0.05	3000	3 7 14 21	19 7.6 2.2 0.14	0.16 0.065 0.018 0.001	0.0083 0.0086 0.0083 0.0071	Kawano (2021a)
Saitama, Japan 2020 (Hokumei)	1	0.05	4000	3 7 14 21	28 13 5.1 0.83	0.20 0.10 0.03 0.005	0.0071 0.0078 0.0062 0.0064	Kawano (2021a)
Shizuoka, Japan 2020 (Yabukita)	1	0.05	4000	3 7 14 21	28 16 6.9 0.69	0.22 0.12 0.057 0.006	0.0077 0.0074 0.0082 0.0082	Kawano (2021a)
Nara, Japan 2020 (Yabukita)	1	0.05	3400	3 7 14 21	19 14 7.1 1.8	0.14 0.11 0.055 0.014	0.0076 0.0080 0.0077 0.0078	Kawano (2021a)
Kochi, Japan 2020 (Yabukita)	1	0.05	3800	3 7 14 21	32 7.2 1.8 0.5	0.23 0.057 0.013 0.003	0.0073 0.0079 0.0071 0.0053	Kawano (2021a)
Miyazaki, Japan 2020 (Yabukita)	1	0.05	3200	3 7 14 21	22 6.5 1.2 0.06	0.18 0.058 0.011 0.001	0.0083 0.0090 0.0094 0.011	Kawano (2021a)
Shizuoka, Japan 2021 (Yabukita)	1	0.05	4000	3 7 14 21	24 21 7.0 5.2	0.22 0.17 0.06 0.04	0.0091 0.0084 0.0085 0.0072	Kawano (2021b)
Miyazaki, Japan 2021 (Minamisayaka)	1	0.05	4000	3 7 14 21	23 16 6.5 1.1	0.20 0.13 0.06 0.01	0.0086 0.0086 0.0085 0.0094	Kawano (2021b)

Table 9 Processing factor from tea leaves to tea infusion (Summary)

RAC	Processed commodity	Calculated processing factors [a]	PF (mean or best estimate)
Tea leaves	Tea infusion	0.0053, 0.0062, 0.0064, 0.0071 (3), 0.0072, 0.0073, 0.0074, 0.0076, 0.0077 (2), 0.0078 (2), 0.0079, 0.0080, 0.0082 (2), 0.0083 (3), 0.0084, 0.0085 (2), 0.0086 (3), 0.0090, 0.0091, 0.0094 (2), 0.011	0.0081

Notes:

[a] Each value represents a separate trial. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

APPRAISAL

Chlorantraniliprole was first evaluated for residues and toxicological aspects by the 2008 JMPR. The 2008 JMPR established an ADI for chlorantraniliprole of 0–2 mg/kg bw and concluded that an ARfD was not necessary.

The 2008 JMPR also established the definition of residue as follows:

Definition of the residue for both compliance with MRL and estimation of dietary intake for plant and animal commodities: *chlorantraniliprole*

The residue is fat soluble.

It was evaluated for additional uses by JMPR in 2010, 2013, 2014 and 2016. At the Fifty-second Session of the CCPR (2021), chlorantraniliprole was listed for consideration of additional uses by the 2022 JMPR.

The current Meeting received new information on method of analysis, storage studies, use patterns, supervised residue trials and processing studies on avocado and tea.

Methods of analysis

The Meeting noted that the analytical method for avocado had been evaluated by the 2008 JMPR.

The Meeting received information on a new analytical method for chlorantraniliprole in tea.

Chlorantraniliprole was extracted from tea leaves (dry) with acetonitrile/water (8:2, v/v), cleaned-up and analysed with LC-MS/MS. Tea infusion, prepared by soaking tea leaves in boiling water and filtered, was cleaned-up and then analysed with the same method. Recovery data supported an LOQ of 0.01 mg/kg. The Meeting agreed that the method is suitable for analysing chlorantraniliprole in dried tea leaves and tea infusion.

Stability of pesticides in stored analytical samples

Stability studies of chlorantraniliprole in tea leaves (dry) were available. The Meeting concluded that chlorantraniliprole in tea leaves stored at ≤-20 °C was stable for at least 4 months. All the residue samples were analysed within this period. Concurrent recoveries for tea leaves in the field trial samples were 73–116 percent.

Results of supervised residue trials on crops

Avocado

The critical GAP for avocado in the United States is two foliar applications at 0.112 kg ai/ha with a minimum interval of 10 days and PHI of 1 day.

In trials matching the GAP, residues of chlorantraniliprole in avocados were (n=5): 0.043, 0.063, 0.083, 0.094 and 0.16 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR value of 0.083 mg/kg for avocado.

Tea, green, black (black, fermented and dried)

The critical GAP for tea in Japan is one foliar application of chlorantraniliprole (5 g ai/hL) before each pick with a PHI of 3 days.

The Meeting noted that the typical interval between picks is 30 days. According to the decline study, 0.3–9.6 percent of the DAT3 residue remained 18 days. The Meeting considered that the residue carry-over from previous treatments would be insignificant and agreed that the trials approximated the cGAP in Japan.

In trials approximating the GAP, residues of chlorantraniliprole in tea leaves (dry) were (n=10): 19 (2), 22, 23, 24, 25, 28 (2), 29 and 32 mg/kg.

The Meeting estimated a maximum residue level of 80 mg/kg and an STMR of 24.5 mg/kg.

Fate of residues during processing

Tea infusion

The Meeting received residue data for chlorantraniliprole in tea infusions.

Residues of chlorantraniliprole in tea infusion, prepared from the tea leaves in trials conducted in 2020 and 2021, were (n=8): 0.14, 0.16, 0.18, 0.20 (2), 0.22 (2) and 0.23 mg/kg.

The Meeting estimated an STMR value of 0.20 mg/kg in tea infusion.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with MRL and for estimation of dietary risk assessment for plant and animal commodities: *chlorantraniliprole*.

The residue is fat-soluble.

Table 10 Maximum residue level Recommendations for chlorantraniliprole

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FI0326	Avocado	0.3		0.083	
DT1114	Tea, green, black (black, fermented and dried)	80		24.5	

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
	Tea infusion			0.20	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for chlorantraniliprole is 0–2 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for chlorantraniliprole were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the current and earlier JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 0–1 percent of the maximum ADI for chlorantraniliprole. The Meeting concluded that long-term dietary exposure to residues of chlorantraniliprole from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for chlorantraniliprole is not necessary. The Meeting concluded that acute dietary exposure to residues of chlorantraniliprole from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Code	Author(s)	Year	Title
Woodward and Tsuchizawa (2010)	Woodward, M and Tsuchizawa, M	2010	Magnitude and decline of chlorantraniliprole residues in tea – Japan, 2007. The Institute of Environmental Toxicology, Japan. Report number DuPont-26802, Revision No. 1. GLP: No. Unpublished.
Kawano (2021a)	Kawano, T	2021	Crop field trial on Japanese green tea with SAMCOR Flowable 10. Japan Plant Protection Association. Japan. Report Number JP2020C253. GLP: Yes. Unpublished.
Kawano (2021b)	Kawano, T	2021	Crop field trial on Japanese green tea with SAMCOR Flowable 10. Japan Plant Protection Association. Japan. Report Number JP2021C049. GLP: Yes. Unpublished.
Shepard (2021)	Shepard, E	2021	Magnitude and Decline of Chlorantraniliprole (E2Y45) Residues in Avocados Following Two Foliar Applications of Chlorantraniliprole 35WG – 5 sites in United States, 2020. Report No. FMC-55093, Eurofins Study No. 90540. GLP: Yes. Unpublished.

CHLORMEQUAT (015)

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EXPLANATION

Chlormequat (usually formulated as the chloride salt) is a plant growth regulator registered for use on cereals and grapes. It acts primarily by reducing cell elongation, as well as by lowering the rate of cell division and by inhibiting the synthesis of gibberellins. Chlormequat was evaluated by the Meeting in 1970, 1972, 1994 (T, R), 1997 (R), 1999 (T, for ARfD), 2000 (R) and 2017 (T, R, periodic re-evaluation).

The 2017 Meeting reaffirmed the ADI of 0–0.05 mg/kg bw (established in 1997) and ARfD of 0.05 mg/kg bw (established in 1999). The 2017 Meeting confirmed residue definitions as follows:

The residue definition (for compliance with the MRL and dietary risk assessment) in plant and animal commodities: *chlormequat cation*.

The residue is not fat soluble.

For chlormequat chloride technical concentrate and soluble concentrate, specifications were established by the Joint Meeting on Pesticides Specifications in 2005.

The Forty-third Session of the Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included chlormequat for evaluation of uses on barley and wheat.

The current Meeting received new information on: GAP in Australia, Canada and the United Kingdom of Great Britain and Northern Ireland, analytical method, supervised residue trials conducted in Canada and the United States, and processing studies, for wheat and barley.

RESIDUE ANALYTICAL METHODS

Analytical methods

The 2017 Meeting evaluated analytical methods for determining chlormequat chloride in plant and animal matrices and their validation data.

The current Meeting received information on an LC-MS/MS method (M01-011) for determining chlormequat chloride in the supervised residue trials and processing studies. This method was similar to the LC-MS/MS method (CEN/TC 275/WG 4N) provided to the 2017 JMPR. In M01-011 method, a homogenized sample of each of wheat or barley matrices (grain, leafy parts and processed commodities) was fortified with a known concentration of chlormequat-D4 chloride as internal standard. Residues of chlormequat/internal standard were extracted from the fortified sample with methanol/water (2:1, v/v) using a high-speed homogenizer. An aliquot of extract was then filtered for determination of chlormequat by LC-MS/MS.

For validation of the method for determining chlormequat chloride in wheat and barley matrices, recovery tests were performed at the fortification levels of 0.01 mg/kg and 0.1 mg/kg, each in five replicates. In these recovery tests, two mass transitions were monitored: m/z 122 → 58 for quantification; and m/z m/z 122 → 59 or m/z 122 → 63 for confirmation as shown in the following table.

At the fortification levels of 0.01 and 0.1 mg/kg in all the wheat and barley matrices tested, the recoveries and RSD values were within the acceptable range and the validated LOQs were 0.01 mg/kg.

Table 1 Summary of recoveries of the LC-MS/MS method for the determination of chlormequat chloride in wheat, barley and their processed commodities

Matrix	Fortification level (mg/kg)	n	Recovery (%)			Recovery (%)		
			Range	Mean	RSD	Range	Mean	RSD
			m/z 122 → 58 (Quantification)			m/z 122 → 59 (Confirmation)		
AA 150722, Norris, F.A., 2016 (calculated as chlormequat chloride)								
Wheat grain	0.0103	5	103–109	105	2.5	89–112	103	8.3
	0.103	5	101–110	107	3.2	94–110	103	6.0
Wheat straw	0.0103	5	98–110	105	5.0	102–109	105	3.0
	0.103	5	94–98	96	1.5	82–95	90	6.8
Wheat hay	0.0103	5	100–116	110	5.7	104–114	110	3.8
	0.103	5	91–103	97	4.8	88–100	95	4.7
Wheat forage	0.0103	5	103–112	108	3.2	106–115	110	3.0
	0.103	5	101–107	103	2.4	88–105	98	8.3
Wheat AGF [a]	0.0103	5	100–114	108	5.6	90–109	102	7.9
	0.103	5	87–98	91	5.3	90–97	93	2.9
			m/z 122 → 58 (Quantification)			m/z 122 → 63 (Confirmation)		
AA 160702, Hoi, S.W., 2017a (calculated as chlormequat cation)								
Wheat bran	0.0103	5	95–105	100	4.2	92–103	97	5.4
	0.103	5	91–95	94	2.1	94–101	97	2.7
Wheat flour	0.0103	5	95–106	102	4.2	97–102	99	2.1
	0.103	5	95–105	100	4.2	94–101	99	2.6
Wheat middlings	0.0103	5	95–102	99	2.8	100–109	105	4.5
	0.103	5	100–105	102	2.0	97–106	101	3.6
Wheat shorts	0.0103	5	99–107	103	3.2	97–106	101	3.5
	0.103	5	101–105	104	1.5	93–102	99	3.7
Wheat germ	0.0103	5	87–118	100	13.1	96–108	101	5.0
	0.103	5	100–109	105	3.4	98–107	101	3.3
AA 160703, Hoi, S.W., 2017b (calculated as chlormequat cation)								
Barley grain	0.0103	5	106–109	108	1.2	106–110	108	1.4
	0.103	5	100–105	102	2.0	96–104	101	3.1
Barley bran	0.0103	5	100–108	104	2.9	100–109	104	3.9
	0.103	5	98–104	102	2.2	97–100	99	1.4
Barley flour	0.0103	5	99–107	104	3.0	96–110	104	5.5
	0.103	5	103–105	104	0.86	99–103	101	1.5
Barley pearled seed	0.0103	5	98–103	101	2.2	92–106	99	4.9
	0.103	5	100–104	103	2.1	99–102	100	1.2
Barley hay	0.0103	5	87–106	99	7.4	91–103	96	4.5
	0.103	5	99–108	102	3.4	97–100	99	1.4
Barley straw	0.0103	5	92–106	99	7.2	91–97	94	3.3
	0.103	5	97–103	100	2.4	97–103	100	2.2

Notes:

[a] Aspirated grain fractions.

Stability of pesticide residues in stored analytical samples

The 2017 Meeting evaluated the storage stability data on cereal matrices (grain, straw and processed matrices). Chlormequat chloride in wheat grain and straw samples fortified at 0.10 and 0.50 mg/kg were

demonstrated to be stable for at least 24 months when stored frozen at approximately -18 or -20 °C. Wheat bran and wholemeal bread, and barley malt and beer from a processing study were re-analysed for chlormequat chloride after about 11–13 months of further frozen storage. Residues of chlormequat chloride were stable for at least 13 months in wheat bran and whole wheat bread, 12 months in barley malt and 11 months in beer, when stored frozen at approximately -18 °C.

The proved stable periods cover the sample storage intervals in the residue trials.

USE PATTERN

Chlormequat has been registered as a plant growth regulator for uses on a number of crops. The 2017 Meeting received labels from many countries and the use patterns (foliar applications) related to the supervised trials submitted to the Meeting were summarized.

The current Meeting received a new label from Australia, Canada and the United Kingdom. As the supervised trial data were submitted to the current Meeting on wheat and barley, the uses on wheat and, barley are summarized below. The labels of Canada and the United Kingdom do not restrict grazing or harvesting forage while that of Australia contains "grazing and cutting for stock feed are not allowed before 21 days after the application".

Table 2 Registered uses of chlormequat chloride on cereals relevant to the current evaluation of supervised trials on wheat and barley

Crop	Country	Conc. g ai/L Form	Application					Min. PHI days
			Method	Growth stage/timing	Rate kg ai/ha	Min Water Volume, L/ha	No	
Barley, spring & winter	Canada	620 SL	Foliar	GS 30–32 [a]	1.426	100	1	NA
				GS 14–32 GS 32–39	0.713 + 0.713		1 + 1	NA
Barley, winter	United Kingdom	750 SL	Foliar	GS 30–Up to and including GS 32	1.50 [c]	200	1	NA
Barley, spring	United Kingdom	750 SL	Foliar	GS 23–Up to and including GS 32	1.125	200	1	NA
Wheat	Argentina	750 SL	Foliar	BBCH 21–31	2.025	NA	1	NA
Wheat	Australia	580 SL	Foliar	Z 25–Z31	0.758	100	1	NA
Wheat, winter	Canada	620 SL	Foliar	GS 31–39	1.116	100	1	NA
				GS 12–30 + GS 31–39	0.620 + 0.496		1 + 1	NA
Wheat, spring & durum	Canada	620 SL	Foliar	GS 31–32 [b]	1.116	100	1	NA
				GS 12–30 + GS 31–32 [b]	0.496 + 0.620		1 + 1	NA
Wheat, winter	United Kingdom	750 SL	Folia	GS 21–Up to and including GS 32	1.50 [c]	150	1	NA
Wheat, spring	United Kingdom	750 SL	Foliar	GS 29–Up to and including GS 32	0.9375	200	1	NA

Notes:

[a] If applications are not possible at Zadoks GS 30 to 32 due to environmental or field conditions, apply up to GS 39.

[b] If applications are not possible at Zadoks GS 31 to 32 due to environmental or field conditions, do not apply later than GS 39.

[c] A maximum dose of 1.33 L/ha (0.9975 kg ai/ha) must not be exceeded when applied before stem elongation (GS 30).

Growth stages for comparison

Zadoks GS	Stage	BBCH	Stage
12	2-leaf	21	beginning of tillering: 1st tiller detectable
14	4-leaf	25	mid-tillering
21	main stem and one tiller	30	beginning of stem elongation
23	main stem and three tillers	31	1st node at least 1 cm above tillering node
29	Main stem and nine or more tillers	32	Node 2 at least 2 cm above node 1
30	beginning of stem elongation	33	Node 3 at least 2 cm above node 2
31	1-node	37	Flag leaf just visible, still rolled
32	2-node	39	Flag leaf stage: flag leaf fully unrolled, ligule just visible
39	flag leaf collar visible		

RESULTS OF SUPERVISED TRIALS ON CROPS

The current Meeting received information on supervised trials using chlormequat chloride on wheat and barley. The results of these supervised trials are summarized in the following table:

Group	Subgroup	Commodity	Table No.
Cereal grains	Wheat, similar grains and pseudocereals without husks	Wheat	Table 3
			Table 4
	Barley, similar grains and pseudocereals with husks	Barley	Table 5
			Table 6
Cereal grains (including pseudocereals) and grass feed products	Cereal grains feed products with high water (≥20%) content (forage and silage) and cereal grains feed products with low water content (<20%) content (hay, straw)	Barley, forage Barley, hay and/or straw	Table 7
		Barley forage	Table 8
		Barley straw	Table 9
		Wheat, forage Wheat, hay and/or straw	Table 10
		Wheat forage	Table 11
		Wheat straw	Table 12

In addition to the descriptions and details of the field trials, each study report includes a summary of the analytical methods, together with the corresponding procedural recoveries, LOQ, LOD, and information on storage of samples. Duration of freezer storage between sampling and analysis were reported for all trials and were covered by the conditions of the freezer storage stability studies.

All appropriate trials are summarized and used. In the trials, where multiple analyses were conducted on a single sample, the mean value is reported. Where multiple samples were taken from a single plot, the individual and mean values are reported. Where results from separate plots with distinguishing characteristics such as different varieties or treatment schedules were reports, results are listed for each plot.

When residues were not quantifiable, they are shown as below the LOQ of the relevant analytical method (e.g., < 0.01 mg/kg). Residues and application rate have generally been rounded to two or three significant figures.

Although control plots were included in the trials, control data are not reported in the following tables unless residues in control samples exceeded the LOQ. Results have not been corrected for concurrent method recoveries.

Residue values from the trials conducted according to the critical GAP were used for the estimation of maximum residue levels, STMR and HR (if applicable). Those results included in the tables are underlined.

Cereal Grains

Wheat, similar grains and pseudocereals without husks–Trials in Canada in 2015

A total of six field trials were conducted on locally grown commercial wheat varieties (one winter wheat and five spring wheat varieties) in Canada during 2015 growing season (AA150722; Norris, F.A., 2016). The wheat was grown under normal agronomic practices for the region and grew and developed normally.

Each trial consisted of three plots: a control plot, and two treated plots. The treated plots for the winter wheat site received either a single foliar application of chlormequat chloride (620 SL) at a growth stage of Zadoks GS 12–30 (target rate of 0.62 kg ai/ha, for forage and grain sample collection) or at a growth stage of Zadoks GS 31–39 target rate of 1.12 kg ai/ha, for hay, grain and straw sample collection. The treated plots for spring wheat sites received single foliar application of chlormequat chloride (620 SL) at a rate of either at a growth stage of Zadoks GS 12–30 (target rate of 0.50 kg ai/ha, for forage-sample collection) or at a growth stage of Zadoks GS 31–32 (target rate of 1.12 kg ai/ha, for hay, grain and straw sample collection).

In the trials, except in two decline trials, one untreated sample and two treated samples were taken at the following growth stages: forage at 15–20 cm stage prior to stem elongation (jointing); hay at early flower (boot) to soft dough stage; and grain and straw at commercial maturity (grains = kernel with hull removed). In the two decline trials, forage, hay, grain and straw samples were collected 7 days before commercial harvest stage, at commercial harvest, and at 7 and 14 days after the commercial harvest stage.

Samples were frozen immediately after collection and shipped frozen at -33 to -10 °C to the analytical laboratory. Upon receipt, the samples were transported at <-5 °C to the laboratory and maintained frozen at <-18 ºC until extraction for a maximum of ca. 4 months (136 days). Extracts were analysed within 7 days. Separate storage stability studies evaluated by the 2017 JMPR demonstrated that chlormequat chloride is stable for at least 24 months in wheat grain and straw when stored frozen.

Residues of chlormequat chloride were determined by LC-MS/MS (method M01-011) with an LOQ of 0.01 mg/kg. Concurrent recoveries (fortification levels: 0.01–2.6 mg/kg) were within the acceptable range with relative standard deviation <20 percent.

Trials in the United States in 2016

A total of 17 supervised trials were conducted on locally grown commercial wheat varieties (winter wheat in 8 sites and spring wheat in nine sites) in the United States during 2016 (AA 160702, Hoi, S.W., 2017a). The wheat was grown under normal agronomic practices for the regions and grew and developed normally.

Each trial consisted of three plots: a control plot, and two treated plots. The treated plot for winter wheat received a single foliar application of chlormequat (620 SL) either at a growth stage of Zadoks GS 20 (target rate of 0.25 kg ai/ha, for forage sample collection) or at a growth stage of Zadoks GS 39 (target rate of 0.45 kg ai/ha, for hay, grain and straw sample collection). The treated plot for spring wheat received a single foliar application of chlormequat chloride (620 SL) either at a growth stage of Zadoks GS 20 (target rate of 0.20 kg ai/ha, for forage sample collection) or at a growth stage of Zadoks GS 32 (target rate of 0.45 kg ai/ha, for hay grain and straw sample collection). In two trials, an additional treated plot received a chlormequat chloride application at a nominal rate of 2.20 kg ai/ha at GS 39 for obtaining raw agricultural commodity (RAC) for a processing study (See the section on processing). These trials, other than those for obtaining the RAC for processing study, were conducted according to the GAP in Canada, which allows one application of chlormequat chloride (620 SL) at a rate of 1.12 kg ai/ha up to and not later than GS 39.

In the trials, except in two decline trials, one untreated sample and two treated samples of wheat grain and straw were collected at the following growth stages (based on > 40 percent of plants within the population): forage, cut between tillering and stem elongation (GS 25–30); hay, early flower (boot) to soft dough stage (GS 45–85) and grain/straw, commercial maturity (GS 92)(grain=kernel with hull intact). In the two decline trials, forage, hay, grain and straw samples were collected 10 days and 5 days before commercial harvest stage, at commercial harvest, and 5 days and 10 days after the commercial harvest stage.

Samples were frozen immediately after collection and shipped at approximately -29 to-6 ºC to the analytical laboratory. Upon receipt, they were maintained frozen at <-18 °C until extraction for a maximum of approximately 10 months (310 days). Extracts were analysed within 5 days. Separate storage stability studies evaluated by the 2017 JMPR demonstrated that chlormequat chloride is stable for at least 24 months in wheat grain and straw when stored frozen.

Residues of chlormequat chloride were determined by LC-MS/MS (method M01-011) with an LOQ of 0.01 mg/kg. Concurrent recoveries (fortification levels: 0.01–5.2 mg/kg) were 89–101 percent, with relative standard deviation < 20 percent.

In the following tables, either chlormequat chloride or chlormequat cation was reported in the "Chloride (Individual)" or "Cation (individual)". As the residue definition is chlormequat cation for both compliance and dietary exposure assessment, the mean concentrations of chlormequat cation were reported in the "Cation (Mean)" column.

Table 3 Residues of chlormequat in wheat grain from supervised trials conducted with single foliar application of chlormequat chloride (620 SL formulation) in Canada in 2015 and in the United States in 2016

Location (Variety) [a/]	Application					DAT Days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/ hL	Water, min, L/ha	No	Timing		Chloride (individual)	Cation (Mean) [b/]	
GAP in Argentina for Wheat	2.025	-	NA	1	BBCH 21–31	NA			
GAP in Canada for Wheat, winter	1.116	-	100	1	GS 31-39	NA			
GAP in Canada for Wheat, spring & durum	1.116	-	100	1	GS 31-32 [a]	NA			
Trials conducted in Canada in 2015 (samples: grain, GS 92 except in decline studies)									
Thorndale,	0.614	0.372	165	1	GS 12-30	79	0.64, 0.67	0.51	AA150722

Location (Variety) [a/]	Application					DAT Days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/ hL	Water, min, L/ha	No	Timing		Chloride (individual)	Cation (Mean) [b/]	
Middlesex, ON (Pioneer 25R39/Winter)[d]	1.140	0.677	169	1	GS 31-39	64	0.91, 1.1	0.78	Trial ON1
Thorndale, Middlesex, ON (Sable/Spring)[d]	0.502	0.299	168	1	GS 12-30	66	1.3, 1.3	1.0	AA150722
	1.090	0.655	166	1	GS 31-32	66 74 [c] 81 87	1.3, 1.3 1.4, 1.6 1.4, 1.5 1.4, 1.3	1.0 1.2 1.1 1.0	Trial ON2
Outlook, RM of Rudy, SK (Utmost/Spring)	0.511	0.332	154	1	GS 12-30	94	0.12, 0.14	0.10	AA150722
	1.050	0.742	142	1	GS 31-32	85	0.37, 0.43	0.31	Trial SK1
Hanley, RM of Rosedale, SK (Cardale/Spring)	0.500	0.444	113	1	GS 12-30	99	0.10, 0.12	0.08	AA150722
	1.090	0.994	110	1	GS 31-32	89	0.62, 0.67	0.50	Trial SK2
Hague, RM of Rosthern, SK (AC Vespar/ Spring)	0.476	0.331	144	1	GS 12-30	84	0.33, 0.32	0.25	AA150722
	1.130	0.743	152	1	GS 31-32	72	1.6, 2.0	1.4	Trial SK3
Glenboro, RM of South Cypress, MB (Cardale/Spring)	0.520	0.330	158	1	GS 12-30	68	0.55, 0.61	0.45	AA150722
	1.110	0.746	149	1	GS 31-32	49 57 [c] 64 71	1.5, 1.2 1.3, 1.3 1.4, 1.2 1.2, 1.3	1.0 1.0 1.0 0.97	Trial MB
Trials conducted in the United States in 2016 (samples: grain, GS 92 except in decline studies)									
Seven Springs, Wayne, NC (26R41/Winter)	1.127	0.791	142	1	GS 39	63	0.455, 0.403	0.43	AA160702 NC
Fisk, Butler, MO (Branson/Winter)	1.102	0.596	185	1	GS 39	45	1.19, 1.33	1.26	AA160702 MO-1
Tronoto, Deuel, SD (ForeFront/Spring)	1.092	0.892	122	1	GS 32	67	0.950, 1.06	1.01	AA160702 SD
Highland, Madison, IL (25R78/Winter)	1.114	0.771	145	1	GS 39	52	0.263, 0.263	0.26	AA160702 IL-1
Uvalde, Hill, TX (Expresso/Spring)	1.132	0.939	120	1	GS 32	64	0.888, 0.839	0.86	AA160702 TX-1
Cleveland, Stutsman, ND (Prosper/Spring)	1.149	0.807	142	1	GS 32	40	1.86, 2.01	1.94	AA160702 ND-1
Carrington, Foster, ND (Faller/Spring)	1.102	0.797	138	1	GS 32	65	0.650, 0.648	0.65	AA160702 ND-2
York, York, NE (Overland HRW/ Winter)	1.119	0.601	186	1	GS 39	55	0.495, .541	0.52	AA160702 NE-1
Hinton, Caddo, OK (Duster/Winter)	1.102	0.713	155	1	GS 39	56	0.553. 0.523	0.54	AA160702 OK-1
Littlefield, Lamb, TX (TAN 111/Winter)	1.102	0.793	139	1	GS 39	56	0.555, 0.518	0.54	AA160702 TX-2
Levelland, Hockley, TX (Express/Spring)	1.105	0.795	139	1	GS 32	58	0.504, 0.504	0.50	AA160702 TX-3

Location (Variety) [a/]	Application					DAT Days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/ hL	Water, min, L/ha	No	Timing		Chloride (individual)	Cation (Mean) [b/]	
Wall, Tom Green, TX (Expresso/Spring)	1.119	0.799	140	1	GS 32	72	0.276, 0.307	0.29	AA160702 TX-4
Weatherford, Custer, OK	1.132	0.741	153	1	GS 39	62	1.58, 1.50	1.54	AA160702 OK-2
(Endurance/Winter)	2.226	1.485	150	1	GS 39	62	2.85, 2.86	2.86	
Richland, Keokuk, IA	1.142	0.674	169	1	GS 39	54	0.863, 0.863	0.85	AA160702 IA-2
(GV662/Winter)	2.273	1.347	169	1	GS 39	54	1.54, 1.40	1.47	
American Falls, Power, ID (Klassic/Spring)	1.102	0.664	166	1	GS 32	61	1.89, 1.81	1.85	AA160702 ID
Kirksville, Adair, MO (Certified Rollag Spring Wheat)	1.119	0.669	167	1	GS 32	41 46 52 [c/] 55 62	2.67, 2.84 2.72, 3.10 2.89, 2.97 2.56, 2.54 2.28, 2.31	2.75 2.91 2.93 2.55 2.30	AA160702 MO-2
Montpelier, Stutsman, ND (Prosper/Spring)	1.124	0.796	141	1	GS 32	37 42 47 [c/] 52 57	1.51, 1.63 1.41, 1.35 1.49, 1.38 1.28, 1.34 1.68, 1.07	1.57 1.38 1.44 1.31 1.38	AA160702 ND-3

Notes:

[a]/ The term "Winter" means winter wheat; "Spring" means spring wheat.

[b]/ Calculated by adjusting for the molecular weights (MW of chlormequat chloride, 158.07; and MW of chlormequat cation, 122.62).

[c]/ Commercial harvest timing (GS 92).

[d]/ Same location with the application dates nine days apart.

RM=Rural Municipality.

Table 4 Residues of chlormequat in wheat grain from supervised trials conducted with single foliar application in European countries and evaluated by the 2017 JMPR (extracted from the 2017 JMPR Evaluation)

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation & Scaled value	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Brunne, Germany (winter wheat, Thasos)	460 SL	37	1.52	150	94	0.33	0.26 0.35	2005/1014176, ACK/03/04
	750 SL	37	1.50	150	94	0.45	0.35 0.47	
Seebach, northern France (winter wheat, Cap Horn)	460 SL	34	1.52	150	68	0.74	0.57 0.76	2005/1014176, FAN/03/04
	750 SL	34	1.50	150	68	0.73	0.57 0.77	
Aussonne, southern France	460 SL	35	1.52	150	80	0.44	0.34 0.45	2005/1014176, FTL/03/04

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation & Scaled value	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
(winter wheat, Autan)								
	750 SL	34	1.50	150	80	0.62	0.48 0.65	
Withington, United Kingdom (spring wheat, Paragon)	460 SL	37	1.52	150	78	0.80	0.62 0.83	2005/1014176, OAT/01/04
	750 SL		1.50	150	78	0.76	0.59 0.80	
D-75233, Niefern-Öschelbronn, Germany (winter wheat, Tores)	750 SL	37	1.67	195	84	0.62	0.48 0.58	2010/1014090, 01
D-71277, Perouse-Rutesheim, Germany (winter wheat, Tommi)	750 SL	37	1.40	163	98	0.30	0.23 0.33	2010/1014090, 02
F-45300, Rouvres-Saint-Jean, France (winter wheat, Campero)	750 SL	37	1.57	204	84	0.96	0.74 0.95	2010/1014090, 03
F-45300, Bouilly-en-Gâtinais, France (winter wheat, Apache)	750 SL	37	1.58	206	71	0.47	0.36 0.46	2010/1014090, 04
North Cave, East Yorkshire, United Kingdom (winter wheat, Oakley)*	750 SL	37	1.56	203	75	1.3 c0.94	1.0 c0.73	2010/1041090, 05
74193 Stetten a. H. Rieslingstrasse 18, Baden-Württemburg, Germany, 2003 (winter wheat, Transit)	350 SL	37	0.70	100	57	0.26	0.20 0.58	2004/1015956, 01
	750 SL	37	1.50	100	57	0.20	0.16 0.22	
82170 Pompignan 30 route de Toulouse, Midi-Pyrenées, France, 2003 (winter wheat, Sagem)#	350 SL	39	0.70	100	50	4.6	3.6 10.4	2004/1015956, 05

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation & Scaled value	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
	750 SL		1.50	100	51	7.9	6.1 8.2	
D-47652 Weeze, Nordrhein-Westfalen, Germany, 2007 (spring wheat, Taifun)	750 SL	32	1.54	200	79	1.3	1.0 1.3	2008/1014941, 01
NL-6595, MS Ottersum, Limburg, Netherlands, 2007 (winter wheat, Limos)	750 SL	32	1.62	210	75	0.88	0.68 0.85	2008/1014941, 02
F-12290, Aveyron, France, 2007 (spring wheat, Florence Aurore)	750 SL	37	1.00	195	98	0.21	0.16 0.32	2008/1014941, 03
F-82100 Tarn et Garonne, France, 2007 (winter wheat, Apache)	750 SL	33	1.04	202	85	0.39	0.30 0.58	2008/1014941, 04
I-40068 Emilia Romagna, Italy, 2007 (spring wheat, Lippo)	750 SL	32	1.05	204	98	0.44	0.34 0.66	2008/1014941, 05
I-40054 Emilia Romagna, Italy, 2007 (winter wheat, Duilio)	750 SL	32	1.07	208	96	0.06	0.05 0.09	2008/1014941, 06
Via Calabria Nuovo No. 3, Quarto Inferiore, 40057 Bologna, Italy, 2007 (spring wheat, Croine)	750 SL	32	1.55	201	87	0.10	0.078 0.10	2008/1014940, 01
Castel S. Pietro, 40024 Bologna, Italy, 2007 (durum wheat, San Carlo)	750 SL	32	1.56	202	99	0.07	0.05 0.06	2008/1014940, 02
82000 Montauban, France, 2007 (winter wheat, Quality)	750 SL	32	1.48	192	65	0.07	0.05 0.07	2008/1014940, 03
82700 Finhan, France, 2007 (durum wheat, Joyaux)	750 SL	37	1.57	204	72	0.61	0.47 0.61	2008/1014940, 04
Granarolo	750 SL	33	1.56	202	62	< 0.05	< 0.04	2009/1021674,

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation & Scaled value	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
dell'Emilia, 40057, Emilia Romagna, Italy, 2008 (spring wheat, Blasco)								01
V. Matteotti 13, Molinella, Bologna 40062, Italy, 2008 (durum wheat, Duilio)	750 SL	32	1.52	198	96	< 0.05	< 0.04	2009/1021674, 02
Barry d'Islemade, 82000 Tarn et Garonne, France, 2008 (winter wheat, Quality)	750 SL	32	1.57	204	95	0.14	0.11 0.14	2009/1021674, 03
Finhan, 82700 Tarn et Garonne, France, 2008 (durum wheat, Dakter)	750 SL	32	1.55	201	106	0.73	0.57 0.74	2009/1021674, 04
Herbert Neumann Dorfstr. 2, 16833 Brunne, Germany, 2004 (winter wheat, Thasos)	460 SL	37	1.52	150	94	0.33	0.26 0.35	2005/1014176, 01
	750 SL	37	1.50	150	94	0.45	0.35 0.47	
30 route de Hunspach, 67160 Seebach, France, 2004 (winter wheat, Cap Horn)	460 SL	34	1.52	150	68	0.74 c0.15	0.57 c0.12 0.76	2005/1014176, 02
	750 SL	34	1.50	150	68	0.73 c0.15	0.57 c0.12 0.77	
Ourmieres 3529, route de Merville 31840 Aussonne, France, 2004 (winter wheat Autan)	460 SL	35	1.52	150	80	0.44	0.34 0.45	2005/1014176, 03
	750 SL	35	1.50	150	80	0.62	0.48 0.65	
Upcote Farm, Withington, GL54 4BL, United Kingdom,	460 SL	37	1.52	150	78	0.80	0.62 0.83	2005/1014176, 04

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation & Scaled value	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
2004 (spring wheat, Paragon)								
	750 SL	37	1.50	150	78	0.76	0.59 0.80	

Notes:

Except where indicated, no residues above the LOQ were found in any of the untreated control samples.

*Trial accidentally oversprayed with an additional application of 1.6 kg ai/ha chlormequat chloride 18 days prior to the trial application.

#Trial flagged by applicant as having abnormally high residues due to extremely low rainfall during the trial, contributing to lowered yields, and use of a durum wheat variety. Noting that this result differs significantly from the rest of the data, it is considered not representative of the residues expected after treatment in accordance with GAP, and has not been included the consideration for MRL estimation.

Barley, similar grains and pseudocereals with husks

A total of 20 supervised trials were conducted on locally grown commercial varieties of barley in Canada (10) and the United States (10) during 2016 (AA160703, Hoi S.W., 2017b). In all of the sites, spring barley were grown under normal agronomic practices for the regions and developed normally. Each trial consisted of two plots: a control plot and a treated plot. Each treated plot received a single foliar application of chlormequat chloride (620 SL) at a growth stage of Zadoks GS 32 at either lower target rate (target rate of 0.58 kg ai/ha in the Canadian trials or 0.71 kg ai/ha in the United States trials) or higher rate (target rate of 1.43 kg ai/ha in the United States trials or 1.15 kg ai/ha in Canadian trials).

In these trials, except the decline studies, the barley matrices were sampled at the following growth stages (based on >50 percent of plants within the population): hay, early flower (boot) to soft dough stage (GS 45–85); and grain/straw, commercial maturity (GS 92)(grain=kernel with hull intact). In the residue decline trials, hay, grain and straw samples were taken at 10 days and 5 days before the normal commercial harvest stage, at commercial harvest, and at 5 and 10 days after the normal commercial harvest stage.

Samples were frozen immediately after sampling and shipped frozen to the analytical laboratory. Samples were maintained frozen at <-18 ºC until extraction for a maximum of 316 days (*ca.* 11 months). Extracts were analysed within 6 days. Separate storage stability studies concluded that chlormequat chloride is stable for at least 24 months under frozen storage conditions in cereal grain and straw.

Residues of chlormequat chloride were determined by LC-MS/MS, using method M01-011. The method was validated with an LOQ of 0.01 mg/kg. Concurrent procedural recoveries were within the acceptable range of 70–110 percent, with relative standard deviation <20 percent.

Table 5 Residues of chlormequat in barley grain (spring barley) from supervised trials conducted with single foliar application of chlormequat chloride (620 SL formulation) in Canada and the United States in 2016

Location (Variety) [a/]	Application					DAT days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/ hL	Water, L/ha	No	Timing		Cation (Individual)	Cation (Mean)	
GAP in the United Kingdom for Winter barley	1.50	-	Min. 200	1	Up to and including GS 32	NA			
GAP in Canada for Barley, spring & winter	1.426	-	Min.100	1	GS 30–32	NA			
Trials conducted in Canada in 2016 (samples: grain, GS 92 except in decline studies)									
Branchton, ON, 2016 (Spring Barley, Dignity)	0.689	0.04	1805	1	GS 32	64	0.850, 0.906	0.88	AA160703-CAN-1
Delisle, SK, 2016 (Spring Barley, Austenson)	0.682	0.05	1380	1	GS 32	74	0.230, 0.229	0.23	AA160703-CAN-2
Taber, AB, 2016 (Spring Barley, CDC Austenson)	0.694	0.05	1368	1	GS 32	65	0.569, 0.656	0.61	AA160703-CAN-3
Saskatoon, SK, 2016 (Spring Barley, Austenson)	0.680	0.05	1369	1	GS 32	64	0.539, 0.493	0.52	AA160703-CAN-4
Coalhurst, AB 2016 (Spring Barley, CDC Austenson)	0.702	0.05	1382	1	GS 32	76	0.058 0.072	0.06	AA160703-CAN-5
Alvena, SK, 2016 (Spring Barley, CDC Austenson)	0.699	0.06	1102	1	GS 32	65	0.224, 0.260	0.24	AA160703-CAN-6
Lamont, AB, 2016 (Spring Barley, Austenson)	0.707	0.05	1403	1	GS 32	77	0.451, 0.486	0.47	AA160703-CAN-7
Carberry, MB, 2016 (Spring Barley, Conlon)	0.709	0.07	1034	1	GS 32	67	0.806, 0.790	0.80	AA160703-CAN-8
Josephburg, AB, 2016 (Spring Barley, Conlon)	0.709	0.05	1407	1	GS 32	51 56 60 [a/] 66 71	0.811, 0.582 0.94, 1.01 0.97, 1.14 1.13, 0.94 0.873, 0.848	0.70 0.98 1.06 1.04 0.860	AA160703-CAN-9
Elgin, MB, 2016 (Spring Barley, Newdale)	0.541	0.04	1511	1	GS 32	65	1.01, 1.05	1.03	AA160703-CAN-10
	1.408	0.10	1478	1	GS 32	65	1.35, 1.58	1.46	
Trials conducted the United States in 2016 (samples: grain, GS 92 except in decline studies)									
North Rose, NY, 2016 (Spring Barley, Ac Minoa)	1.465	0.85	173	1	GS 32	41	1.92, 1.23	1.57	AA160703-NY
Fitchburg, WI, 2016 (Spring Barley, Hazen)	1.438	0.86	167	1	GS 32	49	2.55, 2.62	2.59	AA160703-WI

Location (Variety) [a/]	Application					DAT days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/ hL	Water, L/ha	No	Timing		Cation (Individual)	Cation (Mean)	
Richland, IA, 2016 (Spring Barley, Tradition)	1.421	0.90	159	1	GS 32	44 49 55 [a/] 59 64	4.00, 3.67 3.44, 3.59 3.84, 3.67 3.96, 3.78 3.56, 3.72	3.84 3.52 3.76 3.87 3.64	AA160703-IA
Grand Island, NE, 2016 (Spring Barley, Certified Tradition)	1.428	0.77	187	1	GS 32	55	1.03, 1.03	1.03	AA160703-NE
Fargo, ND, 2016 (Spring Barley, Tradition)	1.502	1.02	147	1	GS 32	52	0.527, 0.551	0.54	AA160703-ND-1
Carrington, ND, 2016 (Spring Barley, Rasmussen)	1.433	1.01	142	1	GS 32	62	1.09, 1.02	1.04	AA160703-ND-2
Fargo, ND, 2016 (Spring Barley, Tradition)	1.48	1.02	145	1	GS 32	51	0.477, 0.482	0.48	AA160703-ND-3
	2.92	2.03	143	1	GS 32	51	1.90, 1.87	1.88	
Smithfield, ID, 2016 (Spring Barley, Golden Eye)	1.359	0.77	176	1	GS 32	47	3.52, 3.08	3.30	AA160703-ID
Porterville, CA, 2016 (Spring Barley, UC937)	1.428	0.83	172	1	GS 32	78	0.118, 0.132	0.12	AA160703-CA
Hermiston, OR, 2016 (Spring Barley, Haybet)	1.436	0.83	173	1	GS 32	49	1.30, 1.72	1.51	AA160703-OR

Notes:

[a]/ Commercial harvest timing (GS 92).

Table 6 Residues of chlormequat in barley grain from supervised trials conducted with single foliar application of chlormequat chloride in European countries and evaluated by the 2017 JMPR (extracted from the 2017 JMPR Evaluation)

Location, Year (variety)	Application					Residues, mg/kg as chlormequat chloride	Residues, mg/kg as chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
F-91150 Erzeville, Roinvillers, France, 2009 (spring barley, Sebastian)	750 SL	37	1.7	219	76	0.84	0.65	2010/1014090, 06
50180 Utebo, Zaragoza, Spain, 2010 (barley, Graphic)	750 SL	32	1.4	182	69	0.40	0.31	2011/1071895, 01
66750 Saint-	750 SL	32	1.6	207	59	0.40	0.31	2011/1071895,

Location, Year (variety)	Application					Residues, mg/kg as chlormequat chloride	Residues, mg/kg as chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Cyprien, Pyrénées-Orientales, France, 2010 (barley, Prestige)								02
50490 Villareal de Huerva, Spain, 2010 (barley, Montage)	750 SL	32	1.5	200	70	0.76	0.59	2011/1071895, 03
01560 St-Jean-sur-Reyssouze, Ain, France, 2010 (barley, Vanessa)	750 SL	32	1.4	187	84	0.08	0.062	2011/1071895, 04
21737 Wischhafen, Niedersachsen, Germany, 2011 (winter barley, Pelikan)	750 SL	37	1.5	202	76	0.16	0.12	2012/1016109, 01
21726 Oldendorf, Niedersachsen, Germany, 2011 (winter barley, Naomie)	750 SL	37	1.6	211	76	0.22	0.17	2012/1016109, 02
45300 Thignonville, Loiret, France, 2011 (spring barley, Sebastian)	750 SL	37	1.5	202	67	0.47	0.36	2012/1016109, 03
91150 Mespuits, Essonne, France, 2011 (spring barley, Sebastian)	750 SL	37	1.4	190	68	0.41	0.32	2012/1016109, 04
82130 Lafrancaise, Midi P., France, 2011 (winter barley, Azurel)	750 SL	32	1.6	220	73	< 0.05	< 0.04	2012/1016109, 05
82700 Bourret, Tarn et Garonne, France, 2011 (winter barley, Azurel)	750 SL	32	1.4	181	70	0.78	0.60	2012/1016109, 06
44492 Fonfria, Teruel, Spain,	750 SL	32	1.5	200	75	1.4	1.1	2012/1016109, 07

Location, Year (variety)	Application					Residues, mg/kg as chlormequat chloride	Residues, mg/kg as chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
2011 (barley, Estrelia)								
22809 Loarre, Aragon, Spain, 2011 (barley, Meseta)	750 SL	32	1.5	200	72	1.2	0.93	2012/1016109, 08
67229 Gerolsheim Römerstrasse 8, Rheinland-Pfalz, Germany, 2003 (spring barley, Scarlett)	350 SL	37	0.70	100	55	1.0	0.78	2004/1015956, 02
	750 SL	37	1.5	100	55	0.99	0.77	
Homelands Farm, Bucknell, Bicester, OX6 9NB, United Kingdom, 2003 (winter barley, Leonie)	350 SL	37	0.70	100	75	0.92	0.71	2004/1015956, 03
	750 SL	37	1.5	100	75	0.64	0.50	
67160 Seeback route de Hunspach, Alsace, France, 2003 (winter barley, Majestic)	350 SL	37	0.70	100	58	0.46	0.36	2004/1015956, 04
	750 SL	37	1.5	100	58	0.49	0.38	

Notes:

No residues above the LOQ were found in any of the untreated control samples.

Animal Feeds

Cereal grains feed products with high water (≥20%) content (forage and silage) and

cereal grains feed products with low water content (<20%) content (hay, straw)

Barley forage; and Barley, hay and/or straw

In a total of 20 supervised trials conducted on barley in Canada (10) and the United States (10) during 2016 (AA160703, Hoi S.W., 2017b), hay samples were taken at earlier growth stages (GS 45–85) and straw samples were taken at the same time as grains (GS 92).

In the following tables, individual analytical results of duplicate samples were reported as “Cation (individual)” followed by the mean analytical results in the “Cation (Mean)” column.

Table 7 Residues of chlormequat in barley hay (H) or straw (S) from supervised trials conducted with foliar application of chlormequat chloride (620 SL formulation) in Canada and the United States in 2016 (residue concentrations expressed on an as-received basis)

Location (Variety) [a/]	Application					DAT days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/ hL	Water, L/ha	No	Timing		Cation (Individual)	Cation (Mean)	
GAP in the United Kingdom for Winter barley	1.50	-	Min. 200	1	Up to and including GS 32	NA			
GAP in Canada for Barley, spring & winter	1.426	-	Min. 100	1	GS 30–32	NA			
Trials conducted in Canada in 2016 (samples: hay, GS 45–85; straw, GS 92)									
Branchton, ON, 2016 (Spring Barley, Dignity)	0.689	0.04	1805	1	GS 32	31 H 64 S	18.3, 6.76 7.08, 5.71	12.5 6.40	AA160703-CAN-1
Delisle, SK [b], 2016 (Spring Barley, Austenson)	0.682	0.05	1380	1	GS 32	28 H 74 S	1.68, 1.75 0.938, 0.840	1.71 0.89	AA160703-CAN-2
Taber, AB, 2016 (Spring Barley, CDC Austenson)	0.694	0.05	1368	1	GS 32	44 H 65 S	4.38, 4.32 1.30, 1.21	4.35 1.25	AA160703-CAN-3
Saskatoon, SK, 2016 (Spring Barley, Austenson)	0.680	0.05	1369	1	GS 32	31 H 64 S	1.00, 1.31 2.93, 2.92	1.16 2.92	AA160703-CAN-4
Coalhurst, AB 2016 (Spring Barley, CDC Austenson)	0.702	0.05	1382	1	GS 32	47 H 76 S	1.72, 1.11 0.568, 0.833	1.41 0.70	AA160703-CAN-5
Alvena, SK [c], 2016 (Spring Barley, CDC Austenson)	0.699	0.06	1102	1	GS 32	35 H 65 S	14.3, 9.75 14.5, 17.9	12.0 16.2	AA160703-CAN-6
Lamont, AB, 2016 (Spring Barley, Austenson)	0.707	0.05	1403	1	GS 32	27 H 77 S	4.35, 3.81 0.624, 0.583	4.08 0.60	AA160703-CAN-7
Carberry, MB, 2016 (Spring Barley, Conlon)	0.709	0.07	1034	1	GS 32	35 H 67 S	14.9, 11.6 8.26, 8.03	13.2 8.15	AA160703-CAN-8
Josephburg, AB, 2016 (Spring Barley, Conlon)	0.709	0.05	1407	1	GS 32	24 H 25 H 32 H 38 H 42 H 51 S 56 S 60 S[a/] 66 S 71 S	25.9, 18.2 22.7, 18.4 12.8, 18.5 9.30, 7.12 7.47, 4.72 1.40, 2.56 1.73, 2.37 5.22, 6.30 3.52, 3.89 2.64, 2.38	22.1 20.5 15.6 8.21 6.09 1.98 2.05 5.76 3.70 2.51	AA160703-CAN-9
Elgin, MB, 2016 (Spring Barley, Newdale)	0.541	0.04	1511	1	GS 32	31 H 65 S	7.07, 5.56 1.68, 170	6.31 1.69	AA160703-CAN-10
Trials conducted the United States in 2016 (samples: hay, GS 45–85; straw, GS 92)									
North Rose, NY, 2016 (Spring Barley, Ac Minoa)	1.465	0.85	173	1	GS 32	15 H 41 S	32.0, 60.8 (c 0.02) 14.2, 12.7 (c 0.01)	46.4 13.5	AA160703-NY

Location (Variety) [a/]	Application					DAT days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/ hL	Water, L/ha	No	Timing		Cation (Individual)	Cation (Mean)	
Fitchburg, WI, 2016 (Spring Barley, Hazen)	1.438	0.86	167	1	GS 32	29 H 49 S	47.4, 47.6 17.6, 15.8	47.5 16.7	AA160703-WI
Richland, IA, 2016 (Spring Barley, Tradition)	1.421	0.90	159	1	GS 32	20 H 23 H 29 H 34 H 38 H 44 S 49 S 55 S[a/] 59 S 64 S	34.7, 31.6 16.5, 19.6 15.2, 16.5 16.4, 21.8 14.6, 20.2 26.3, 23.5 18.2, 16.9 16.1, 14.9 20.0, 15.7 17.1, 13.8	33.1 18.1 15.8 19.1 17.4 24.9 17.6 15.5 17.9 15.4	AA160703-IA
Grand Island, NE, 2016 (Spring Barley, Certified Tradition)	1.428	0.77	187	1	GS 32	32 H 55 S	17.9, 17.4 2.29, 2.57	17.7 2.43	AA160703-NE
Fargo, ND [d], 2016 (Spring Barley, Tradition)	1.502	1.02	147	1	GS 32	30 H 52 S	13.8, 24.2 3.25, 4.26	19.0 3.76	AA160703-ND-1
Carrington, ND, 2016 (Spring Barley, Rasmussen)	1.433	1.01	142	1	GS 32	24 H 62 S	14.6, 12.3 5.86, 7.54	13.5 6.70	AA160703-ND-2
Fargo, ND [d], 2016 (Spring Barley, Tradition)	1.48	1.02	145	1	GS 32	31 H 51 S	34.8, 36.6 11.0, 8.49 (c 0.04)	35.7 9.76	AA160703-ND-3
Smithfield, ID, 2016 (Spring Barley, Golden Eye)	1.359	0.77	176	1	GS 32	26 H 47 S	47.7, 72.7 31.8, 27.1	60.2 29.5	AA160703-ID
Porterville, CA, 2016 (Spring Barley, UC937)	1.428	0.83	172	1	GS 32	34 H 78 S	5.46, 5.28 6.63, 5.24	5.37 5.93	AA160703-CA
Hermiston, OR, 2016 (Spring Barley, Haybet)	1.436	0.83	173	1	GS 32	21 H 49 S	39.2, 38.4 4.35, 4.65	38.8 4.50	AA160703-OR

Notes:

[a]/ Commercial harvest timing (GS 92).

[b]/ Delise is 34 miles from Saskatoon, 71 miles from Alvena.

[C]/ Avena is 43 miles from Saskatoon.

[d]/ Two sites in Fargo, ND were 25 miles apart (based on the longitude and latitude information).

Table 8 Residues of chlormequat in barley forage from supervised trials conducted with a single foliar application in European countries and evaluated by the 2017 JMPR (results reported on a fresh weight basis)(extracted from the 2017 JMPR Evaluation; matrices not related to forage were removed from the original table)

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/ kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
F-91150 Erzeville,	750 SL	37	1.7	219	0	Whole plant w/o roots	27	21	2010/1014090, 06

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
Roinvillers, France, 2009 (spring barley, Sebastian)					14	Whole plant w/o roots	16	12	
					28	Whole plant w/o roots	12	9.3	
50180 Utebo, Zaragoza, Spain, 2010 (barley, Graphic)	750 SL	32	1.4	182	0	Whole plant w/o roots	28	22	2011/1071895, 01
					14	Whole plant w/o roots	2.5	1.9	
					28	Whole plant w/o roots	0.70	0.54	
66750 Saint-Cyprien, Pyrénées-Orientales, France, 2010 (barley, Prestige)	750 SL	32	1.6	207	0	Whole plant w/o roots	33	26	2011/1071895, 02
					14	Whole plant w/o roots	8.6	6.7	
					28	Whole plant w/o roots	7.3	5.7	
21737 Wischhafen, Niedersachsen, Germany, 2011 (winter barley, Pelikan)	750 SL	37	1.5	202	0	Whole plant w/o roots	19	15	2012/1016109, 01
					14	Whole plant w/o roots	3.8	2.9	
					28	Whole plant w/o roots	2.8	2.2	
21726 Oldendorf, Niedersachsen, Germany, 2011 (winter barley, Naomie)	750 SL	37	1.6	211	0	Whole plant w/o roots	24	19	2012/1016109, 02
45300 Thignonville, Loiret, France, 2011 (spring barley, Sebastian)	750 SL	37	1.5	202	0	Whole plant w/o roots	38	29	2012/1016109, 03
					14	Whole plant w/o roots	4.6	3.6	
					27	Whole plant w/o roots	2.5	1.9	
91150 Mespuits, Essonne, France, 2011 (spring barley, Sebastian)	750 SL	37	1.4	190	0	Whole plant w/o roots	24	19	2012/1016109, 04
82130 Lafrancaise, Midi P., France, 2011 (winter barley, Azurel)	750 SL	32	1.6	220	0	Whole plant w/o roots	41	32	2012/1016109, 05
					15	Whole plant w/o roots	4.3	3.3	
					29	Whole plant w/o roots	2.1	1.6	

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
82700 Bourret, Tarn et Garonne, France, 2011 (winter barley, Azurel)	750 SL	32	1.4	181	0	Whole plant w/o roots	30	23	2012/1016109, 06
44492 Fonfria, Teruel, Spain, 2011 (barley, Estrelia)	750 SL	32	1.5	200	0	Whole plant w/o roots	96	74	2012/1016109, 07
					13	Whole plant w/o roots	4.8	3.7	
					28	Whole plant w/o roots	2.0	1.6	
22809 Loarre, Aragon, Spain, 2011 (barley, Meseta)	750 SL	32	1.5	200	0	Whole plant w/o roots	30	23	2012/1016109, 08
67229 Gerolsheim Römerstrasse 8, Rheinland-Pfalz, Germany, 2003 (spring barley, Scarlett)	350 SL	37	0.70	100	0	Whole plant w/o roots	46.4	36	2004/1015956, 02
	750 SL	37	1.5	100	0	Whole plant w/o roots	32.4	25	
Homelands Farm, Bucknell, Bicester, OX6 9NB, United Kingdom, 2003 (winter barley, Leonie)	350 SL	37	0.70	100	0	Whole plant w/o roots	35.1	27	2004/1015956, 03
	750 SL	37	1.5	100	0	Whole plant w/o roots	41.8	32	
67160 Seebach route de Hunspach, Alsace, France, 2003 (winter barley, Majestic)	350 SL	37	0.70	100	0	Whole plant w/o roots	18.1	14	2004/1015956, 04
	750 SL	37	1.5	100	0	Whole plant w/o roots	27.5	21	

Notes:

No residues above the LOQ were found in any of the untreated control samples.

Table 9 Residues of chlormequat in barley straw from supervised trials conducted with a single application in European countries and evaluated by the 2017 JMPR (results reported on an as-is basis)(Extracted from the 2017 JMPR Evaluation)

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
F-91150 Erzeville, Roinvillers, France, 2009 (spring barley, Sebastian)	750 SL	37	1.7	219	76	34	26	2010/1014090, 06
50180 Utebo, Zaragoza, Spain, 2010 (barley, Graphic)	750 SL	32	1.4	182	69	0.80	0.62	2011/1071895, 01
66750 Saint-Cyprien, Pyrénées-Orientales, France, 2010 (barley, Prestige)	750 SL	32	1.6	207	59	39	30	2011/1071895, 02
50490 Villareal de Huerva, Spain, 2010 (barley, Montage)	750 SL	32	1.5	200	70	1.6	1.2	2011/1071895, 03
01560 St-Jean-sur-Reyssouze, Ain, France, 2010 (barley, Vanessa)	750 SL	32	1.4	187	84	1.6	1.2	2011/1071895, 04
21737 Wischhafen, Niedersachsen, Germany, 2011 (winter barley, Pelikan)	750 SL	37	1.5	202	76	6.7	5.2	2012/1016109, 01
21726 Oldendorf, Niedersachsen, Germany, 2011 (winter barley, Naomie)	750 SL	37	1.6	211	76	7.1	5.5	2012/1016109, 02
45300 Thignonville, Loiret,	750 SL	37	1.5	202	67	3.5	2.7	2012/1016109, 03

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
France, 2011 (spring barley, Sebastian)								
91150 Mespuits, Essonne, France, 2011 (spring barley, Sebastian)	750 SL	37	1.4	190	68	4.1	3.2	2012/1016109, 04
82130 Lafrancaise, Midi P., France, 2011 (winter barley, Azurel)	750 SL	32	1.6	220	73	< 0.5	< 0.39	2012/1016109, 05
82700 Bourret, Tarn et Garonne, France, 2011 (winter barley, Azurel)	750 SL	32	1.4	181	70	3.3	2.6	2012/1016109, 06
44492 Fonfria, Teruel, Spain, 2011 (barley, Estrelia)	750 SL	32	1.5	200	75	2.4	1.9	2012/1016109, 07
22809 Loarre, Aragon, Spain, 2011 (barley, Meseta)	750 SL	32	1.5	200	72	7.6	5.9	2012/1016109, 08
67229 Gerolsheim Römerstrasse 8, Rheinland-Pfalz, Germany, 2003 (spring barley, Scarlett)	350 SL	37	0.70	100	55	8.7	6.7	2004/1015956, 02
	750 SL	37	1.5	100	55	7.3	5.7	
Homelands Farm, Bucknell, Bicester, OX6 9NB,	350 SL	37	0.70	100	75	5.8	4.5	2004/1015956, 03

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
United Kingdom, 2003 (winter barley, Leonie)								
	750 SL	37	1.5	100	75	9.1	7.1	
67160 Seeback route de Hunspach, Alsace, France, 2003 (winter barley, Majestic)	350 SL	37	0.70	100	58	6.6	5.1	2004/1015956, 04
	750 SL	37	1.5	100	58	5.2	4.0	

Notes:

No residues above the LOQ were found in any of the untreated control samples.

Wheat forage; and Wheat, hay and/or straw

Trials in Canada in 2015

In a total of six field trials conducted on wheat (one on winter wheat and the other five on spring wheat) in Canada during 2015 growing season (AA150722; Norris, F.A., 2016), samples of forage and hay were taken at earlier growth stages (forage, GS 25–30; and hay, GS 45–85). Straw samples were taken at the same time as grains (GS 92). Details of sampling and analysis are described earlier.

Trials in the United States in 2016

In a total of 17 supervised trials conducted on wheat in the United States during 2016 (AA 160702, Hoi, S.W., 2017a), samples of forage and hay were taken at earlier growth stages (forage, GS 25–30; and hay, GS 45–85). Straw samples were taken at the same time as grains (GS 92).

In the following tables, either chlormequat chloride or chlormequat cation was reported in the "Chloride (Individual)" or "Cation (individual)". As the residue definition is chlormequat cation for both compliance and dietary exposure assessment, the mean concentrations of chlormequat cation were reported in the "Cation (Mean)" column.

Table 10 Residues of chlormequat in wheat forage (F), hay (H) or straw (S) from supervised trials conducted with foliar application of chlormequat chloride (620 SL formulation) in Canada in 2015 and in the United States in 2016 (residue concentrations expressed on an as-received basis)

Location (Variety) [a/]	Application					DAT days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/hL	Water, min, L/ha	No	Timing		Chloride (individual)	Cation (Mean) [b/]	
GAP in AR for Wheat	2.025	-	NA	1	BBCH 21–31	NA			
GAP in CA for Wheat, winter	1.116	-	100	1	GS 31-39	NA			

Location (Variety) [a/]	Application					DAT days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/ hL	Water, min, L/ha	No	Timing		Chloride (individual)	Cation (Mean) [b/]	
GAP in Canada for Wheat, spring & durum	1.116	-	100	1	GS 31-32 [a]	NA			
Trials conducted in Canada in 2015 (samples: forage, GS 25–30; hay, GS 45–85; straw, GS 92)									
Thorndale, Middlesex, ON (Pioneer 25R39/Winter)	0.614	0.372	165	1	GS 12-30	19 F	0.86, 0.36	0.47	AA150722
	1.140	0.677	169	1	GS 31-39	36 H 64 S	3.9, 38 14, 32	16 18	Trial ON1
Thorndale, Middlesex, ON (Sable/Spring)	0.502	0.299	168	1	GS 12-30	20 F 28 F 33 F 40 F	4.1, 5.1 2.1, 2.3 2.3, 1.8 1.3, 1.3	3.6 1.7 1.6 1.0	AA150722 Trial ON2
	1.090	0.655	166	1	GS 31-32	21 H 28 H 36 H 42 H 66 S 74 S[c/] 81 S 87 S	74, 81 55, 50 44, 36 31, 34 23, 27 30, 32 25, 28 26, 24	60 41 31 25 19 24 20 19	
Outlook, RM of Rudy, SK (Utmost/Spring)	0.511	0.332	154	1	GS 12-30	3 F	15, 13	11	AA150722
	1.050	0.742	142	1	GS 31-32	15 H 85 S	29, 24 7.1, 6.8	21 5.4	Trial SK1
Hanley, RM of Rosedale, SK (Cardale/Spring)	0.500	0.444	113	1	GS 12-30	6 F	7.5, 6.8	5.5	AA150722
	1.090	0.994	110	1	GS 31-32	40 H 89 S	4.9, 5.5 5.4, 4.8	4.0 4.0	Trial SK2
Hague, RM of Rosthern, SK (AC Vespar/ Spring)	0.476	0.331	144	1	GS 12-30	8 F	5.0, 5.4	4.0	AA150722
	1.130	0.743	152	1	GS 31-32	22 H 72 S	0.58, 0.34 14, 15	0.36 11	Trial SK3
Glenboro, RM of South Cypress, MB (Cardale/Spring)	0.520	0.330	158	1	GS 12-30	6 F 12 F 20 F 28 F	10, 8.0 2.9, 2.9 1.9, 2.1 1.4, 1.6	7.0 2.2 1.6 1.2	AA150722 Trial MB
	1.110	0.746	149	1	GS 31-32	10H 17 H 23 H 32 H 49 S 57 S[c/] 64 S 71 S	80, 82 42, 47 (c 0.26) 40, 35 37, 42 43, 48 34, 38 39, 38 29, 31	63 35 29 30 36 28 30 23	

Location (Variety) [a/]	Application					DAT days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/ hL	Water, min, L/ha	No	Timing		Cation (individual)	Cation (Mean) [b/]	
Trials conducted in the United States in 2016 (samples: forage, GS 25–30; hay, GS 45–85; straw, GS 92)									
Seven Springs, Wayne, NC	0.620	0.395	157	1	GS 20	15 F	4.46, 4.67	4.6	AA160702
(26R41/Winter)	1.127	0.791	142	1	GS 39	27 H 63 S	25.0, 22.0 3.18, 1.01	23.5 2.09	NC
Fisk, Butler, MO (Branson /Winter)	0.618	0.332	186	1	GS 20-39	24 F	0.783, 0.698 (c 0.01)	0.74	AA160702 MO-1
	1.102	0.596	185	1	GS 39	32 H 45 S	38.9, 34.4 22.6, 20.1	36.5 21.5	
Tronoto,Deuel, SD	0.487	0.303	160	1	GS 20	13 F	1.02, 0.680	0.85	AA160702
(ForeFront/Spring)	1.092	0.892	122	1	GS 32	20 H 67 S	36.6, 22.3 11.9, 13.2	29.5 12.6	SD
Highland, Madison, IL	0.598	0.412	145	1	GS 20	22F	0.970, 1.01	0.99	AA160702
(25R78/Winter)	1.114	0.771	145	1	GS 39	15 H 52 S	15.2, 15.9 5.90, 8.97	15.6 7.44	IL-1
Uvalde, Hill, TX	0.489	0.443	110	1	GS 20	14 F	10.2, 12.9	11.5	AA160702
(Expresso/Spring)	1.132	0.939	120	1	GS 32	28 H 64 S	15.3, 18.0 10.3, 8.60	16.7 9.46	TX-1
Cleveland, Stutsman, ND	0.504	0.372	135	1	GS 20	14 F	5.71, 5.03 (c 0.04)	5.37	AA160702
(Prosper/Spring)	1.149	0.807	142	1	GS 32	20 H 40 S	34.8, 37.2 (c 0.04) 13.2, 14.2 (c 0.02)	36.0 13.7	ND-1
Carrington, Foster, ND	0.499	0.355	141	1	GS 20	14 F	1.52, 1.42	1.47	AA160702
(Faller/Spring)	1.102	0.797	138	1	GS 32	20 H 65 S	20.4, 19.5 13.9, 11.2	20.0 12.6	ND-2
York, York, NE	0.623	0.430	145	1	GS 20	8 F	16.5, 13.4	14.9	AA160702
(Overland HRW/ Winter)	1.119	0.601	186	1	GS 39	34 H 55 S	17.5, 19.7 9.13, 9.01	18.6 9.07	NE-1
Hinton, Caddo, OK	0.623	0.339	184	1	GS 20	32 F	0.580, 0.492	0.54	AA160702
(Duster/Winter)	1.102	0.713	155	1	GS 39	21 H 56 S	47.8, 38.9 13.0, 10.9	43.4 11.9	OK-1
Littlefield, Lamb, TX	0.640	0.447	143	1	GS 20	18 H	2.66, 0.870	1.77	AA160702
(TAN 111/ Winter)	1.102	0.793	139	1	GS 39	40 H 56 S	20.2, 16.3 12.6, 9.96	18.3 11.3	TX-2
Levelland, Hockley, TX	0.504	0.353	143	1	GS 20	14 F	1.51, 1.36	1.44	AA160702
(Express/Spring)	1.105	0.795	139	1	GS 32	45 H 58 S	3.73, 3.75 8.55, 7.31	3.74 7.93	TX-3
Wall, Tom Green, TX	0.502	0.350	143	1	GS 20	14 F	3.54, 4.71	4.71	AA160702
(Expresso/Spring)	1.119	0.799	140	1	GS 32	35 H 72 S	12.0, 11.0 9.32, 10.2	11.5 9.76	TX-4
Weatherford, Custer, OK	0.625	0.338	185	1	GS 20	22 F	3.40, 3.64	3.52	AA160702

Location (Variety) [a/]	Application					DAT days	Chlormequat mg.kg		Reference Trial No
	kg ai/ha	kg ai/ hL	Water, min, L/ha	No	Timing		Cation (individual)	Cation (Mean) [b/]	
(Endurance/Winter)	1.132	0.741	153	1	GS 39	22 H 62 S	42.8, 40.5 12.3, 9.78	41.6 11.0	OK-2
Richland, Keokuk, IA (GV662/Winter)	0.625	0.387	161	1	GS 20	21 F	3.14, 2.68	2.91	AA160702
	1.142	0.674	169	1	GS 39	17 H 54 S	19.8, 59.2 10.1, 8.76	39.5 9.42	IA-2
American Falls, Power ID (Klassic/Spring)	0.499	0.294	170	1	GS 20	19 F	2.68, 0.878	1.78	AA160702
	1.102	0.664	166	1	GS 32	28 H 61 S	30.6, 36.6 19.7, 20.4	33.6 20.1	ID
Kirksville, Adair, MO (Certified Rollag Spring Wheat)	0.492	0.298	165	1	GS 20	4 F 9 F 14 F[d/] 19 F 24 F	37.5, 34.3 14.7, 19.7 3.99, 4.33 2.45, 2.98 1.62, 2.58	35.9 17.2 4.16 2.71 2.10	AA160702 MO-2
	1.119	0.669	167	1	GS 32	16 H 21 H 26 H[e/] 31 H 35 H 41 S 46 S 52 S[c/] 55 S 62 S	41.8, 36.4 23.5, 20.6 23.0, 24.8 23.5, 20.6 22.4, 15.8 19.9, 20.3 22.5, 20.6 20.1, 19.6 (c 0.02) 12.7, 15.7 19.0, 19.1	39.1 25.2 23.9 22.1 19.1 20.1 21.5 19.9 14.2 19.0	
Montpelier, Stutsman, ND (Prosper/Spring)	0.487	0.371	131	1	GS 20	10 F 15 F 20 F[d/] 25 F 30 F	4.56, 6.21 1.76, 1.66 1.09, 0.875 0.497, 0.787 0.427, 0.557	5.39 1.71 0.983 0.642 0.492	AA160702 ND-3
	1.124	0.796	141	1	GS 32	11 H 19 H 21 H[e/] 26 H 32 H 37 S 42 S 47 S[c/] 52 S 57 S	52.5, 45.7 33.9, 28.0 30.2, 27.2 31.6, 33.8 21.4, 38.6 22.7, 25.6 19.1, 9.62 21.3, 17.9 19.7, 18.6 10.9, 26.4	49.1 31.0 28.7 32.7 30.0 24.2 14.4 19.6 19.1 18.7	

Notes:

[a]/ The term "Winter" means winter wheat; "Spring" means spring wheat.

[b/] Calculated by adjusting for the molecular weights (MW of chlormequat chloride, 158.07; and MW of chlormequat cation, 122.62).

[c/] Commercial harvest timing (GS 92).

[d/] GS 30 forage (commercial harvest).

[e/] GS 45–80 hay (commercial harvest).

RM=Rural Municipality.

Table 11 Residues of chlormequat in wheat forage from trials conducted with a single foliar application in European countries and evaluated by the 2017 JMPR (results reported on a fresh weight basis)(Extracted from the 2017 JMPR Evaluation; matrices not related to forage were removed from the original table)

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
D-75233, Niefern-Öschelbronn, Germany (winter wheat, Tores)	750 SL	37	1.67	195	0	Whole plant w/o roots	26	20	2010/1014090, 01
					14	Whole plant w/o roots	5.5	4.3 5.2	
					28	Whole plant w/o roots	4.6	3.6	
D-71277, Perouse-Rutesheim, Germany (winter wheat, Tommi)	750 SL	37	1.40	163	0	Whole plant w/o roots	30	23	2010/1014090, 02
F-45300, Rouvres-Saint-Jean, France (winter wheat, Campero)	750 SL	37	1.57	204	0	Whole plant w/o roots	44	34	2010/1014090, 03
F-45300, Bouilly-en-Gâtinais, France (winter wheat, Apache)	750 SL	37	1.58	206	0	Whole plant w/o roots	59	46	2010/1014090, 04
North Cave, East Yorkshire, United Kingdom (winter wheat, Oakley)*	750 SL	37	1.56	203	0	Whole plant w/o roots	78 c46	60 c36	2010/1041090, 05
					15	Whole plant w/o roots	42	33	
					27	Whole plant w/o roots	24	19	
74193 Stetten a. H. Rieslingstrasse 18, Baden-Württemburg, Germany, 2003 (winter wheat, Transit)	350 SL	37	0.70	100	0	Whole plant w/o roots	15.2	12	2004/1015956, 01
	750 SL	37	1.50	100	0	Whole plant w/o roots	20.9	16	
82170 Pompignan 30 route de Toulouse, Midi-Pyrenées, France, 2003	350 SL	39	0.70	100	0	Whole plant w/o roots	10.5	8.1	2004/1015956, 05

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
(winter wheat, Sagem)#									
	750 SL		1.50	100	0	Whole plant w/o roots	27.2	21	
D-47652 Weeze, Nordrhein-Westfalen, Germany, 2007 (spring wheat, Taifun)	750 SL	32	1.54	200	0	Whole plant w/o roots	80	62	2008/1014941, 01
					15	Whole plant w/o roots	10	7.8 10	
					28	Whole plant w/o roots	4.2	3.3	
I-40068 Emilia Romagna, Italy, 2007 (spring wheat, Lippo)	750 SL	32	1.05	204	0	Whole plant w/o roots	52	40	2008/1014941, 05
					14	Whole plant w/o roots	17	13 25	
					28	Whole plant w/o roots	5.4	4.2	
Via Calabria Nuovo No. 3, Quarto Inferiore, 40057 Bologna, Italy, 2007 (spring wheat, Croine)	750 SL	32	1.55	201	0	Whole plant w/o roots	126	98	2008/1014940, 01
					14	Whole plant w/o roots	5.7	4.4 5.7	
					29	Whole plant w/o roots	0.36	0.28	
Granarolo dell'Emilia, 40057, Emilia Romagna, Italy, 2008 (spring wheat, Blasco)	750 SL	33	1.56	202	0	Whole plant w/o roots	60	47	2009/1021674, 01
					14	Whole plant w/o roots	8.6	6.7 8.7	
					29	Whole plant w/o roots	0.27	0.21	
V. Matteotti 13, Molinella, Bologna 40062, Italy, 2008 (durum wheat, Duilio)	750 SL	32	1.52	198	0	Whole plant w/o roots	68	53	2009/1021674, 02
Barry d'Islemade, 82000 Tarn et Garonne, France, 2008 (winter wheat, Quality)	750 SL	32	1.57	204	0	Whole plant w/o roots	30	23	2009/1021674, 03
Finhan, 82700 Tarn et Garonne, France, 2008 (durum wheat, Dakter)	750 SL	32	1.55	201	0	Whole plant w/o roots	27	21	2009/1021674, 04

Notes:

Except where indicated, no residues above the LOQ were found in any of the untreated control samples.

*Trial accidentally oversprayed with an additional application of 1.6 kg ai/ha chlormequat chloride 18 days prior to the trial application.

#Trial flagged by applicant as having abnormally high residues due to extremely low rainfall during the trial, contributing to lowered yields, and use of a durum wheat variety.

Table 12 Residues of chlormequat in wheat straw from supervised trials conducted with a single foliar application in European countries and evaluated by the 2017 JMPR (results reported on a fresh weight basis)(Extracted from the 2017 JMPR Evaluation)

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Brunne, Germany (winter wheat, Thasos)	460 SL	37	1.52	150	94	26	20	2005/1014176, ACK/03/04
	750 SL	37	1.50	150	94	31	24 32	
Seebach, northern France (winter wheat, Cap Horn)	460 SL	34	1.52	150	68	4.1	3.2 4.3	2005/1014176, FAN/03/04
	750 SL	34	1.50	150	68	3.1	2.4	
Aussonne, southern France (winter wheat, Autan)	460 SL	35	1.52	150	80	27	21 28	2005/1014176, FTL/03/04
	750 SL	35	1.50	150	80	14	11	
Withington, United Kingdom (spring wheat, Paragon)	460 SL	37	1.52	150	78	14	11	2005/1014176, OAT/01/04
	750 SL	37	1.50	150	78	19	15 20	
D-75233, Niefern-Öschelbronn, Germany (winter wheat, Tores)	750 SL	37	1.67	195	84	8.1	6.3 7.6	2010/1014090, 01
D-71277, Perouse-Rutesheim, Germany (winter wheat, Tommi)	750 SL	37	1.40	163	98	9.4	7.3 11	2010/1014090, 02
F-45300, Rouvres-Saint-Jean, France (winter wheat, Campero)	750 SL	37	1.57	204	84	6.2	4.8 6.2	2010/1014090, 03

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
F-45300, Bouilly-en-Gâtinais, France (winter wheat, Apache)	750 SL	37	1.58	206	71	24	19 24	2010/1014090, 04
North Cave, East Yorkshire, United Kingdom (winter wheat, Oakley)*	750 SL	37	1.56	203	75	38 c28	29 c22	2010/1041090, 05
74193 Stetten a. H. Rieslingstrasse 18, Baden-Württemburg, Germany, 2003 (winter wheat, Transit)	350 SL	37	0.70	100	57	16.7	13 37	2004/1015956, 01
	750 SL	37	1.50	100	57	13.4	10 14	
82170 Pompignan 30 route de Toulouse, Midi-Pyrenées, France, 2003 (winter wheat, Sagem)#	350 SL	39	0.70	100	50	23.7	18 53	2004/1015956, 05
	750 SL	39	1.50	100	51	52.9	41 55	
D-47652 Weeze, Nordrhein-Westfalen, Germany, 2007 (spring wheat, Taifun)	750 SL	32	1.54	200	79	13	10 13	2008/1014941, 01
NL-6595, MS Ottersum, Limburg, The Netherlands, 2007 (winter wheat, Limos)	750 SL	32	1.62	210	75	9.5	7.4 9.2	2008/1014941, 02
F-12290, Aveyron, France, 2007 (spring wheat, Florence	750 SL	37	1.00	195	98	10	7.8 16	2008/1014941, 03

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Aurore)								
F-82100 Tarn et Garonne, France, 2007 (winter wheat, Apache)	750 SL	33	1.04	202	85	4.2	3.3 6.4	2008/1014941, 04
I-40068 Emilia Romagna, Italy, 2007 (spring wheat, Lippo)	750 SL	32	1.05	204	98	1.9	1.5 2.9	2008/1014941, 05
I-40054 Emilia Romagna, Italy, 2007 (winter wheat, Duilio)	750 SL	32	1.07	208	96	< 0.50	< 0.39	2008/1014941, 06
Via Calabria Nuovo No. 3, Quarto Inferiore, 40057 Bologna, Italy, 2007 (spring wheat, Croine)	750 SL	32	1.55	201	87	< 0.50 (0.15)	< 0.39 (0.12)	2008/1014940, 01
Castel S. Pietro, 40024 Bologna, Italy, 2007 (durum wheat, San Carlo)	750 SL	32	1.56	202	99	< 0.50 (0.36)	< 0.39 (0.28)	2008/1014940, 02
82000 Montauban, France, 2007 (winter wheat, Quality)	750 SL	32	1.48	192	65	9.0	7.0 9.6	2008/1014940, 03
82700 Finhan, France, 2007 (durum wheat, Joyaux)	750 SL	37	1.57	204	72	16	12 15	2008/1014940, 04
Granarolo dell'Emilia, 40057, Emilia Romagna, Italy, 2008 (spring wheat, Blasco)	750 SL	33	1.56	202	62	< 0.50	< 0.39	2009/1021674, 01
V. Matteotti 13, Molinella, Bologna 40062, Italy, 2008 (durum wheat, Duilio)	750 SL	32	1.52	198	96	0.61	0.47 0.63	2009/1021674, 02
Barry	750 SL	32	1.57	204	95	4.1	3.2	2009/1021674,

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
d'Islemade, 82000 Tarn et Garonne, France, 2008 (winter wheat, Quality)							4.1	03
Finhan, 82700 Tarn et Garonne, France, 2008 (durum wheat, Dakter)	750 SL	32	1.55	201	106	< 0.50 (0.32)	< 0.39 (0.25)	2009/1021674, 04
Herbert Neumann Dorfstr. 2, 16833 Brunne, Germany, 2004 (winter wheat, Thasos)	460 SL	37	1.52	150	94	26	20 27	2005/1014176, 01
	750 SL	37	1.50	150	94	31	24 32	
30 route de Hunspach, 67160 Seebach, France, 2004 (winter wheat, Cap Horn)	460 SL	34	1.52	150	68	4.1	3.2 4.3	2005/1014176, 02
	750 SL	34	1.50	150	68	3.1	2.4 3.2	
Ourmieres 3529, route de Merville 31840 Aussonne, France, 2004 (winter wheat Autan)	460 SL	35	1.52	150	80	27	21 28	2005/1014176, 03
	750 SL	35	1.50	150	80	15	12 16	
Upcote Farm, Withington, GL54 4BL, United Kingdom, 2004 (spring wheat, Paragon)	460 SL	37	1.52	150	78	14	11 15	2005/1014176, 04
	750 SL	37	1.50	150	78	19	15 20	

Notes:

Except where indicated, no residues above the LOQ were found in any of the untreated control samples. Values in italics have been proportionally adjusted for application rate in order to match the Argentine GAP for wheat.

*Trial accidentally oversprayed with an additional application of 1.6 kg ai/ha chlormequat chloride 18 days prior to the trial application.

#Trial flagged by applicant as having abnormally high residues due to extremely low rainfall during the trial, contributing to lowered yields, and use of a durum wheat variety.

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Effect of processing on the nature of residue

The 2017 JMPR evaluated the high-temperature hydrolysis study simulating processing procedures, and processing studies on barley, oat, rye and wheat. Chlormequat was not hydrolysed during simulated hydrolysis. The 2017 Meeting estimated processing factors for processed commodities of barley and wheat from harvested grains.

The current Meeting received the results of new processing studies on barley and wheat as follows. Processing factors are estimated in the same way as in 2017 Meeting.

Barley

The 2017 JMPR calculated processing factors for processing of barley: cleaned grain (pot barley), offal (pot barley), pearling dust, pot barley, cleaned grain (malting), offal (malting), steeping water, malt sprouts, malt, spent grain, condensate, flocs, wort, yeast, green beer and beer, for which no data were provided to the current Meeting. The current Meeting received information on processing of barley to bran, pearled barley and flour.

Two trials were conducted in Canada and the United States in 2016 for collecting bulk samples for processing studies (AA 160703, Hoi, S.W., 2017b). Chlormequat chloride (620 SL) was applied once as a foliar spray at a rate of 1.41 kg ai/ha (Canadian trial) or at an exaggerated rate of 2.92 kg ai/ha (United State trial) at GS 32. Barley grain was harvested in duplicate at normal commercial harvest (GS 92) and kept in the processing laboratory at ≤ -12 ºC until processing. Before weighing and cleaning, some grain samples were collected and kept in frozen storage. Grains were processed using laboratory-scale equipment representative of commercial practices to pearled barley, flour and bran, but samples were processed by batch rather than continuous, as in commercial operation. Each grain sample was processed independently.

Barley grain samples were weighed, and their moisture contents were adjusted to 11.0–13.5 percent with drying as necessary. Sampled were cleaned by aspiration to remove grain dust, and screening to separate large and small foreign particles from the barley grain. Cleaned barley was hulled and separated in a laboratory huller into blocked barley and husk (hulls). The resulting husk were discarded.

For production of pearled barley, blocked barley was processed in an abrasive testing mill. After milling, the material was separated with a sifter equipped with a 24-mesh sieve. Material on top of the sieve was pearled barley. Fine material (pearlings) passed through the sieve and was weighed and the discarded. Pearled barley fractions were collected and placed in frozen storage.

For production of flour and bran, the moisture content of the blocked barley was adjusted to 14.5 percent. The sample was mixed for a minimum of 15 minutes and allowed to equilibrate for a minimum of 12 hours before milling. Conditioned blocked barley was fed through the break side of a mill with three break rolls. After passing through the break rolls, the material was fed onto the break sifter

screens (140 and 800 µm). Material passing through the 140 µm screen was break flour. Material not passing through 140 µm screen but passing through the 800 µm screen was middlings. Material exiting the end of sifter was coarse bran. Middlings were then fed into the reduction side of the mill with to reduction rolls. After passing through the reduction rolls, the material was passed over a 160 µm screen. Material passing through the screen was reduction flour. Material remaining on top of the screen was shorts. Shorts were passed through the reduction roll two additional times. Break and reduction flours were combined and mixed for 13–17 minutes. Resulting barley flour fractions were collected and placed into frozen storage.

Coarse bran exiting the break sieve was placed into the reduction side of the mill and conveyed by beater bars over a 128 µm screen. Material passing though the screen was shorts and was added to shorts from the reduction mill. Material passing over the screen and exiting the end was bran. After weighing, shorts were discarded. Resulting bran fractions were collected and placed into frozen storage.

Samples were maintained frozen at <-18 ºC until extraction for a maximum of 316 days. Extracts were analysed within 6 days. Residues of chlormequat chloride were determined by LC-MS/MS method M01-011 with an LOQ of 0.01 mg/kg. Concurrent procedural recoveries were within the acceptable range of 70–110 percent, with relative standard deviation < 20 percent.

The residues in the RAC and processed commodities are shown in Table 13.

Table 13 Residues of chormequat in barley grains and their processed commodities

Trial No. (Variety)	Application		Sample	Chlormequat cation, mg/kg [a]	Processing factor
	kg ai/ha	No			
AA160703-CAN-10 (Spring barley, Newdale)	1.41	1	Grain [b]	1.35, 1.58	-
			Pre-processing grain	1.59, 1.42	1.18, 0.899
			Bran	1.38, 1.48	1.02, 0.937
			Pearled barley	0.425, 0.337	0.315, 0.213
			Flour	0.309, 0.298	0.229, 0.189
AA160703-ND-3 (Spring barley, Tradition)	2.92	1	Grain [b]	1.90, 1.87	-
			Pre-processing grain	1.76, 1.87	0.926, 1.00
			Bran	1.68, 1.71	0.884, 0.914
			Pearled barley	0.425, 0.337	0.224, 0.180
			Flour	0.309, 0.298	0.163, 0.159

Notes:

[a] Chlormequat chloride was analysed and expressed in chlormequat cation equivalents. Each sample was analysed in duplicate, and the mean analytical result is included in the above table.

[b] At harvest (same values as in Table 3)

Wheat

The 2017 JMPR calculated processing factors for wheat offal, epidermis coarse bran, fine bran, straight flour, low grade meal, flour (type 550), total bran (whole meal flour), straight flour (whole meal flour), whole meal flour, Dough and wholegrain bread. The current Meeting received information on processing of wheat grain to aspirated grain fraction, middlings, shorts, flour, germ and bran.

Two trials were conducted in the United States in 2016 for collecting bulk samples for processing studies (AA 160702, Hoi, S.W., 2017a). Chlormequat chloride (620 SL) was applied once as a foliar spray at a rate of 1.13–1.14 kg ai/ha or at an exaggerated rate of 2.23 kg ai/ha at GS 32. Wheat grain was harvested in duplicate at normal commercial harvest (GS 92) and kept frozen in the processing laboratory

at < -1 ºC until processing. Before weighing, cleaning and processing, some grain samples were collected and kept in frozen storage. Grains were processed using laboratory-scale equipment representative of commercial practices. Grain samples were processed to the aspirated grain fraction (AGF), middlings, shorts, flour and bran, germ and bran. Each grain sample was processed independently.

Generation of AGF simulated industrial practices used in terminal elevators to remove grain dust. Samples were processed in batch rather than continuous, as in commercial operation. The bulk grain samples from AA 160702 OK-2 trial with the application at 1.1 kg ai/ha were weighed and their moisture content was determined to be below 13.0 percent. Therefore, no additional drying was carried out. Each sample was placed in a dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. As the sample moved in the system (120 minutes), the sample was aspirated to remove grain dust. The grain dust moving through the 2360 µm (8 mesh) sieve was combined to produce one AGF sample and the sample was frozen for storage.

Bulk wheat grain samples from AA 160702 OK-2 trial and IA-2 for processing were taken from frozen storage and weighed. The moisture content was determined for each sample. The samples from IA-2 trial showed moisture content above 13.5 percent, they were dried in a tray oven at 54–71 ºC until their moisture content became 11.0–13.5 percent. Then samples were cleaned by aspiration to remove grain dust and screening to separate large and small foreign particles from the cleaned wheat samples.

Cleaned wheat, after adjusting to moisture content of 16 percent for 1–1.5 hours (tempering), was passed through a disc mill and sifted. The germ fraction remained on top of the 30-mesh sieve was aspirated to remove bran. The germ (with endosperm) was passed through a reduction mill. The germ and reduced endosperm were sifted with sieves to separate the germ from the endosperm. The germ fraction was also aspirated again to remove additional bran and milled/sieved to remove additional endosperm. Resulting germ fractions were collected and placed in frozen storage.

The wheat varieties used for processing studies were determined to be intermediate structure (between floury and vitreous)(OK-2) or floury (IA-2) and were tempered accordingly for flour production. Tempered wheat was fed through the spout on the break side of a mill equipped with three break rolls, and then fed onto the break sifter screens (140 µm and 800 µm). Materials passing through the 800 µm screen is middlings. Material not passing though was conveyed to the end of the sifter. Material exiting the end of the sifter is bran (coarse). After sampling, the remaining middlings were poured into the feed hopper of the reduction system (to mill middlings into flour and by-products) with two reduction rolls. After passing through the reduction rolls, the material was fed to the reduction sifter screen of 160 µm. Material passing through the screen is reduction flour. Material not passing through and conveyed to the end of the sifter is shorts (low-grade mill product, containing principally germ and fine bran particles). The break and reduction flours were mixed using a mixer to produce standard mill run flour. Flour fractions were collected and placed into frozen storage.

Bran exiting the break sieve is placed in the reduction side of the mill, but not reduced with the rollers. The coarse bran is conveyed by beater bars over a 128 µm screen. Material passing through the screen is shorts and is added to shorts from the reduction mill. Material passing over the screen and exiting the end is bran. Shorts and bran fractions were collected and placed into frozen storage.

Samples of processed commodities were maintained frozen at < -18 ºC until extraction for a maximum of 310 days. Exacts were analysed within 5 days of extraction. Residues of chlormequat chloride were determined by LC-MS/MS method M01-011. The method was validated with an LOQ of 0.01 mg/kg. Concurrent procedural recoveries were within the acceptable range of 70–110 percent with relative standard deviation below 20 percent.

The residues in the RAC and processed commodities are shown in Table 14.

Table 14 Residues of chormequat in wheat grains and their processed commodities

Trial No. (Variety)	Application		Sample	Chlormequat cation, mg/kg [a]	Processing factor
	kg ai/ha	No			
AA160702-OK-2 (Winter Wheat, Endurance)	1.13	1	Grain [b]	1.58, 1.50	-
			AGF	13.4, 11.5 (untreated, 0.01)	8.48, 7.67
	2.23	1	Grain [b]	2.85, 2.86	-
			Pre-processing grain	2.31, 2.77	0.811, 0.969
			Bran	3.32, 3.29	1.16, 1.15
			Flour	0.323, 0.330	0.113, 0.115
			Middlings	0.947, 0.980	0.332, 0.343
			Shorts	1.34, 1.37	0.470, 0.479
			Germ	13.7, 12.4	4.81, 4.34
AA160702-IA-2 (Winter Wheat, GV662)	2.30	1	Grain [b]	1.54, 1.40	-
			Pre-processing grain	1.74, 1.72	1.13, 1.23
			Bran	3.42, 3.33	2.22, 2.38
			Flour	0.132, 0.125	0.086, 0.089
			Middlings	1.13, 1.17	0.734, 0.835
			Shorts	2.46, 2.48	1.60, 1.77
			Germ	8.25, 8.70	5.36, 6.21

Notes:

[a] Chlormequat chloride was analysed and expressed in chlormequat cation equivalents. Each sample was analysed in duplicate, and the mean analytical result is included in the above table.

[b] At harvest (same values as in Table 3).

Table 15 shows the summary of processing factors calculated from the studies provided to the current Meeting.

Table 15 Processing factors of chlormequat for barley and wheat processed commodities

Commodity	n	Processing factor	
		Individual	Best estimate
Barley			
Grain at harvest	4	-	-
Pre-processing grain	4	0.899, 0.926, 1.00, 1.18	0.96
Bran	4	0.884, 0.914, 0.937, 1.02	0.93
Pearled barley	4	0.180, 0.213, 0.224, 0.315	0.22
Flour	4	0.159, 0.163, 0.189, 0.229	0.18
Wheat			
Grain at harvest	4	-	-
Pre-processing grain	4	0.811, 0.969, 1.13, 1.23	1.0
Bran	4	1.15, 1.16, 2.22, 2.38	1.7
Flour	4	0.086, 0.089, 0.113, 0.115	0.10
Middlings	4	0.332, 0.343, 0.734, 0.835	0.54
Shorts	4	0.470, 0.479, 1.60, 1.77	1.0

Commodity	n	Processing factor	
		Individual	Best estimate
Germ	4	4.34, 4.81, 5.36, 6.21	5.1
AGF	2	7.67, 8.48	8.1

APPRAISAL

Chlormequat is a plant growth regulator and usually formulated as the chloride salt. It acts primarily by reducing cell elongation, as well as by lowering the rate of cell division and by inhibiting the synthesis of gibberellins. Chlormequat was evaluated by the Meeting in 1970, 1972, 1994 (T, R), 1997 (R), 1999 (T, for ARfD), 2000 (R) and 2017 (T, R, periodic re-evaluation).

The 2017 Meeting reaffirmed the ADI of 0–0.05 mg/kg bw (established in 1997) and ARfD of 0.05 mg/kg bw (established in 1999). The 2017 Meeting confirmed residue definitions as follows:

The residue definition (for compliance with the MRL and dietary risk assessment) in plant and animal commodities: chlormequat cation.

The residue is not fat soluble.

The Forty-third Session of the Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included chlormequat for evaluation of uses on barley and wheat.

The current Meeting received new information on: GAP in Canada, analytical methods, supervised residue trials and processing studies, for wheat and barley.

Analytical methods

The 2017 Meeting evaluated two LC-MS/MS methods and one GC method for plant commodities, and a LC-MS/MS method and an ion chromatography method for animal commodities for determining chlormequat chloride.

The current Meeting received information on a new LC-MS/MS method (M01-011), which was similar to the LC-MS/MS method (CEN/TC 275/WG 4N) provided to the 2017 JMPR, for determining chlormequat chloride in the supervised residue trials and processing studies. In this method, a homogenized samples of wheat or barley matrices (grain, leafy parts and processed commodities) were fortified with a known concentration of chlormequat-D4 chloride as internal standard. Residues of chlormequat/internal standard were extracted from the fortified sample with methanol/water (2:1, v/v). An aliquot of extract was then filtered for determination of chlormequat chloride by LC-MS/MS.

At the fortification levels of 0.01 and 0.1 mg/kg in grain, straw, hay, forage and processed commodities of wheat and barley, the mean recoveries were 90–110 percent and the RSD values were < 13 percent. The validated LOQ was 0.01 mg/kg for chlormequat chloride in these matrices.

Stability of pesticide residues in stored analytical samples

The 2017 Meeting evaluated the stability data on chlormequat chloride in cereal matrices (grain, straw and processed matrices) stored at approximately -18 or -20 °C. The proved stable periods cover the sample storage intervals in the residue trials.

Results of supervised residue trials on crops

The Meeting received supervised trial data for chlormequat on wheat and barley conducted in Canada and the United States in 2015–2016.

Cereal grains

Wheat

The 2017 JMPR evaluated supervised trials conducted in France, Germany and Italy on winter, spring and durum wheat against the critical GAP in Argentina (one foliar application at the maximum rate of 2.025 kg ai/ha during BBCH 21–31) and estimated the maximum residue level of 2 mg/kg and STMR of 0.58 mg/kg for wheat.

The GAP of Argentina as shown above is valid at the time of this evaluation and is more critical than GAP of Canada for wheat (one foliar application at a maximum rate of 1.12 kg ai/ha, up to GS 39).

The current Meeting received information on supervised residue trials conducted in Canada (5) and the United States (17) on spring and winter wheat. In the United State trials, grains were harvested with hulls intact while the definition of the subgroup covering wheat states "without husks". The processing study using the wheat grains obtained in the trials conducted in the United States showed that the residues in pre-processing grain and in the grains at harvest were similar with the best estimate of processing factor of 1.0. Therefore, the Meeting decided to use the data on residues in grains with husk from these United State trials for evaluation.

As in each trial in Canada and the United States, a single application made at GS 32 or 39 at rates of 1.0–1.1 kg ai/ha while the GAP in Argentina allows a maximum rate of 2.025 kg ai/ha, the Meeting decided to use the proportionality principle for estimating a maximum residue level and an STMR. Scaled residues were calculated using the formula below.

Scaled residue (mg/kg) = (residue in the trial, mg/kg) × 2.025 (kg ai/ha) / (rate used in the trial, *kg ai/ha*).

After each scaled residue value, the residue found in each trial and respective application rate are indicated in a pair of parentheses, e.g., (residue value in mg/kg, application rate in kg ai/ha).

Residues of chlormequat cation in spring wheat grain in the trials in Canada and the United States with the application at GS 32 approximating the GAP in Argentina with scaling were in rank order (n=14): 0.52 (0.29, 1.119), 0.60 (0.31, 1.050), 0.92 (0.50, 1.105), 0.93 (0.53, 1.090), 1.2 (0.65, 1.102), 1.5 (0.86, 1.132), 1.8 (1.0, 1.110), 1.9 (1.01, 1.092), 2.2 (1.2, 1.090), 2.5 (1.4, 1.130), 2.6 (1.44, 1.124), 3.4 (1.85, 1.102), 3.4 (1.94, 1.149) and 5.3 (1.44, 1.124) mg/kg.

Residues of chlormequat cation in winter wheat grain in the trials in Canada and the United States with the application at GS 39 approximating the GAP in Argentina with scaling were in rank order were in rank order (n=9): 0.47 (0.26, 1.114), 0.77 (0.43, 1.127), 0.94 (0.52, 1.119), 0.99 (0.54, 0.102), 0.99 (0.54, 0.102), 1.4 (0.78, 1.140), 1.5 (0.85, 1.142), 2.3 (1.26, 1.102) and 2.8 (1.54, 1.132) mg/kg.

As the application timing in the trials on spring wheat was GS 32 and on winter wheat was GS 39 while the GAP in Argentina specifies BBCH 31, influence of the application timing (GS 32 vs GS 39) on the residues were tested by Mann-Whitney test, which indicates that there is no significant difference between the two residue populations. The Meeting decided to combine the data from the two different application timing to derive a maximum residue level and STMR.

Combined residue populations of chloride cation from the independent trials (one trial used for comparison above was not independent) in Canada and the United States on spring and winter wheat were in rank order (n=22): 0.47, 0.52, 0.59, 0.77, 0.91, 0.92, 0.94, 0.99, 0.99, 1.2, 1.5, 1.5, 1.8, 1.9, 2.2, 2.3, 2.5, 2.6, 2.8, 3.4, 3.4 and 5.3 mg/kg.

The residue population from the trials in Canada and the United States would lead to a higher estimates of maximum residue level and STMR. Based on the residue population from the Canadian and United States trials, the Meeting estimated a maximum residue level of 7 mg/kg and an STMR of 1.5 mg/kg for wheat.

IESTI calculation for wheat grain and its processed products resulted in a maximum of 110 percent of ARfD for children (consumption of wheat flakes; the processing factor of 0.8 estimated by the 2017 JMPR for the processing of oat its flakes was used). The Meeting therefore used an alternative GAP approach for estimating maximum residue level and STMR, and decided to evaluate the trial data against the GAP of Canada for wheat.

The combined residues of chlormequat cation in wheat grain from the trials in Canadian and United States, matching the GAP in Canada, were in rank order (n=22): 0.26, 0.29, 0.31, 0.43, 0.50, 0.50, 0.52, 0.54, 0.54, 0.65, 0.85, 0.86, 1.0, 1.0, 1.2, 1.3, 1.4, 1.4, 1.5, 1.9, 1.9 and 2.9 mg/kg.

The Meeting estimated a maximum residue level of 4 mg/kg for wheat to replace the previous recommendation of 2 mg/kg. The Meeting estimated an STMR of 0.855 mg/kg.

Barley

The 2017 JMPR evaluated supervised trials conducted in France, Germany and Spain on barley with the application timing at BBCH 32–37 against the critical GAP in the United Kingdom (one foliar application at the maximum rate of 1.65 kg ai/ha during BBCH 25–30) and estimated a maximum residue level of 2 mg/kg and STMR of 0.37 mg/kg for barley.

The current critical GAP was from the United Kingdom for barley which allows one foliar application at the maximum rate of 1.50 kg ai/ha up to and including GS 32. This GAP is similar to GAP in Canada (one foliar application at the maximum rate of 1.43 kg ai/kg up to GS 39).

The current Meeting received information on supervised residue trials conducted in Canada (10) and the United States (10) on barley in 2016 with a single application at GS 32 at rates 1.36–1.50 kg ai/ha.

Residues of chlormequat cation in barley grains in the independent trials conducted in Canada and the United States approximating GAP in the United Kingdom were in rank order (n=10): 0.12, 0.54, 1.0, 1.0, 1.5, 1.5, 1.6, 2.6, 3.3 and 3.9 mg/kg.

The Meeting estimated a maximum residue level of 7 mg/kg and an STMR of 1.5 mg/kg for barley.

IESTI calculation for barley grain and its processed products resulted in a maximum of 110 percent of ARfD for children (consumption of barley flakes; the processing factor of 0.8 estimated by the 2017 JMPR for the processing of oat its flakes was used). The Meeting looked for an alternative GAP that would lead to a smaller estimate of maximum residue level and STMR. GAP of Canada for wheat is similar to GAP of the United Kingdom and it would lead to the same maximum residue level. There was insufficient information available on the validity of GAP contained in the use pattern table of the Evaluation of the 2017 JMPR, the Meeting decided to maintain the previous recommendation of 2 mg/kg (STMR 0.37 mg/kg) until information becomes available on valid GAPs for barley in countries which allow lower application rates than the GAP used by this Meeting to enable the alternative GAP approach.

Residues in animal feeds

Barley forage

The 2017 JMPR received data on barley forage taken after one application of chlormequat at BBCH 32 or 37 in the trials conducted in Europe. However, as feeding cereal grain forage was not common in Europe unless specified on the label, the data were not evaluated.

The critical GAP is in the United Kingdom for barley allows one foliar application up to and including GS 32 at the maximum rate of 1.50 kg ai/ha. Neither the United Kingdom label nor the Canadian label contains restrictions regarding grazing or cutting for feed. Generally, forage can be used for grazing or cutting for feed 2 to 3 weeks after the application of pesticides.

In the trials conducted on barley in Canada and the United States and provided to the current Meeting, barley forage samples were not taken or analysed. The Meeting therefore evaluated the European trials against the critical GAP in the United Kingdom.

Residues of chlormequat cation in forage taken 14 to 21 days after the application in the European trials approximating GAP in Canada were in rank order:

Application at BBCH 37 (n=3):2.9, 3.6 and 12 mg/kg; and

Application at BBCH 32 (n=4): 1.9, 3.3, 3.7 and 6.7 mg/kg.

Mann-Whitney U-test indicates that these two populations were not significantly different. The Meeting combined the residue populations to estimate a median residue and highest residue.

The combined residues of chlormequat cation in barley forage were in rank order (n=7): 1.9, 2.9, 3.3, 3.6, 3.7, 6.7 and 12 mg/kg.

The Meeting estimated a median residue of 3.6 mg/kg (as received) and highest residue of 12 mg/kg (as received) for barley forage.

Wheat forage

The 2017 JMPR evaluated supervised trials conducted in France, Germany and Italy against the critical GAP in Argentina (one foliar application at the maximum rate of 2.025 kg ai/ha during BBCH 21–31) and estimated a median residue of 8.7 mg/kg and highest residue of 25 mg/kg for wheat forage (as received).

The current Meeting received information on residues of chlormequat cation in wheat forage, samples of which were taken at around GS 30 after one application at GS 12–30 at rates of 0.48–0.64 kg ai/ha, much lower rates than in the critical GAP in Argentina. As residues of chlormequat cation at 14–21 DAT were in a range of 0.85–12 mg/kg, the Meeting concluded that the median and highest residues recommended by the 2017 JMPR cover the residues found in wheat forage from the trials in Canada and the United States.

Barley, hay and/or straw

The 2017 JMPR evaluated supervised trials conducted in Europe on barley against the critical GAP in the United Kingdom (one foliar application at the maximum rate of 1.65 kg ai/ha during BBCH 25–30; since expired) and estimated a maximum residue level of 50 mg/kg (dw), and a median residue of 4.15 mg/kg (as received) and highest residue of 30 mg/kg for barley straw and fodder, dry.

Residues of chlormequat cation in barley hay in the trials conducted in the United States approximating the GAP in the United Kingdom were in rank order (n=10):5.4, 14, 18, 19, 33, 36, 39, 46, 48 and 60 mg/kg (as received), or 6.1, 15, 20, 22, 38, 41, 53, 54, and 68 mg/kg (dw).

Residues of chlormequat cation in barley straw in the trials conducted in the United States approximating the GAP in the United Kingdom were in rank order (n=10): 2.4, 3.8, 4.5, 5.9, 6.7, 9.8, 14, 16, 17, and 30 mg/kg (as received) or 2.8, 4.3, 5.1, 6.7, 7.6, 11, 15, 18, 19 and 34 mg/kg (dw).

The residue population of hay from the United States trials would lead to a higher maximum residue level, and median and highest residue than the residue population of straw and those estimated by the 2017 JMPR based on the European trials. Using the residue population of hay from the United States trials, the Meeting estimated a maximum residue level of 150 mg/kg (dw) for barley hay and/or straw and withdrew the previous recommendation for barley straw and fodder, dry of 50 mg/kg, dw. The Meeting also estimated a median residue and highest residue of 34.5 mg/kg and 73 mg/kg (as received) for barley hay and 8.25 mg/kg and 32 mg/kg (as received) for barley straw.

Wheat hay and/or straw

The 2017 JMPR evaluated residue trials on wheat conducted in Europe against the critical GAP in Argentina and estimated a maximum residue level of 80 mg/kg (dw), median residue of 13 mg/kg (as received) and highest residue of 55 mg/kg for wheat straw and fodder, dry.

Residues of chlormequat cation in wheat hay from the trials in Canada and the United States approximating the GAP in Argentina after scaling were in rank order (including one pair of not-independent trials for comparison of application timing):

Application at GS 39 (n=9): 28 (15.6, 1.114), 28 (15.6, 1.114), 34 (18.3, 1.102), 34 (18.6, 1.119), 42 (23.5, 1.127), 67 (36.5, 1.102), 70 (39.5, 1.142), 74 (41.6, 1.132) and 80 (43.4, 1.102) mg/kg; and

Application at GS 32 (n=14): 0.64 (0.36, 1.13), 6.9 (3.73, 1.105), 7.4 (4, 1.090), 21 (11.2, 1.119), 30 (16.7, 1.132), 37 (20, 1.102), 41(21, 1.050), 43 (23.9, 1.119), 55 (29.5, 1.092), 59 (32.7, 1.124), 62 (33.6, 1.102), 63 (36, 1.149), 64 (35, 1.111) and 111 (0, 1.090) mg/kg.

Mann-Whitney U-test indicates that these two data populations are not significantly different.

The combined data set from independent trials in Canada and the United States were in rank order (n=22): 0.64, 6.9, 7.4, 21, 28, 30, 34, 34, 37, 41, 42, 43, 55, 59, 62, 63, 64, 67, 70, 74, 80 and 111mg/kg (as received) or 0.73, 7.8, 8.4, 23, 32, 34, 38, 38, 42, 46, 48, 49, 62, 67, 70, 73, 72, 76, 80, 85, 91 and 127 mg/kg (dw).

Residues of chlormequat cation in wheat straw from the trials in Canada and the United States approximating GAP in Argentina after scaling were:

Application at GS 39 (n=9): 3.8 (2.09, 1.127), 14 (7.44, 1.114), 16 (9.07, 1.119), 17 (9.42, 1.142), 20 (11.0, 1.132), 21 (11.3, 1.102), 22 (11.9, 1.102), 32 (18, 1.140) and 40 (21.5, 1.102) mg/kg

Application at GS 32 (n=14):7.4 (4.0, 1.090), 10 (5.4, 1.050), 15 (7.93, 1.105), 17 (9.46, 1.132), 18 (9.76, 1.119), 20 (11, 1.130), 23 (12.6, 1.102), 23 (12.6, 1.102), 24 (13.7, 1.149), 35 (19.6, 1.124), 36 (19.9, 1.119), 37 (20.1, 1.102), 45 (24, 1.109) and 55 (30, 1.110) mg/kg,

Mann-Whitney U-test indicates that these two populations are not significantly different. The Meeting decided to combine these datasets.

The combined data set from independent trials in Canada and the United States were in rank order (n=22): 3.8, 7.4, 10, 14, 15, 16, 17, 17, 18, 20, 20, 21, 22, 23, 23, 24, 35, 36, 37, 40, 45 and 55 mg/kg

(as received) or 4.3, 8.4, 11, 16, 17, 18, 19, 19, 20, 23, 23, 24, 25, 26, 26, 27, 40, 41, 42, 45, 51 and 62 mg/kg. (dw).

The residue population of hay from trials conducted in Canada and the United States would lead to a higher maximum residue level than that from the residue population in straw from the Canadian and United States trials and the previous recommendation made in 2017 based on straw. Using the data population of residues in hay, the Meeting estimated a maximum residue level of 200 mg/kg (dw) for wheat hay and/or straw. The previous recommendation on wheat straw or fodder, dry (80 mg/kg, dw) was withdrawn. The Meeting estimated a median residue and highest residue of 42.5 and 117 mg/kg (as received) for wheat hay and 20.5 and 55 mg/kg (as received) for wheat straw.

Since barley hay/straw and wheat hay/straw are not distinguishable in trade, the Meeting agreed that the higher maximum residue level of wheat of 200 mg/kg should also apply to barley hay and/or straw.

Fate of residues during processing

Processing

The Meeting received information on processing of wheat to bran, flour, middlings, shorts, Germ and aspirated grain fractions; and of barley to bran, pearled barley and flour. For the processing studies, samples of wheat and barley grains were obtained from the supervised trials with the applications at the critical GAP rate and its 2-fold rate, and chlormequat cation was analysed. Processing factors of wheat to its processed commodities and barley to its processed commodities are summarized below together with the processing factors derived by the 2017 JMPR for additional processed commodities. Using the best estimates of processing factors and the STMR values for wheat and barley, the STMR-P values were calculated for processed commodities of wheat and barley. For estimation of acute dietary exposure from flakes of wheat and barley, the Meeting used the processing factor of 0.80 for the processing of oat to its flakes estimated by the 2017 JMPR.

Processing factors of chlormequat for wheat to its processed commodities and barley to its processed commodities are shown below.

Table 16 Calculated STMR-Ps for processed food and feed commodities

Commodity (Food/feed)	N	Processing factor		STMR/ STMR-P/ Median
		Individual	Best estimate	
Wheat grain at harvest	4	-	-	0.855
Bran	4+6	1.15, 1.16, 2.22, 2.38 2.5, 2.8, 2.9, 3.1, 3.4, 4.6	2.65	2.3
Flour	4+5	0.086, 0.089, 0.113, 0.115 0.19, 0.28, 0.29, 0.30, 0.41	0.19	0.16
Germ	4	4.34, 4.81, 5.36, 6.21	5.1	4.3
Wholemeal [a]	6	0.86, 0.91, 1.0, 1.0, 1.1, 1.4	1.0	0.855
Wholemeal bread	6	0.49, 0.51, 0.53, 0.55, 0.63, 0.79	0.54	0.46
Barley grain at harvest	4	-	-	0.37
Bran	4	0.884, 0.914, 0.937, 1.02	0.93	0.34
Pearled barley	4+5	0.180, 0.213, 0.224, 0.315 0.06, 0.8, 0.9, 1.0, 1.0	0.32	0.12
Flour	4	0.159, 0.163, 0.189, 0.229	0.18	0.066
Malt	5	0.69, 0.9, 0.9, 0.9, 1.0	0.9	0.33
Beer	5	0.015, 0.1, 0.2, 0.2, 0.2	0.2	0.074
Wheat grain at harvest	4	-	-	0.855

Commodity (Food/feed)	N	Processing factor		STMR/ STMR-P/ Median
		Individual	Best estimate	
Middlings	4	0.33, 0.34, 0.73, 0.84	0.54	0.46
Shorts	4	0.47, 0.48, 1.6, 1.8	1.0	0.89
AGF	2	7.67, 8.48	8.1	6.9
Barley grain at harvest	4	-	-	0.37
Spent grain	4	0.01, 0.02, 0.02, 0.03	0.02	0.007

Notes:

NB: Values in italics were from the 2017 JMPR.

[a] The processing factors calculated separately for wheat wholemeal flour and wholemeal in the 2017 JMPR Report were combined under wheat "wholemeal".

Based on the processing factors and maximum residue level of wheat grain of 4 mg/kg, the Meeting estimated maximum residue levels for wheat bran (unprocessed) at 10 mg/kg, replacing the previous recommendation of 7 mg/kg, and for wheat germ at 20 mg/kg.

Residues in animal commodities

Livestock dietary burden

Dietary burden calculations for cattle and poultry are provided below. The dietary burdens were estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO Manual.

Table 17 Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden: chlormequat, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	max	Mean	max	Mean	max	Mean	Max	Mean
Beef cattle	22.4	9.72	32.6	12.9	133.0	48.3	3.22	3.22
Dairy cattle	28.5	11.5	32.6	12.9	99.8	38.8	3.05	2.78
Poultry broiler	3.17	3.17	2.00	2.00	1.92	1.92	0.34	0.34
Poultry layer	3.17	3.17	15.3	6.83	1.88	1.88	1.47	1.47

The calculated highest maximum dietary burden was 133.0 ppm (chlormequat cation) based on the Australian diet, which is more than 30 percent higher than the highest dose rate in the cattle feeding study. Therefore, it was not possible to estimate residues in mammalian commodities at the highest maximum burden.

Chlormequat was registered in Australia and, according to the current product labels, approved for use on wheat with one application at a maximum rate of 0.758 kg ai/ha from Z 25 to Z 31, but not for other cereal grains. Grazing and cutting for stock feed are not allowed before 21 days after the application. Most of forage and hay data and all of straw data used for estimating median and highest residue were from the samples taken at 20 days or later after application in the trials. Taking into consideration the registration of chlormequat for wheat in Australia, no importation of hay or straw into Australia, and that the application timing in the GAP in Australia is comparable to the GAP of Argentina, the Meeting decided to apply the proportionality principle to the median and highest residue values of wheat feed items for re-calculating the maximum and mean dietary burden based on the Australian diet.

Based on the maximum rate of 2.025 kg ai/ha in Argentinian GAP for wheat and that of 0.758 kg ai/ha in the Australian GAP for wheat, the median and highest residue in wheat derived feed were scaled for the purpose of calculating dietary burdens based on the Australian diet. The scaled

median and highest residue were: 15.9 and 28.8 mg/kg in wheat hay, 7.67 and 20.6 mg/kg for wheat straw, and 3.26 and 9.36 mg/kg for wheat forage.

After recalculation using adjusted median and highest residue for wheat and removing forage, hay and straw derived from other cereal grains, animal dietary burdens based on the Australian diet, together with other diets, were as follows.

Table 18 Recalculated maximum and mean dietary burdens of chlormequat (for adjustment for the Australian diet)

	United States-Canada		European Union		Australia		Japan	
	Max	Mean	max	Mean	max	Mean	Max	Mean
Beef cattle	22.4	9.72	32.6	12.9	37.4❶	18.1❷	3.22	3.22
Dairy cattle	28.5	11.5	32.6❸	12.9❹	26.0	12.3	3.05	2.78
Poultry broiler	3.17	3.17	2.00	2.00	3.10	3.10	0.34	0.34
Poultry layer	3.17	3.17	15.3❺	6.83❻	2.99	2.99	1.47	1.47

Notes:

❶Highest maximum dietary burden for beef cattle suitable for estimation of maximum residue levels for mammalian meat, fat and offal.

❷Highest mean dietary burden for beef cattle suitable for estimation of STMRs for mammalian meat, fat and offal.

❸Highest maximum dietary burden for dairy cattle suitable for estimation of maximum residue level for milks.

❹Highest mean dietary burden for dairy cattle suitable for estimation of STMRs for milks.

❺Highest maximum dietary burden for broiler and layer suitable for estimation of maximum residue levels for poultry meat, fat, offal, and eggs.

❻Highest mean dietary burden for broiler and layer suitable for estimation of STMRs for poultry meat, fat, offal and eggs.

Animal commodity maximum residue levels.

Mammals

The highest maximum dietary burden of chlormequat cation for dairy cattle was 32.6 ppm and the highest mean dietary burden was 12.9 ppm.

	Feed level (ppm, cation)	Residue in milk (mg/kg as the cation)
MRL		
Feeding study	28	0.15
	93	0.26
Dietary burden (max) & highest residue	32.6	0.16
STMR		
Feeding study	28	0.15
Dietary burden (mean)	12.9	0.069

The Meeting estimated a maximum residue level of 0.2 mg/kg for milks to replace the previous recommendation of 0.3 mg/kg, and an STMR of 0.069 mg/kg for milks.

The highest maximum dietary burden of chlormequat cation for beef cattle was 37.4 ppm and the highest mean dietary burden was 18.1 ppm.

Table 19 Residues in tissues from cattle dosed with chlormequat in the diet

	Feed level (ppm, cation)	Residues (mg/kg, as chlormequat cation) Meat	Fat	Liver	Kidney
MRL					
Feeding study	28	0.085	0.040	0.078	0.36
	93	0.085 [a]	0.078	0.39	0.82
Dietary burden, HR	37.4	0.085	0.043	0.11	0.40
STMR					
Feeding study	28	< 0.04	< 0.04	0.062	0.31
Dietary burden, STMR	18.1	< 0.04	< 0.04	0.036	0.20

Notes:

[a] This value is from the dose level of 28 ppm as the cation. A higher highest residue was observed among the cows fed at this level than those fed 93 ppm as the cation.

The Meeting estimated maximum residues level of 0.2 mg/kg for meat from mammals other than marine mammals, confirming the previous recommendation, and an STMR and an HR of 0.04 and 0.085 mg/kg. respectively.

The Meeting estimated a maximum residue level of 0.1 mg/kg for mammalian fat, confirming the previous recommendation, and an STMR and an HR of 0.04 and 0.043 mg/kg, respectively.

Based on the residue data for kidney, higher than those for liver, the Meeting estimated a maximum residue level of 0.5 mg/kg for edible offal, mammalian, replacing the previous recommendation of 1 mg/kg. The Meeting estimated an STMR and an HR of 0.036 and 0.11 mg/kg for liver, and 0.20 and 0.40 mg/kg for kidney, respectively.

Poultry

The highest maximum dietary burden of chlormequat cation for poultry was 15.3 ppm and the highest mean dietary burden was 6.83 ppm. No residues of chlormequat cation above the LOQ (0.05 mg/kg as chlormequat chloride) were found at the highest feeding level of 46.5 ppm (as the cation) in meat or fat. Therefore, the Meeting estimated maximum residue levels of 0.04(*) mg/kg for poultry meat and fat confirming the previous recommendations. The Meeting estimated STMR and HR for meat and fat of 0.04 mg/kg.

Table 20 Residues in eggs and tissues from poultry dosed with chlormequat in the diet

	Feed level (ppm, cation)	Residues in liver (mg/kg, cation)	Residues in eggs (mg/kg, cation)
MRL			
Feeding study	14.0	0.078	0.093
	46.5	0.26	0.12
Dietary burden, HR	15.3	0.085	0.094
STMR			
Feeding study	4.65	0.04	< 0.04
	14	0.054	0.078
Dietary burden, STMR	6.83	0.043	0.049

The Meeting estimated a maximum residue level of 0.2 mg/kg for poultry edible offal, replacing the previous recommendation, and an STMR and an HR of 0.043 and 0.085 mg/kg respectively. The

Meeting also estimated a maximum residue level of 0.2 mg.kg for eggs, replacing the previous recommendation, and an STMR and an HR of 0.049 and 0.094 mg/kg respectively.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue (for compliance with the MRL and dietary exposure assessment) in plant and animal commodities: *chlormequat cation*.

The residue is not fat soluble.

Table 21 Maximum residue level recommendations for chlormequat

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
GC 0640	Barley	2	2	0.37	-
AS 0640	Barley, hay and/or straw	200 (dw)		Median: 34.5 (hay) 8.25 (straw) (ar)	Highest: 73 (hay) 32 (straw) (ar)
	Barley, straw and fodder, dry	W	50 (dw)		
MO 0105	Edible offal (mammalian)	0.5	1	0.036 (liver) 0.20 (kidney)	0.11 (liver) 0.40 (kidney)
PE 0269	Eggs	0.2	0.1	0.049	0.094
MF 0100	Mammalian fats (except milk fats)	0.1	0.1	0.04	0.043
MM 0095	Meat (from mammals other than marine mammals)	0.2	0.2	0.04 (muscle) 0.04 (fat)	0.085 (muscle) 0.043 (fat)
ML 0095	Milks	0.2	0.3	0.069	-
PF 0111	Poultry fats	0.04*	0.04*	0.04	0.04
PM 0111	Poultry meat	0.04*	0.04*	0.04 (muscle, fat)	0.04 (muscle, fat)
PO 0111	Poultry, edible offal of	0.2	0.1	0.043	0.085
GC 0654	Wheat	4	2	0.855	-
CM 0654	Wheat bran, unprocessed	10	7	2.3	-
AS 0654	Wheat, hay and/or straw	200 (dw)	80 (dw)	Median: 42.5 (hay) 20.5 (straw) (ar)	Highest: 117 (hay) 55 (straw) (ar)
CF 1210	Wheat germ	20	-	4.3	
CF 1211	Wheat, flour			0.16	
CF 1212	Wheat wholemeal			0.855	
	Wheat wholemeal bread			0.46	
CF 0640	Barley bran, processed			0.34	
CM 0640	Barley, pearled (pot barley)			0.12	
CF 3511	Barley, flour			0.066	
	Barley malt			0.33	
	Barley beer			0.074	

Notes:

(ar) – as received; (dw) – dry weight.

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The International Estimated Daily Intakes (IEDIs) of chlormequat cation were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting. The results are shown in Annex 3 of the 2022 Report.

The ADI for chlormequat chloride is 0–0.05 mg/kg bw/day (or 0–0.0388 mg/kg bw/day expressed as chlormequat cation). The calculated IEDIs for chlormequat cation were 1–20 percent of the maximum ADI for chlormequat expressed as cation. The Meeting concluded that the long-term dietary exposure to residues of chlormequat cation, when chlormequat chloride is used in accordance with GAPs that have been considered by JMPR, are unlikely to pose a public health concern.

Acute dietary exposure

The International Estimated Short-Term Intakes (IESTIs) of chlormequat cation were calculated for food commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting. The results are shown in Annex 4 to the 2022 Report.

The ARfD for chlormequat chloride is 0.05 mg/kg bw (or 0.0388 mg/kg bw expressed as chlormequat cation).

The calculated IESTIs for chlormequat ranged from 0–60 percent of the ARfD for children, and 0–30 percent for the general population. The Meeting concluded that the acute dietary exposure to residues of chlormequat cation, when chlormequat chloride is used in accordance with GAPs that have been considered by JMPR, are unlikely

REFERENCES

Reference No. (Study No.)	Author(s)	Year	Title, Date, etc.
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AA160702.	Hoi, Sio Wai	2017a	Magnitude of the Residue of Chlormequat Chloride in Wheat. American Agricultural Services, Inc. Cary, North Carolina. 09 August 2017 GLP, unpublished
AA160703	Hoi, Sio Wai	2017b	Magnitude of the Residue of Chlormequat Chloride in Barley. American Agricultural Services, Inc., Cary, North Carolina. 09 August 2017 GLP, unpublished

DIAZINON (022)

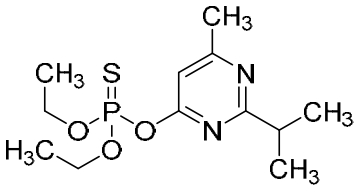
First draft prepared by Dr J Cudmore, Chemicals Regulation Division of the Health and Safety Executive, United Kingdom

EXPLANATION

Diazinon is a contact organophosphorus insecticide with a wide range of insecticidal activity. It is effective against sucking, chewing and boring insects, including soil-living insects. Diazinon has been evaluated on numerous occasions by the JMPR commencing in 1963. The most recent periodic review was in 1993. Following public health concerns identified by the International Agency for Research on Cancer (IARC), the JMPR in 2016 evaluated all previously considered toxicological data in addition to new studies. The 2016 JMPR recommended an ADI of 0–0.003 mg/kg bw and an ARfD of 0.03 mg/kg bw. Diazinon was scheduled at the Fifty-first Session of the CCPR (2019) for Periodic Review for residues by the 2020 JMPR and re-scheduled for the 2022 JMPR.

The Meeting received information from the manufacturer on physical and chemical properties, animal and plant metabolism, rotational crop studies, environmental fate in soil, analytical methods, storage stability, use patterns, supervised residue trials, processing studies and livestock feeding studies.

IDENTITY

ISO Common Name	Diazinon	
Chemical name	IUPAC:	*O,O*-diethyl-*O*-(2-isopropyl-6-methylpyrimidin-4-yl)phosphorothioate
	CAS:	*O,O*-diethyl-*O*-[6-methyl-2-(1-methylethyl)-4-pyrimidinyl]phosphorothioate
CIPAC No.	15	
CAS No	333-41-5	
Structural formula		
Molecular formula	$C_{12}H_{21}N_2O_3PS$	
Molecular mass	304.34 g/mol	

PHYSICAL AND CHEMICAL PROPERTIES

The physical and chemical properties of diazinon are summarised in Table 1.

Table 1 Physical and chemical properties of the pure and technical grade diazinon

Property	Results	Test material purity and specification	Reference
Appearance and odour	Pale yellow liquid at 20 °C Faint organophosphate odour	Pure (99.3%)	R-14219
	Yellow liquid at 20 °C	Technical (96%)	R-14213
Freezing point	<-25 °C	Pure (99.3%)	R-14219
Vapour pressure	1.197×10^{-2} Pa at 25 °C	Pure (99.4%)	R-2093
Volatility	Henry's Law constant = 6.1×10^{-2} Pa m^3 mol^{-1}	Not specified	R-2251
Octanol/water partition coefficient	Log P_{ow} = 3.30 at 25 °C	Pure, radiolabelled (99% radiochemical purity)	R-2095
	Log P_{ow} = 3.81 at 25 °C, pH 6.58	Pure (97.8%)	R-4599
	Log P_{ow} = 3.69 at 24 °C	Technical (96.3%)	R-6502
Solubility in water	0.0655 g/L in water at 25 °C	Technical (purity not stated)	R-6500
	0.060 g/L at pH 7 and 22 °C	Pure (99.4%)	R-2094
	0.0595 g/L in pure water and 25 °C. Solubility in water is not pH-dependent at pH 5, 7 and 9.	Pure (97.8%)	R-4597
Surface tension	49.5 mN/m (90 percent saturated solution)	Pure (99.3%)	R-14219
Solubility in organic solvents	>9000 g/L in acetonitrile, acetone, methanol, carbon tetrachloride, heptane, toluene and n-octanol at 25 °C	Technical (93%)	R-4628
	>2000 g/L in methanol and hexane at 25 °C	Technical	R-6500
Explosive properties	No thermal or mechanical (shock) sensitivity exhibited. Not explosive.	Technical (96%)	R-14213
Hydrolysis in water	pH / Temp. (° C) / K_{obs} ($days^{-1}$) / DT_{50} (days)	Not specified	R-228
	5.0 / 30 / 4.56×10^{-6} / 1.8		
	7.0 / 30 / 3.43×10^{-7} / 23		
	9.0 / 30 / 6.84×10^{-7} / 12		
	5.0 / 50 / 1.46×10^{-5} / 0.5		
	7.0 / 50 / 2.55×10^{-6} / 3.2		
	9.0 / 50 / 4.98×10^{-6} / 1.6		
	5.0 / 70 / 5.52×10^{-5} / 0.15		
	7.0 / 70 / 1.88×10^{-5} / 0.4		
	9.0 / 70 / 3.75×10^{-5} / 0.2		
	Hydrolytically stable at neutral pH. Hydrolysis occurs under acidic and alkaline conditions. Major hydrolysis products are parent, G-27550 and an unknown.	Pure, radiolabelled (96.1 percent radiochemical purity)	R-231
	pH / Temp. (° C) / K_{obs} ($days^{-1}$) / DT_{50} (days)		
	5 / 25 / 5.6×10^{-2} / 12		
	7 / 25 / 5.0×10^{-3} / 138		

Property	Results				Test material purity and specification	Reference
	9		9×10^{-3}	77		
Aqueous photolysis	Photolysis product: G-27550 (average over 2 solvent systems)				Pure, radiolabelled (99% radiochemical purity)	G24480/0239
	pH	Temp. (° C)	K_{obs} ($hours^{-1}$)	DT_{50} (days)		
	7	12-49	1.18×10^{-3}	25		
Photo-transformation	Upper limit of the quantum efficiency of direct photochemical transformation = ≤0.3 Real lifetime $\tau = 2.09 \times 10^7$ s Estimation of photochemical half-life in aqueous solution: $t_{1/2}$ = ≥ 0.2×10^4 (1 cm distilled water) $t_{1/2}$ = ≥ 0.1×10^6 (100 cm river (Neckar) water)				Pure (99.4%)	G24480/2088
Stability in water	Stable over 5 days at 21.7 µg/mL				Technical (purity not stated)	R-6500
Stability in air	$t_{1/2}$ = 1.3-1.5 hours (calculated, Atkinson method)				Not specified	R-2376
Boiling point	212 °C Starts to decomposes at >140 °C				Technical (purity not stated)	R-6498
Relative Density	1.11				Technical (not stated)	R-6492
	1.11				Technical (not stated)	R-6499
Auto-ignition temperature	Not found <400 °C				Technical (96%)	R-14213
Flash point	92 °C				Technical (96%)	R-14213
Dissociation constant	Data is consistent with the protonation of a heteroaromatic nitrogen at low pH values. The compound is a weak base.				Pure (99.3%)	R-14219
	pK_a = 2.60 at 20 °C				Pure (purity not stated)	R-2092
Photochemical oxidative degradation	Half-life = 1.3 hours in air				Pure (99.3%)	R-14219
Spectra of active substance	UV-vis (cyclohexane solution), IR, ^{1}H-NMR spectroscopy and mass spectrometry (electron impact) performed. λ_{max} = 246 nm, ε = 4050 L/mol/cm				Pure (99.3%)	R-14219
	λ = 290 nm, ε = 20.86 L/mol/cm				Pure (99.4%)	G24480/2088
Impurity spectra (Potentially present in the technical material)	UV-vis, IR, ^{1}H-NMR spectroscopy and mass spectrometry (electron impact) performed. UV-vis characteristics:				*O,O,O′,O′*-tetraethyl-thiopyrophosphate Purity = (98.1%)	R-14365
	Solvent	λ (nm)	ε (L/mol/cm)			
	Acetonitrile:H_2O (9:1, v/v)	276	15.0			

Property	Results			Test material purity and specification	Reference
	Acetonitrile: 1 M HCl (9:1, v/v)	275	6.4		
	Acetonitrile: 1 M NH_3 (9:1, v/v)	274	26.1		
	UV-vis, IR, ^{1}H-NMR spectroscopy and mass spectrometry (electron impact) performed. UV characteristics:			*O,O,O',O'*-tetraethyl-dithiopyrophosphate (S,S-TEPP) Purity = (98%)	R-14366
	Solvent	λ (nm)	ε (L/mol/cm)		
	Acetonitrile:H_2O (9:1, v/v)	280	19.7		
	Acetonitrile: 1 M HCl (9:1, v/v)	281	2.77		
	Acetonitrile: 1 M NH_3 (9:1, v/v)	248	68.5		
		277	51.0		

Formulation

Formulations of diazinon are available as wettable powders, emulsion, oil in water and emulsion concentrates.

Formulation type	Active substance content	Application type
WP (Wettable Powder)	400 g/kg	Foliar applications
EW (Emulsion, oil in water)	500 g/L	Foliar applications
EC (Emulsion concentrate)	600 g/L	Foliar applications

METABOLISM AND ENVIRONMENTAL FATE

Radiolabel Position

The fate and behaviour of diazinon in animals, plants and the environment were investigated using ^{14}C - diazinon labelled in the pyrimidine ring as shown in Figure 1.

^{14}C-labelled diazinon

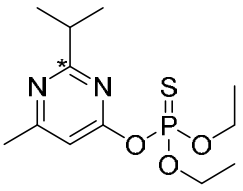
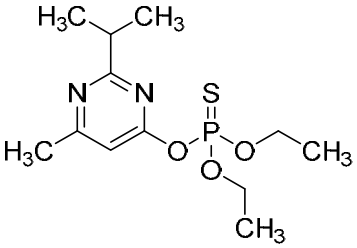
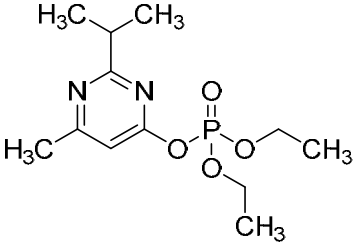
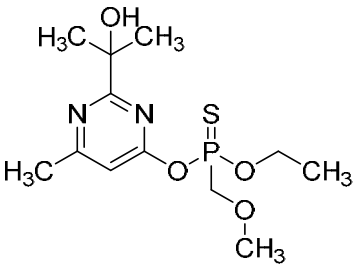
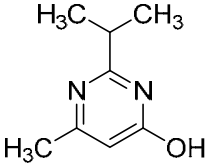
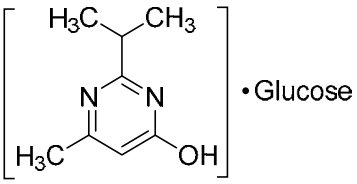
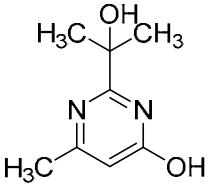
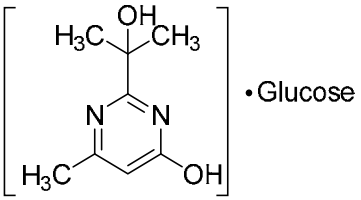
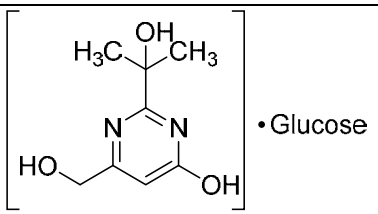
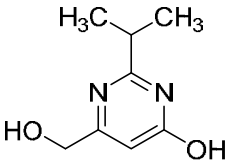
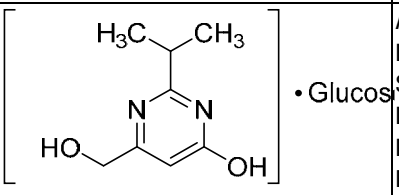
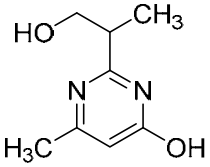
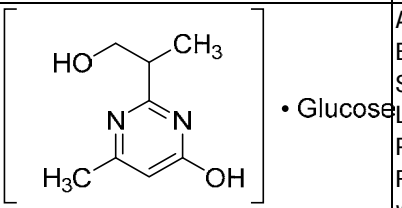
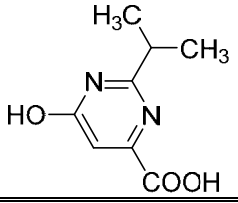
^{14}C -diazinon pyrimidine ring	 * position of ^{14}C radiolabel

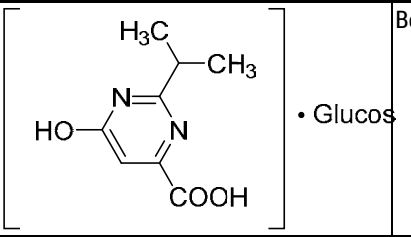
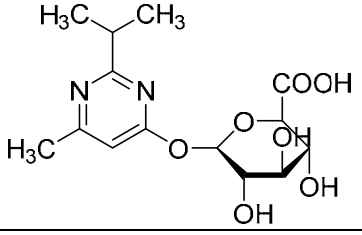
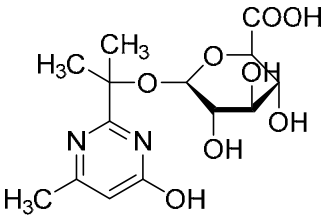
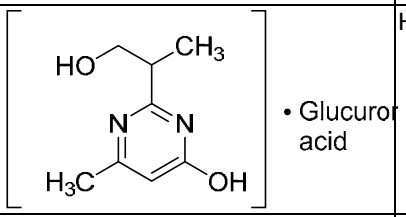
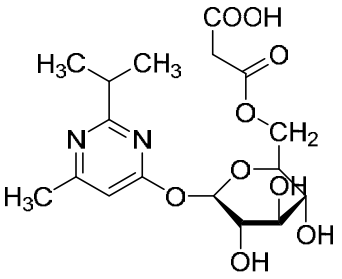
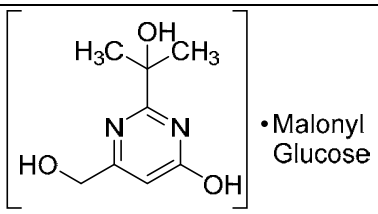
Figure 1 [^{14}C]-labelled test material used in animal metabolism, plant metabolism and environmental fate studies

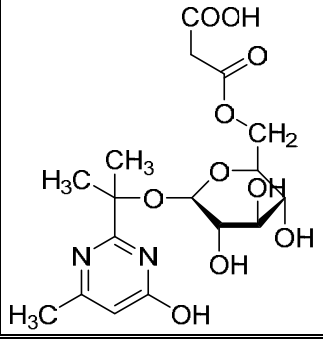
The chemical structures and code names of the major degradation compounds from the metabolism of diazinon are summarized in Table 2.

Table 2 Structure of compounds and code names appearing in metabolism and environmental fate studies

Name/Code	Chemical name	Chemical structure	Occurrence in metabolism studies
Diazinon	*O,O*-diethyl-*O*-(2-isopropyl-6-methyl-4-pyrimidinyl)-phosphorothioate		Apple Beans Sweet corn Lettuce Potatoes Goat Hen Rotated spring wheat
G-24576 (diazoxon)	*O,O*-diethyl-*O*-(2-isopropyl-6-methyl-4-pyrimidinyl) phosphorate		Goat Hen
CGA-14128 (hydroxydiazinon)	*O,O*-diethyl-*O*-(2-[2-hydroxy-2-isopropyl]-6-methyl-4-pyrimidinyl)phosphorothioate		Goat Hen
G-27550 B_1	6-methyl-2-(1-methyl-ethyl)-4-pyrimidinol		Apple Beans Sweet corn Lettuce Potatoes Goat Hen Rotated Spring wheat
Glucose conjugate of G-27550	6-methyl-2-(1-methyl-ethyl)-4-pyrimidinol glucose conjugate		Bean, vines only
GS-31144 C	2-(2-hydroxyisopropyl)-6-methyl-4-pyrimidinol		Apple Beans Sweet corn Lettuce Potatoes Goat Hen Rotated spring

Name/Code	Chemical name	Chemical structure	Occurrence in metabolism studies
			wheat
Glucose conjugate of GS-31144 E_2	2-(2-hydroxyisopropyl)-6-methyl-4- pyrimidinol glucose conjugate		Apple Beans Sweet corn Lettuce Potatoes
Two glucose conjugates of trihydroxy pyrimidinyl moiety† G and H	2-(2-hydroxyisopropyl)-6-hydroxymethyl-4- pyrimidinol glucose conjugate		Apple Beans Sweet corn Lettuce Potatoes Rotated spring wheat
JAK-III-57 D	2-(1-methylethyl)-6-hydroxymethyl-4- pyrimidinol		Apple Beans Sweet corn Lettuce Potatoes Rotated spring wheat
Glucose conjugate of JAK-III-57 F_2	2-(1-methylethyl)-6-hydroxymethyl-4- pyrimidinol glucose conjugate		Apple Beans Sweet corn Lettuce Potatoes Rotated spring wheat
CL-XIX-29 E_1 (also referred to as M3 in some studies)	2-(1-hydroxypropan-2-yl)-6-methyl-4-pyrimidinol		Apple Beans Sweet corn Lettuce Potatoes Hen Rotated spring wheat
Glucose conjugate of CL-XIX-29 F1	2-(1-hydroxypropan-2-yl)-6-methyl-4-pyrimidinol glucose conjugate		Apple Beans Sweet corn Lettuce Potatoes Rotated spring wheat
JAK-IV-23	2-isopropyl-6-oxo-1,6-dihydropyrimidine-4-carboxylic acid		Beans

Name/Code	Chemical name	Chemical structure	Occurrence in metabolism studies
Glucose conjugate of JAK-IV-23	2-isopropyl-6-oxo-1,6-dihydropyrimidine-4-carboxylic acid glucose conjugate		Beans
Glucuronic acid conjugates of G-27550	6-methyl-2-(1-methyl-ethyl)-4-pyrimidinol glucuronic acid conjugate		Hen
Glucuronic acid conjugates of GS-31144	2-(2-hydroxyisopropyl)-6-methyl-4- pyrimidinol glucuronic acid conjugate		Hen
Glucuronic acid conjugates of CL-XIX-29	2-(1-hydroxypropan-2-yl)-6-methyl-4-pyrimidinol malonyl glucuronic acid conjugate		Hen
A conjugate of G-27550§ (postulated to be a malonyl glucose conjugate)	6-methyl-2-(1-methyl-ethyl)-4-pyrimidinol malonyl glucose conjugate		Beans Potatoes Lettuce
A conjugate of JAK-III-57§ (postulated to be a malonyl glucose conjugate)	2-(1-methylethyl)-6-hydroxymethyl-4- pyrimidinol malonyl glucose conjugate		Beans Potatoes Lettuce

Name/Code	Chemical name	Chemical structure	Occurrence in metabolism studies
A conjugate of GS-31144§ (postulated to be a malonyl glucose conjugate)	2-(2-hydroxyisopropyl)-6-methyl-4-pyrimidinol malonyl glucose conjugate		Beans Potatoes Lettuce

Notes:

† The structure of the two metabolites G and H were not fully elucidated. The mass spectral analysis did not confirm the positions of the hydroxyl groups. The metabolites were susceptible to hydrolysis with β-glucosidase but no identification work was undertaken on the aglycone.

§ The identity of the conjugates were not established. The aglycones released from enzymatic or acid hydrolysis were confirmed.

Plant metabolism

The meeting received information on metabolism of diazinon in apples, green beans, sweet corn, lettuce and potatoes.

Apples (Study ABR-89058)

Apple trees (variety Empire hybrid) grown outdoors in a loam soil were treated with [^{14}C]-pyrimidine-diazinon (specific activity = 9.8 µCi/mg, radiochemical purity 98 percent).

The test material was formulated as a WG formulation and applied three times. The first application was made when the buds were in the early tight cluster stage (approximately BBCH 55) at a rate of 3.36 kg ai/ha. This application was split between a soil application (3.024 kg ai/ha) and a foliar application to a single branch (0.336 kg ai/ha). The second and third applications were foliar applications to the same branch made when the apples were 5 and 7.5 cm in diameter at a rate of 10.09 kg ai/ha. The second application was made 104 days after the first application and the third application was made 29 days later.

Mature apples were harvested 14 days after the last treatment. Foliage samples were also collected. All samples were stored frozen at ≤ -15 °C for up to 15 months prior to analysis. The total radioactivity in the samples was determined by combustion with LSC. Leaves, peel and pulp were extracted twice with methanol: Water (9:1,v/v) and the extracts were then partitioned with ethyl acetate. Characterisation and identification of the organosoluble and aqueous extracts was performed using 2D-TLC and/or HPLC analysis with mass spectral identification on specific metabolites. Extracts were also treated with ß-glucosidase for 12 hours at 37 °C to support the identification of the glucose conjugates.

The total radioactive residues and the distribution of radioactivity in apples are shown in Table 3. The TRR ranged from 0.126 mg eq/kg (apple pulp) to 51.1 mg eq/kg (apple leaves). The TRR was higher in apple peel (3.44 mg eq/kg) compared to apple pulp (0.126 mg eq/kg).

Solvent extractabilities with methanol: water (9: 1, v/v) ranged from 89.7 percent TRR for peel to 91.9 percentTRR for leaves. The majority of the extracted residue was found to be organosoluble.

Table 3 TRR and distribution of radioactivity in apple treated with ^{14}C-diazinon

Fraction	Apple leaves		Whole apple		Apple peel		Apple pulp	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR by combustion	100	51.1	100	1.29	100	3.44	100	0.126
Methanol: water (9:1, v/v)	91.9	46.9	-	-	89.7	3.09	91.0	0.115
Aqueous phase	36.3	18.5	-	-	8.26	0.284	37	0.0467
Organic phase	55.6	28.4	-	-	81.7	2.81	54.0	0.0680
Non-extracted	8.1	4.14	-	-	10.4	0.358	9.0	0.011

The identification/characterization of residues in apple crop fractions are outlined in table 4.

The non-extracted residue ranged from 8.1 percent TRR (4.1 mg eq/kg) for leaves to 11.6 percent TRR (0.15 mg eq/kg) for whole apple.

Table 4 Identification/characterization of radioactivity in apples

Fraction	Apple leaves		Apple Peel		Apple pulp		Whole apple†	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR by combustion	100	51.1	100	3.44	100	0.126	100	1.29
Total extracted	91.9	46.9	89.7	3.09	91.0	0.115	N/A	N/A
Diazinon (A)	43.7	22.3	73.3	2.5	16.1	0.02	69.0	0.89
G-27550 (B_1)	5.9	3.0	11.9	0.4	60.7	0.08	14.7	0.19
GS-31144 (C)	2.4	1.2	0.3	0.01	2.6	0.003	0.39	0.005
JAK-111-57 (D)	-	-	0.1	0.003	0.5	0.001	0.16	0.002
CL-XIX-29 (E_1)	-	-	0.6	0.02	0.8	0.001	0.54	0.007
Glucose conjugate of GS-31144 (E_2)	8.3	4.2						
Glucose conjugate of CL-XIX-29 (F_1)	1.6	0.8	0.7	0.02	3.9	0.005	0.78	0.01
Glucose conjugate of JAK-111-57 (F_2)	2.8	1.4						
Glucose conjugate of Trihydroxy pyrimidinyl moiety (G)	4.2	2.1	1.6	0.06	1.3	0.002	1.6	0.02
Glucose conjugate of Trihydroxy pyrimidinyl moiety (H)	3.5	1.8						
Unknown	3.2	1.6	0.1	0.003	-	-	0.078	0.001
B_1 (G-27550) and metabolite B_2 §	2.1	1.1	0.4	0.01	0.7	0.01	0.31	0.004
Aqueous metabolites not identified #	5.5	2.8	1.0	0.03	2.3	0.003	0.78	0.01
Total identified**	72.4	36.8	88.5	3.01	85.9	0.112	87.2	1.12
Non-extracted	8.1	4.1	10.4	0.4	9.0	0.01	11.6	0.15

Notes:

† The values for whole apples were estimated by multiplying the mg/kg values in the peel by 0.351 and mg/kg values in the flesh by 0.649 and summing the two values. The percent TRR values have been calculated from these mg/kg estimates.

§ B2 is an unidentified metabolite.

No information is given in the study on the number of individual metabolites found in this fraction.

**Includes metabolites that have been quantified together. It does not include fractions that contain identified and unidentified metabolites.

Beans with pods (study ABR-90040)

Beans with pods (variety provider) grown outdoor in a loam soil were treated with [^{14}C]-pyrimidine-diazinon (specific activity = 9.8 µCi/mg, radiochemical purity 98 percent).

The test material was formulated as an EC formulation and applied three times. The first application was made at a rate of 4.48 kg ai/ha applied pre-emergence 1 day after sowing. A further two foliar applications were made at a rate of 1.4 kg ai/ha. The first foliar application was made 34 days after the pre-emergence use and the second foliar application was made 15 days later.

A sample of vines was taken 31 days after the pre-emergence application (i.e. 3 days before the first foliar application). A sample of vines with beans was taken 7 days after the first foliar application. Samples of beans with pods and vines were taken 14 days after the last application, which is stated to represent crop maturity. The growth stages are not stated.

Samples were stored frozen at ≤ -15 °C for up to 21 months prior to analysis.

The total radioactivity in the samples was determined by combustion with LSC. Vines and beans were extracted twice with methanol: Water (9:1, v/v) and the extracts were then partitioned with ethyl acetate. Characterization and identification of the organosoluble and aqueous extracts was performed using 2D-TLC and/or HPLC analysis with mass spectral identification on specific metabolites. Proton NMR was used to assist in the structural elucidation of metabolite E_2. Extracts were also treated with ß-glucosidase for 12 hours at 37 °C to support the identification of the glucose conjugates.

The total radioactive residues and the distribution of radioactivity in beans are shown in Table 5.

The TRR ranged from 0.425 mg eq/kg (vines harvested 32 DAFT) to 3.53 mg eq/kg (vines harvested 14 DALA). The solvent extractabilities with methanol: water (9:1, v/v) ranged from 54.3 percent TRR for vines harvested 32 DAFT to 89.2 percent TRR for vines with beans. The majority of the extracted radioactivity was found to be aqueous soluble.

Table 5 TRR and distribution of radioactivity in beans treated with ^{14}C-diazinon

Fraction	Vines harvested 32 DAFT		Vines and beans harvested 41 DAFT		Beans with pods harvested 14 DALA		Vines harvested 14 DALA	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR by combustion	100	0.425	100	4.45	100	0.456	100	3.53
Methanol: water (9:1, v/v)	54.3	0.231	89.2	3.96	76.1	0.347	81.9	2.89
Aqueous phase	47.1	0.200	54.7	2.43	54.0	0.246	75.9	2.68
Organic phase	7.17	0.0305	34.4	1.53	22.1	0.100	6.06	0.214
Non-extracted	45.7	0.194	10.8	0.480	23.9	0.109	18.1	0.639

Notes:

DAFT: Days after first treatment.

DALA: Days after last treatment.

The identification/characterization of residues in the crop fractions are outlined in Table 6.

The identification of metabolites in the vines and beans harvested 41 days after sowing was not reported. It is stated that the identification/characterization was unsuccessful, and this was attributed to co-extraction of sugars and multiple overlapping of components in the HPLC analysis.

Diazinon was not found in the vine samples. In beans, harvested 14 DALA, diazinon was found at 2.1 percent TRR (0.01 mg/kg). In a number of cases the individual levels of the metabolites were not determined.

For vines, harvested 32 DAFT, the highest TRR was 17.3 percent TRR (0.07 mg eq/kg) which was a mixture of two glucose conjugates and an unknown metabolite. The metabolites CL-XIX-29 and a glucose conjugate of GS-31144 occurred at 15.5 percent TRR (0.07 mg eq/kg). All other metabolites identified occurred at levels below 10 percent TRR.

In vines, harvested 14 DALA, the metabolites CL-XIX-29 and a glucose conjugate of GS-31144 were found at a level of 27.5 percent TRR (0.97 mg eq/kg), an unknown metabolite occurred at 19.5 percent TRR (0.69 mg eq/kg) and two glucose conjugates were found at a level of 12.5 percent TRR (0.44 mg eq/kg). All other metabolites identified were < 10 percent TRR.

In beans, harvested 14 DALA, the major metabolites/fractions were G-27550 (26.7 percent TRR, 0.12 mg eq/kg) and a mixture of two glucose conjugates of the trihydroxy pyrimidinyl moiety and an unknown metabolite (19.5 percent TRR, 0.09 mg eq/kg). All other metabolites identified were < 10 percent of the TRR.

Table 6 Identification/characterization of radioactivity in beans with pods

	Vines harvested 32 DAFT		Beans with pods harvested 14 DALA		Vines harvested 14 DALA	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR by combustion	100	0.425	100	0.456	100	3.53
Total extracted	54.3	0.231	76.1	0.347	81.9	2.89
Diazinon (A)	-	-	2.1	0.01	-	-
G-27550 (B_1)	5.4	0.02	26.7	0.12	5.5	0.19
GS-31144 (C)	9.8	0.04	6.0	0.03	7.0	0.25
JAK-111-57 (D)	1.5	0.01	4.4	0.02	2.5	0.09
CL-XIX-29 (E_1)	15.5	0.07	6.7	0.03	27.5	0.97
Glucose conjugate of GS-31144 (E_2)						
Glucose conjugate of CL-XIX-29 (F_1)	4.8	0.02	7.0	0.03	12.5	0.44
Glucose conjugate of JAK-111-57 (F_2)	2.6	0.01				
Glucose conjugate of a Trihydroxy pyrimidinyl moiety (G)	17.3	0.07	19.5	0.09	2.6	0.09
Glucose conjugate of a Trihydroxy pyrimidinyl moiety (H)						
Unknown (I)					19.5	0.69
Glucose conjugate of G-27550	-	-	-	-	0.2	0.01
TLC unknown metabolite	-	-	2.9	0.01	-	-
Unknown metabolites#	0.0	< 0.01	1.3	< 0.01	2.8	0.11
Total Identified**	39.6	0.17	52.9	0.24	57.8	2
Non-extracted	45.7	0.194	23.9	0.109	18.1	0.639

Notes:

DAFT: Days after first treatment.

DALA: Days after last treatment.

Number of individual metabolites not reported.

** Includes metabolites that have been quantified together.

In a supplementary study, retained crop fractions of beans with pods (14 DALA) and vines (14 DALA) were subject to analysis after 69 months of freezer storage. The total radioactivity in the samples was determined by combustion with LSC. The samples were extracted with methanol: water (9: 1, v/v) and partitioned with ethyl acetate.

The HPLC profiles of a mature bean sample and a mature vine sample, extracted after 19 months of storage, were compared to the HPLC profiles of a mature bean sample and mature vine sample extracted after 69 months of storage. The HPLC profiles of the extracts from 19 months of storage compared to 69 months of storage were qualitatively similar. For the vine samples, the percent TRR of the main fractions were quantitatively similar. For the bean samples, the percent TRR of the main fractions were quantitatively similar, except the region containing the glucose conjugates of CL-XIX-29 and JAK-111-57 was no longer present.

The TRR determined by combustion and the solvent extractabilities for the bean samples extracted after 69 months of storage are shown in Table 7.

Table 7 TRR and distribution of radioactivity in beans treated with ^{14}C-diazinon

Fraction	Beans with pods harvested 14 DALA		Vines harvested 14 DALA	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR	100	0.509	100	3.755
Methanol: water (9:1, v/v)	74.9	0.381	78.2	2.94
Aqueous phase	50.1	0.255	68.4	2.57
Organic phase	24.8	0.126	9.78	0.367
Non-extracted	25.1	0.128	21.8	0.819

Notes:

DALA: Days after last treatment.

After extraction with methanol: water and partitioning with ethyl acetate, the aqueous fractions were subject to sample clean-up, derivatization and enzyme hydrolysis steps in order to aid the identification of residues. Separate fractions were treated with cellulase (12 hours at 37 °C), ß-glucosidase (12 hours at 37 °C), HCl (6 M HCl for 1 hour at 95 °C), acetic anhydride: pyridine (90: 10 v/v, room temperature for 12 hours) and 3 M HCl in butanol (1 hour at 95 °C). Metabolites were identified using TLC, HPLC, MS and H-NMR.

The metabolites in mature beans and vines were identified as G-27550, which co-chromatographed with an unknown metabolite (B_2), GS-31144, JAK-III-57, CL-XIX-29 and various conjugates. The results are summarized in Table 8.

In mature beans, the main fraction represented 20.3 percent TRR. This fraction contained conjugates of JAK-IV-23, JAK-III-57, G-27550 and GS-31144 as well as JAK-IV-23 and two unknown metabolites. The individual levels were not stated. Treatment with cellulase released JAK-IV-23 and the

acid hydrolysis treatment released GS-31144 and G-27550. Based on the characterization (derivatization via acetylation) and a comparison to descriptions in the literature, it was postulated that the conjugates of JAK-IV-23, GS-31144 and G-27550 were malonyl glucose conjugates. No further details were provided.

All other fractions in mature beans were present at levels not exceeding 9.3 percent TRR (0.047 mg eq/kg).

In vines, the main fraction represented 27 percent TRR. The two metabolites identified in this fraction were CL-XIX-29 and a conjugate of GS-31144. Two further fractions accounted for over 10 percent of the TRR. A fraction of 17.4 percent TRR, that contained conjugates of JAK-IV-23, JAK-III-57, G-27550 and GS-31144 as well as two unknowns, and a fraction of 10.9 percent TRR that contained a conjugate of CL-XIX-29 and a conjugate of JAK-III-57.

All other fractions for the vines were present at levels not exceeding 6.4 percent TRR (0.239 mg eq/kg).

Table 8 Characterization and identification of aqueous soluble metabolites in green beans with pods and vines

	Beans with pods harvested 14 DALA				Vines harvested 14 DALA			
	0.509 mg/kg†				3.755 mg/kg†			
	Percent in aqueous fraction	Percent in extracted residue	Percent TRR	mg eq/kg	Percent in aqueous fraction	Percent in extracted residue	Percent TRR	mg eq/kg
G-27550 (B_1) Unknown (B_2)	18.5	12.4	9.3	0.047	4.0	3.5	2.7	0.103
GS-31144 (C)	9.2	6.1	4.6	0.023	6.0	5.2	4.1	0.154
JAK-III-57 (D)	13.1	8.8	6.6	0.033	9.3	8.1	6.4	0.239
CL-XIX-29 (E_1) Glucose conjugate of GS-31144 (E_2)	13.8	9.3	6.9	0.035	39.4	34.5	27.0	1.013
Glucose conjugate of CL-XIX-29 (F_1) Glucose conjugate of JAK-III-57 (F_2)	4.9	3.3	2.5	0.013	15.9	13.9	10.9	0.408
JAK-IV-23 (free and conjugated), Glucose conjugate of a Trihydroxy pyrimidinyl moiety Conjugate of G-27550 §, Conjugate of JAK-III-57 §, Conjugate of GS-31144 §, 2 unknowns #	40.4	27.0	20.3	0.103	25.4	22.2	17.4	0.652
Total identified**	-	-	29.9	0.151	-	-	51.1	1.917

Notes:

DAFT: Days after first treatment.

DALA: Days after last treatment.

†The characterization and identification work was undertaken on an additional subsample of each matrix.

The individual levels, including of the two unknowns, are not stated.

**Includes metabolites that have been quantified together. It does not include fractions that contain identified and unidentified metabolites.

§ Aglycones confirmed but the identity of the conjugate only postulated to be malonyl glucose conjugate.

In addition, the post-extraction solids from beans with pods were subjected to further extraction procedures (methanol/water reflux, NaCl reflux, enzyme hydrolysis). The results are summarized in Table 9.

Table 9 Summary of the radioactivity following treatment of the PES of Beans with pods harvested 14 DALA

Fraction	Percent TRR	mg eq/kg
Initial PES	25.1	0.128†
9: 1 v/v MeOH/H_2O	2.1	0.011
Organic	0.3	0.001
Aqueous	0.6	0.003
1 percent NaCl	3.0	0.015
Organic	0.5	0.003
Aqueous	1.8	0.009
Cellulase hydrolysis	8.5	0.044
Organic	0.1	0.000
Aqueous	6.7	0.034
Protease hydrolysis	2.9	0.015
Final PES	6.4	0.033

Notes:

†The characterization and identification work was undertaken on an additional subsample of green beans.

The extraction procedures individually released 2.1–8.5 percent TRR (0.011–0.044 mg eq/kg), leaving 6.4 percent TRR (0.033 mg eq/kg) in the final post-extraction solids.

The methanol/water and NaCl organosoluble fractions were analysed by 2D-TLC. In the methanol/water organosoluble fraction, parent diazinon and G-27550 were present as the major metabolites, whilst the aqueous fraction comprised of polar metabolites. The NaCl organosoluble fraction was found to contain G-27550 and GS-31144 as the major components. The individual metabolite levels were not quantified and the solubilised radioactivity following enzyme hydrolysis was too viscous for chromatographic profiling.

Sweet corn (Study ABR-89057)

Sweet corn (variety not stated) grown in a greenhouse was treated with [^{14}C]-pyrimidine-diazinon (specific activity = 9.8 µCi/mg, radiochemical purity 98 percent).

The test material was formulated as an EC formulation and applied three times. The first application was made pre-emergence at a rate of 4.48 kg ai/ha applied the day of sowing. The second and third applications were foliar applications at a rate of 3.5 kg ai/ha. The second application was made 50 days after the pre-emergence application and the third application was made 24 days later.

Samples of crop fractions were taken 72 DAFT (days after first treatment) i.e. 2 days before the second foliar application. Further crop fractions were taken 14 days after the last application. The growth stages are not stated.

The samples were stored frozen for up to 18 months prior to analysis.

The total radioactivity in the samples was determined by combustion with LSC. Samples were extracted twice with methanol: Water (9:1, v/v) and the extracts were then partitioned with ethyl acetate. Extracts from the mature forage samples were also treated with ß-glucosidase for 12 hours at 37 °C to support the identification of the glucose conjugates extracted. PES were also subjected to enzymatic hydrolysis; a sample was incubated at 37 °C with sodium acetate buffer with amyloglucosidase.

Characterization and identification was performed using 2D-TLC and/or HPLC analysis with mass spectral identification on specific metabolites. The total radioactive residues and the distribution of radioactivity in sweet corn fractions are shown in Table 10.

The TRR ranged from 0.087 mg eq/kg for the ears harvested 72 DAFT to 3.89 mg eq/kg for forage harvested 14 DALA. The solvent extractabilities with methanol: water (9:1, v/v) ranged from 20.3 percent TRR for stalks harvested 72 DAFT to 74.5 percent TRR for forage harvested 14 DALA. The majority of the radioactivity was shown to be aqueous soluble.

Table 10 TRR and distribution of radioactivity in sweet corn fractions treated with ^{14}C-diazinon

Fraction	Forage sampled 72 DAFT		Ears sampled 72 DAFT		Stalks sampled 72 DAFT	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR by combustion	100	2.070	100	0.087	100	0.810
Methanol: water (9:1, v/v)	69.1	1.43	59.4	0.0517	20.3	0.164
Aqueous phase	56.9	1.18	55.2	0.0480	16.7	0.135
Organic phase	12.0	0.248	4.04	0.00352	3.57	0.0289
Non-extracted	30.9	0.640	40.6	0.0353	79.7	0.646

	Forage sampled 14 DALA		Cob sampled 14 DALA		Grain sampled 14 DALA	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR	100	3.891	100	0.250	100	0.453
Methanol: water (9:1, v/v)	74.5	2.90	47.5	0.119	26.4	0.120
Aqueous phase	57.5	2.24	44.3	0.111	25.0	0.113
Organic phase	17.0	0.661	3.18	0.0795	1.40	0.00634
Non-extracted	25.5	0.992	52.5	0.131	73.6	0.333
Amyloglucosidase	-	-	-	-	58.9	0.267

Notes:

DAFT: Days after first treatment.

DALA: Days after last application.

The identification/characterization of residues in the organosoluble fractions are shown in Table 11.

Table 11 Identification/characterization of radioactivity in the organosoluble residues in sweet corn fractions

Fraction	Forage sampled 72 DAFT		Stalks sampled 72 DAFT	
	Percent TRR	mg eq/ kg	Percent TRR	mg eq/ kg
TRR by combustion	100	2.07	100	0.81
Total extracted	69.1	1.43	20.3	0.164
Diazinon (A)	0.5	0.0104	-	-
G-27550 (B1)	7	0.145	7.7	0.0624
GS-31144 (C)	0.7	0.0145	1.3	0.0105
JAK-111-57 (D)	1.3	0.0269	1.1	0.009
Unknown (I)	-	-	0.6	0.005
Unknown (II)	0.4	0.008	1.5	0.0122
Unknown (III)	0.4	0.008	-	-
Total identified	9.5	0.2	10.1	0.0819
Total non-extracted	30.9	0.64	79.7	0.646

	Forage sampled 14 DALA		Cob sampled 14 DALA		Grain sampled 14 DALA	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR by combustion	100	3.891	100	0.250	100	0.453
Total extracted	74.5	2.90	47.5	0.119	26.4	0.120
Diazinon (A)	1.8	0.07	-	-	-	-
G-27550 (B_1)	10.8	0.42	1.6	0.004	0.9	0.004
GS-31144 (C)	1.2	0.0467	1.2	0.003	0.1	0.0005
JAK-111-57 (D)	1.9	0.0739	1.2	0.003	0.1	0.0005
Unknown (I)	-	-	-	-	-	-
Unknown (II)	0.3	0.0117	1.2	0.003	0.1	0.0005
Unknown (III)	-	-	2	0.005	0.1	0.0005
Unknown (IV)	-	-				
Total Identified	15.7	0.611	4	0.01	1.1	0.005
Total non-extracted	25.5	0.992	52.5	0.131	73.6	0.333
Amyloglucosidase	-	-	-	-	58.9	0.267

Notes:

DALA: Days after last application.

Diazinon was identified in forage harvested 72 DAFT at a level of 0.5 percent TRR (0.01 mg/kg) and in forage harvested 14 DALA at 1.8 percent TRR (0.07 mg/kg). In both forage and stalks harvested 72 DAFT, all identified metabolites were < 10 percent TRR. The highest TRR was identified as G-27550 which occurred at a level of 7 percent and 7.7 percent TRR in forage and stalks respectively. For forage harvested 22 DAFT only 9.5 percent TRR was identified and 30.9 percent of the TRR remained

unextracted. For stalks harvested 22 DAFT, only 10.1 percent TRR was identified and 79.7 percent of the TRR remained unextracted.

For forage harvested 14 DALA, G-27550 was identified as a major metabolite (10.8 percent TRR, 0.42 mg eq/kg).

For cob and grain, of the metabolites identified none exceeded 10 percent TRR. The non-extracted residue was 52.5 percent and 73.6 percent TRR for mature cob and mature grain respectively.

Treatment of the PES from grain with amyloglucosidase solubilized around 60 percent of the TRR. The majority of this radioactivity was highly polar in nature, based on its retention time in HPLC analysis, comprising of a number of analytes. Following a series of clean-up steps, the residue separated into two peaks by HPLC. Attempts were made to precipitate sugars that may be present in the fraction, but these were unsuccessful. Therefore, the isolated peaks were acetylated, and TLC analysis indicated the presence of a complex mixture of sugar conjugates.

For the aqueous fractions, only the extraction from the sweet corn forage harvested 14 DALA were subject to further identification work. The identification/characterization of both aqueous and organic metabolites in mature sweet corn forage is shown in Table 12.

In a number of cases, the individual levels of the metabolites was not determined. The major fractions identified in mature forage were G-27550 (14.5 percent TRR, 0.56 mg eq/kg), a mixture of 4 glucose conjugates (12.4 percent TRR, 0.48 mg eq/kg) and an unknown (11.8 percent TRR, 0.46 mg eq/kg).

Table 12 Identification/characterization of organosoluble and aqueous metabolites in sweet corn forage

	Sweet corn forage harvested 14 DALA	
	Percent TRR	mg eq/kg
TRR by combustion	100	3.891
Total extracted	74.5	2.90
Diazinon (A)	1.8	0.07
G-27550 (B_1)	14.5	0.56
GS-31144 (C)	3.0	0.12
JAK-111-57 (D)	5.6	0.22
CL-XIX-29 (E_1)	4.0	0.16
Glucose conjugate of GS-31144 (E_2)		
Glucose conjugate of CL-XIX-29 (F_1)	12.4	0.48
Glucose conjugate of JAK-111-57 (F_2)		
Glucose conjugate of trihydroxy pyrimidinyl moiety (G)		
Glucose conjugate of trihydroxy pyrimidinyl moiety (H)		
Unknown (I)	11.8	0.46
B_1 (G-27550) and metabolite B_2†	7.5	0.29
Aqueous metabolites not identified§	11.6	0.45
Total identified**	41.3	1.61
Non-extracted	25.5	0.992

Notes:

DALA: Days after last application.

§ HPLC and TLC showed at least two metabolites.

† B_2 is an unidentified metabolite.

** Includes metabolites that have been quantified together. It does not include the fraction that contain identified and unidentified metabolites.

Lettuce (ABR-90039)

Lettuce (variety Simpson) grown outdoor was treated with [^{14}C]-pyrimidine-diazinon (specific activity = 9.8 µCi/mg, radiochemical purity 98 percent).

The test material was formulated as an EC formulation and applied three times. The first application was made pre-emergence at a rate of 4.48 kg ai/ha, one day after sowing. The second and third applications were foliar applications at a rate of 1.4 kg ai/ha. The second application was made 34 days after the pre-emergence application with the third application being made 7 days later.

Immature leaves were harvested just before the third application was made. Mature leaves were harvested 14 days after the last application. Samples were stored frozen for up to 19 months prior to analysis.

Samples were extracted twice with methanol: Water (9:1, v/v) and the extracts were then partitioned with ethyl acetate. Characterization and identification was performed using 2D-TLC and/or HPLC analysis with mass spectral identification on specific metabolites. Extracts were also treated with ß-glucosidase for 12 hours at 37 °C to support the identification of the glucose conjugates extracted.

The total radioactive residues and the distribution of radioactivity in lettuce are shown in Table 13.

The TRR was 1.885 mg eq/kg and 0.656 mg eq/kg in immature lettuce and mature lettuce respectively. For immature lettuce, the solvent extractability using methanol: water (9: 1, v/v) was 87.2 percent TRR. The extracted residue was found to consist of approximately equal amounts of aqueous and organosoluble residue. For mature lettuce, the solvent extractability using methanol: water (9:1, v/v) was 78.4 percent TRR. The majority of the extracted residue was found to be aqueous soluble.

Table 13 TRR and distribution of radioactivity in lettuce treated with ^{14}C-diazinon

Fraction	Immature lettuce leaves		Mature lettuce leaves	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR	100	1.885	100	0.656
Methanol: water (9:1, v/v)	87.2	1.64	78.4	0.514
Aqueous phase	47.1	0.888	54.9	0.36
Organic phase	40.1	0.756	23.5	0.154
Non-extracted	12.8	0.241	21.6	0.142

The identification/characterization of residues in the organosoluble fraction for immature and mature lettuce is outlined in Table 14.

Table 14 Identification/characterization of radioactivity in the organosoluble fraction from lettuce samples

Fraction	Immature lettuce leaves		Mature lettuce leaves	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR	100	1.885	100	0.656
Organic phase	*40.1*	*0.756*	*23.5*	*0.154*
Diazinon	18.6	0.35	8.9	0.06

Fraction	Immature lettuce leaves		Mature lettuce leaves	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
G-27550	18.9	0.36	10.2	0.07
GS 31144	2.3	0.04	4.3	0.03
Unknown metabolites	0.4	< 0.01	0.1	< 0.01
Total identified	39.8	0.75	23.4	0.16

In the organic fraction, G-27550 was the highest metabolite identified (18.9 percent TRR in immature lettuce and 10.2 percent TRR in mature lettuce).

Table 15 summarizes the characterization and identification of residues in the organo and aqueous fractions for mature lettuce leaves.

Diazinon was identified at a level of 11.8 percent TRR (0.08 mg eq/kg). In a number of cases the individual levels of the metabolites were not determined. The major metabolites/ identified were G-27550 (17.5 percent TRR, 0.12 mg eq/kg), an unknown metabolite (12.7 percent TRR, 0.08 mg eq/kg) and GS-31144 (11.7 percent TRR, 0.08 mg eq/kg).

For mature lettuce leaves, 63.5 percent TRR (0.42 mg eq/kg) was identified and 21.6 percent TRR (0.142 mg eq/kg) remained unextracted.

Table 15 Identification/characterization of radioactivity in mature lettuce leaves

	Mature lettuce leaves	
	Percent TRR	mg eq/kg
TRR by combustion	100	0.656
Total extracted	78.4	0.514
Diazinon (A)	11.8	0.08
G-27550 (B_1)	17.5	0.12
GS-31144 (C)	11.7	0.08
JAK-111-57 (D)	1.3	0.01
CL-XIX-29 (E_1)	3.0	0.02
Glucose conjugate of GS-31144 (E_2)		
Glucose conjugate of CL-XIX-29 (F_1)	9.4	0.06
Glucose conjugate of JAK-111-57 (F_2)		
Glucose conjugate of trihydroxy pyrimidinyl moiety (G)	6.7	0.04
Glucose conjugate of trihydroxy pyrimidinyl moiety (H)	2.1	0.01
Unknown metabolite	12.7	0.08
Unknown metabolites (at least two metabolites)	3.4	< 0.03
Total identified**	63.5	0.42
Unextracted	21.6	0.142

Notes:

** Includes metabolites that have been quantified together.

In a supplementary study, retained crop fractions of lettuce were subject to analysis after 69 months of freezer storage. The total radioactivity in the samples was determined by combustion with LSC. The samples were extracted with methanol: water (9: 1, v/v) and partitioned with ethyl acetate.

The HPLC profile of the extracts of lettuce from 19 months of storage and from 69 months of storage were compared. The HPLC profiles were qualitatively similar and the percent TRR of the main fractions were quantitatively similar.

The TRR determined by combustion and the solvent extractabilities for lettuce samples extracted after 69 months of storage are shown in Table 16.

Table 16 TRR and distribution of radioactivity in lettuce treated with ^{14}C-diazinon

Fraction	Immature lettuce leaves		Mature lettuce leaves	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR	100	2.178	100	0.751
Methanol: water (9:1, v/v)	81.5	1.78	72.2	0.542
Aqueous phase	33.2	0.723	48	0.36
Organic phase	48.3	1.05	24	0.18
Non-extracted	18.5	0.403	27.8	0.209

After extraction with methanol: water (9: 1 v/v) the aqueous fractions were subject to sample clean-up, derivatization and enzyme hydrolysis steps in order to aid the identification of residues. Separate fractions were treated with cellulase (12 hours at 37 °C), ß-glucosidase (12 hours at 37 °C), HCl (6 M HCl for 1 hour at 95 °C), acetic anhydride: pyridine (90: 10 v/v, room temperature for 12 hours) and 3 M HCl in butanol (1 hour at 95 °C). Metabolites were identified using TLC and HPLC.

Diazinon was not identified. In a number of cases the individual levels of the metabolites were not determined. For immature lettuce, the major metabolite/fraction identified was a mixture of G-27550 and unknown metabolite B_2 (11.7 percent TRR, 0.255 mg eq/kg). A total of 24.4 percent TRR (0.522 mg eq/kg) was identified.

For mature lettuce, the major metabolites/fractions identified were mixture of conjugates along with 3 unknown metabolites (16.7 percent TRR, 0.125 mg eq/kg) and a mixture of two conjugates (10.1 percent TRR, 0.076 mg eq/kg). The aglycones of the conjugates were identified as G-27550 and GS-31144. The study postulates that the conjugates are malonyl glucose conjugates based on the successfully derivatization, via acetylation, and information from the literature. No further details are provided.

The results are summarized in Table 17.

Table 17 Identification/characterization of aqueous metabolites in lettuce

	Immature lettuce			Mature lettuce		
TRR by combustion	2.178 mg/kg†			0.751 mg/kg†		
	Percent Aqueous	Percent TRR	mg eq/kg	Percent Aqueous	Percent TRR	mg eq/kg
G-27550 (B_1) / Unknown (B_2)	35.2	11.7	0.255	9.8	4.7	0.035
G-27550 (B_1)	24.2	8.0	0.175	6.7	3.2	0.024
GS-31144 (C)	17.1	5.7	0.124	16.2	7.8	0.059
JAK-III-57 (D)	-	-	-	2.7	1.3	0.010
Glucose conjugate of GS-31144 E_2)	3.7	1.2	0.027	7.4	3.6	0.027
Glucose conjugate of CL-XIX-29 (F_1)	10.9	3.6	0.079	21.0	10.1	0.076

	Immature lettuce			Mature lettuce		
TRR by combustion	2.178 mg/kg†			0.751 mg/kg†		
	Percent Aqueous	Percent TRR	mg eq/kg	Percent Aqueous	Percent TRR	mg eq/kg
Glucose conjugate of JAK-III-57 (F_2)						
Two glucose conjugates of trihydroxy pyrimidinyl moiety (G+H)	7.5	2.5	0.054	8.4	4.0	0.030
Conjugate of: G-27550 § GS-31144 § And three unknown metabolites	25.6	8.5	0.185	34.5	16.7	0.125
Conjugate of G-27550 §	8.9	3.0	0.064	12.0	5.8	0.044
Total identified**	-	35.7	0.778	-	40.5	0.305

Notes:

**Includes metabolites that have been quantified together. It does not include fractions that contain identified and unidentified metabolites.

† The characterization and identification work was undertaken on an additional subsample of lettuce.

§ Aglycones confirmed but the identity of the conjugate only postulated to be malonyl glucose conjugate.

In addition, the PES from mature lettuce was subject to further extraction procedures (methanol/water reflux, NaCl reflux, enzyme hydrolysis). The results are summarized in Table 18.

Table 18 Summary of the radioactivity following treatment of the PES of mature lettuce

Fraction	Percent TRR	mg eq/kg
Initial PES	27.8	0.209
9/1 v/v MeOH/H_2O	9.1	0.068
Organic	2.3	0.017
Aqueous	2.6	0.019
1 percent NaCl	6.7	0.050
Organic	1.3	0.010
Aqueous	4.1	0.030
Cellulase hydrolysis	2.5	0.018
Organic	0.2	0.001
Aqueous	1.7	0.013
Protease hydrolysis	2.6	0.020
Final PES	6.4	0.040

The extraction procedures individually released 2.5–9.1 percent TRR (0.018–0.068 mg eq/kg), leaving 6.4 percent of the TRR (0.040 mg eq/kg) in the post-extraction solids.

The methanol/water and NaCl organosoluble fractions were analysed by HPLC and 2D-TLC. In the methanol/water organosoluble fraction, parent diazinon (1 percent TRR), G-27550 (< 0.5 percent TRR) and an unknown metabolite (0.6 percent TRR) were identified.

The NaCl organosoluble and aqueous fractions were found to contain G-27550 (1 percent TRR in each fraction), GS-31144 (< 0.1 percent TRR) and an unknown (0.2 percent TRR).

Following cellulase hydrolysis, G-27550 (0.1 percent TRR) was identified in the organic phase and the aqueous phase fraction was found to contain polar metabolites, the number and levels were not stated.

The protease hydrolysis fractions were too viscous for chromatographic profiling.

Potatoes (study ABR-89059)

Potatoes (variety Katahdim) grown outdoor were treated with [^{14}C]-pyrimidine-diazinon (specific activity = 9.8 µCi/mg, radiochemical purity 98 percent).

The test material was formulated as an EC formulation and applied three times. The first application was made pre-emergence at a rate of 4.48 kg ai/ha and applied three days after planting of seed potatoes. The second and third applications were foliar applications at a rate of 1.4 kg ai/ha. The second application was made 75 days after the pre-emergence application with the third application being made 7 days later.

Foliage and tuber samples were harvested 74 days after the pre-emergence use (i.e. 1 day before the first foliar application). Further samples of foliage and tubers were harvested 15 DALA. Samples were stored frozen for up to 16 months prior to analysis.

The total radioactivity in the samples was determined by combustion with LSC. Samples were extracted twice with methanol: water (9:1, v/v) and the extracts were then partitioned with ethyl acetate. Extracts were also treated with ß-glucosidase for 12 hours at 37 °C to support the identification of the glucose conjugates extracted. PES were also subjected to enzymatic hydrolysis; a sample was incubated at 37 °C with sodium acetate buffer with amyloglucosidase. In addition, the PES were subject to acid hydrolysis; samples were refluxed with 3 M HCl for 2 hours (temperature not stated).

Characterization and identification was performed using 2D-TLC and/or HPLC analysis with mass spectral identification on specific metabolites. The total radioactive residues and the distribution of radioactivity in lettuce are shown in Table 19.

The TRR ranged from 0.059 mg eq/kg for immature foliage to 1.93 mg eq/kg for mature foliage. The solvent extractabilities using methanol: water (9:1, v/v) ranged from 15.8 percent TRR for mature tubers to 81.5 percent TRR for mature foliage. The majority of the extracted residue was found to be aqueous soluble.

Table 19 TRR and distribution of radioactivity in potato foliage and tubers treated with ^{14}C-diazinon

Fraction	Immature foliage (-1 DALA)		Mature foliage (15 DALA)		Immature tubers (-1 DALA)		Mature tubers (15 DALA)	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR by combustion	100	0.059	100	1.930	100	0.068	100	0.275
Methanol: water (9:1, v/v)	32.0	0.0189	81.5	1.57	16.8	0.011	15.8	0.0435
Aqueous phase	29.3	0.0173	75.9	1.46	15.2	0.01	14.6	0.0402
Organic phase	2.69	0.00159	5.62	0.108	1.65	0.001	1.23	0.003
Non-extracted	68	0.04	18.5	0.357	83.2	0.0566	84.2	0.232
Amyloglucosidase	-	-	-	-	-	-	32.8	0.0902

Fraction	Immature foliage (-1 DALA)		Mature foliage (15 DALA)		Immature tubers (-1 DALA)		Mature tubers (15 DALA)	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
3M HCl	-	-	-	-	-	-	71.6	0.197

Notes:

DALA: Days after last application

Treatment of the PES from potato tubers with amyloglucosidase solubilized around 33 percent TRR. The majority of this radioactivity was polar, based on its retention time in HPLC analysis and possibly contained various sugar conjugates. Acid hydrolysis released around 72 percent of the TRR with 11.5 percent of the TRR identified as G-27550.

The identification/characterization of residues was only determined in mature potato foliage samples with the summary outlined in Table 20. Diazinon was found at 14.2 percent TRR (0.27 mg/kg). In a number of cases the individual levels of the metabolites were not determined. The major metabolites/fractions identified in potato foliage were a mixture of two glucose conjugates (20.8 percent TRR, 0.4 mg eq/kg), a trihydroxy glucose conjugate (14.1 percent TRR, 0.27 mg eq/kg) and CL-XIX and a glucose conjugate of GS-31144 (11.5 percent TRR, 0.22 mg eq/kg). The structure of the trihydroxy glucose conjugate was not fully elucidated.

Table 20 Identification/characterization of residues in mature potato foliage

	Mature potato foliage (15 DALA)	
	Percent TRR	mg eq/kg
TRR by combustion	100	1.930
Total extracted	81.5	1.57
Diazinon (A)	14.2	0.27
G-27550 (B_1)	1.3	0.03
GS-31144 (C)	2.1	0.04
JAK-111-57 (D)	4.3	0.08
CL-XIX-29 (E_1)	11.5	0.22
Glucose conjugate of GS-31144 (E_2)		
Glucose conjugate of CL-XIX-29 (F_1)	20.8	0.40
Glucose conjugate of JAK-111-57 (F_2)		
A glucose conjugate of trihydroxy pyrimidinyl moiety (H)	14.1	0.27
Unknown (I)	7.4	0.14
Unresolved metabolite G-27550 (B_1) and B_2†	1.9	0.04
Total identified**	68.3	1.31
Total unextracted	18.5	0.357

Notes:

†B_2 is an unidentified metabolite.

**Includes metabolites that have been quantified together. It does not include fractions that contain identified and unidentified metabolites.

A literature paper from 1972 outlining the metabolism in rice and peas was also provided to the Meeting. This has not been evaluated as the paper did not include sufficient details to contribute to the metabolic pathway and residue definitions for plants.

In Figure 2 an overall proposal for the metabolic pathway of diazinon in plants is outlined.

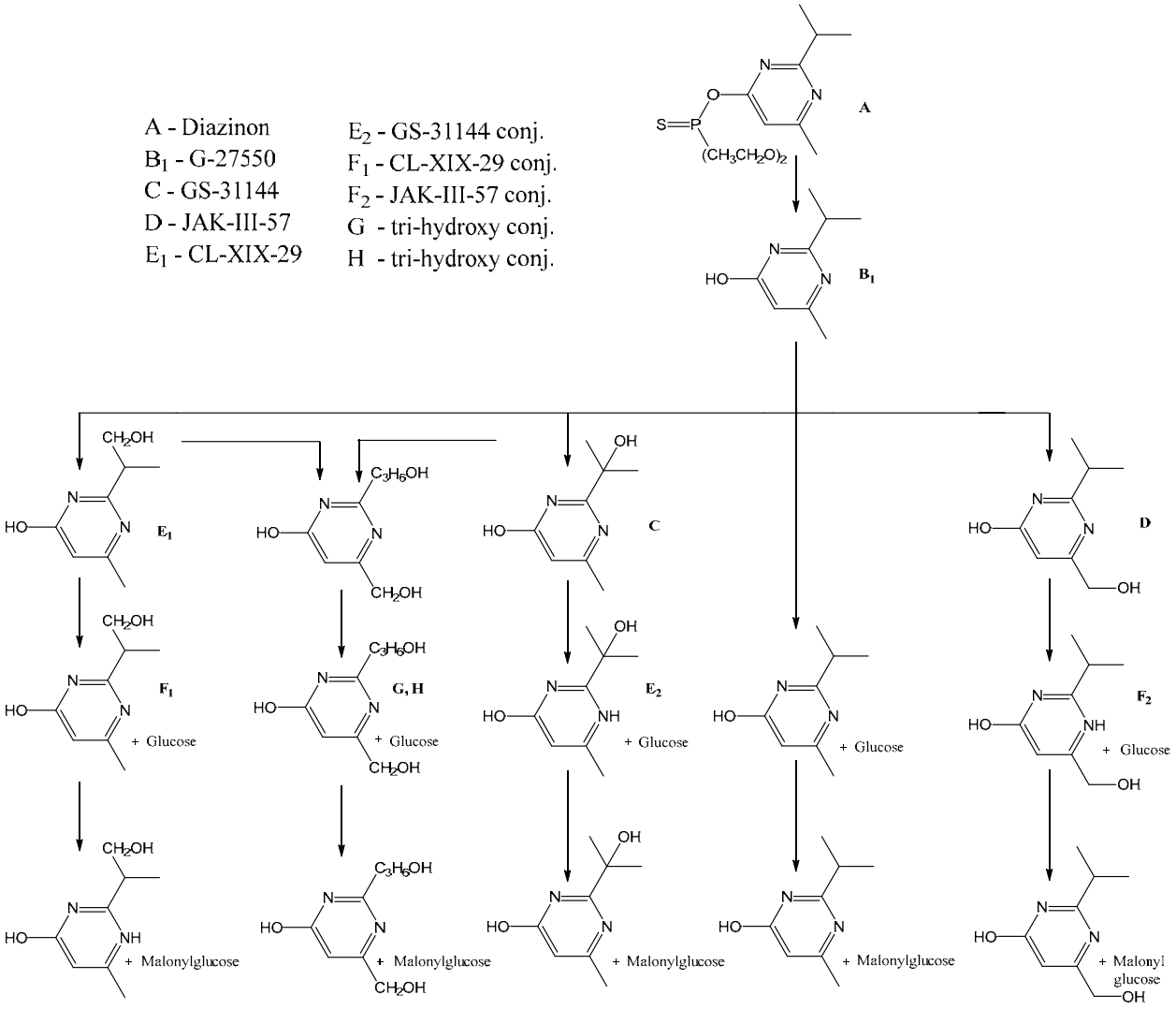


Figure 2 Proposed Metabolic pathway in plants

Environmental fate in soil

The meeting received information on aerobic degradation in soil, photolysis in soil, hydrolytic degradation, anaerobic degradation and soil leaching. Only the data on aerobic degradation in soil, photolysis in soil and the hydrolytic degradation, which are relevant to MRL setting, are reported here.

Aqueous hydrolysis

The aqueous hydrolysis of diazinon was investigated in two studies.

In the first study (R-228) aqueous hydrolysis was investigated for a concentration of 10 mg/L diazinon, at pH 5, 7 and 9 and temperatures of 30, 50 and 70 °C. Quantification was performed by GC-FID. The percentage of diazinon at each time interval is outlined in Table 21.

Table 21 Hydrolysis of diazinon at different temperatures and pH

Hydrolysis period	percent diazinon								
	pH 5			pH 7			pH 9		
	30 °C	50 °C	70 °C	30 °C	50 °C	70 °C	30 °C	50 °C	70 °C
0 hours	100	100	100	100	100	100	100	100	100
2 hours	-	-	63	-	-	82	-	-	70
4 hours	-	83	44	-	-	71	-	-	52
6 hours	-	-	30	-	-	63	-	-	39
8 hours	-	59	20	-	-	55	-	-	30
24 hours	67	28	-	-	81	19	-	76	4
32 hours	-	18	-	-	-	12	-	-	-
48 hours	44	8	-	-	70	4	-	49	-
3 days	31	-	-	-	53	-	-	29	-
4 days	21	-	-	-	43	-	-	19	-
7 days	7	-	-	86	22	-	63	5	-
8 hours	4	-	-	-	17	-	-	-	-
14 hours	-	-	-	74	-	-	42	-	-
21 hours	-	-	-	55	-	-	28	-	-
28 hours	-	-	-	44	-	-	19	-	-

The study showed that diazinon degraded in aqueous media, assuming first order kinetics, with the rate constants and half-lives outlined in Table 22.

Table 22 Rate constants and DT_{50} values for diazinon degradation in aqueous media

pH	Temp. (°C)	Kobs (days-1)	DT_{50} (days)
5.0	20 (extrapolated)	2.03 × 10-6	3.8
7.0		1.03 × 10-7	78
9.0		2.03 × 10-7	40
5.0	30	4.56 × 10-6	1.8
7.0		3.43 × 10-7	23
9.0		6.84 × 10-7	12
5.0	50	1.46 × 10-5	0.5
7.0		2.55 × 10-6	3.2
9.0		4.98 × 10-6	1.6
5.0	70	5.52 × 10-5	0.15
7.0		1.88 × 10-5	0.4
9.0		3.75 × 10-5	0.2

Notes:

The degradation products were not determined.

In a second study (R-231) the rate of hydrolysis of [^{14}C]-pyrimidine-diazinon was investigated at 23-25°C and at a pH of 5, 7 and 9. The concentration of diazinon was approximately 11 µg/ml. Total radioactive residues were determined by combustion and LSC. Quantification was performed at intervals between 0 and 32 days by HPLC and TLC.

[^{14}C]-pyrimidine-diazinon degraded over time at pH 5, 7 and 9. HPLC analysis identified the presence of three components: diazinon, G-27550 and an unknown component. Levels of G-27550

reached a maximum of 67.4 percent AR at pH 5 (day 21), 6.9 percent AR at pH 7 (day 32) and 18.8 percent AR at pH 9 (day 32). The unknown component accounted for no more than 7.5 percent AR in all samples. The levels of the degradation products of diazinon at the varying pH values over time are summarized in Table 23.

Table 23 Distribution of the radioactivity applied to buffer samples

pH buffer	Time interval (days)	percent of applied radioactivity		
		Diazinon	G-27550	Unknown
5.0	0	92.7, 89.4 (91.1)	2.4, 2.4 (2.4)	ND, ND (ND)
	2	82.7, 82.1 (82.4)	10.2, 10.2 (10.2)	0.3. ND (0.2)
	5	69.5, 69.1 (69.3)	23.6, 22.3 (23.0)	0.6, 0.6 (0.6)
	8	57.5, 57.2 (57.4)	36.1, 35.2 (35.7)	1.2, 1.1 (1.2)
	11	46.7, 49.7 (48.2)	45.1, 46.1 (45.6)	0.8, 1.1 (1.0)
	14	41.2, 40.9 (41.1)	52.8, 51.2 (52.0)	0.6, 0.7 (0.7)
	21	29.9, 27.2 (28.6)	66.5, 68.2 (67.4)	0.8, 0.8 (0.8)
7.0	0	90.5, 91.6 (91.1)	0.5, 0.5 (0.5)	ND, ND (ND)
	5	88.3, 83.9 (86.1)	1.1, 1.5 (1.3)	1.4, 1.0 (1.2)
	11	85.5, 87.5 (86.5)	2.7, 2.7 (2.7)	2.9, 3.1 (3.0)
	21	81.9, 83.0 (82.5)	4.9, 4.7 (4.8)	4.9, 4.9 (4.9)
	29	79.1, 78.3 (78.7)	6.1, 6.5 (6.3)	6.6, 6.5 (6.6)
	32	75.9, 75.6 (75.8)	7.0, 6.8 (6.9)	7.4, 7.5 (7.5)
9.0	0	91.5, 91.0 (91.3)	0.7, 0.6 (0.7)	0.3, ND (0.2)
	5	84.9, 86.9 (85.9)	3.6, 4.0 (3.8)	1.1, 1.2 (1.2)
	11	85.2, 85.4 (85.3)	7.9, 7.4 (7.7)	2.6, 3.0 (2.8)
	21	75.4, 74.0 (74.7)	12.7, 12.8 (12.8)	5.2, 4.7 (5.0)
	29	70.9, 70.1 (70.5)	17.2, 17.4 (17.3)	6.8, 6.4 (6.6)
	32	69.0, 67.6 (68.3)	18.8, 18.8 (18.8)	7.2, 6.6 (6.9)

Notes:

ND Not detected.

The calculated rate constants and degradation half-lives (DT_{50}) are summarized in Table 24.

Table 24 Rate constants and DT_{50} values for diazinon degradation in aqueous media

pH	Temp. (°C)	K_{obs} ($days^{-1}$)	DT_{50} (days)
5	25	5.6×10^{-2}	12
7		5.0×10^{-3}	138
9		9×10^{-3}	77

Aqueous photolysis

The photolysis of diazinon in aqueous solutions was investigated in two studies.

In the first study (G24480/2088), non-radio-labelled diazinon at a concentration of 50 µg/mL in deionised water was exposed to artificial light (1000 W mercury lamp) for a period of 12 days at 20–25 °C. The concentration of diazinon was determined by HPLC-UV at the start and end of the exposure and remained at 50 µg/mL.

In a second study (G24480/0239), ^{14}C-diazinon at a concentration of 6 mg/L in phosphate buffer at pH 7 was exposed to 360 hours of natural sunlight over 30 days. On average the daily sunlight was 12 hours and the temperatures ranged from 12–49 °C. Aliquots of the buffer solution were sampled at intervals from 0–360 hours. The TRR were determined directly by LSC and are summarized in Table 25.

Table 25 Total radioactivity recovered on exposure of diazinon to sunlight

Time (hours)	Total radioactivity recovered (percent)		
	Dark control	Replicate 1	Replicate 2
5	100	100	100
12	111	106	106
24	107	106	102
48	109	102	103
72	96	90	100
146	90	87	93
240	85	82	84
360	72	74	82

Aliquots were directly placed on TLC plates and separated using two solvent systems. In addition, the test vessels from the 360 hour samples were extracted with hexane to account for any residues that had bound to the walls of the test vessels. The results are summarized in Tables 26 and 27.

Table 26 Distribution of radioactivity (percent AR) following exposure of diazinon to sunlight (TLC developed using solvent system 1 - toluene: chloroform: ethanol: formic acid (8:8:2:1, v/v).

Time (hours)	percent AR (replicate 1, replicate 2)				
	Origin	Region 1	G-27550	Region 2	Diazinon
Exposure to light					
5	0, 0	0, 0	3.49, 0.96	0, 0	96.46, 99.02
12	0, 0.73	0.04, 0.44	2.08, 3.14	1.27, 2.05	102.05, 97.43
24	0, 0	0, 0.12	3.82, 3.30	1.26, 0.97	100.83, 97.47
48	1.5, 1.28	2.29, 1.19	7.5, 9.34	4.0, 1.85	84.1, 88.99
72	1.53, 0.81	0, 0	9.98, 10.42	1.18, 1.05	76.72, 87.88
146	4.54, 4.78	2.32, 1.51	17.79, 18.71	3.38, 3.55	59.19, 64.46
240	7.24, 7.57	1.17, 1.85	25.21, 24.34	3.45, 5.02	45.13, 45.04
Hexane extract					
360	1.38, 0.03	0.94, 0.26	2.07, 0.33	0.37, 0.1	11.4, 8.34
Dark controls					
5	0	0	1.65	0	98.30
12	0	0	1.55	0	109.74
24	0	0	1.78	0	105.74
48	1.1	2.79	3.96	1.3	98.0
72	0	0	2.84	0	93.53
146	1.85	2.05	7.46	0	78.46
240	3.57	0.32	9.05	0	72.15
360	4.73	4.85	14.4	0	48.25
Hexane extract					
360	0.05	0.14	0.21	0.06	7.39

Table 27 Distribution of radioactivity (percent AR) following exposure of diazinon to sunlight (TLC developed using solvent system 2 - hexane: ethyl acetate (8:2, v/v).

Time (hours)	percent AR (replicate 1, replicate 2)	
	Region 1	Diazinon
Exposure to light		
5	1.22, 2.42	98.24, 95.23
12	1.51, 2.05	102.69, 101.95
24	0.98, 3.02	102.70, 96.59
48	5.66, 5.85	87.53, 87.93
72	2.83, 3.52	78.36, 86.63
146	7.54, 6.18	59.64, 65.07
240	12.95, 12.48	43.69, 45.61
360	14.89, 15.19	22.29, 26.12
Hexane extract		
360	0.26, 0.10	15.74, 9.00
Dark controls		
5	0	99.97
12	0.17	111.12
24	0.51	107.05
48	2.94	99.01
72	0	92.83
146	1.03	78.84
240	0.32	72.48
360	2.11	43.66
Hexane extracts		
360	0.06	7.68

The samples from 360 hours of exposure were also analysed by two dimensional TLC. The results are summarised in Table 28.

Table 28 Distribution of radioactivity (percent AR) from two dimensional TLC

	percent of AR	
	Solvent system 1	Solvent system 2
Origin	8.51	10.16
Unknown 1	2.86	3.39
Unknown 2	2.71	3.14
Unknown 3	7.22	5.23
G-27550	15.57	18.54
Unknown 4	1.52	2.05
Unknown 5	8.24	8.46
Unknown 6	2.25	2.31
Unknown 7	0.18	0.19
Unknown 8	0.93	1.14
Unknown 9	2.90	3.00
Diazinon	18.71	21.59
Unknown 10	0.80	1.64
Unknown 11	0.75	0.62
Unknown 12	0.83	0.49
Unknown 13	0.73	0.40
Total	73.71	82.35

Half-lives of diazinon were calculated as 23.3 days for solvent system 1 and 25.8 days for solvent system 2.

Soil photolysis

The photodegradation of diazinon in soil was investigated in two studies.

In the first study (R-236) [^{14}C]-pyrimidine-diazinon was applied to a sandy loam soil. The characteristic of the soil are outlined below:

Soil	percent sand	percent silt	percent clay	percent organic matter	pH	Cation exchange capacity (meq/100g)
Sandy loam	54.8	29.4	15.8	2	5.4	15

A water/soil slurry was applied to TLC plates which were dried and duplicate aliquots (4.6 µg ai/10 µL) of diazinon was applied to each soil band. Plates were irradiated using an artificial light source (10 hours) or using natural sunlight (32.6 hours) at 25 ° C. Control samples were stored in the dark. At each sampling interval duplicate TLC plates were developed and analysed by LSC and UV.

Mass balances of the radioactivity applied were 93.7–101 percent AR. Significant degradation of diazinon (approximately 62 percent AR) occurred within 24 hours of sunlight exposure; a similar amount of degradation of diazinon (approximately 65 percent) occurred after 9 days following exposure to artificial light. In the dark control samples, diazinon degradation was approximately 34 percent AR after 10 days.

G-27550 represented an average of 41 percent, 24 percent and 16 percent AR in artificially irradiated, natural sunlight and dark control samples, respectively after the exposure period. GS-31144 accounted for up to 3.6 percent AR after 32.6 hours of natural sunlight but was not found in the dark or artificial sunlight samples.

During the course of the experiments no attempt was made to collect volatiles.

The distribution of the radioactivity after the application of diazinon in soil is summarized in Tables 29 to 31.

Table 29 Distribution of radioactivity (percent AR) after application of diazinon in soil and photolysis by sun light

	Sample interval (hours)							
	0	1	8	21	27.25	32.6	32.6	32.6
Replicate 1								
Origin	1.3	2	10.5	20.9	23.1	34.4	32.3	27.1
Diazinon	97.4	93.6	64	34.9	32.2	23.1	19.5	27.8
G-27550	0.5	1.4	10	23.7	22.5	21.2	24.4	24.4
GS-31144	ND	ND	1.6	2.8	2.7	1.2	3.6	2.8
Unknown 1	ND	ND	7	9.2	9.6	2.9	9.8	9.1
Unknown 2	0.4	0.3	2.2	3.3	4	4.1	4.8	4.5
Unknown 3	1.2	1.5	3.1	2.6	3.2	6.7	2.6	1.9
Unknown 4	a	0.1	a	0.1	a	ND	0.1	ND
Recovery	101	98.9	98.3	97.3	97.5	93.7	97.1	97.3
Replicate 2								
Origin	1.6	2.3	1.1	20.4	18.9	28.5	31.3	27.1
Diazinon	96.3	92.8	63.4	35.7	39.6	31.4	22.6	28.7
G-27550	0.6	1.3	9.7	23.4	21.8	23.6	24.4	24.3
GS-31144	ND	ND	1.6	3	2.2	0.9	3.5	2.6

	Sample interval (hours)							
	0	1	8	21	27.25	32.6	32.6	32.6
Unknown 1	ND	ND	6.8	9.5	8.9	3.1	9.2	8.5
Unknown 2	0.4	0.4	2.7	3.2	3	3.2	4.1	4.7
Unknown 3	2.3	2.2	3	2.5	2.8	4.9	2.3	2.1
Unknown 4	a	0.1	a	ND	a	0.1	0.1	ND
Recovery	101	99.2	98.2	97.8	97.3	95.6	97.5	98.1

Notes:

ND = Not detected.

[a] = No information available.

Table 30 Distribution of radioactivity (percent AR) after application of diazinon in soil and photolysis by artificial light

	Sample interval							
	0 hours	1 hour	8 hours	1 day	3 days	7 days	9 days	10 day
Replicate 1								
Origin	1.3	1.5	2.3	4.5	6.3	11	9.7	10.1
Diazinon	97.4	95.7	89.7	81.8	58.8	42.4	35.9	27.9
G-27550	0.5	0.8	3.2	7.5	19.8	27.4	33.2	38.5
GS-31144	ND	ND	ND	ND	ND	ND	ND	ND
Unknown 1	ND	ND	1.4	3.2	8.2	11.3	12.7	15
Unknown 2	0.4	0.3	0.5	1.1	2	2.6	2.7	2.1
Unknown 3	1.2	1	1.4	2.1	2.2	3.8	3.4	2.8
Unknown 4	a	ND	ND	0.1	a	0.1	a	ND
Recovery	101	99.2	98.6	100	97.4	98.5	96.6	96.5
Replicate 2								
Origin	1.6	1.3	2.5	4.7	7.1	11.5	11.1	10.9
Diazinon	96.3	95.2	89.9	80.1	50.6	33.7	28.6	21.8
G-27550	0.6	0.9	3.7	8.2	24.2	33.2	38.6	43.3
GS-31144	ND	ND	ND	ND	ND	ND	ND	ND
Unknown 1	ND	ND	1.5	3	8.3	12.8	14.2	15.4
Unknown 2	0.4	0.3	0.5	1.2	2.4	3.4	2.9	2.3
Unknown 3	2.3	1.2	1.3	2.3	2.1	3.9	3.2	3.3
Unknown 4	a	ND	0.1	ND	a	0.1	a	ND
Recovery	101	98.8	99.4	99.5	94.7	98.7	98.6	97.1

Notes:

ND = Not detected.

[a] = No information available.

Table 31 Distribution of radioactivity (percent AR) after application of diazinon in soil – Dark controls

	Sample interval							
	0 hours	1 hour	8 hours	1 day	3 days	7 days	9 days	10 day
Replicate 1								
Origin	1.3	1.1	1.6	1.6	2.6	5	4.7	4.4
Diazinon	97.4	90.4	94.6	90	82.3	82.3	61.7	61.9
G-27550	0.5	0.5	1.4	4.2	7.2	7.2	17.3	16.5
GS-31144	ND	ND	ND	ND	ND	ND	ND	ND
Unknown 1	ND	ND	0.7	ND	5.9	5.9	13.5	13.7
Unknown 2	0.4	0.3	0.3	1.2	0.5	0.5	1.3	1.1
Unknown 3	1.2	0.6	0.8	2	0.9	0.9	1.5	1.4

	Sample interval							
	0 hours	1 hour	8 hours	1 day	3 days	7 days	9 days	10 day
Unknown 4	[a]	[a]	[a]	0.1	[a]	[a]	[a]	[a]
Recovery	101	92.8	99.4	99.2	99.4	99.4	100	99
Replicate 2								
Origin	1.6	1.6	1.8	2.1	2.7	4.9	4.8	4.3
Diazinon	96.3	95.5	92.4	91.1	82.9	64.8	57.6	63.9
G-27550	0.6	0.7	1.3	3.7	6.9	16.1	17.5	16.4
GS-31144	ND	ND	ND	ND	ND	ND	ND	ND
Unknown 1	ND	ND	0.8	ND	5.9	11	17.2	13.2
Unknown 2	0.4	0.3	0.4	1.3	0.5	1	1.3	1
Unknown 3	2.3	0.8	0.8	1.6	0.9	1.7	1.6	1.1
Unknown 4	[a]	[a]	[a]	0.1	[a]	ND	[a]	[a]
Recovery	101	99	97.5	100	99.9	100	99.9	99.7

Notes:

ND = Not detected.

[a] = No information available.

Half-lives of diazinon were calculated as 17.3 hours (natural sunlight), 5.5 days (artificial light) and 14.7 days (dark control).

In a second study (6015-208) [^{14}C]-pyrimidine-diazinon was applied to a sandy loam soil.

Soil samples were either exposed to continuous artificial light for 210 hours or natural sun light for 58.5 hours at 25 °C.

A water/soil slurry was applied to TLC plates which were dried and duplicate aliquots of 7–51 mg/kg diazinon was applied to each soil band. Plates were irradiated using an artificial light source or using natural sunlight. Control samples were stored in the dark. At each sampling interval TLC plates were developed and analysed by LSC and UV.

During the course of the experiments, volatiles were only collected from the samples irradiated with artificial light.

The distribution of the radioactivity after the application of diazinon in soil is summarized in Tables 32–34.

Table 32 Distribution of radioactivity (percent AR) after application of diazinon in soil and photolysis by artificial light

Identified region	Replicate	Sampling interval (hours)			
		90	141	190	210
Diazinon	A	52.82	38.27	40.13	37.59
	B	47.78	40.28	35.93	34.56
	C	57.87	44.86	42.29	35.37
G-27550	A	12.68	15.20	18.74	29.95
	B	25.25	18.53	9.46	9.73
	C	10.35	17.00	17.40	11.68
Unknown	A	0.12	7.88	0.14	2.96
	B	0.20	4.09	6.10	9.86
	C	0.12	5.38	0.20	6.92
Origin - unknown	A	28.99	33.63	38.91	26.27
	B	19.56	34.45	40.66	30.64
	C	21.88	21.22	29.58	25.51
Total	A	94.60	94.98	97.92	96.77

Identified region	Replicate	Sampling interval (hours)			
		90	141	190	210
radioactivity*	B	92.79	97.36	91.14	84.79
	C	90.21	98.46	89.47	79.48
Average recovery		92.53	96.93	92.84	87.01

Notes:

ND Not detected.

- Quantification not possible.

* Sum of all radioactive regions.

Table 33 Distribution of radioactivity (percent AR) after application of diazinon in soil and photolysis by sun light

Identified region	Replicate	Sampling interval (hours)			
		5	13.5	21	35.5
Diazinon	A	90.01	41.12	45.72	35.57
	B	65.68	52.72	46.52	-
	C	69.67	52.56	41.78	37.64
G-27550	A	10.49	21.50	13.05	15.36
	B	10.14	19.17	9.68	-
	C	8.24	18.15	18.75	11.02
Unknown	A	0.52	0.28	0.31	2.24
	B	0.35	0.30	7.61	-
	C	0.33	0.72	7.24	13.37
Origin - unknown	A	16.06	15.80	25.08	34.34
	B	11.28	17.74	25.89	-
	C	8.99	17.22	26.97	25.77
Total radioactivity*	A	89.24	78.70	84.16	87.51
	B	87.45	89.93	89.71	-
	C	87.22	88.65	94.73	87.80
Average recovery		87.97	85.76	89.53	87.66

Notes:

ND Not detected.

- Quantification not possible.

* Sum of all radioactive regions.

Table 34 Distribution of radioactivity (percent AR) after application of diazinon in soil – Dark controls

Identified region	Replicate	Sampling interval (hours)				
		24	48	120	168	264
Diazinon	A	91.14	92.87	84.79	83.86	78.33
	B	85.44	93.67	86.62	86.99	77.15
	C	93.47	88.28	90.75	85.31	75.54
G-27550	A	ND	ND	ND	3.97	9.58
	B	ND	ND	ND	3.67	7.70
	C	ND	ND	1.74	4.69	12.10
Unknown	A	2.51	2.68	3.41	0.43	0.35
	B	1.18	1.88	3.79	0.42	4.12
	C	1.63	1.82	1.31	0.55	0.30
Origin - unknown	A	2.51	1.81	4.94	6.57	10.75
	B	1.37	1.98	4.75	6.43	10.98
	C	1.38	2.53	2.46	5.87	11.28

Identified region	Replicate	Sampling interval (hours)				
		24	48	120	168	264
Total radioactivity*	A	96.15	97.36	93.14	94.82	99.00
	B	87.99	97.52	95.16	97.51	99.95
	C	96.47	92.64	96.25	96.42	99.22
Average recovery		93.54	95.84	94.85	96.25	99.39

Notes:

ND Not detected.

- Quantification not possible.

* Sum of all radioactive regions.

Average recoveries were 92.3 percent, 87.7 percent and 96.0 percent AR for the artificial light, sunlight and dark control samples, respectively and remained consistent throughout the study, indicating minimal evolution of volatile radioactive components. Radioactivity in the traps accounted for no more than 0.2 percent AR after 210 hours of artificial light exposure. After 210 hours of artificial light exposure, the radioactivity profile was qualitatively similar to the profile of the samples exposed to 58.5 hours of natural sunlight.

Only one major degradation product was identified, G-27550. Two minor unknown metabolites were also determined.

DT_{50} values were determined by linear regression analysis of the data. Diazinon was found to degrade under natural sunlight with a half-life of 37.4 hours. However, the DT_{50} for samples exposed to artificial light could not be calculated due to limited degradation occurring. The DT_{50} for diazinon in the dark control samples was 926 hours.

Aerobic degradation in soil

Three studies have investigated the aerobic degradation of diazinon.

In the first study (R-259) diazinon, [14C]-pyrimidine-diazinon was applied at a rate of 10 mg ai/kg dry weight of soil to a sandy loam soil. The soil characteristics are outlined below:

Soil Characteristic	Sandy loam
Sand	47 percent
Silt	49.1 percent
Clay	3.9 percent
Organic carbon	1.1 percent
Cation exchange capacity	7.3 meq/100 g soil
pH	7.5

The degradation in the laboratory under aerobic conditions was investigated. The soil was maintained at a 75 percent moisture holding capacity and maintained in the dark at 25 °C. Volatiles were collected in trapping solutions. Samples of soil were removed for analysis from day 0 to 166 days after treatment. The TRR were determined by combustion with LSC.

Samples were extracted five times with methanol: water (8: 2, v/v). The combined methanol: water extracts were partitioned with dichloromethane. The remaining sediment was further extracted by refluxing for 6 hours with methanol. Extracts were analysed by TLC, GC-FID and GC-MS.

Total recoveries ranged from 74–105 percent AR. Diazinon decreased from 100 percent AR to 0.3 percent AR after 166 days. The volatile radioactivity was almost entirely attributed to CO_2. The levels of

CO_2 increased to 55.6 percent AR after 166 days. The major metabolite, G-27550 reached a maximum level of 72.9 percent AR after 14 days and then decreased to 4.7 percent AR after 166 days. Non-extracted radioactivity levels increased during the study, reaching 15.1 percent AR after 166 days

The distribution of radioactivity in soil is outlined in Table 35.

Table 35 Distribution of radioactivity (percent of AR)

Metabolite	Days after treatment					
	0	14	28	56	84	166
Diazinon	101.6	12.3	5.3	3.2	2.0	0.3
G-27550	3.1	72.9	55.9	69.6	49.0	4.7
GS-31144	ND	ND	ND	ND	ND	1.5
Unknown	ND	ND	ND	ND	0.5	4.8
$^{14}CO_2$	-	2.6	6.9	15.6	20.4	55.6
Unknown volatiles	-	0.4	0.4	0.5	0.4	0.8
Non-extracted	0.4	4.2	5.5	9.1	9.1	15.1
Total extracted	104.7	85.2	61.2	72.8	51.0	11.3
Total recovery	105.1	92.4	74.0	98.0	81.4	82.8

Notes:

ND = not detected.

The degradation rate of diazinon was investigated in a second study (R014085) in which [^{14}C]-pyrimidine-diazinon was applied at a rate of 1.2 mg ai/kg dry weight of soil to three different soils types. The soil characteristics are outlined below:

Soil Characteristic	Soil 1	Soil 2	Soil 3
Particle size distribution (percent) 63 µm – 2mm (sand) 2 µm – 63 µm (silt) < 2 µm (clay)	 64.54 23.20 12.27	 83.13 7.83 9.04	 47.30 25.28 27.43
Texture class	Sandy loam	Loamy sand	Clay loam
Organic carbon (percent)	1.9	0.8	2.8
pH	6.4	6.2	6.5
Cation exchange capacity (meq/100 g)	16.5	7.0	25.6
Water holding capacity (percent)	55.8	38.8	59.2

The soil samples were incubated in the dark at 20 °C for 76 days. Volatiles were collected in trapping solutions. The TRR were determined by combustion with LSC. Samples were extracted with acetonitrile and acetonitrile: water (4:1, v/v). The extraction procedure was repeated three times. The TRR in extracts were determined by LSC. Extracts were analysed by HPLC-UV and TLC. The TRR in non-extracted material was not determined.

The total recoveries ranged from 76.7 to 98.7 percent. The distribution of radioactivity is outlined in Table 36.

Table 36 Distribution of radioactivity (percent of AR)

Metabolite	Days after treatment							
	0	1	3	7	14	30	55	76
Sandy loam								
Diazinon	92.1	87.0	79.5	53.5	31.3	6.9	2.6	1.8
GS-31144	ND	ND	0.2	1.0	1.4	2.3	3.5	4.5
G-27550	1.1	5.6	11.8	40.8	57.6	75.2	70.6	65.8
Diazoxon	< 0.2	< 0.3	< 0.4	0.2	< 0.3	< 0.3	< 0.3	0.6
Others	1.4	1.4	1.4	0.9	1.1	1.5	1.5	1.2
Organic volatiles	-	< 0.1	< 0.1	0.1	0.2	0.2	0.3	0.3
CO_2	-	< 0.1	0.1	0.4	1.3	3.8	7.4	9.9
Total volatiles	-	< 0.1	0.1	0.5	1.5	4.0	7.7	10.2
Total extracted	94.2	93.4	92.4	96.1	90.9	85.3	78.5	73.0
Total recovery	94.2	93.4	92.5	96.6	92.4	89.3	86.2	83.2
Loamy sand								
Extracts	98.7	92.6	94.4	91.7	85.4	76.8	71.6	62.2
Diazinon	96.6	88.0	87.3	68.1	56.1	32.9	17.0	9.1
GS-31144	ND	ND	0.1	0.4	0.7	1.6	3.0	4.2
G-27550	0.7	3.6	6.2	22.2	27.7	40.6	49.1	48.1
Diazoxon	< 0.2	< 0.2	< 0.3	< 0.2	< 0.3	< 0.3	< 0.4	< 0.4
Others	1.6	1.1	1.4	1.3	1.3	2.1	3.3	1.6
Organic volatiles	-	< 0.1	< 0.1	0.1	0.3	0.8	1.1	1.3
CO_2	-	< 0.1	0.1	0.2	0.6	1.8	4.0	5.8
Total volatiles	-	< 0.1	0.1	0.3	0.9	2.6	5.1	7.1
Total extracted	98.7	92.6	94.4	91.7	85.4	76.8	71.6	62.2
Total recovered	98.7	92.6	94.5	92.0	86.3	79.4	76.7	69.3
Clay loam								
Extracts	95.6	90.5	94.9	93.5	91.1	82.6	76.3	68.9
Diazinon	93.0	83.4	78.7	52.3	33.7	11.3	6.4	4.1
GS-31144	ND	ND	0.2	0.8	1.2	2.2	3.6	4.7
G-27550	1.4	6.5	15.5	38.6	55.6	67.6	63.8	59.1
Diazoxon	< 0.2	< 0.3	< 0.3	0.2	0.3	< 0.3	0.7	0.6
Others	1.4	1.2	1.2	1.9	0.8	2.1	2.2	1.0
Organic volatiles	-	< 0.1	0.1	0.3	0.7	0.8	0.8	0.8
CO_2	-	< 0.1	0.1	0.4	1.2	3.2	6.8	9.3
Total volatiles	-	< 0.1	0.2	0.7	1.9	4.0	7.6	10.1
Total extracted	95.6	90.5	94.9	93.5	91.1	82.6	76.3	68.9
Total recovered	95.6	90.5	95.1	94.2	93.0	86.6	83.9	79.0

Extractability from all samples declined with time, with 62.2–73 percent AR extracted at day 76.

The metabolite G-27550 accounted for a maximum of 67.6 percent and 75.2 percent AR in the clay loam and sandy loam soils, respectively at day 30 and 49.1 percent AR in loamy sand soil at day 55. Another identified degradate, GS-31144, was present at 4.2–4.7 percent AR at day 76. Volatile radioactivity accounted for a maximum of 10.2 percent AR at day 76, with $^{14}CO_2$ accounting for up to 9.9 percent AR. No other degradates of diazinon detected exceeded 2.5 percent AR.

The half-time (DT_{50}) and DT_{90} values were determined using single first order kinetics (SFO) and are outlined in Table 37.

Table 37 Summary of DT_{50} and DT_{90} values for diazinon

Test Soil	DT_{50} Diazinon (days)	DT_{90} Diazinon (days)
Sandy loam	8.0	26
Loamy sand	23	75
Clay loam	9.9	33

In a third study (R-14086) the degradation rate of the metabolite G-27550 was investigated. The same soils as outlined in the second study above (sandy loam, loamy sand and clay loam) were used. G-27550, labelled in the pyrimidine ring, was applied at a rate of 0.6 mg/kg dw of soil. The soil samples were incubated in the dark at 20 °C for 76 days. Samples were extracted with acetonitrile and acetonitrile: water (4:1, v/v). The extraction procedure was repeated three times. The TRR were determined in the extracts by combustion with LSC. Extracts were analysed by HPLC-UV and TLC. The amount of radioactivity in non-extracted material was not determined in the study.

The distribution of radioactivity is outlined in Table 38.

Table 38 Distribution of radioactivity (percent of AR)

Metabolite	Days after treatment							
	0	1	3	7	14	30	59	120
Sandy loam								
GS-31144	< 0.2	< 0.1	0.3	0.4	0.7	0.4	4.2	4.1
G-27550	104.4	104.0	104.9	95.2	92.8	86.6	71.2	53.6
Unknown 4	< 0.2	< 0.1	< 0.1	< 0.2	< 0.2	< 0.4	0.5	0.4
Unknown 5	< 0.2	< 0.1	< 0.1	< 0.2	< 0.2	< 0.4	0.5	< 0.2
Unknown 6	< 0.2	< 0.1	< 0.1	< 0.2	< 0.2	< 0.4	0.3	0.6
Others	< 0.2	< 0.1	< 0.1	< 0.2	< 0.2	< 0.4	< 0.3	1.3
Total extracted	104.4	104.0	105.2	95.6	93.5	88.3	77.1	60.6
Loamy sand								
GS-31144	< 0.2	0.3	0.4	0.7	1.0	1.7	4.8	6.2
G-27550	103.8	103.1	102.5	94.6	92.6	84.7	74.5	54.0
Unknown 4	< 0.2	< 0.1	< 0.1	< 0.2	< 0.2	< 0.3	< 0.2	< 0.3
Unknown 5	< 0.2	< 0.1	< 0.1	< 0.2	< 0.2	< 0.3	< 0.2	< 0.3
Unknown 6	< 0.2	< 0.1	< 0.1	< 0.2	< 0.2	< 0.3	< 0.2	2.7
Others	< 0.2	< 0.1	< 0.1	< 0.2	< 0.2	< 0.3	0.3	1.3
Total extracted	103.8	103.4	102.9	95.3	93.6	88.2	80.4	64.8
Clay loam								
GS-31144	0.2	0.4	0.3	0.5	0.5	0.4	1.8	5.7
G-27550	101.0	103.1	100.0	92.7	92.5	85.3	71.4	51.6
Unknown 4	< 0.2	0.1	< 0.2	< 0.2	< 0.2	0.3	0.6	0.9
Unknown 5	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.3	< 0.2	< 0.2
Unknown 6	< 0.2	0.1	< 0.2	< 0.2	< 0.2	< 0.3	< 0.2	< 0.2
Others	< 0.2	< 0.1	< 0.2	< 0.2	< 0.2	< 0.3	< 0.2	< 0.2
Total extracted	101.2	103.7	100.3	93.2	93.0	86.8	76.4	58.6

The amount of G-27550 declined from 101–104.4 percent AR to 71.2–74.5 percent AR after 59 days and 51.6-54.0 percent after 120 days. A minor metabolite, GS-31144, represented a maximum of 6.2 percent AR after 120 days. No other radioactive components exceeded 2.7 percent AR at 120 days.

Degradation was slow in all three soils. The calculated DT_{50} and DT_{90} values (assuming first-order kinetics) are presented in Table 39.

Table 39 Summary of DT_{50} and DT_{90} values for G-27550

Test Soil	DT_{50} G-27550 (days)	DT_{90} G-27550 (days)
Sandy loam	124	411
Loamy sand	131	434
Clay loam	124	411

Residues in Succeeding or Rotational crops

Confined rotational crop studies

The nature of the residue in rotational crops has been investigated two studies.

Study 1 (ABR-90064)

Rotational crops of spring wheat, lettuce, sugar beet and soya beans were planted following the harvest of greenhouse grown maize treated with diazinon.

The primary crop of maize was treated with [^{14}C]-pyrimidine-diazinon (specific activity 9.7 µCi/mg, radiochemical purity of 98.8 percent) at a rate of 4.48 kg ai/ha, applied pre-emergence, followed by two foliar applications, at a rate of 3.5 kg ai/ha, made at growth stage of BBCH 30–39. The two foliar applications were made 50 and 74 days after sowing of the primary crop. The rotational crops were planted 98 days after the last foliar application to maize.

Rotational crop plant samples were harvested 156 days (lettuce), 206 days (spring wheat), 226 days (soya beans) and 288 days (sugar beets) after the last application. These crop samples are reported as representing crop maturity. Crops were also harvested at 25 percent and/or 50 percent maturity. The growth stages were not reported. Wheat samples were separated into tops, stalks, hulls and grain, sugar beet samples were separated into leaves and roots and soya bean plants were separated into stalks, pods and beans.

Total radioactive residues were determined by combustion with LSC. All samples were extracted two times with methanol: water (9:1, v/v). For spring wheat stalks and mature soya bean stalks the extracts were partitioned with hexane followed by ethyl acetate. Aqueous extracts from these samples were also subject to enzyme hydrolysis using ß-glucosidase (no experimental conditions were given).

Characterisation and identification was performed using 2D-TLC and/or HPLC analysis with mass spectral identification on specific metabolites. Samples were stored frozen prior to analysis. The length of storage is not stated.

The total radioactive residues and the distribution of radioactivity in the rotational crops is shown in Table 40.

The TRR ranged from 0.016 mg eq/kg for both mature sugar beet roots and tops to 0.624 mg eq/kg for mature wheat stalks. The solvent extractabilities with methanol: water (9:1, v/v) ranged from 11.6 percent TRR for mature wheat grain to 90.7 percent TRR for 50 percent mature sugar beet leaves.

The results of the partitioning were not presented in the report.

Table 40 Distribution of radioactive residues in rotational crops

Commodity	TRR (mg eq /kg)	Extracted (Methanol: water, 9: 1, v/v)		Non-extracted	
		(mg eq/kg)	Percent TRR	mg eq/kg	Percent TRR
Lettuce					
50 percent Mature Foliage	0.072	-	-	-	-
Mature Foliage	0.0385	0.0278	72.1	0.0107	27.9
Sugar beets					
25 percent Mature leaves	0.061	-	-	-	-
50 percent Mature leaves	0.0403	0.0366	90.7	0.0037	9.3
50 percent Mature Roots	0.048	-	-	-	-
Mature Tops	0.016	-	-	-	-
Mature Roots	0.016	-	-	-	-
Spring wheat					
25 percent Mature foliage	0.1363	0.0910	66.8	0.0453	33.2
50 percent Mature foliage	0.243	-	-	-	-
Mature Stalks	0.6242	0.438	70.1	0.187	29.9
Mature Hulls	0.4987	0.290	58.2	0.208	41.8
Mature Grain	0.2416	0.0280	11.6	0.214	88.5
Soybeans					
25 percent Mature Stalks	0.124	-	-	-	-
50 percent Mature Stalks	0.1609	0.101	63.0	0.0595	37.0
Mature Stalks	0.1866	0.137	73.2	0.0500	26.8
Mature Pods	0.2332	0.100	42.9	0.133	57.1
Mature Beans	0.1905	0.031	16.1	0.160	83.9

The identification of metabolites was only undertaken for wheat samples. Table 41 shows the identification of diazinon and its metabolites in wheat. Diazinon was only identified in mature stalks (7.21 percent TRR, 0.045 mg/kg). In a number of cases the individual levels of the metabolites were not determined.

In 25 percent mature foliage, the major metabolites/fractions identified were an unknown plus G-27550 (22 percent TRR, 0.03 mg eq/kg) and an unknown metabolite (10.3 percent TRR, 0.014 mg eq/kg).

In mature stalks, the major metabolites/fractions identified were an unknown plus G-27550 (21 percent TRR, 0.131 mg eq/kg) and an unknown metabolite (14.3 percent TRR, 0.089 mg eq/kg).

In mature hulls, the major metabolites/fractions identified were an unknown plus G-27550 (17 percent TRR, 0.085 mg eq/kg) and CL-XIX-29 plus three conjugates (11 percent TRR, 0.055 mg eq/kg).

In mature grain, none of the metabolites/fractions identified exceeded 3 percent TRR.

Table 41 Identification of diazinon and its metabolites in rotational crops of wheat

Fraction	25 percent Mature foliage		Mature Stalks		Mature Hulls		Mature Grain	
	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
TRR	100	0.1363	100	0.6242	100	0.4987	100	0.2416
Unknowns	10.3	0.014	14.3	0.089	6.62	0.033	2.48	0.006
Glucose conjugate of trihydroxy pyrimidinyl moiety I	8.80	0.012	5.77	0.036	11.0	0.055	0.823	0.002
CL-XIX-29 and JAK-III-57 glucose conjugate	3.67	0.005	-	-			-	-
CL-XIX-29	2.93	0.004	3.68	0.023	1.60	0.008	-	-
JAK-III-57	2.93	0.004	-	-	1.60	0.008	-	-
GS 31144	6.02	0.008	4.65	0.029	4.61	0.023	0.414	< 0.001
Unknown plus G-27550	22.0	0.03	21.0	0.131	17.0	0.085	0.828	0.002
Diazinon	-	-	7.21	0.045	-	-		-
Unknown (II)	2.20	0.003	-	-	-	-		-
Total identified**	24.35	0.033	21.31	0.133	18.8	0.094	1.237	0.002

Notes:

**Includes metabolites that have been quantified together. It does not include fractions that contain identified and unidentified metabolites.

Study 2 (ABR-90065)

Rotational crops of winter wheat, soya bean and sugar beet were planted outdoors following the harvest of primary crops treated with diazinon.

Primary crops of lettuce, beans with pods and potatoes were all treated with [^{14}C]-pyrimidine-diazinon (specific activity 9.8 µCi/mg, radiochemical purity of 98 percent) at a rate of 4.48 kg ai/ha, applied pre-emergence, followed by two foliar applications at a rate of 1.4 kg ai/ha. In all cases the pre-emergence application was made 1 day after sowing of the primary crop. For lettuce, the first foliar application was made 34 days after the pre-emergence application with the second foliar application being made 7 days later. For beans with pods, the the first foliar application was made 34 days after the pre-emergence application with the second foliar application being made 15 days later. For potatoes the first foliar application was made 75 days after the pre-emergence application with the second foliar application being made 7 days later.

The foliar applications to the primary crops occurred when there was approximately 25 percent crop interception for lettuce and beans with pods and 60 percent crop interception for potatoes.

Following the harvest of the primary crop of treated lettuce, rotational crops of winter wheat and lettuce were planted 90 DALA and 308 DALA respectively. Following harvest of the primary crop of treated beans with pods, a rotational crop of soya bean was planted 327 DALA and following the harvest of the primary crop of treated potatoes, a rotational crop of sugar beet was planted 294 DALA.

Plant samples of the rotational crops were taken at various time points following planting as outlined in Table 42. The growth stages of the crop are not reported. Wheat samples were separated into

tops, stalks, hulls and grain, sugar beet samples were separated into tops and roots and soya bean plants were separated into stalks, pods and beans.

Table 42 Time periods for harvesting of rotational crops

Crop fraction	Days after planting the rotational crops	Days after soil treatment
Wheat – planted 90 DALA		
Forage	48	379
25 percent mature stalks	233	364
50 percent mature stalks	260	391
Mature stalks	296	427
Mature hulls	296	427
Mature grain	296	427
Lettuce – planted 308 DALA		
50 percent maturity	42	391
Mature	56	405
Soya beans – planted 327 DALA		
25 percent mature stalks	39	415
50 percent mature stalks	70	446
Mature stalks	135	511
Mature pods	135	511
Mature beans	135	511
Sugar beets – planted 294 DALA		
25 percent mature tops	44	420
50 percent mature tops	70	446
50 percent mature beets	70	446
Mature tops	135	511
Mature beets	135	511

Total radioactive residues were determined by combustion with LSC. All rotational crop samples were extracted two times with methanol: water (9:1, v/v). Samples were stored at ≤ -12 °C for 15 months.

The total radioactive residues and the distribution of radioactivity in the rotational crops is shown in Table 43. The lowest TRR was 0.003 mg eq/kg for 25 percent and 50 percent mature stalks of winter wheat and for mature sugar beet tops. The highest TRR was 0.027 mg eq/kg for mature soya bean stalks.

The solvent extractabilities using methanol: water (9:1, v/v) ranged from 10.1 percent TRR for mature soya bean pods to 61.4 percent TRR for 50 percent mature soybean stalks. Owing to the TRR in sugar beet leaves and tops being low (0.003–0.007 mg eq/kg) no extraction was undertaken.

Table 43 Distribution of radioactive residues in rotational crops

Primary crop	Commodity	Total (mg eq/kg)	Percent Extracted (Methanol: water, 9: 1, v/v)		Percent Non-extracted	
			Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
Lettuce	Winter wheat – 90 DALA					
	Fall Grazing Leaves	0.014	-	-	-	-
	25 percent Mature Stalks	0.003	-	-	-	-
	50 percent Mature Stalks	0.003	-	-	-	-
	Mature Stalks	0.012	46.1	0.005	53.9	0.006

Primary crop	Commodity	Total (mg eq/kg)	Percent Extracted (Methanol: water, 9: 1, v/v)		Percent Non-extracted	
			Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
	Mature Hulls	0.011	26.4	0.003	73.6	0.008
	Mature Grain	0.008	-	-	-	-
Lettuce	Lettuce – 308 DALA					
	50 percent Mature Foliage	0.004	-	-	-	-
	Mature Foliage	0.006	-	-	-	-
Beans with pods	Soya beans – 327 DALA					
	25 percent Mature Stalks	0.009	-	-	-	-
	50 percent Mature Stalks	0.011	61.4	0.007	38.6	0.004
	Mature Stalks	0.027	28.7	0.008	71.3	0.019
	Mature Pods	0.012	10.1	0.001	89.9	0.011
	Mature Beans	0.015	16.3	0.002	83.7	0.013
Potatoes	Sugar beets – 294 DALA					
	25 percent Mature Tops	0.004	-	-	-	-
	50 percent Mature Tops	0.006	-	-	-	-
	50 percent Mature Roots	0.007	-	-	-	-
	Mature Tops	0.003	-	-	-	-
	Mature Roots	0.006	-	-	-	-

Notes:

No identification work was undertaken.

Field Rotational Crop Studies (G24880-2514)

Eight rotational crop field trials were conducted in the United States. Rotational crops of lettuce, turnips and wheat were planted 30, 60 and 180 days after the last application to primary crops of lettuce, squash (variety not stated), melon and tomato.

The primary crops were all treated with one application at 4.48 kg ai/ha, applied pre-emergence using a granular formulation, followed by five foliar applications at a rate of 0.56 kg ai/ha using an EC formulation. Table 44 summarises the application timings for the foliar applications to the primary crops.

Table 44 Application timings for the foliar applications to the primary crop

Trial number	Primary crop	Application No	Growth stage	Days after planting
1	Lettuce	1	Seedling	14
		2	Seeding	21
		3	Vegetative	28
		4	Vegetative	35
		5	Vegetative	42
2	Tomato	1	Immature flowering	59
		2	Immature flowering	66
		3	Immature flowering	73
		4	Immature flowering	80

Trial number	Primary crop	Application No	Growth stage	Days after planting
		5	Immature fruit	87
3	Lettuce	1	Seedling	43
		2	Seedling	51
		3	Seedling	57
		4	Immature plant	64
		5	Mature plant	71
4	Squash	1	Pre-bloom	20
		2	Bud set, pre-bloom	27
		3	Blooming fruit set	34
		4	Blooming fruit set	41
		5	Bloom, fruit set mature	48
5	Melon	1	Blooming	16
		2	Blooming fruit set	23
		3	Blooming fruit set	30
		4	Blooming	37
		5	Limited fruit and blooms	42
6, 7 and 8	Lettuce	1	Vegetative	14
		2	Vegetative	21
		3	Vegetative	28
		4	Vegetative	35
		5	Vegetative	42

Rotational crop samples were taken and stored frozen for a maximum period of 15 months prior to analysis.

Samples were analysed for diazinon, G-24576 and CGA-14128 using method AG-550A. Procedural recoveries were conducted at fortification levels of 0.01–1 mg/kg. Recoveries ranged from 80–131 percent for diazinon, 75–137 percent for G-24576 and 75–120 percent for CGA-14128.

The residues determined in rotational crops are outlined in Table 45. All residues of diazinon, G-24576 and CGA-14128 were less than 0.01 mg /kg in all rotational crops at all plant back intervals.

Table 45 Residues in rotational crops from trials conducted in the United States following an application of 1 × 4.48 kg ai/ha (applied pre-emergence) plus 5 × 0.56 kg ai/ha (applied as a foliar application post emergence) to the primary crops

Trial Number	Primary crop	Rotational crop	Portion analysed	DALA	Diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
1	Lettuce	Wheat	Forage	180	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Hay		< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Straw		< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Grain		< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
2	Tomato	Lettuce	Leaves	181	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)

Trial Number	Primary crop	Rotational crop	Portion analysed	DALA	Diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
		Turnips	Leaves	181	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Roots	181	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Wheat	Forage	30	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Hay	30	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Straw	30	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Grain	30	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Forage	60	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Hay	60	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Straw	60	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Grain	60	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
3	Lettuce	Turnip	Leaves	31	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Roots	31	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Leaves	60	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Roots	60	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
4	Squash	Lettuce	Leaves	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Turnip	Leaves	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Roots	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Wheat	Forage	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Hay	182	< 0.01, < 0.01	< 0.01,	< 0.01,

Trial Number	Primary crop	Rotational crop	Portion analysed	DALA	Diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
					(< 0.01)	< 0.01 (< 0.01)	< 0.01 (< 0.01)
			Straw	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Grain	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
5	Melon	Lettuce	Leaves	34	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Turnips	Leaves	34	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Roots	34	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Leaves	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Roots	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Wheat	Forage	34	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Hay	34	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Straw	34	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Grain	34	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Forage	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Hay	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Straw	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Grain	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
6	Lettuce	Lettuce	Leaves	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Turnip	Leaves	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01	< 0.01, < 0.01

Trial Number	Primary crop	Rotational crop	Portion analysed	DALA	Diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
						(< 0.01)	(< 0.01)
			Roots	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Wheat	Forage	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Hay	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Straw	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Grain	61	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
7	Lettuce	Lettuce	Leaves	33	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Turnip	Leaves	33	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Roots	33	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Wheat	Forage	33	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Hay	33	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Straw	33	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Grain	33	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
8	Lettuce	Lettuce	Leaves	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Turnip	Leaves	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Roots	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
		Wheat	Forage	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Hay	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Straw	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)

Trial Number	Primary crop	Rotational crop	Portion analysed	DALA	Diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
			Grain	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
			Leaves	182	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Animal metabolism

The meeting received information on metabolism of diazinon in ruminants (lactating goat) and poultry (laying hens).

Lactating goat (studies ABR-88117, ABR-8818, ABR-88135)

Two lactating goats (unstated breed) were orally dosed by capsule with [^{14}C]-pyrimidine-diazinon (specific activity 9.7 µCi/mg, radiochemical purity 98.8 percent). The goats were dosed daily for four consecutive days at a nominal rate of 4.1 mg/kg bw/day (109–114 ppm feed). The biological phase and the initial analytical phase were reported in one study with subsequent analytical work performed in a further two studies.

Milk, urine and faeces were collected daily. The animals were sacrificed approximately 24 hours after administration of the last dose and samples of liver, kidney, fat and muscle were taken. Samples were homogenised and the total radioactivity in the samples was determined by combustion with LSC.

Milk samples were extracted with acetonitrile: water (1:1, v/v) and partitioned with hexane. The aqueous phase was then partitioned with butanol followed by ethyl acetate. Tissue samples were extracted three times with methanol: water (9:1, v/v) and partitioned with hexane. The aqueous phase was then partitioned with butanol followed by ethyl acetate.

Characterisation and identification was performed using 2D-TLC and/or HPLC analysis with mass spectral identification on specific metabolites. Samples were stored frozen for up to 5 months prior to analysis.

The TRR in mg eq/kg and as a percentage of the total dose applied are shown in Tables 46, 47 and 48 for urine and faeces, milk and tissues respectively.

Table 46 Radioactive residues in ruminant urine and faeces from lactating goats administered with [^{14}C]-pyrimidine-diazinon

Sampling day	percent of dose			
	Urine		Faeces	
	Animal 1	Animal 2	Animal 1	Animal 2
Day 1	1.86	14.17	0.43	0.92
Day 2	13.23	16.23	3.02	5.58
Day 3	36.18	18.33	2.98	2.97
Day 4	12.37	15.8	2.35	2.57
Total recovery	63.64	64.53	8.78	12.04

Table 47 Radioactive residues in ruminant milk from lactating goats administered with [^{14}C]-pyrimidine-diazinon

Sampling day	Animal 1		Animal 2		Mean	
	mg eq/kg	percent of dose	mg eq/kg	percent of dose	mg eq/kg	percent of dose
Day 1	0.453	0.08	0.2	0.05	0.327	0.07
Day 2	0.613	0.1	0.228	0.06	0.421	0.08
Day 3	0.687	0.1	0.242	0.06	0.465	0.08
Day 4	0.687	0.09	0.236	0.06	0.462	0.08
Total recovery	2.44	0.37	0.906	0.23	1.675	0.31

Table 48 Radioactive residues in ruminant tissues from lactating goats administered with [^{14}C]-pyrimidine-diazinon

	Animal 1		Animal 2	
	mg eq/kg	percent of dose	mg eq/kg	percent of dose
Tissue				
Muscle (tenderloin)	0.406	0.02	0.166	0.01
Muscle (leg)	0.448	1.02	0.142	0.29
Liver	1.566	0.16	0.878	0.09
Kidney	3.019	0.04	0.998	0.02
Omental fat	0.363	0.08	0.150	0.03
Perirenal fat	0.356	0.03	0.112	ND
Total	6.158	1.35	2.446	0.44

The distribution of the radioactivity in tissues is shown in Table 49.

The TRR ranged from 0.112 mg eq/kg for perirenal fat to 3.019 mg eq/kg in kidney. The solvent extractability for milk (acetonitrile: water, 1:1, v/v) was 91.5 percent TRR. The majority of the extracted residue was found to be organosoluble. The solvent extractabilities for tissues (methanol: water, 9:1, v/v) ranged from 79 percent TRR for kidney to 99 percent TRR for muscle (leg). The majority of the extracted residue was found to be organosoluble.

Table 49 Distribution of radioactivity in samples from lactating goats administered with [^{14}C] pyrimidine-diazinon (animal 1)

Tissue	TRR by combustion (mg eq/kg)	Aqueous extracted		Organic extracted		Non-extracted	
		Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
Liver	1.566	33	0.52	46	0.72	21	0.33
Kidney	3.019	31	0.94	64	1.93	6	0.18
Omental fat	0.363	1.9	0.007	95	0.34	5	0.02
Perirenal fat	0.356	0.99	0.004	95	0.34	4	0.014
Muscle (Tenderloin)	0.406	9.5	0.04	86	0.35	5	0.020
Muscle (leg)	0.448	0.99	0.004	98	0.44	1	0.004
Milk (day 4)	0.687	5.46	0.04	86	0.59	9	0.06

The identification and characterization of metabolites is shown in Table 50. In a number of cases the individual levels of the metabolites were not determined.

Table 50 Identification of diazinon and its metabolites in combined aqueous and organic extracts of samples from lactating goats administered with [^{14}C]-pyrimidine-diazinon (animal 1)

Matrix	Liver	Kidney	Fat (omental)	Fat (perirenal)	Muscle (tender-loin)	Muscle (leg)	Milk
TRR by combustion (mg eq/kg)	1.566	3.019	0.363	0.356	0.406	0.448	0.687 (Milk day 4)
Percent TRR [mg eq/kg]							
Organic soluble	46	64	95	95	86	98	86
	[0.72]	[1.93]	[0.34]	[0.34]	[0.35]	[0.44]	[0.59]
Peak eluting at origin	4.5	10.2	1.3	0.7	0.8	0.9	1.3
	[0.071]	[0.308]	[0.005]	[0.03]	[0.003]	[0.004]	[0.009]
Unidentified peaks#	2.4	3.1	2	1.8	9.3	8.5	7.4
	[0.038]	[0.094]	[0.007]	[< 0.008]	[0.039]	[0.037]	[0.051]
GS-31144	19	30.6	6.8	4.2	39.4	40.4	37.3
	[0.298]	[0.924]	[0.025]	[0.015]	[0.16]	[0.181]	[0.256]
G-27550	19.2	19.8	9.3	4.3	26	35.3	39.3
	[0.301]	[0.598]	[0.034]	[0.015]	[0.106]	[0.158]	[0.270]
G-24576	0.3	0.3	4.1	0.8	1	< 0.1	0.15
	[0.005]	[0.009]	[0.015]	[0.003]	[0.004]	[< 0.001]	[0.001]
CGA-14128	0.2	< 0.1	12.8	12.3	1.4	0.4	0.1
	[0.003]	[< 0.003]	[0.047]	[0.044]	[0.006]	[0.002]	[< 0.001]
Diazinon	0.2	< 0.1	67.8	64	6.2	1.6	0.15
	[0.003]	[< 0.003]	[0.246]	[0.228]	[0.025]	[0.007]	[0.001]
Aqueous soluble	33	31	1.9	0.99	9.5	0.99	5.46
	[0.52]	[0.94]	[0.007]	[0.004]	[0.04]	[0.004]	[0.04]
Peak eluting at origin	18	2.8	-	-	0.8	1.6	-
	[0.282]	[0.085]			[0.003]	[0.007]	
Unidentified peaks#	14.6	26.7	-	-	6.5	9.1	-
	[0.23]	[0.807]			[0.027]	[0.041]	
GS-31144	< 0.01	0.4	-	-	1.1	< 0.1	-
G-27550	[0.001]	[0.012]	-	-	[0.005]	[< 0.001]	-
G-24576	< 0.01	0.2	-	-	1.1	< 0.1	-
CGA-14128	[0.001]	[0.006]	-	-	[0.005]	[< 0.001]	-
Diazinon			-	-			-
Total extracted	79	94	100	96	95	99	91
	[1.24]	[2.84]	[0.367]	[0.342]	[0.386]	[0.444]	[0.625]
Total identified**	38.9	51.5	100	85.6	76.2	78	77
	[0.61]	[1.56]	[0.367]	[0.305]	[0.309]	[0.349]	[0.529]
Unextracted (PES)	21	6	5	4	5	1	9
	[0.33]	[0.18]	[0.02]	[0.014]	[0.02]	[0.004]	[0.06]

Notes:

Consists of 1-5 discrete peaks, with maximum individual TRR ≤ 15 percent.

** Includes metabolites that have been quantified together.

Tissue samples, from the initial extraction with methanol: water (9: 1, v/v), were also subjected to acid hydrolysis (6 M HCl at 85 °C, left overnight). The results are summarised in Table 51. In a number of cases the individual levels of the metabolites were not determined.

Table 51 Identification of diazinon and its metabolites following acid hydrolysis of selected tissue extracts

Matrix	Liver	Kidney	Muscle (tender-loin)	Muscle (leg)
Percent TRR [mg/kg]				
TRR extracted	79 [1.24]	94 [2.84]	95 [0.386]	99 [0.444]
Peak eluting at origin	6.8 [0.107]	1.7 [0.051]	1.6 [0.007]	3.7 [0.017]
Unidentified peaks	22.9 [0.358]	28 [0.845]	18.7 [0.076]	21.5 [0.097]
GS-31144	24.1 [0.377]	41.1 [1.241]	72.7 [0.295]	73.3 [0.328]
G-27550	24.7 [0.387]	22.8 [0.688]		
G-24576 CGA-14128 Diazinon	0.5 [0.008]	0.4 [0.012]	< 0.1 [< 0.001]	0.4 [0.002]

Liver and kidney organosoluble extracts and kidney aqueous soluble extracts were also treated with β-glucuronidase (incubated at 37 °C, left overnight). No detailed results are reported; but it is stated that increased levels of GS-31144 and G-27550 were observed indicating the potential presence of glucuronic conjugates of these metabolites.

Poultry (studies ABR-88116, ABR-119, ABR-88135 and ABR-89040)

Four laying hens (leghorn) were orally dosed by capsule with [^{14}C]-pyrimidine-diazinon (specific activity 30.3 µCi/mg, radiochemical purity of 99.1 percent) once daily for seven consecutive days at a nominal rate of 1.6 mg/kg bw/day (25 ppm feed). There was one control animal. The biological phase and initial analytical phase was undertaken in one study. The additional three studies undertook further analytical work.

Samples of excreta and eggs (separated into whites and yolk) were collected daily. At sacrifice, approximately 24 hours after the last dose, samples of liver, kidney, muscle , skin with attached fat and peritoneal fat. The samples were homogenised, combusted and analysed by LSC. Samples were stored frozen for up to 12 months prior to analysis.

Egg and tissue samples were extracted three times with methanol: water (9:1, v/v) and where then partitioned with hexane followed by butanol. Characterisation and identification was performed using 2D-TLC and/or HPLC analysis with mass spectral identification on specific metabolites.

The TRR in mg eq/kg and as a percentage of the total radioactivity applied are shown in Table 52 for excreta and eggs and in table 53 for tissue samples.

Table 52 Radioactive residues in eggs and excreta from laying hens administered[^{14}C]-pyrimidine-diazinon

Sampling day	Excreta	Egg yolk		Egg white		Whole egg†	
	percent of dose	mg eq/kg	percent of dose	mg eq/kg	percent of dose	mg eq/kg	percent of dose
Day 1	12.71	0.006	< 0.01	0.044	0.01	0.03	0.01
Day 2	9.43	0.013	< 0.01	0.045	0.01	0.034	0.01
Day 3	10.83	0.027	< 0.01	0.051	0.01	0.043	0.01
Day 4	11.88	0.043	< 0.01	0.048	0.01	0.047	0.01
Day 5	9.99	0.053	< 0.01	0.042	0.01	0.046	0.01
Day 6	12.22	0.061	< 0.01	0.038	0.01	0.046	0.01
Day 7	11.58	0.065	< 0.01	0.066	0.01	0.066	0.01
Total recovery	78.64	0.268	< 0.01	0.334	0.07	0.312	0.07

Notes:

† Determined based on the total weights of separated egg white and yolk.

Table 53 Radioactive residues tissues from laying hens administered [^{14}C] pyrimidine-diazinon

Sample	mg eq/kg	percent of dose
Liver	0.110	0.02
Kidney	0.148	0.01
Muscle	0.025	0.05
Skin with attached fat	0.018	0.01
Peritoneal fat pad	0.010	< 0.01
Total	0.311	0.09

The distribution of the radioactivity in tissues is shown in Table 54.

The TRR ranged from 0.01 mg eq/kg for perirenal fat to 0.148 mg eq/kg for kidney. The solvent extractability for egg yolk (methanol: water, 9:1, v/v) was 67 percent TRR. The extracted residue was found to be mostly organosoluble. For egg whites the solvent extractability (methanol: water, 9:1, v/v) was 98 percent TRR. The majority of the extracted residue was found to be organosoluble. The solvent extractabilities for tissues (methanol: water, 9:1, v/v) ranged from 31 percent TRR for peritoneal fat to 76 percent TRR for kidney.

Table 54 Distribution of radioactivity in samples from laying hens administered with [^{4}C]-pyrimidine-diazinon

Tissue	TRR by combustion (mg eq/kg)	Aqueous extracted		Organic extracted		Non-extracted	
		Percent TRR	mg eq/kg	Percent TRR	mg eq/kg	Percent TRR	mg eq/kg
Egg yolk (Day 7)	0.065	8	0.005	59	0.0384	33	0.0215
Egg white (day 7)	0.066	13	0.009	85	0.0561	2	0.001
Liver	0.110	32	0.0352	31	0.0341	37	0.0407
Kidney	0.148	40	0.0592	36	0.0533	24	0.0355
Lean meat	0.025	33	0.008	31	0.008	36	0.009
Skin plus fat	0.018	17	0.003	27	0.005	56	0.01
Peritoneal fat	0.01	12	0.001	19	0.002	69	0.007

The identification and characterization of metabolites is shown in Table 55. In a number of cases the individual levels of the metabolites were not determined.

Table 55 Identification of diazinon and its metabolites in combined aqueous and organic extracts of samples from laying hens administered with [^{14}C]-pyrimidine-diazinon

Matrix	Egg Yolk (day 7)	Egg white (day 7)	Liver	Kidney	Lean meat	Skin with fat	Peritoneal fat
TRR (mg eq/kg)	0.065	0.066	0.110	0.148	0.025	0.018	0.01
Percent TRR [mg eq/kg]							
Organic soluble	59.0 [0.038]	85.3 [0.056]	30.9 [0.034]	36.5 [0.054]	31.4 [0.008]	26.8 [0.005]	19.2 [0.002]
Unidentified peaks eluting at the origin	18.2 [0.012]	6 [0.004]	16.1 [0.018]	4 [0.006]	6.8 [0.002]	4.8 [< 0.001]	5.7 [< 0.001]
Unidentified peaks	10.7 [0.008]	35.9 [0.024]	9.4 [0.01]	26.3 [0.039]	16.1 [0.006]	7.2 [< 0.003]	4.2 [< 0.001]
GS-31144	18.6 [0.012]	33.3 [0.022]	3.5 [0.004]	3.7 [0.006]	6.5 [0.002]	4.2 [< 0.001]	3.1 [< 0.001]
G-27550	11.1 [0.007]	9.4 [0.006]	0.6 [< 0.001]	2.3 [0.003]	2 [< 0.001]	2.6 [< 0.001]	0.7 [< 0.001]
G-24576	0.4 [< 0.001]	1.3 [< 0.001]	0.9 [0.001]	0.2 [< 0.001]	0.3 [< 0.001]	0.13 [< 0.001]	4.3 [< 0.001]
CGA-14128	0.06 [< 0.001]	0.05 [< 0.001]	< 0.001 [< 0.001]	0.1 [< 0.001]	< 0.1 [< 0.001]	0.9 [< 0.001]	1.4 [< 0.001]
Diazinon	0.02 [0.001]	0.03 [< 0.001]	0.03 [< 0.001]	< 0.01 [< 0.001]	< 0.1 [< 0.001]	< 0.01 [< 0.001]	2 [< 0.001]
Aqueous soluble	8.0 [0.005]	12.7 [0.008]	32.1 [0.035]	39.5 [0.058]	32.6 [0.008]	17.2 [0.003]	11.8 [0.001]
Unidentified peaks eluting at the origin	1.5 [0.001]	0.5 [< 0.001]	16 [0.018]	10.8 [0.016]	7.5 [0.002]	1.8 [< 0.001]	0.8 [< 0.001]
Unidentified peaks	4.9 [0.004]	10.9 [0.008]	11.1 [0.012]	28.4 [0.042]	24.7 [0.007]	14.7 [0.004]	7.3 [< 0.003]
GS-31144	< 0.01 [< 0.001]	1.3 [< 0.001]	5 [0.006]	0.2 [< 0.001]	0.1 [< 0.001]	0.5 [< 0.001]	3.2 [< 0.001]
G-27550							
G-24576	1.6 [0.001]	0.19 [< 0.001]	0.01 [< 0.001]	0.2 [< 0.001]	0.3 [< 0.001]	0.1 [< 0.001]	0.5 [< 0.001]
CGA-14128							
Diazinon							

All the initial extracts with methanol/water, except fat, were subjected to acid hydrolysis (6 M HCl at 85 °C, left overnight). The summary of these results are shown in Table 56. In a number of cases the individual levels of the metabolites were not determined.

Table 56 Identification of diazinon and its metabolites following acid hydrolysis of selective tissue extracts

Matrix	Egg Yolk (day 7)	Egg white (day 7)	Liver	Kidney	Lean meat	Skin with fat
Percent TRR [mg/kg]						
TRR extracted	67 [0.0434]	98 [0.0651]	63 [0.0693]	76 [0.1125]	64 [0.016]	44 [0.008]
Unidentified peaks eluting at the origin	4.62 [0.003]	6.06 [0.004]	5.45 [0.006]	9.46 [0.014]	8.00 [0.002]	5.56 [< 0.001]
Unidentified peaks	52.3 [0.034]	50 [0.033]	39.1 [0.043]	41.9 [0.062]	52 [0.013]	16.7 [0.003]

Matrix	Egg Yolk (day 7)	Egg white (day 7)	Liver	Kidney	Lean meat	Skin with fat
GS-31144	10.8 [0.007]	42.4 [0.028]	7.27 [0.008]	23.0 [0.034]	4 [0.001]	16.7 [0.003]
G-27550			7.27 [0.008]			
G-24576	1.5 [< 0.001]	1.5 [< 0.001]	3.64 [0.004]	2.03 [0.003]	4 [< 0.001]	5.56 [< 0.001]
CGA-14128						
Diazinon						

In a follow up study, the aqueous extracts were partitioned with hexane, then butanol followed by ethyl acetate. The resulting organic and aqueous fractions were treated with β-glucuronidase (incubated at 37 °C, left overnight). The metabolites GS-31144, G-27550 and CL-XIX-29 were identified following treatment with β-glucuronidase.

The overall identification and characterization of metabolites is shown in Table 57.

Table 57 Identification of diazinon and its metabolites in combined aqueous and organic extracts of samples from laying hens administered with [^{14}C]-pyrimidine-diazinon

Matrix	Egg Yolk (day 7)	Egg white (day 7)	Liver	Kidney	Muscle	Skin with fat	Peritoneal fat
TRR (mg eq/kg)	0.065	0.066	0.110	0.148	0.025	0.018	0.01
Percent TRR [mg eq/kg]							
Organosoluble							
Unknown	2.89 [0.002]	-	-	-	-	6.30 [0.001]	1 [< 0.001]
Glucuronide conjugates†	25.2 [0.016]	41.3 [0.027]	23.5 [0.026]	24.6 [0.036]	22.39 [0.006]	2.31 [< 0.001]	9.71 [0.001]
CL-XIX-29			1.98 [0.002]	5.73 [0.008]		9.69 [0.002]	
GS-31144	18.6 [0.012]	33.3 [0.022]	3.46 [0.004]	3.69 [0.006]	6.52 [0.002]	4.16 [0.001]	3.06 [0.001]
G-27550	11.1 [0.007]	9.38 [0.006]	0.59 [< 0.001]	2.26 [0.003]	1.98 [< 0.001]	2.61 [< 0.001]	0.73 [< 0.001]
G-24576	0.42 [,0.001]	1.28 [< 0.001]	0.89 [0.001]	0.18 [< 0.001]	0.24 [< 0.001]	1.25 [< 0.001]	0.77 [< 0.001]
CGA-14128	0.06 [< 0.001]	0.05 [< 0.001]	< 0.01 [< 0.001]	0.11 [< 0.001]	0.03 [< 0.001]	0.02 [< 0.001]	1.37 [< 0.001]
Diazinon	0.02 [< 0.001]	0.03 [< 0.001]	0.03 [< 0.001]	0.08 [< 0.001]	0.04 [< 0.001]	0.89 [< 0.001]	2 [< 0.001]
Aqueous soluble							
Glucuronide conjugates†	3.25 [< 0.003]	2.53 [< 0.003]	23.1 [0.026]	31.7 [0.047]	30.7 [0.008]	8.31 [< 0.004]	3.47 [< 0.003]
CL-XIX-29	2.2 [0.001]	8.78 [0.006]	4.02 [0.004]	7.51 [0.011]	1.50 [< 0.001]	8.24 [0.002]	4.64 [< 0.001]
GS-31144	2.57 [0.002]	1.45 [0.001]	5.01 [0.006]	0.36 [< 0.001]	0.17 [0.001]	0.63 [< 0.001]	3.67 [0.001]
G-27550							
G-24576							
CGA-14128							
Diazinon							
Total extracted	67	98	63	76	64	46	31

Matrix	Egg Yolk (day 7)	Egg white (day 7)	Liver	Kidney	Muscle	Skin with fat	Peritoneal fat
	[0.0435]	[0.065]	[0.0693]	[0.1125]	[0.016]	[0.008]	[0.003]
Total identified#	63.4 [0.0412]	98 [0.065]	62.6 [0.0689]	76.2 [0.113]	63.6 [0.0159]	38.1 [0.007]	29.4 [0.003]
Total unextracted (PES)	33 [0.0215]	2 [0.001]	37 [0.0407]	24 [0.0355]	36 [0.009]	56 [0.01]	69 [0.007]

Notes:

† G-27550, GS-31144, CL-XIX-29 identified following treatment with β-glucuronidase.

Includes metabolites that have been quantified together.

In a further follow up study, the PES from egg yolk and tissues were subjected to enzymatic hydrolysis (incubated with protease at 37 °C and left overnight). The post-enzyme (protease) extraction radioactivity distributions are summarized in Table 58.

Table 58 Distribution of radioactivity released from the PES of egg and tissue samples following treatment with protease

Matrix	Percent TRR released by enzymatic treatment [mg eq/kg]	Percent TRR remaining in solids [mg eq/kg]
Egg yolk (day 7) - PES	N/A	33 [0.021]
Protease	21 [0.014]	12 [0.008]
Liver - PES	N/A	37 [0.041]
Protease	19 [0.021]	18 [0.02]
Kidney - PES	N/A	24 [0.036]
Protease	22 [0.033]	2 [0.003]
Muscle - PES	N/A	36 [0.009]
Protease	30 [0.008]	6 [0.002]
Skin with fat - PES	N/A	56 [0.01]
Protease	56 [0.01]	0
Peritoneal fat	N/A	69 [0.007]
Protease	69	0

Notes:

N/A Not applicable.

Further analysis of the radioactivity released following protease treatment was only undertaken for the liver samples. It is reported that residues of diazinon, CGA-14128, G-24576, G-27550, GS-31144 and CL-XIX-29 were found. No further detailed are available.

In Figure 3 an overall proposal for the metabolic pathway of diazinon in goat and hens is outlined.

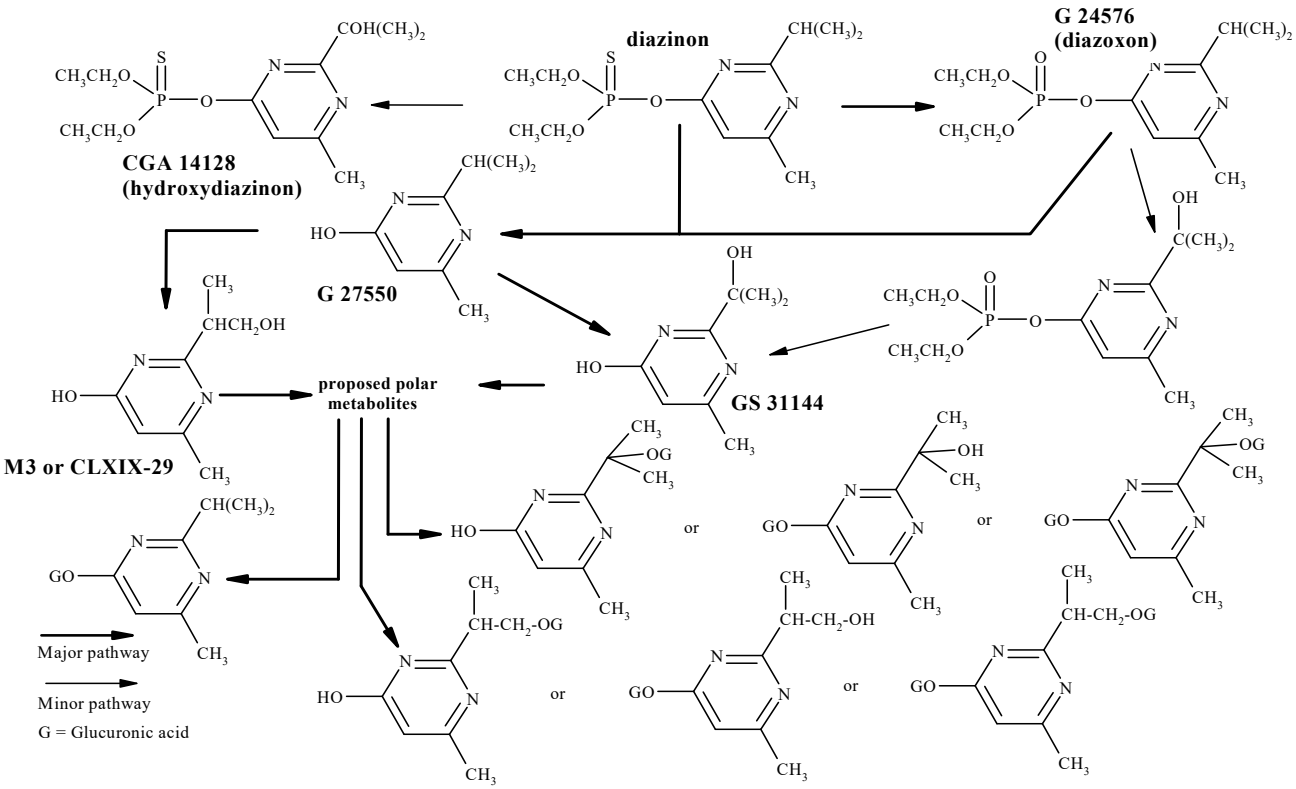


Figure 3 Proposed metabolic pathway in goats and hens

RESIDUE ANALYSIS

Analytical methods

Method MAK 824/042610

Residues of diazinon were extracted from pineapple pulp and peel using acetonitrile: water (9: 1, v/v) followed by centrifugation. The supernatant was concentrated under vacuum and 5 percent w/v sodium chloride solution and hexane was added. The upper hexane layer was partitioned twice with acetonitrile. The two acetonitrile phases were combined and the solvent removed under vacuum. The residue was redissolved in 5 ml of methanol: water (1:1, v/v). Final determination was by LC-MS/MS using a C8 column, gradient elution and using the ion transition m/z 305.4 → 169.2. An ILV of the method was also undertaken.

A summary of the recovery data are outlined in table 59. The method was linear over the range 1–50 ng/mL.

Table 59 Recovery data for analytical method MAK 824/042610 used to determine residues of diazinon in pineapple

Crop Study reference	Fraction	Fortification level [mg/kg]	Individual recoveries [percent]	Range of recoveries [percent]	Mean recovery [percent]	RSD
Pineapple MAK 824/042610 –	Peel	0.01	79, 79, 80, 82, 84	79 - 84	81	2.7
		0.1	75, 81, 81, 82, 78	75 - 82	79	3.6

Crop Study reference	Fraction	Fortification level [mg/kg]	Individual recoveries [percent]	Range of recoveries [percent]	Mean recovery [percent]	RSD
Primary validation data						
	Pulp	0.01	80, 80, 82, 80, 79	79 - 82	80	1.4
		0.1	86, 74, 84, 75, 88	74 - 88	81	7.9
Pineapple 20051340/01-RVP - ILV	Peel	0.01	84, 86, 92, 86, 80	80 - 92	86	5
		0.1	96, 91, 85, 79, 93	79 - 96	89	8
	Pulp	0.01	83, 81, 83, 88, 83	81 - 88	84	3
		0.1	85, 85, 84, 91, 92	84 - 92	89	6

AG-550 and AG-550 A

AG-550 and AG-550 A are the same method, with AG-550 A representing an update to include additional validation data.

Diazinon, G-24576 and CGA-14128 were extracted from animal tissues (excluding fat), milk, eggs, and crops, including processed fractions, using acetone: water (9:1, v/v) followed by extraction with petroleum ether: dichloromethane: water (5: 5: 1, v/v/v). The residues were then partitioned with dichloromethane. The dichloromethane layer was evaporated to dryness and reconstituted in acetone.

Residues of diazinon, G-24576 and CGA-14128 present in fat were extracted using acetonitrile and partitioned into hexane. The solvent was concentrated to dryness and reconstituted in acetone: water (9:1, v/v).

Sample clean-up was achieved by solid-phase extraction using a florisil Sep-Pak cartridge prior to analysis. Final determination was achieved by GC-FPD

Additional validation for the determination of diazinon in products of animal origin was undertaken using the same extraction procedures outlined above but with final determination achieved using GC-NPD.

A summary of the recovery data are outlined in Tables 60, 61 and 62 for diazinon, G-2465 and CGA-14128 respectively. For the GC-FPD method, the individual recoveries were not reported. The method was linear over the range 5–100 ng/mL for the GC-FPD method and 10–200 ng/mL for the GC-NPD method.

Table 60 Recovery data for analytical method AG-550 used to determine residues of diazinon

Method Study reference	Commodity	Fortification level [mg/kg]	Individual recoveries [percent]	Range of recoveries [percent]	Mean recovery [percent]	RD (n)
	Tomatoes				105	5 (4)
AG-550/ GC-FPD	Carrots				75	5 (4)
G 24480/723	Cucumbers				107	10 (4)
	Peas				104	10 (4)
	Apples				102	5 (4)
	Peppers				98	13 (4)
	Radishes				116	4 (4)
	Potatoes				117	5 (4)
	Cabbage				117	7 (4)
	Cantaloupe				108	4 (4)
	Squash	0.01			114	9 (4)

Method Study reference	Commodity	Fortification level [mg/kg]	Individual recoveries [percent]	Range of recoveries [percent]	Mean recovery [percent]	RD (n)
	Broccoli	0.05	Not reported	Not reported	83	5 (4)
	Bulb onions	0.5			93	14 (4)
	Raspberries				107	6 (4)
	Green beans				110	21 (4)
	Almonds				80	6 (4)
	Refined corn oil				84	20 (4)
	Crude corn oil				90	18 (4)
	Refined bleached corn oil				111	8 (4)
	Bovine liver				105	8 (4)
AG-550/GC-FPD	Bovine fat	0.01			114	14 (4)
G 24480/723	Milk	0.05	Not reported	Not reported	103	16 (4)
	Poultry muscle	1			100	12 (4)
	Eggs				100	13 (4)
AG-550 A/GC-FPD	Pears				103	7 (4)
G 24480/1690						
	Almond hulls				109	15 (4)
	Plums		Not reported	Not reported	103	6 (4)
	Strawberries				92	13 (4)
	Hops	0.01			104	27 (4)
	Field corn	0.05			128	4 (4)
	Lettuce	1			125	4 (4)
	Bovine muscle	0.005	101, 90, 85, 80, 80	80-101	87	10 (5)
AG-550 A/ GC- NPD		0.05	96, 81, 111, 101, 86	83-95	89	6 (5)
2418	Bovine kidney	0.005	81, 94, 1018, 108, 88	81-108	96	13 (5)
		0.05	93, 109, 103, 90, 94	90-94	98	8 (5)
	Swine liver	0.005	96, 81, 111, 101, 86	81-111	95	13 (5)
		0.05	85, 112, 109, 115, 110	85-115	106	11 (5)

Table 61 Recovery data for analytical method **AG-550** used to determine residues of G-24576

Method Study reference	Commodity	Fortification level [mg/kg]	Individual recoveries [percent]	Range of recoveries [percent]	Mean recovery [percent]	RSD (n)
AG-550/GC-FPD G 24480/723	Tomatoes				94	8 (4)
	Carrots				92	6 (4)
	Cucumbers				112	10 (4)
	Peas				101	2 (4)
	Apples				95	8 (4)
	Peppers				117	4 (4)

Method Study reference	Commodity	Fortification level [mg/kg]	Individual recoveries [percent]	Range of recoveries [percent]	Mean recovery [percent]	RSD (n)
	Radishes	0.01 0.05 0.5	Not reported	Not reported	106	8 (4)
	Potatoes				113	5 (4)
	Cabbage				114	7 (4)
	Cantaloupe				104	6 (4)
	Squash				106	5 (4)
	Broccoli				100	0.4 (4)
	Bulb onions				91	5 (4)
	Raspberries				85	8 (4)
	Green beans				113	5 (4)
	Almonds				85	5 (4)
	Refined corn oil				99.5	14 (4)
	Crude corn oil				102	12 (4)
	Refined bleached corn oil				91.7	7 (4)
AG-550/GC-FPD G 24480/723	Bovine liver	0.01 0.05 1	Not reported	Not reported	68	9 (4)
	Bovine fat				125	11 (4)
	Milk				110	1.5 (4)
	Poultry muscle				96	11 (4)
	Eggs				123	19 (4)
AG-550 A/GC-FPD G 24480/1690	Pears	0.01 0.05 1.0	Not reported	Not reported	94	6 (4)
	Almond hulls				82	25 (4)
	Plums				94	6 (4)
	Strawberries				86	5 (4)
	Hops				54	18 (4)
	Field corn				104	18 (4)
	Lettuce				109	19 (4)

Table 62 Recovery data for analytical method **AG-550** used to determine residues of CGA-14128

Method Study reference	Commodity	Fortification level [mg/kg]	Individual recoveries [percent]	Range of recoveries [percent]	Mean recovery [percent]	RSD (n)
AG-550/GC-FPD G 24480/723	Tomatoes	0.01 0.05 0.5	Not reported	Not reported	88	3 (4)
	Carrots				80	14 (4)
	Cucumbers				117	13 (4)
	Peas				96	9 (4)
	Apples				81	9 (4)
	Peppers				107	5 (4)
	Radishes				108	12 (4)
	Potatoes				114	4 (4)
	Cabbage				112	8 (4)
	Cantaloupe				107	4 (4)
	Squash				112	8 (4)
	Broccoli				96	10 (4)
	Bulb onions				97	4 (4)
	Raspberries				107	2 (4)
	Green beans				114	6 (4)

Method Study reference	Commodity	Fortification level [mg/kg]	Individual recoveries [percent]	Range of recoveries [percent]	Mean recovery [percent]	RSD (n)
	Almonds				87	5 (4)
	Refined corn oil				87	5 (4)
	Crude corn oil				83	7 (4)
	Refined bleached corn oil				76	14 (4)
AG-550/GC-FPD G 24480/723 – Primary Validation	Bovine liver	0.01 0.05 1	Not reported	Not reported	107	8 (4)
	Bovine fat				118	8 (4)
	Milk				118	6 (4)
	Poultry muscle				102	8 (4)
	Eggs				95	12 (4)
AG-550 A/GC-FPD G 24480/1690-primary validation	Pears	0.01 0.05 1.0	Not reported	Not reported	98	10 (4)
	Almond hulls				82	11 (4)
	Plums				107	8 (4)
	Strawberries				117	10 (4)
	Hops				90	16 (4)
	Field corn				111	4 (4)
	Lettuce				121	8 (4)

Method REM 4/81

This method was used in the storage stability study for animal matrices.

Samples of muscle, liver and kidney were macerated with methanol. The extracts were diluted with water and partitioned with dichloromethane. The solvent was evaporated and the residue cleaned up via SPE prior to chromatographic analysis.

Samples of fat were ground with anhydrous sodium sulphate and refluxed in hexane. The hexane extract was cleaned up following partitioning with acetonitrile followed by SPE.

Extracts were analysed by gas chromatography using nitrogen-phosphorus or flame ionisation detection. The limit of determination of the method is stated to be 0.01 mg/kg for all matrices. No validation data were provided.

Stability of residues in stored analytical samples

The meeting received freezer storage stability data for diazinon, G-24576, CGA-14128 and G-27550 in a variety of plant and animal matrices.

Plant commodities

Corn, tomato, potato, apple, strawberry, lettuce, processed commodities

Samples of corn, tomato, apple, strawberry, lettuce and processed fractions were homogenised and then separate samples were fortified at 1 mg/kg with diazinon or G-24576 or CGA-1412. The samples were stored frozen at ≤ -12 °C. At each time point two stored samples, two freshly prepared procedural recoveries samples and an unfortified control sample were analysed.

Samples were analysed using method AG-550. The results are presented in Tables 63 to 65 for diazinon, G-24576 and CGA-14128.

Table 63 Storage stability data for diazinon residues in frozen plant matrices

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Corrected recovery † (mean,%)	Mean uncorrected recovery, percent†	Mean procedural recovery (n=2) (percent)
High water content	Tomato	Diazinon	0	1	116, 108 (112)	112	105
			99		82, 83 (83)	81	97
			179		74, 81 (78)	73	94
			448		81, 82 (82)	82	146
			782		86, 82 (84)	84	104
High water content	Apple	Diazinon	0	1	106, 106 (106)	99	93
			108		83, 88 (86)	75	87
			184		84, 83 (84)	84	121
			541		98, 102 (100)	88	88
			784		86, 76 (81)	66	81
High acid content	Strawberry	Diazinon	0	1	109, 85 (97)	97	103
			99		56, 60 (58)	53	91
			179		38, 44 (41)	41	101
			541		47, 57 (52)	41	78
			792		28, 30 (29)	27	93
High water content	Lettuce	Diazinon	0	1	101, 108 (105)	98	93
			106		95, 93 (94)	93	99
			184		100, 94 (97)	92	95
			433		88, 101 (95)	95	105
			792		95, 114 (105)	88	84
High starch content	Field corn grain	Diazinon	0	1	117, 89 (103)	80	78
			114		123, 91 (107)	87	81
			189		96, 95 (96)	95	99
			418		91, 88 (90)	88	98
			782		106, 137 (122)	111	91
High starch content	Potato	Diazinon	0	1	100, 100 (100)	96	96
			114		136, 102 (119)	98	82
			189		96, 101 (99)	91	92
			418		96, 100 (98)	98	108
			784		101, 94 (98)	91	93

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Corrected recovery † (mean,%)	Mean uncorrected recovery, percent†	Mean procedural recovery (n=2) (percent)
Processed commodity	Refined corn oil	Diazinon	0	1	93, 87 (90)	84	93
			106		110, 108 (109)	93	85
			197		103, 108 (106)	96	91
			458		98, 93 (96)	95	99
			799		120, 119 (120)	103	86
Processed commodity	Soybean dried beans	Diazinon	0	1	109, 111 (110)	98	89
			34		103, 117 (110)	102	93
			111		90, 91 (91)	86	94
			271		199, 174 (187)[c]	155	83
			467		101, 107 (104)	104	101
Processed commodity	Tomato paste	Diazinon	0	1	97, 96 (97)	89	92
			34		84, 90 (87)	67	77
			111		82, 90 (86)	83	96
			271		106, 112 (109)	107	98
			467		111, 102 (107)	102	95
Processed commodity	Sugar beet molasses	Diazinon	0	1	95, 113 (104)	75	72
			41		96, 95 (96)	96	104
			114		96, 90 (93)	91	98
			281		156, 142 (149)	127	85
			470		106, 99 (103)	103	102

Notes:

† In the report where recoveries were < 100 percent they were corrected on the basis of the procedural recovery. The uncorrected recoveries were not reported. The mean uncorrected recovery has been calculated on the basis of the mean procedural recovery. The mean recoveries which did not require correction have also been included in this column for completeness.

Table 64 Storage stability data for G-24576 residues in frozen plant matrices

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Recovery (mean,%)†	Mean uncorrected recovery, percent†	Mean procedural recovery (n=2) (%)
High water content	Tomato	G-24576	0	1	121, 108 (115)	115	100
			99		<10, <10 (<10)	<10	93
			179		<10, <10	<10	98

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Recovery (mean,%)†	Mean uncorrected recovery, percent†	Mean procedural recovery (n=2) (%)
					(<10)		
			448		<10, <10 (<10)	<10	107
			782		-	-	-
High water content	Apple	G-24576	0	1	92, 97 (95)	87	92
			108		<10, <10 (<10)	<10	99
			184		<10, <10 (<10)	<10	95
			541		<10, <10 (<10)	<10	74
			784		-	-	-
High acid content	Strawberry	G-24576	0	1	108, 102 (105)	105	103
			99		<10, <10 (<10)	<10	97
			179		<10, <10 (<10)	<10	99
			541		<10, <10 (<10)	<10	-
			792		-	-	-
High water content	Lettuce	G-24576	0	1	118, 109 (114)	113	99
			106		19, 24 (22)	22	101
			184		32, 16 (24)	24	119
			433		<10, <10 (<10)	<10	109
			792		<10, <10 (<10)	-	100
High starch content	Field corn grain	G-24576	0	1	106, 105 (106)	106	102
			114		56, 62 (59)	57	96
			189		40, 39 (40)	40	106
			418		33, 30 (32)	32	106
			782		27, 26 (27)	24	88
High starch content	Potato	G-24576	0	1	111, 110 (111)	111	111
			114		<10, <10 (<10)	<10	100
			189		<10, <10 (<10)	<10	103
			418		<10, <10 (<10)	<10	101
			784		-	-	-
Processed commodity	Refined corn oil	G-24576	0	1	131, 114 (123)	123	121
			106		94, 104 (99)	99	103
			197		101, 104 (101)	93	92
			458		90, 118 (104)	77	74
			799		102, 93 (98)	98	107

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Recovery (mean,%)†	Mean uncorrected recovery, percent†	Mean procedural recovery (n=2) (%)
Processed commodity	Soybean dried beans	G-24576	0	1	103, 79 (91)	77	85
			34		11, 12 (12)	10	87
			111		<10, <10 (<10)	<10	106
			271		23, 14 (19)	16	86
			467		12, 10 (11)	10	87
Processed commodity	Tomato paste	G-24576	0	1	92, 109 (101)	85	84
			34		<10, <10 (<10)	<10	87
			111		<10, <10 (<10)	<10	98
			271		<10, <10 (<10)	<10	77
			467		<10, <10 (<10)	<10	95
Processed commodity	Sugar beet molasses	G-24576	0	1	109, 93 (101)	90	89
			41		15, <10 (13)	13	101
			114		<10, 23 (17)	17	109
			281		<10, <10 (<10)	<10	89
			470		<10, <10 (<10)	<10	98

Notes:

† In the report where recoveries were < 100 percent they were corrected on the basis of the procedural recovery. The uncorrected recoveries were not reported. The mean uncorrected recovery has been calculated on the basis of the mean procedural recovery. The mean recoveries which did not require correction have also been included in this column for completeness.

Table 65 Storage stability data for CGA-14128 residues in frozen plant matrices

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Recovery (mean,%)	Mean uncorrected recovery, percent†	Mean procedural recovery (n=2) (percent)
High water content	Tomato	CGA-14128	0	1	130, 115 (123)	123	114
			99		77, 91 (84)	84	102
			179		86, 86 (86)	86	125
			448		59, 54 (57)	57	107
			782		60, 60 (60)	53	88
High water content	Apple	CGA-14128	0	1	104, 101 (103)	103	103
			108		51, 61 (56)	51	91
			184		53, 65 (59)	59	121
			541		41, 46 (44)	38	86
			784		43, 41 (42)	40	96
High acid content	Strawberry	CGA-14128	0	1	111, 112 (112)	112	121
			99		43, 43 (43)	43	106

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Recovery (mean,%)	Mean uncorrected recovery, percent†	Mean procedural recovery (n=2) (percent)
			179		31, 35 (33)	33	120
			541		24, 29 (27)	25	94
			792		12, 15 (14)	13	91
High water content	Lettuce	CGA-14128	0	1	87, 95 (91)	87	96
			106		89, 79 (84)	84	101
			184		98, 96 (97)	97	102
			433		104, 91 (98)	92	94
			792		94, 105 (100)	100	102
High starch content	Field corn grain	CGA-14128	0	1	106, 111 (109)	101	93
			114		93, 99 (96)	96	110
			189		98, 86 (92)	92	102
			418		88, 117 (103)	103	114
			782		107, 108 (108)	108	104
High starch content	Potato	CGA-14128	0	1	129, 134 (132)	132	133
			114		107, 89 (98)	89	91
			189		111, 99 (105)	105	113
			418		102, 102 (102)	82	80
			784		105, 115 (110)	94	85
Processed commodity	Refined corn oil	CGA-14128	0	1	102, 116 (109)	109	113
			106		94, 98 (96)	91	95
			197		112, 105 (109)	97	89
			458		130, 99 (115)	85	74
			799		114, 119 (117)	116	99
Processed commodity	Soybean dried beans	CGA-14128	0	1	129, 126 (128)	108	84
			34		101, 91 (96)	96	109
			111		83, 94 (89)	89	113
			271		139, 138 (139)	101	73
			467		115, 115 (115)	97	84
Processed commodity	Tomato paste	CGA-14128	0	1	93, 93 (93)	93	100
			34		71, 71 (71)	71	111
			111		75, 85 (80)	78	98
			271		46, 57 (52)	52	102
			467		74, 71 (73)	73	102
Processed commodity	Sugar beet molasses	CGA-14128	0	1	111, 116 (114)	84	74
			41		85, 80 (83)	76	92

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Recovery (mean,%)	Mean uncorrected recovery, percent†	Mean procedural recovery (n=2) (percent)
			114		120, 137 (129)	106	82
			281		47, 51 (49)	46	94
			470		121, 135 (128)	128	111

Notes:

† In the report where recoveries were < 100 percent they were corrected on the basis of the procedural recovery. The uncorrected recoveries were not reported. The mean uncorrected recovery has been calculated on the basis of the mean procedural recovery. The mean recoveries which did not require correction have also been included in this column for completeness.

Strawberry

Samples of strawberry were homogenised, fortified at 10 mg/kg and stored frozen at ≤ -20 °C. The following samples were prepared:

- 20 samples fortified with 10 mg/kg diazinon
- 20 samples fortified with 10 mg/kg G-24576
- 20 samples fortified with 10 mg/kg CGA-14128 and fortified with 10 mg/kg G-27550

At each time point two stored samples and two procedural recoveries were analysed along with an untreated control sample. Samples were analysed using method AG-550. The stored samples fortified with G-24576 were also analysed for G-27550. The results are shown in Tables 66 to 69.

Table 66 Storage stability data for diazinon residues in strawberry

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Recovery (mean,%)	Procedural recovery (percent)†
High acid content	Strawberry	Diazinon	0	10	96, 96 (96)	Mean = 105 at 10 mg/kg (n=14)
			1		95, 99 (97)	
			3		98, 103 (101)	
			7		96, 100 (98)	
			13		97, 98 (98)	
			28		94, 94 (94)	
			56		91, 94 (93)	

Notes:

† The procedural recoveries at each time point are not stated.

Table 67 Storage stability data for G-24576 residues in strawberry

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Recovery (mean,%)	Procedural recovery (percent)†
High acid content	Strawberry	G-24576	0	10	21, 19.6 (20)	Mean = 120 at 0.1 mg/kg (n=6)
			1		10.7, 7 (9)	

			3		13.2, 12.7 (13)	Mean = 105 at 0.5 mg/kg (n=4) Mean =94 at 10 mg/kg (n=4)
			7		2, 3 (3)	
			13		1, <1 (1)	
			28		< 1, < 1 (1)	
			56		<1, < 1 (<1)	

Notes:

† The procedural recoveries at each time point are not stated.

Table 68 Storage stability data for CGA-14128 residues in strawberry

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Recovery (mean,%)	Procedural recovery (percent)†
High acid content	Strawberry	CGA-14128	0	10	98, 131 (115)	Mean = 110 at 10 mg/kg (n=14)
			1		162, 104 (133)	
			3		101, 103 (102)	
			7		87, 94 (91)	
			13		126, 105 (116)	
			28		89, 92 (91)	
			56		90, 82 (86)	

Notes:

† The procedural recoveries at each time point are not stated.

Table 69 Storage stability data for G-27550 residues in strawberry

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level (mg/kg)	Recovery (mean,%)	Procedural recovery (percent)†
High acid content	Strawberry	G-27550	0	10	96, 121 (109)	Mean = 101 at 0.1 mg/kg (n=5) Mean = 101 at 0.5 mg/kg (n=2) Mean =97 at 10 mg/kg (n= 14)
			1		167, 95 (131)	
			3		101, 103 (102)	
			7		87, 90 (89)	
			13		105, 144 (125)	
			28		92, 92 (92)	
			56		102, 96 (99)	

Notes:

† The procedural recoveries at each time point are not stated.

Residues of diazinon, CGA-14128 and G-27550 were found to be stable in strawberry for the 56 days of frozen storage. Residues of G-24576 were not stable over the 56 days of frozen storage. Recoveries of only 20 percent were obtained in day zero samples, which were analysed approximately 4 hours after fortification, in contrast to procedural recoveries which were prepared and analysed within a shorter time interval. It has been postulated that G-24576 degrades to G-27550 and therefore G-27550 was determined in the strawberry samples fortified with G-24576. The results are shown in Table 70.

Table 70 Analysis for G-27550 residues in samples of strawberry fortified with G-24576

Commodity category	Crop/commodity	Analyte	Storage period (days)	Fortification level of G-24576 (mg/kg)	Concentration, mg/kg, of G-27550 (mean)	Procedural recovery (%)†	G-24576 equivalent recoveries, percent §
High acid content	Strawberry	G-27550	0	10	4.3 , 4.3 (4.3)	Mean = 120 at 0.1 mg/kg (n=6)	81, 81 (81)
			1		5.5, 5.9 (5.7)		104, 112 (108
			3		5.3, 58 (5.6)		100, 110 (106)
			7		4.4, 4.5 (4.5)	Mean = 105 at 0.5 mg/kg (n=4)	83, 85 (85)
			13		6.4, 5.7 (6.1)		121, 108 (115)
			28		4.5, 4.6 (4.6)	Mean =94 at 10 mg/kg (n=4)	85, 87 (87)
			56		5.6, 52 (5.4)		106, 98 (103)

Notes:

† The procedural recoveries at each time point are not stated.

§ MW of G-24576 = 288 g/mol.

MW G-27550 = 152 g/mol.

G-24576 equivalent levels = 1.89 × G-27550 levels.

Pineapple

Samples of pineapple pulp and peel were homogenised and fortified with 0.1 mg/kg diazinon. Samples were stored at ≤ - 18 °C. At each time point two stored samples, one freshly prepared procedural recovery sample and an unfortified control sample were analysed. The samples were analysed using method MAK 824/042610. The results are summarised in Table 71.

Table 71 Storage stability data for diazinon residues in pineapple

Commodity category	Crop/commodity	Analyte	Storage period (months)	Fortification level (mg/kg)	Analysed concentration (mean, mg/kg)	Recovery (mean,%)	Procedural recovery (percent)
High acid content	Pineapple peel	Diazinon	0	0.1	0.085, 0.080 (0.083)	85, 80 (83)	-
			1		0.073, 0.073 (0.073)	73, 73 (73)	86
			3		0.069, 0.070 (0.070)	69, 70 (70)	85
	Pineapple pulp		0	0.1	0.089, 0.083 (0.086)	89, 83 (86)	-
			1		0.074, 0.068 (0.071)	74, 68 (71)	84
			3		0.067, 0.068 (0.068)	67, 68 (68)	85

Diazinon residues were found to be stable in pineapple pulp and peel for the three-month storage period when stored at ≤ -18 °C.

Animal commodities

Study SPR 19-81

The storage stability of diazinon was investigated in the tissue samples from sheep. Three sheep were plunge dipped once in a dip containing a nominal concentration of 500 mg diazinon/L. The animals were slaughtered the day after treatment and samples were homogenised before being frozen at -20 °C. Samples of muscle, liver, kidney and fat were analysed on the day of storage and after 8 months for freezer storage. Samples were analysed using method REM 4/81. The results are summarised in Table 72.

Table 72 Storage stability of diazinon residues in sheep tissues

Commodity category	Crop/commodity	Analyte	Storage period (months)	Analysed concentration (mg/kg)			Percentage recovery, relative to day 0 (percent)			Mean procedural recovery (percent)
				Animal 1	Animal 2	Animal 3	Animal 1	Animal 2	Animal 3	
Animal commodity	Muscle	Diazinon	0	0.88	0.58	1.9	-	-	-	-
			8	0.72	0.60	1.4	82	103	74	98
	Liver	Diazinon	0	0.12	< 0.1	< 0.1	-	-	-	-
			8	0.09	N.A	N.A	75	-	-	90
	Kidney	Diazinon	0	0.083	0.25	0.45	-	-	-	-
			8	0.25	0.18	0.44	301	72	98	94
	Fat	Diazinon	0	2.8	3.1	5.0	-	-	-	-
			8	3.2	2.6	5.6	114	84	112	79

Notes:

N.A= not analysed

USE PATTERNS

Table 73 represents a summary of the GAPs submitted for consideration in this Meeting.

Table 73 List of uses of diazinon submitted for this meeting

Crop	Country	Indoor/ outdoor	Formulation	Method	Rate, kg ai/ha	Number	Re-treatment Interval, days	PHI, days
Apples, pears, quince	Chile	Outdoor	EW	Foliar	0.07 kg ai/hL	3	15	21
Apples, pears, quince	Chile	Outdoor	WP	Foliar	0.056 kg ai/hL	3	15	30
Cherries	Chile	Outdoor	WP	Foliar	0.06 kg ai/hL	3	15	15
Cherries	Chile	Outdoor	EW	Foliar	0.07 kg ai/hL	3	15	15
Pineapples	United States	Outdoor	WP	Foliar	1.12	2	28	7
Onions	Costa Rica	Outdoor	EW	Foliar	0.75	3	8	10
Cabbage	Chile	Outdoor	EW	Foliar	0.6	2	Not stated	7
Cabbage	Costa Rica	Outdoor	EW	Foliar	0.75	3	8	10
Cabbage	United States	Outdoor	WP	Pre-planting	4.48	1	-	-

Crop	Country	Indoor/ outdoor	Formulation	Method	Rate, kg ai/ha	Number	Re-treatment Interval, days	PHI, days
Cabbage	United States	Outdoor	SL	Pre-planting	4.48	1	-	-
Cabbage	United States	Outdoor	SL	Pre-planting	4.48	1	-	-
Tomatoes	Costa Rica	Outdoor	EW	Foliar	0.75	3	8	10
Potatoes	Costa Rica	Outdoor	EW	Foliar	0.75	3	8	10
Maize	Costa Rica	Outdoor	EC	Foliar	0.75	3	8	10
Maize	Costa Rica	Outdoor	EC	Foliar	0.39	Not stated	7	14
Wheat	Russia	Outdoor	EC	Foliar	1.08	1	-	60
Barley	Russia	Outdoor	EC	Foliar	0.9	1	-	30
Barley	Russia	Outdoor	EC	Foliar	0.3	1	-	60

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Pome fruits

Apple and pear

Six trials on apple and seven trials on pear were conducted in the United States in 1991. Two of the apple trials and two of the pear trials (conducted Yakima Country, Washington) are regarded as replicate trials and therefore there are five independent apple trials and six independent pear trials.

At each trial site different application rates were investigated.

Three foliar applications were made using a WP formulation at application rates between 0.053–0.96 kg ai/hL. The retreatment intervals were between 13 and 17 days and samples were collected for analysis between 21 and 28 days after the last application.

Samples were stored frozen at -20 °C for up to 160 days.

Residues of diazinon, G-24576 and CGA-14128 were determined using the analytical method AG-550A. Procedural recoveries were undertaken for each analyte at fortification levels of 0.01–1 mg/kg. For diazinon recoveries ranged from 79–99 percent, for G-24576 recoveries ranged from 66–91 percent and for CGA-14128 recoveries ranged from 80–107 percent.

A summary of the residue trials in apples and pears are shown in Tables 74 and 75 respectively.

Table 74 Residues in Apple from supervised trials in United States involving foliar applications of diazinon

Location, Country Year, Crop/Variety	Rate (kg ai/hL)	Water volume (L/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
GAP Chile Foliar application	0.07 ×3	2000 - 2500	15	-	-	21	-		
Fresno County, California 1991 Apple / Granny Smith	0.053 0.053 0.053	2805 2805 2805	- 14 14	Fruit 6 cm	fruit	21	0.02, < 0.01 (0.02)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)

Location, Country Year, Crop/Variety	Rate (kg ai/hL)	Water volume (L/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
						28	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.078 0.078 0.078	2805 2805 2805	- 14 14	Fruit 6 cm	fruit	21	0.01, 0.01 (0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
Columbia, County, New York 1991 Apple / Empire	0.17 0.17 0.17	977 977 977	- 17 13	Fruit 6 cm	fruit	22	0.06, 0.03; (0.05)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.01, 0.02; (0.02)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.46 0.46 0.46	977 977 977	- 17 13	Fruit 6 cm	fruit	22	0.03, 0.02; (0.03)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.04, 0.01; (0.03)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.46 0.46 0.46	977 977 977	- 17 13	Fruit 6 cm	fruit	22	< 0.01	< 0.01	< 0.01
						28	0.04	< 0.01	< 0.01
Johnston County, N. Carolina 1991 Apple / Golden Delicious	0.087 0.087 0.087	1945 1945 1945	- 15 14	Not stated	fruit	21	0.03, 0.04; (0.04)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	< 0.01, 0.02; (0.02)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.11 0.11 0.11	1945 1945 1945	- 15 14	Not stated	fruit	21	0.05, 0.08; (0.07)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.01, < 0.01; (0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.23 0.23	1945 1945	- 15	Not stated	fruit	21	0.10	< 0.01	< 0.01

Location, Country Year, Crop/Variety	Rate (kg ai/hL)	Water volume (L/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
	0.23	1945	14			28	0.07	< 0.01	< 0.01
Yakima County, Washington 1991 Apple / Red Delicious **Replicate trial 1a**	0.079 0.079 0.079	2151 2151 2151	- 16 13	Fruit 6-7 cm	fruit	21	0.08, 0.06; (0.07)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.05, 0.08; (0.07)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.1 0.1 0.1	2151 2151 2151	- 16 13	Fruit 6-7 cm	fruit	21	0.11, 0.13; (0.12)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.24, 0.18; (0.21)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.21 0.21 0.21	2151 2151 2151	- 16 13	Fruit 6-7 cm	fruit	21	0.42	< 0.01	< 0.01
						28	0.32	< 0.01	< 0.01
Yakima County, Washington 1991 Apple / Red Delicious **Replicate trial 1b**	0.079 0.079 0.079	2151 2151 2151	- 16 13	Fruit 6-7 cm	fruit	21	0.13, 0.13 (0.13)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.10, 0.07; (0.09)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.1 0.1 0.1	2151 2151 2151	- 16 13	Fruit 6-7 cm	fruit	21	0.06, 0.10; (0.08)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.05, 0.03; (0.04)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
Allegan County, Michigan 1991 Apple / Red Delicious	0.061 0.061 0.061	2806 2806 2806	- 15 14	Immature fruit	fruit	21	0.04, < 0.01; (0.03)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.02, 0.04; (0.03)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)

Location, Country Year, Crop/Variety	Rate (kg ai/hL)	Water volume (L/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
	0.078 0.078 0.078	2806 2806 2806	- 15 14	Immature fruit		21	0.03, 0.03; (0.03)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.04, 0.04; (0.04)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)

Notes:

MID Maximum individual dose.

MTD Maximum total dose.

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets.

Table 75 Residues in pear from supervised trials in United States involving foliar applications of diazinon

Location, Country Year, Crop/Variety	Rate (kg ai/hL)	Water volume (L/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
GAP Chile Foliar application	0.07 × 3	2000 - 2500	15	-	-	21	-		
Fresno County California 1991 Pear/ Bartlett	0.061 0.061 0.061	2806 2806 2806	- 14 15	Fruit 7.5 cm diameter	fruit	21	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
Fresno County California 1991 Pear/ Bartlett	0.078 0.078 0.078	2806 2806 2806	- 14 15	Fruit 7.5 cm diameter	Fruit	21	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.16 0.16 0.16	2806 2806 1806	- 14 15	Fruit 7.5 cm diameter	Fruit	21	0.02	< 0.01	< 0.01
						28	< 0.01	< 0.01	< 0.01
Washington County, Oregon 1991 Pear/ Bartlett	0.36 0.36 0.36	468 468 468	- 14 15	Fruit 5.5-6 cm diameter	Fruit	21	0.04, 0.02; (0.03)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.04, 0.04; (0.04)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.47 0.47	468 468	- 14	Fruit 5.5-6 cm diameter	Fruit	21	0.04, 0.04; (0.04)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)

Location, Country Year, Crop/Variety	Rate (kg ai/hL)	Water volume (L/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
	0.47	468	15			28	< 0.01, 0.02 (0.02)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.96 0.96 0.96	468 468 468	- 14 15	Fruit 5.5-6 cm diameter	Fruit	21 28	0.04 0.02	< 0.01 < 0.01	< 0.01 < 0.01
Stanislaus County, California 1991 Pear/ Bartlett	0.34 0.18 0.17	652 1188 1291	- 14 14	Mature fruit	Fruit	21 28	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)
	0.34 0.18 0.17	652 1188 1291	- 14 14	Mature fruit	Fruit	21 28	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)
	0.69 0.38 0.35	652 1188 1291	- 14 14	Mature fruit	Fruit	21 28	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Yakima County, Washington 1991 Pear/ Bartlett **Replicate trial 1a**	0.18 0.18 0.18	935 935 935	- 15 13	Green fruit	Fruit	21 28	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)
	0.24 0.24 0.24	935 935 935	- 15 13	Green fruit	Fruit	21 28	< 0.01, 0.02; (0.02) < 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01) < 0.01, < 0.01; (< 0.01)
Yakima County, Washington 1991	0.079 0.079	2151 2151	- 12	Fruit 5-6 cm	Fruit	21	0.05, 0.11; (0.08)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)

Location, Country Year, Crop/Variety	Rate (kg ai/hL)	Water volume (L/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
Pear/ Bosc **Replicate trial 1b**	0.079	2151	16			28	0.11, 0.11; (0.11)	0.02, 0.01; (0.02)	< 0.01, < 0.01; (< 0.01)
	0.1 0.1 0.1	2151 2151 2151	- 12 16	Fruit 5-6 cm	Fruit	21	0.11, 0.11; (0.11)	< 0.01, 0.01; (0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.07, 0.12; (0.10)	0.01, 0.01; (0.01)	< 0.01, < 0.01; (< 0.01)
	0.21 0.21 0.21	2151 2151 2151	- 12 16	Fruit 5-6 cm	Fruit	21	0.43	0.02	< 0.01
						28	0.26	0.02	< 0.01
Allegan County, Michigan 1991 Pear / Bartlett	0.61 0.61 0.61	2806 2806 2806	- 15 14	Immature fruit	Fruit	21	0.05, 0.06; (0.06)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	0.06, < 0.01; (0.04)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.078 0.078 0.078	2806 2806 2806	- 15 14	Immature fruit	Fruit	21	0.01, 0.11; (0.06)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
Wayne County, New York 1991 Pear / Bartlett	0.18 0.18 0.18	2806 2806 2806	- 14 14	Fruit 5.2-5.6 cm	Fruit	21	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.24 0.24 0.24	935 935 935	- 14 14	Fruit 5.2-5.6 cm	Fruit	21	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
						28	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)	< 0.01, < 0.01; (< 0.01)
	0.48 0.48 0.48	2806 2806 2806	- 14 14	Fruit 5.2-5.6 cm	Fruit	21	0.01	< 0.01	< 0.01
						28	< 0.01	< 0.01	< 0.01

Notes:

MID Maximum individual dose.

MTD Maximum total dose.

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets.

Assorted tropical and sub-tropical fruits – inedible peel

Pineapples

Four trials on pineapple were conducted in Costa Rica in 2004. The trials were conducted in two separate locations at the same time and therefore there are only two independent trials.

Six foliar applications were made using an EC formulation at application rates between 0.997–1.13 kg ai/ha. The retreatment intervals were between 9–11 days and samples were collected for analysis 7 days after the last application.

Samples were stored frozen at ≤ - 18 °C for up to 21 days.

Residues of diazinon were determined in pulp and peel using the analytical method MAK/824. Procedural recoveries were undertaken at fortification levels of 0.1 and 0.5 mg/kg. Recoveries ranged from 72–91 percent for peel and recoveries of 91 percent were obtained for pulp.

Residues in the whole fruit were calculated based on the weights and residues present in the pulp and peel samples. A summary of the residue trials are shown in Table 76.

Table 76 Residues in pineapples from supervised trials in Costa Rica involving foliar applications of diazinon

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)
GAP United States Foliar application	1.12 × 2	28	-	-	7	-
Volcan, Costa Rica 2004 Pineapple/ MD-2 **Replicate trial 1a**	1.063 1.106 1.054 1.050 1.054 1.041	- 10 10 10 9 9	BBCH 49	Peel	0 7	0.198 0.016
				Pulp	0 7	< 0.01 < 0.01
				Whole fruit	0 7	0.069 < 0.01
Volcan, Costa Rica 2004 Pineapple/ MD-2 **Replicate trial 1b**	1.024 0.997 1.0068 1.036 1.129 1.035	- 10 10 10 9 9	BBCH 49	Peel	0 7	0.223 0.014
				Pulp	0 7	< 0.01 < 0.01
				Whole fruit	0 7	0.076 < 0.01
Guacimo, Costa Rica 2004	1.040 1.051 1.031 1.073	- 10 9 10	BBCH 49	Peel	0 7	0.292 < 0.01
				Pulp	0 7	< 0.01 < 0.01

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)
Pineapple/ Mayan Gold 3 **Replicate trial 2a**	1.073 1.051	10 9		Whole fruit	0 7	0.102 < 0.01
Guacimo, Costa Rica 2004 Pineapple/ Mayan Gold 3 **Replicate trial 2b**	1.033 1.094 1.090 1.051 1.061 1.035	- 10 9 10 10 9	BBCH 91	Peel	0 7	0.409 0.022
				Pulp	0 7	< 0.01 < 0.01
				Whole fruit	0 7	0.139 < 0.01

Notes:

MID Maximum individual dose.

MTD Maximum total dose.

Brassica vegetables

Cabbage

Seven trials on cabbage were conducted in the United States in 1991. At each trial site two replicate trials were conducted, in some of the trials the replicates were undertaken at a different application rate. In each trial one application of 4.5 kg ai/ha was made using a granular formulation followed by 5 foliar applications at 0.56 kg ai/ha using a WP formulation. The interval between the granular application and the first foliar application ranged from 34–283 days. The retreatment interval for the foliar applications was 7 days.

Samples were stored frozen at ≤ -18 °C for up to 12 months.

Residues of diazinon, G-24576 and CGA-14128 were determined using the analytical method AG-550A. Procedural recoveries were undertaken for each analyte at fortification levels of 0.1, 0.5, 1 and 5 mg/kg. For diazinon recoveries ranged from 74–144 percent, for G-24576 recoveries ranged from 74–140 percent and for CGA-14128 recoveries ranged from 77–151 percent.

A summary of the residue trials in cabbage are shown in Table 77.

Table 77 Residues in cabbage from supervised trials in United States involving foliar applications of diazinon

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
GAP Chile Foliar application	0.6 × 2	Not stated	-	-	**7**	-		
GAP Costa Rica Foliar application	0.75 × 3	8	-	-	**10**			
GAP United States Soil, pre-planting	4.48	N/A	Applied pre-planting	-	-			
Fresno County, California	Pre-plant: 4.5		Not reported	Heads, untrimmed	8	0.13, 0.09 (0.11)	-	< 0.01, < 0.01 (< 0.01)

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
1991 Cabbage/ Copenhagen MKT	Foliar: 0.56 0.56 0.56 0.56 0.56	- 34 7 7 7 7		Heads, untrimmed	14	0.02, 0.01 (0.02)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	0.01, < 0.01 (0.01)	-	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	8	< 0.01, < 0.01 (< 0.01)	-	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	14	< 0.01, < 0.01 (< 0.01)	0.01, < 0.01 (0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	-	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	8	0.21, 0.10 (0.16)	-	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	14	0.03, 0.05 (0.04)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	21	0.03, 0.04 (0.04)	-	< 0.01, < 0.01 (< 0.01)
	Pre-plant: 4.5 Foliar: 0.56 0.56 0.56 0.56 0.56	- 34 7 7 7 7	Not reported	Heads, untrimmed	8	0.10, 0.05 (0.08)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	14	0.05, 0.03 (0.04)	-	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	< 0.01, < 0.01 (< 0.01)	-	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	8	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	14	< 0.01, < 0.01 (< 0.01)	-	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	-	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	8	0.28, 0.12 (0.20)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	14	0.08, 0.04 (0.06)	-	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	21	0.03, 0.02 (0.03)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
Columbia County, New York	Pre-plant: 4.5 Foliar:		Not reported	Heads, untrimmed	7	0.36, 0.13 (0.25)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
1991 Cabbage/ Market Price	0.56 0.56 0.56 0.56 0.56	- 62 7 7 7 8		Heads, untrimmed	13	0.05, 0.15 (0.10)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	0.08, 0.02 (0.05)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	7	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	13	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	7	0.55, 0.78 (0.67)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	13	0.24, 0.25 (0.25)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	21	0.11, 0.13 (0.12)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
	Pre-plant: 9 Foliar: 1.1 1.1 1.1 1.1 1.1	- 62 7 7 7 8	Not reported	Heads, untrimmed	7	1.1	< 0.01	< 0.01
				Heads, untrimmed	13	0.31	< 0.01	< 0.01
				Heads, untrimmed	21	0.34	< 0.01	< 0.01
				Heads, trimmed	7	< 0.01	< 0.01	< 0.01
				Heads, trimmed	13	< 0.01	< 0.01	< 0.01
				Heads, trimmed	21	< 0.01	< 0.01	< 0.01
				Wrapper leaves	7	1.6	< 0.01	< 0.01

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
				Wrapper leaves	13	0.43	< 0.01	< 0.01
				Wrapper leaves	21	0.31	< 0.01	< 0.01
Indian River County, Florida 1991 Cabbage/ Bravo	Pre-plant: 4.5 Foliar: 0.56 0.56 0.56 0.56 0.56	- 37 7 7 7 7		Heads, untrimmed	7	0.33, 0.52 (0.43)	< 0.01, < 0.01 (< 0.01)	-
				Heads, untrimmed	14	0.22, 0.30 (0.26)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	0.35, 0.24 (0.30)	< 0.01, < 0.01 (< 0.01)	-
				Heads, trimmed	7	0.04, 0.02 (0.03)	< 0.01, < 0.01 (< 0.01)	-
				Heads, trimmed	14	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	-
				Wrapper leaves	7	3.3, 2.7 (3.0)	< 0.01, < 0.01 (< 0.01)	-
				Wrapper leaves	14	1.8, 1.2 (1.5)	< 0.01, < 0.01 (< 0.01)	< 0.01, 0.01 (0.01)
				Wrapper leaves	21	0.51, 1.0 (0.76)	< 0.01, < 0.01 (< 0.01)	-
	Pre-plant: 4.5 Foliar: 0.56 0.56 0.56 0.56 0.56	- 37 7 7 7 7		Heads, untrimmed	7	0.69, 0.55 (0.62)	< 0.01, < 0.01 (< 0.01)	-
				Heads, untrimmed	14	0.27, 0.12 (0.20)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	0.25, 0.26 (0.26)	< 0.01, < 0.01 (< 0.01)	-
				Heads, trimmed	7	0.09, 0.03 (0.06)	< 0.01, < 0.01 (< 0.01)	-

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
				Heads, trimmed	14	< 0.01, 0.01 (0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	-
				Wrapper leaves	7	2.8, 3.6 (3.2)	< 0.01, < 0.01 (< 0.01)	-
				Wrapper leaves	14	1.4, 1.1 (1.3)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	21	0.65, 0.91 (0.78)	< 0.01, < 0.01 (< 0.01)	-
Cameron County, Texas 1990-1991 cabbage/ Solid Blue 760/Abbott	Pre-plant: 4.5 Foliar: 0.56 0.56 0.56 0.56 0.56	- 283 7 7 7 7	Not reported	Heads, untrimmed	7	0.15, 0.17 (0.16)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	15	0.03, 0.04 (0.04)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	0.03, 0.05 (0.04)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	7	0.01, 0.09 (0.05)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	15	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	7	0.47, 0.42 (0.45)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	15	0.23, 0.24 (0.24)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	21	0.07, 0.07 (0.07)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
	Pre-plant: 9 Foliar:		Not reported	Heads, untrimmed	7	0.54	< 0.01	< 0.01

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
	1.1 1.1 1.1 1.1 1.1	- 283 7 7 7 7		Heads, untrimmed	15	0.12	< 0.01	< 0.01
				Heads, untrimmed	21	0.16	< 0.01	< 0.01
				Heads, trimmed	7	0.04	< 0.01	< 0.01
				Heads, trimmed	15	0.01	< 0.01	< 0.01
				Heads, trimmed	21	< 0.01	< 0.01	< 0.01
				Wrapper leaves	7	2.1	< 0.01	< 0.01
				Wrapper leaves	15	1.4	< 0.01	< 0.01
				Wrapper leaves	21	0.42	< 0.01	< 0.01
Franklin County, North Carolina 1991 Cabbage/ Market Price	Pre-plant: 4.5 Foliar: 0.56 0.56 0.56 0.56 0.56	- 54 7 7 7 7	Not reported	Heads, untrimmed	7	0.16, 0.20 (0.18)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	14	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	< 0.01, < 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	7	0.07, 0.07 (0.07)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	14	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	7	0.41, 0.59 (0.50)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
				Wrapper leaves	14	0.02, 0.03 (0.03)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
	Pre-plant: 4.5 Foliar: 0.56 0.56 0.56 0.56 0.56	- 54 7 7 7 7	Not reported	Heads, untrimmed	7	0.09, 0.04 (0.07)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	14	0.02, 0.02 (0.02)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	7	0.07, 0.08 (0.08)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	14	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	7	0.91, 0.85 (0.88)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	14	0.14, 0.13 (0.14)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	21	0.07, 0.11 (0.09)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
Columbia County, Wisconsin 1991 Cabbage/ Little Rock	Pre-plant: 4.5 Foliar: 0.56 0.56 0.56 0.56 0.56	- 54 7 7 7 7	Not reported	Heads, untrimmed	7	0.97, 0.57 (0.77)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	14	0.20, 0.31 (0.26)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	0.24, 0.19 (0.22)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	7	0.01, 0.09 (0.05)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
				Heads, trimmed	14	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	7	1.2, 1.3 (1.3)	< 0.01, < 0.01 (< 0.01)	0.01, < 0.01 (0.01)
				Wrapper leaves	14	1.9, 1.1 (1.5)	< 0.01, < 0.01 (< 0.01)	0.01, < 0.01 (0.01)
				Wrapper leaves	21	0.86, 0.86 (0.86)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
	Pre-plant: 4.5 Foliar: 0.56 0.56 0.56 0.56 0.56	- 54 7 7 7 7	Not reported	Heads, untrimmed	7	1.9	< 0.01	0.02
				Heads, untrimmed	14	0.48	< 0.01	< 0.01
				Heads, untrimmed	21	0.53	< 0.01	< 0.01
				Heads, trimmed	7	0.05	< 0.01	< 0.01
				Heads, trimmed	14	< 0.01	< 0.01	< 0.01
				Heads, trimmed	21	< 0.01	< 0.01	< 0.01
				Wrapper leaves	7	4.7	< 0.01	0.03
				Wrapper leaves	14	2.7	< 0.01	0.03
				Wrapper leaves	21	2.2	< 0.01	< 0.01
Fayette County, Ohio	Pre-plant: 4.5 Foliar:		Not reported	Heads, untrimmed	7	0.11, 0.30 (0.21)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
1991 Cabbage/prize	0.56 0.56 0.56 0.56 0.56	- 47 8 6 7 7		Heads, untrimmed	14	< 0.01, 0.03 (0.02)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	7	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	14	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	7	0.35, 0.34 (0.35)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	14	0.09, 0.21 (0.15)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	21	0.05, 0.10 (0.08)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
	Pre-plant: 4.5 Foliar: 0.56 0.56 0.56 0.56 0.56	- 47 8 6 7 7	Not reported	Heads, untrimmed	7	0.07, 0.24 (0.16)	-	-
				Heads, untrimmed	14	0.04, 0.01 (0.03)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, untrimmed	21	< 0.01, 0.01 (0.01)	-	-
				Heads, trimmed	7	< 0.01, < 0.01 (< 0.01)	-	-
				Heads, trimmed	14	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Heads, trimmed	21	< 0.01, < 0.01 (< 0.01)	-	-
				Wrapper leaves	7	0.19, 0.51 (0.35)	-	-

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)	G-24576 (mg/kg)	CGA-14128 (mg/kg)
				Wrapper leaves	14	0.09, 0.07 (0.08)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
				Wrapper leaves	21	0.04, 0.06 (0.05)	-	-

Notes:

MID Maximum individual dose.

MTD Maximum total dose.

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets.

Cereal grains

Wheat

One trial on wheat was conducted in the Russian Federation in 1996. The trial was conducted with one application of 1.08 kg ai/ha using an EC formulation. Residues were determined 278 days after the application.

Only a brief translation of the Russian report has been provided and full details of the field phase and analytical phase were not provided. Samples were stored for up to 4 months prior to analysis. The conditions of storage are not stated. Residues of diazinon were determined in wheat grain and straw using a GC-NPD method. Validation data and procedural recoveries were not available.

A summary of the residue trials in wheat are shown in Table 78.

Table 78 Residues in wheat from supervised trials in United States involving foliar applications of diazinon

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)
GAP Russia Foliar application	1.08 × 1	N/A	-	-	60	-
Rostov Region, Russia 1996 Wheat/ Donskaya Yubileynaya	1.08	-	Sprouts	Grain	278	< 0.005

Notes:

MID Maximum individual dose.

MTD Maximum total dose.

N/A Not applicable.

Animal feed items

Wheat straw

One trial on wheat was conducted in Russia in 1996. The trial was conducted with one application of 1.08 kg ai/ha using an EC formulation. Residues were determined 278 days after the application.

Only a brief translation of the Russian report has been provided and full details of the field phase and analytical phase were not provided. Samples were stored for up to 4 months prior to analysis. The conditions of storage are not stated. Residues of diazinon were determined in wheat grain and straw using a GC-NPD method. Validation data and procedural recoveries were not available.

A summary of the residue trials in wheat are shown in Table 79.

Table 79 Residues in wheat from supervised trials in United States involving foliar applications of diazinon

Location, Country Year, Crop/Variety	Rate (kg ai/ha)	Interval (days)	Growth stage at last application	Crop part	DALA (days)	diazinon (mg/kg)
GAP Russia Foliar application	1.08 × 1	N/A	-	-	60	-
Rostov Region, Russia 1996 Wheat/ Donskaya Yubileynaya	1.08	-	Sprouts	Straw	278	< 0.005

Notes:

MID Maximum individual dose.

MTD Maximum total dose.

N/A Not applicable.

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of the residue on processing

No information was received by the Meeting on the nature of the residue on processing.

Magnitude of the residue on processing

The meeting received information on the fate of diazinon residues during processing of apples and pears.

In the first study apples and pears were treated with diazinon at a rate of 3 × 2.2 kg ai/ha or a rate of 4 × 4.5 kg ai/ha. The fruits were harvested 21 days after the last application and residues of diazinon, G-24576 and CGA-14128 were determined in unwashed fruits, the wash water and washed fruits. Washed fruits were then peeled and baked. The conditions employed for processing were not stated.

Processed fractions were stored for up to 7 months at ≤ -18 °C prior to analysis. Samples were analysed using method AG-550.

Residues of diazinon, G-24576 and CGA-14128 on processing are summarised in Tables 80 and 81 for apples and pears respectively.

Table 80 Residues in apple and processed fractions

Crop/processed commodity	Diazinon residue (mg/kg)	Pf for diazinon	G-24576 residue (mg/kg)	CGA-14128 residue (mg/kg)
Trial 1: 3 × 2.2 kg ai/ha				
Whole fruit, unwashed	0.04	-	< 0.01	< 0.01
Wash water	< 0.01	-	< 0.01	< 0.01
Whole fruit, washed	0.02	0.5	< 0.01	< 0.01
Fruit cores	0.02	0.5	< 0.01	< 0.01
Peel, washed	0.32	8	< 0.01	< 0.01
Whole fruit, washed and peeled	< 0.01	< 0.25	< 0.01	< 0.01
Fruit, sliced, peeled and baked	< 0.01	< 0.25	< 0.01	< 0.01
Whole fruit, peeled and baked	0.05	1.3	< 0.01	< 0.01
Trial 2: 3 × 4.5 kg ai/ha				
Whole fruit, unwashed	0.07	-	< 0.01	< 0.01
Wash water	< 0.01	-	< 0.01	< 0.01
Whole fruit, washed	0.10	1.4	< 0.01	< 0.01
Fruit cores	0.02	0.29	< 0.01	< 0.01
Peel, washed	0.43	6	< 0.01	< 0.01
Whole fruit, washed, peeled and peeled	< 0.01	< 0.14	< 0.01	< 0.01
Fruit, sliced, peeled and baked	< 0.01	< 0.14	< 0.01	< 0.01
Whole fruit, peeled and baked	0.08	1.1	< 0.01	< 0.01

Notes:

Pf = Residue level in processed commodity (mg/kg) ÷ residue level in RAC (mg/kg.

Table 81 Residues in pear and processed fractions

Crop/processed commodity	Residues (mg/kg)		
	diazinon	G-24576	CGA-14128
Trial 1: 3 × 2.2 kg ai/ha			
Whole fruit, unwashed	< 0.01	< 0.01	< 0.01
Wash water	< 0.01	< 0.01	< 0.01
Whole fruit, washed	< 0.01	< 0.01	< 0.01
Fruit cores	< 0.01	< 0.01	< 0.01
Peel, washed	0.01	< 0.01	< 0.01
Whole fruit, washed and peeled	< 0.01	< 0.01	< 0.01
Fruit, sliced and baked	< 0.01	< 0.01	< 0.01
Whole fruit, baked	< 0.01	< 0.01	< 0.01
Trial 2: 3 × 4.5 kg ai/ha			
Whole fruit, unwashed	< 0.01	< 0.01	< 0.01
Wash water	< 0.01	< 0.01	< 0.01
Whole fruit, washed†	0.02	< 0.01	< 0.01
Fruit cores	< 0.01	< 0.01	< 0.01
Peel, washed	0.07	< 0.01	< 0.01
Whole fruit, washed and peeled	< 0.01	< 0.01	< 0.01
Fruit, slice, peeled and baked	< 0.01	< 0.01	< 0.01
Whole fruit, peeled and baked	< 0.01	< 0.01	< 0.01

Notes:

† No explanation for the positive residue in the washed fruit was provided.

In a second study two processing trials on apples were undertaken. Residues of diazinon, G-24576 and CGA-14128 were determined in apple on processing into culls, pomace, juice, sauce and canned apple. The conditions employed for processing were not stated.

Apples were treated at a rate of 6 × 3.4 kg ai/ha or 6 × 6.7 kg ai/ha and harvested immediately after the last application. Samples were stored frozen for up to 9 months prior to analysis. Residues were determined using method AG-550.

Residues of diazinon, G-24576 and CGA-14128 on processing are summarised in Table 82.

Table 82 Residues in apple and processed fractions

Crop/processed commodity	Diazinon residue (mg/kg)	Pf diazinon	G-24576 residues (mg/kg)	CGA-14128 residues (mg/kg)
Trial 1: 6 × 3.4 kg ai/ha				
Fruit (RAC)	0.98	-	< 0.01	< 0.01
Culls	2.2	2.2	< 0.01	< 0.01
Wet pomace	0.40	0.41	< 0.01	< 0.01
Dry pomace	1.4	1.4	< 0.01	< 0.01
Fresh juice	0.02	0.02	< 0.01	< 0.01
Canned juice	< 0.01	< 0.01	< 0.01	< 0.01
Canned slices	< 0.01	< 0.01	< 0.01	< 0.01
Frozen slices	< 0.01	< 0.01	< 0.01	< 0.01
Apple sauce	< 0.01	< 0.01	< 0.01	< 0.01
Trial 2: 6 × 6.7 kg ai/ha				
Fruit (RAC)	2.3	-	< 0.01	< 0.01
Culls	3.8	1.7	< 0.01	< 0.01
Wet pomace	1.2	0.52	< 0.01	< 0.01
Dry pomace	3.9	1.7	< 0.01	< 0.01
Fresh juice	0.04	0.02	< 0.01	< 0.01
Canned juice	< 0.01	< 0.01	< 0.01	< 0.01
Canned slices	< 0.01	< 0.01	< 0.01	< 0.01
Frozen slices	0.02	< 0.01	< 0.01	< 0.01
Apple sauce	< 0.01	< 0.01	< 0.01	< 0.01

Notes:

Pf = Residue level in processed commodity (mg/kg) ÷ residue level in RAC (mg/kg)

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Dairy cattle

Nine dairy cows (Holstein, 3–10 years old, 411–678 kg bw) were divided into three dose groups, each containing three cows, with a further one cow serving as a control for all dose groups. Animals were dosed with diazinon once daily for 28- 30 consecutive days by means of a gelatine capsule administered with a balling gun.

The doses administered to the three dose groups were 41, 124 and 414 ppm. During the study, daily feed consumption and milk production were constant and were on average 21.2 kg/day and 17.0 kg/day respectively.

Milk samples were taken twice daily with the morning sample being kept refrigerated and then combined with the evening sample for each cow. Tissue samples were collected at sacrifice which occurred within 24 hours of the last dose. Milk and tissue samples were stored frozen and analysed within 5 months.

Samples were analysed for diazinon, G-24576 and CGA-14128 using method AG-550A (GC-FPD). Procedural recoveries were undertaken at fortification levels of 0.01–0.5 mg/kg and were acceptable for all commodity/ analyte combinations except milk/ CGA-14128 for which the recoveries were 105–137 percent.

Residues of diazinon were < 0.01 mg/kg in milk from the lowest dose study. Residues of G-24576 and CGA-14128 were < 0.01 mg/kg at all dose rates. The residues of diazinon from the two higher feeding rates are shown in Table 83.

Table 83 Residues of diazinon in whole milk following administration of diazinon to dairy cows once daily at a rate of 124 ppm and 414 ppm

Day	Residue level (mg/kg)	
	124 ppm	414 ppm
1	< 0.01, < 0.01, < 0.01	< 0.01, 0.05, 0.02 (0.03)
3	< 0.01, < 0.01, < 0.01	< 0.01, 0.05, 0.06 (0.04)
7	< 0.01, < 0.01, < 0.01	0.02, 0.08, 0.02 (0.04)
14	< 0.01, < 0.01, < 0.01	< 0.01, 0.06, 0.01 (0.03)
21	< 0.01, < 0.01, 0.01	< 0.01, 0.03, 0.02 (0.02)
27	< 0.01, < 0.01, < 0.01	< 0.01, 0.03, 0.01 (0.03)
Mean residue over the plateau period (mg/kg)	< 0.01	0.03

In tissues residues of G-24576 and CGA-14128 were < 0.01 mg/kg at each dose rate. Residues of diazinon were found in muscle, fat, liver and kidney. A summary of the residues found in tissues is outlined in Table 84.

Table 84 Residues in tissues following administration of diazinon to dairy cows once daily at a rate of 41, 124 and 414 ppm

Tissue	Residue level at day 28, day 29 and day 30 (mg/kg)								
	41 ppm			121 ppm			414 ppm		
	Diazinon	G-24576	CGA-14128	Diazinon	G-24576	CGA-14128	Diazinon	G-24576	CGA-14128
Liver	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, 0.02	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, 0.06, 0.02	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
Mean	< 0.01	< 0.01	< 0.01	0.01	< 0.01	< 0.01	0.03	< 0.01	< 0.01
Kidney	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
Mean	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.01	< 0.01	< 0.01
Perirenal fat	0.02, 0.02, 0.03	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	0.06, 0.05, 0.08	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	0.15, 0.49, 0.58	< 0.01, < 0.01, < 0.01	0.01, 0.04, 0.05
Mean	0.02	< 0.01	< 0.01	0.06	< 0.01	< 0.01	0.41	< 0.01	0.03
Omental fat	0.04, 0.02,	< 0.01,	< 0.01,	0.08, 0.07,	< 0.01,	< 0.01,	0.20, 0.84,	< 0.01,	0.03,

Tissue	Residue level at day 28, day 29 and day 30 (mg/kg)								
	41 ppm			121 ppm			414 ppm		
	Diazinon	G-24576	CGA-14128	Diazinon	G-24576	CGA-14128	Diazinon	G-24576	CGA-14128
	0.03	< 0.01, < 0.01	< 0.01, < 0.01	0.10	< 0.01, < 0.01	< 0.01, < 0.01	0.64	< 0.01, < 0.01	0.06, 0.06
Mean	0.03	< 0.01	< 0.01	0.08	< 0.01	< 0.01	0.56	< 0.01	0.05
Round muscle	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, 0.01, 0.02	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
Mean	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.01	< 0.01	< 0.01
Tenderloin muscle	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	0.01, 0.01, 0.02	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
Mean	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.01	< 0.01	< 0.01

Laying hen

Forty five laying hens (leghorn, 1.4–1.6 kg bw) were divided into three dose groups. For each dose group there were 15 hens that were divided into three replicates (i.e. five hens per replicate, 15 hens per dose group). For each dose group a further five hens were maintained as controls.

Animals were dosed with diazinon once daily for 28 consecutive days by means of a gelatine capsule administered with a balling gun. The doses administered to the three dose groups were 0.5, 1.5 and 5 ppm. During the study, daily feed consumption and egg production were constant. The average feed consumption was 0.11 kg/day.

Egg samples were taken daily, with eggs within each replicate, for each dose group, being pooled to form one composite sample. Tissue samples were collected at sacrifice which occurred 19–23 hours after the last dose. Egg and tissue samples (muscle, skin plus attached fat, peritoneal fat and liver) were stored frozen and analysed within 5 months.

Samples were analysed for diazinon, G-24576 and CGA-14128 using method AG-550A. Procedural recoveries were undertaken at fortification levels of 0.01–0.5 mg/kg and were acceptable for all commodity/ analyte combinations except for one set for recoveries performed at 0.2 mg/kg for tissue samples. The recoveries for tissues at 0.2 mg/kg were 141 percent for diazinon, 150 percent for G-24576 and 172 percent for CGA-14128. The identity of the specific tissue used foe the procedural recoveries is not stated.

Residues of diazinon, G-24576 and CGA-14128 were < 0.01 mg/kg in eggs and all tissue samples for all dose levels.

APPRAISAL

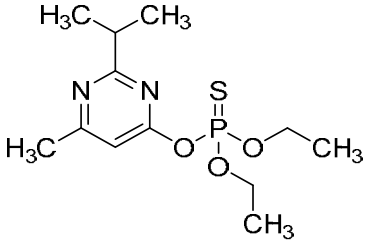
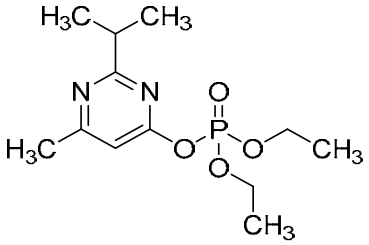
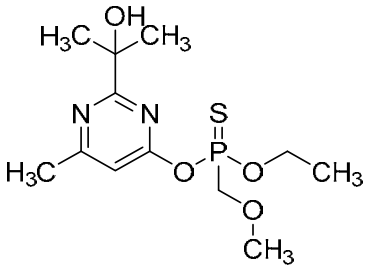
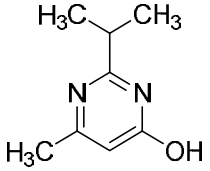
Diazinon is a contact organophosphorus insecticide with a wide range of insecticidal activity. It is effective against sucking, chewing and boring insects, including soil-living insects. Diazinon has been evaluated on numerous occasions by the JMPR commencing in 1963. The Most recent periodic review

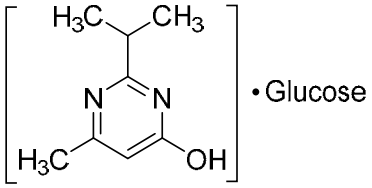
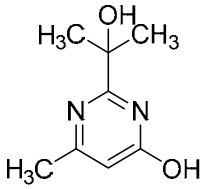
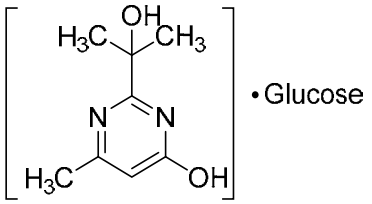
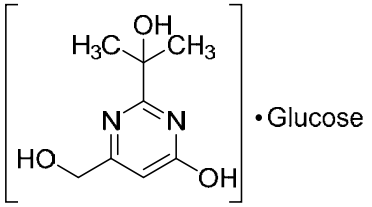
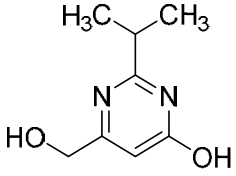
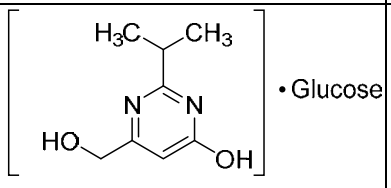
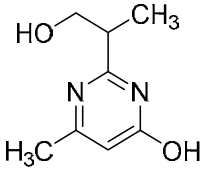
was in 1993. Following public health concerns identified by the International Agency for Research on Cancer (IARC), the JMPR in 2016 evaluated all previously considered toxicological data in addition to new studies. The 2016 JMPR recommended an ADI of 0–0.003 mg/kg bw and an ARfD of 0.03 mg/kg bw. Diazinon was scheduled at the Fifty-first Session of the CCPR (2019) for Periodic Review for residues by the 2020 JMPR and re-scheduled for the 2022 JMPR.

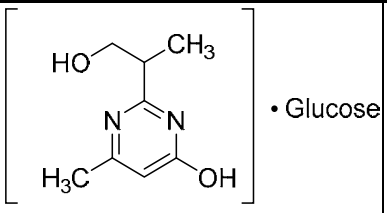
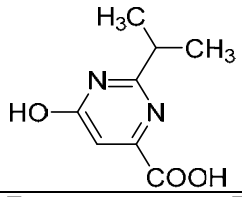
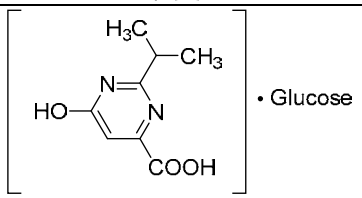
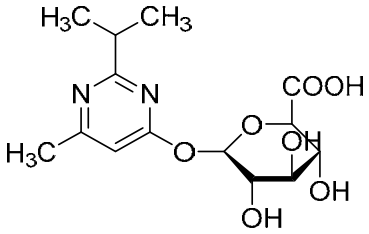
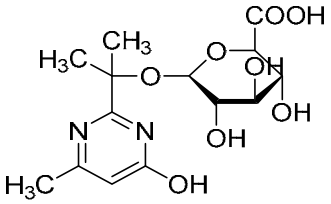
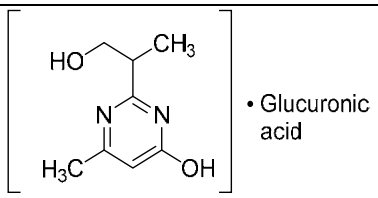
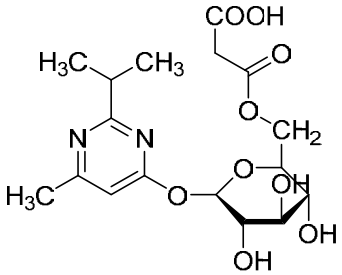
The Meeting received information from the manufacturer on physical and chemical properties, animal and plant metabolism, rotational crop studies, environmental fate in soil, analytical methods, storage stability, use patterns, supervised residue trials, processing studies and livestock feeding studies.

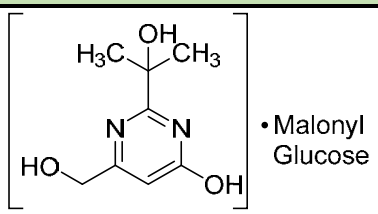
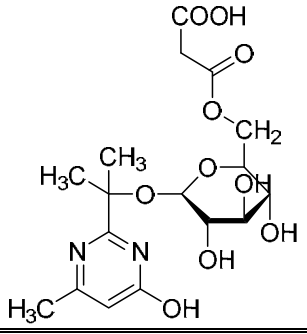
In this document, the common names, chemical structures and chemical names of the metabolites are shown below.

Table 85 Summary information on compounds referred to in the appraisal

Name/Code	Chemical name	Chemical structure	Occurrence in metabolism studies
Diazinon	*O,O*-diethyl-*O*-(2-isopropyl-6-methyl-4-pyrimidinyl)-phosphorothioate		Apple Beans Sweet corn Lettuce Potatoes Goat Hen Rotated spring wheat
G-24576 (diazoxon)	*O,O*-diethyl-*O*-(2-isopropyl-6-methyl-4-pyrimidinyl) phosphorate		Goat Hen
CGA-14128 (hydroxydiazinon)	*O,O*-diethyl-*O*-(2-[2-hydroxy-2-isopropyl]-6-methyl-4-pyrimidinyl)phosphorothioate		Goat Hen
G-27550 B_1	6-methyl-2-(1-methyl-ethyl)-4-pyrimidinol		Apple Beans Sweet corn Lettuce Potatoes Goat Hen Rotated Spring wheat

Name/Code	Chemical name	Chemical structure	Occurrence in metabolism studies
Glucose conjugate of G-27550	6-methyl-2-(1-methyl-ethyl)-4-pyrimidinol glucose conjugate		Bean, vines only
GS-31144 C	2-(2-hydroxyisopropyl)-6-methyl-4-pyrimidinol		Apple Beans Sweet corn Lettuce Potatoes Goat Hen Rotated spring wheat
Glucose conjugate of GS-31144 E_2	2-(2-hydroxyisopropyl)-6-methyl-4- pyrimidinol glucose conjugate		Apple Beans Sweet corn Lettuce Potatoes
Two glucose conjugates of trihydroxy pyrimidinyl moiety† G and H	2-(2-hydroxyisopropyl)-6-hydroxymethyl-4-pyrimidinol glucose conjugate		Apple Beans Sweet corn Lettuce Potatoes Rotated spring wheat
JAK-III-57 D	2-(1-methylethyl)-6-hydroxymethyl-4- pyrimidinol		Apple Beans Sweet corn Lettuce Potatoes Rotated spring wheat
Glucose conjugate of JAK-III-57 F_2	2-(1-methylethyl)-6-hydroxymethyl-4- pyrimidinol glucose conjugate		Apple Beans Sweet corn Lettuce Potatoes Rotated spring wheat
CL-XIX-29 E_1 (also referred to as M3 in some studies)	2-(1-hydroxypropan-2-yl)-6-methyl-4-pyrimidinol		Apple Beans Sweet corn Lettuce Potatoes Hen Rotated spring wheat

Name/Code	Chemical name	Chemical structure	Occurrence in metabolism studies
Glucose conjugate of CL-XIX-29 F1	2-(1-hydroxypropan-2-yl)-6-methyl-4-pyrimidinol glucose conjugate		Apple Beans Sweet corn Lettuce Potatoes Rotated spring wheat
JAK-IV-23	2-isopropyl-6-oxo-1,6-dihydropyrimidine-4-carboxylic acid		Beans
Glucose conjugate of JAK-IV-23	2-isopropyl-6-oxo-1,6-dihydropyrimidine-4-carboxylic acid glucose conjugate		Beans
Glucuronic acid conjugates of G-27550	6-methyl-2-(1-methyl-ethyl)-4-pyrimidinol glucuronic acid conjugate		Hen
Glucuronic acid conjugates of GS-31144	2-(2-hydroxyisopropyl)-6-methyl-4- pyrimidinol glucuronic acid conjugate		Hen
Glucuronic acid conjugates of CL-XIX-29	2-(1-hydroxypropan-2-yl)-6-methyl-4-pyrimidinol malonyl glucuronic acid conjugate		Hen
A conjugate of G-27550 [§] (postulated to be a malonyl glucose conjugate)	6-methyl-2-(1-methyl-ethyl)-4-pyrimidinol malonyl glucose conjugate		Beans Lettuce

Name/Code	Chemical name	Chemical structure	Occurrence in metabolism studies
A conjugate of JAK-III-57 § (postulated to be a malonyl glucose conjugate)	2-(1-methylethyl)-6-hydroxymethyl-4- pyrimidinol malonyl glucose conjugate		Beans Lettuce
A conjugate of GS-31144 § (postulated to be a malonyl glucose conjugate)	2-(2-hydroxyisopropyl)-6-methyl-4- pyrimidinol malonyl glucose conjugate		Beans Lettuce

Notes:

† The structure of the two metabolites G and H were not fully elucidated. The mass spectral analysis did not confirm the positions of the hydroxyl groups. The metabolites were susceptible to hydrolysis with β-glucosidase but no identification work was undertaken on the aglycone.

§ The identity of the conjugates were not established. The aglcones released from enzymatic or acid hydrolysis were confirmed.

With respect to the physical and chemical properties that may impact residues in crops, diazinon is regarded as moderately volatile, it has a higher solubility in organic solvents, compared to its solubility in water, and the partition coefficient (3.3–3.8) indicates its potential to sequester in fat.

Plant metabolism

Plant metabolism data, conducted via pre-emergence/soil directed applications followed by foliar applications, were provided for apple, beans with pods, sweet corn, lettuce and potatoes. In all the studies, the extraction procedure resulted in fractions that contained identified metabolites that were not individually quantified.

Apple

Apples, grown outdoors, were treated with [^{14}C]-pyrimidine-diazinon three times. The first application was at a rate of 3.36 kg ai/ha applied in the early tight cluster stage (approximately BBCH 55); this application was split between a soil application (3.024 kg ai/ha) and a foliar application to a single branch (0.336 kg ai/ha). The second and third applications were foliar applications at a rate of 10.09 kg ai/ha, made to the same branch when the apples were present, 104 days and 133 days after the first application.

Mature apples were harvested 14 days after the last treatment. Foliage samples were also collected. The foliage, pulp and peel were analysed. The TRR in leaves, peel, pulp and whole apple were 51.1 mg eq/kg, 3.44 mg eq/kg, 0.126 mg eq/kg and 1.29 mg eq/kg respectively. Solvent extractabilities, methanol: water (9: 1), ranged from 89.7 percent of the TRR for peel to 91.9 percent of the TRR for leaves.

The major residue identified in leaves, peel and whole apple was diazinon (22.3 mg/kg, 43.7 percent TRR for leaves, 2.5 mg/kg, 73.3 percent TRR for peel and 0.89 mg/kg, 69 percent TRR for whole apple). Metabolite G-27550 was found at 11.9 percent TRR (0.4 mg eq/kg) in peel, at 5.9 percent TRR (3 mg eq/kg) in leaves and at 14.7 percent TRR (0.19 mg/kg) in whole apple.

In the pulp the major residue was G-27550 (60.7 percent TRR, 0.08 mg eq/kg). Diazinon was found at a level of 16.1 percent TRR (0.02 mg/kg).

Glucose conjugates of GS-31144, CL-XIX-29, JAK-111-57 and of the trihydroxy pyrimidyl moiety were identified. For peel, pulp and whole apple the individual levels of these metabolites were not reported.

The PES accounted for 8.1 percent TRR (4.1 mg eq/kg) in leaves, 10.4 percent TRR (0.4 mg eq/kg) in peel, 9 percent TRR (0.01 mg eq/kg) in pulp and 11.6 percent TRR (0.15 mg eq/kg) in whole apple. The TRR identified was 72 percent TRR for leaves, 89 percent TRR for peel, 86 percent TRR for pulp and 87 percent TRR for whole apple.

Beans with pods

Beans with pods, grown outdoors, were treated with [^{14}C]-pyrimidine-diazinon three times. A pre-emergence application was made at a rate of 4.48 kg ai/ha applied 1 day after sowing. Two foliar applications were made at a rate of 1.4 kg ai/ha. The first foliar application was made 34 days after the pre-emergence application and the 2nd foliar application was made 15 days later.

Samples of beans with pods and vines were taken 14 days after the last application, which was stated to represent crop maturity. Immature crop fractions were also sampled: vines were taken 3 days before the first foliar application, 31 days after first treatment (DAFT), and vines with beans were taken 7 days after the first foliar application (41 DAFT). The growth stages of the various crop fractions sampled and analysed were not stated.

The TRR in beans with pods harvested 14 DALA, was 0.456 mg eq/kg. The solvent extractability, methanol: water (9:1), was 76 percent TRR. Diazinon was found at a level of 2.1 percent TRR (0.01 mg/kg). The predominant residue was G-27550 at 26.7 percent TRR (0.12 mg eq/kg). The metabolite JAK-111-57 (4.4 percent TRR, 0.02 mg eq/kg) was also identified. The metabolite CL-XIX-29 and glucose conjugates of GS-31144, CL-XIX-29, JAK-111-57 and of the trihydroxy pyrimidinyl moiety were also identified. However, the individual levels were not reported. The TRR identified in beans with pods was 53 percent TRR. The PES accounted for 24 percent TRR (0.109 mg eq/kg).

The TRR in the vines with beans, harvested 41 DAFT, was 4.45 mg eq/kg. The identification and characterisation of the TRR was unsuccessful.

The TRR in vines was 0.425 mg eq/kg and 3.53 mg eq/kg for vines harvested 32 DAFT and 14 DALA respectively. Solvent extraction was undertaken with methanol: water (9:1) and was 54 percent TRR for the vines harvested 32 DAFT. The solvent extractability was higher for the vines harvested 14 DALA at 82 percent TRR. Diazinon was not identified in either of the vine samples. In vines, harvested 32 DAFT, the highest contribution to the TRR was 17.3 percent TRR (0.07 mg eq/kg) which was attributed to a mixture of two glucose conjugates of the trihydroxy pyrimidinyl moiety and an unknown metabolite. The metabolites CL-XIX-29 and a glucose conjugate of GS-31144 accounted for 15.5 percent TRR (0.07 mg eq/kg), with the individual levels not reported. In the vines harvested 14 DALA, the highest contribution to the TRR (27.5 percent TRR, 0.97 mg eq/kg) was attributed to a mixture of CL-XIX-29 and a glucose conjugate of GS-31144. An unknown metabolite occurred at a level of 19.5 percent TRR (0.69 mg eq/kg). Glucose conjugates of GS-31144 and CL-XIX-29 were identified and together accounted

for 12.5 percent TRR (0.44 mg eq/kg), with the individual levels not stated. The TRR identified in the vines was 58 percent TRR. The PES accounted for 18 percent TRR (0.639 mg eq/kg).

In a supplementary study, retained samples of beans with pods and vines harvested 14 DALA were re-extracted after 69 months of storage. The TRR and solvent extractability (methanol: water (9:1)) for the beans with pods was 0.509 mg eq/kg and 75 percent TRR. This was comparable to the earlier extractions for which the TRR was 0.456 mg eq/kg and the solvent extractability was 76 percent TRR. For the vines the TRR and the solvent extractability was 3.76 mg eq/kg and 78 percent TRR respectively which was comparable to the earlier extraction for which the TRR was 3.53 mg eq/kg and the solvent extractability was 82 percent TRR.

For both beans with pods and vines the extracted residue was partitioned with ethyl acetate and only the aqueous fraction was subjected to further analysis. In beans with pods, the highest contribution to the TRR was 20.3 percent TRR (0.103 mg eq/kg) which was found to contain JAK-IV-23, glucose conjugates of the trihydroxy pyrimidinyl moiety and unidentified conjugates of G-27550, JAK-III-57 and GS-31144. In vines, this fraction accounted for 17.4 percent TRR (0.652 mg eq/kg). The individual levels of the metabolites were not reported for either beans with pods or vines. In vines the highest contribution to the TRR was 27 percent TRR (1.013 mg eq/kg) which was found to contain CL-XIX-29 and a glucose conjugate of GS-31133, with the individual levels not being specified. Free G-27550, GS-31144 and JAK-III-59 were also identified in both beans with pods and vines.

The PES, from the beans with pods, from the supplementary study were subject to further extraction with methanol: water (9:1), 1 percent NaCl, cellulase hydrolysis and protease hydrolysis. The extraction procedures individually released 2.1–8.5 percent TRR (0.011–0.044 mg eq/kg), leaving 6.4 percent TRR (0.033 mg eq/kg) in the final post-extraction solids. In the released radioactivity, diazinon, G-27550 and GS-31144 were identified along with a number of unidentified polar fractions, with the individual levels of the analytes not specified

Sweetcorn

Sweetcorn, grown in a greenhouse, was treated with [^{14}C]-pyrimidine-diazinon three times. The first application was made pre-emergence, on the day of sowing, at a rate of 4.48 kg ai/ha. The second and third applications were foliar applications at a rate of 3.5 kg ai/ha applied 50 and 74 DAFT. Samples of various sweet corn fractions were taken 2 days prior to the last application (72 DAFT) and 14 DALA.

The TRR in the crop fractions sampled 72 DAFT were 2.070 mg eq/kg, 0.087 mg eq/kg and 0.810 mg eq/kg for forage, ears and stalks respectively. The solvent extractabilities (methanol: water (9:1) were 69 percent TRR for forage, 59 percent TRR for ears and 20 percent TRR for stalks.

The TRR in cob and grain harvested 14 DALA were 0.250 mg eq/kg and 0.453 mg eq/kg respectively. Solvent extraction was undertaken with methanol: water (9:1) and was 47.5 percent TRR, for the cob, and 26.4 percent TRR for the grain.

Following solvent extraction, the residue was partitioned with ethyl acetate. Although the majority of the TRR was shown to be aqueous soluble, only the organo-soluble fractions of forage (72 DAFT), stalks (72 DAFT), cobs and grain were subject to further analysis. For forage (14 DALA) both the organo-soluble and aqueous fractions were analysed.

In grain, diazinon was not identified. The metabolites G-27550, GS-31144 and JAK-111-57 were identified at levels of < 1 percent TRR (< 0.004 mg eq/kg). Two unknown metabolites at 0.1 percent TRR (0.0005 mg eq/kg) were also found in the grain. A total of 1.1 percent TRR (0.005 mg eq/kg) was identified in grain. The PES accounted for 74 percent TRR (0.33 mg eq/kg). Subsequent treatment of the

PES with amyloglucosidase released around 60 percent TRR (0.28 mg eq/kg) which was found to be highly polar in nature and likely to comprise of a complex mixture of sugar conjugates. The identities of the aglycone were not established.

In the stalks, harvested 72 DAFT, the predominant residue identified was G-27550 (7.7 percent TRR, 0.0624 mg eq/kg). The metabolites GS-31144 (1.3 percent TRR, 0.01 mg eq/kg) and JAK-111-57 (1.1 percent TRR, 0.009 mg eq/kg) were also identified. Two unknown metabolites were found at levels of 0.6 percent TRR (0.005 mg eq/kg) and 1.5 percent TRR (0.0122 mg eq/kg). Diazinon was not identified. A total of 10 percent TRR (0.0819 mg eq/kg) was identified. The PES accounted for 80 percent TRR (0.646 mg eq/kg).

In the forage samples harvested 72 DAFT, diazinon was identified at a level of 0.5 percent TRR (0.01 mg/kg). The predominant residue was G-27550 (7 percent TRR, 0.145 mg eq/kg). The metabolites GS-31144 (0.7 percent TRR, 0.0145 mg eq/kg) and JAK-111-57 (1.3 percent TRR, 0.0269 mg eq/kg) were also identified. Two unknown metabolites occurred at levels of < 1 percent TRR (< 0.026 mg eq/kg). The TRR identified was 9.5 percent TRR (0.2 mg eq/kg). The PES accounted for 31 percent TRR (0.64 mg eq/kg).

In sweet corn forage (14 DALA), the predominant residue was G27550 (14.5 percent TRR, 0.56 mg eq/kg). Conjugates of CL-XIX-29, JAK-111-57 and of the trihydroxy pyrimidinyl moiety accounted for 12.4 percent TRR (0.48 mg eq/kg), with the individual levels not specified. Unknown metabolite was found at 11.8 percent TRR (0.46 mg eq/kg). Approximately 41 percent TRR (1.6 mg eq/kg) was identified. The PES accounted for 25.5 percent TRR (0.992 mg eq/kg).

Lettuce

Lettuce, grown outdoors, was treated with [^{14}C]-pyrimidine-diazinon three times. The first application was made pre-emergence at a rate of 4.48 kg ai/ha. The second and third applications were foliar applications at a rate of 1.4 kg ai/ha applied at 7 days intervals. Immature leaves were harvested prior to the last application (0 DALA). Mature leaves were harvested 14 DALA.

The TRR were 1.885 mg eq/kg and 0.656 mg eq/kg for immature and mature lettuce respectively. Solvent extraction was undertaken using methanol: water (9:1, v/v). The solvent extractabilities were 87.2 percent of the TRR and 78.4 percent of the TRR for immature and mature lettuce respectively.

For immature lettuce, 47 percent TRR was found to be aqueous soluble and 40 percent TRR was found to be organosoluble. Only the organosoluble residue was subjected to further analysis. The main components identified in the organosoluble fraction from immature lettuce were G-27550 (18.9 percent TRR, 0.36 mg eq/kg) and diazinon (18.6 percent TRR, 0.35 mg/kg). The PES, remaining after solvent extraction, was 12.8 percent TRR (0.241 mg eq/kg).

For mature lettuce both the organosoluble and aqueous soluble extracts were subject to further identification/characterization. The extracts were also treated with ß-glucosidase to confirm the presence of the conjugates identified. The main metabolites identified were G-27550 (17.5 percent TRR, 0.12 mg eq/kg), diazinon (11.8 percent TRR, 0.08 mg/kg) and GS-31144 (11.7 percent TRR, 0.08 mg eq/kg). An unknown metabolite accounted for 12.7 percent TRR (0.08 mg eq/kg). Glucose conjugates of GS-31144, CL-XIX-29, JAK-111-57 as well as free CL-XIX-29 were also identified, with the individual levels not being specified. Two glucose conjugates of the trihydroxy pyrimidinyl moiety were also identified. In total 63.5 percent of the TRR was identified. Following solvent extraction, the PES for mature lettuce accounted for 21.6 percent TRR (0.142 mg eq/kg).

In a supplementary study, retained samples of immature and mature lettuce, after 69 months of storage, were re-extracted with methanol: water (9: 1) followed by partitioning with ethyl acetate. The resulting aqueous phase was subjected to further analysis.

For immature lettuce the TRR and the solvent extractability were 2.2 mg eq/kg and 82 percent TRR. This compared to the original extraction for which the TRR was 1.9 mg eq/kg and 87 percent TRR. For mature lettuce the TRR was 0.75 mg eq/kg and the solvent extractability was 72 percent TRR. This was also comparable to the original extraction for which the TRR and solvent extractability was 0.66 mg eq/kg and 78 percent TRR respectively.

For immature lettuce, the main fraction accounted for 11.7 percent TRR (0.255 mg eq/kg). This fraction contained G-27550 and an unknown metabolite, with the individual levels not stated. The metabolite G-27550 was also identified separately at a level of 8 percent TRR (0.175 mg eq/kg). The metabolites GS-31144 (5.7 percent TRR, 0.124 mg eq/kg) and glucose conjugates of GS-31144 (1.2 percent TRR, 0.027 mg eq/kg), CL-XIX-29/ JAK-III-57 (3.6 percent TRR, 0.079 mg eq/kg) and of the trihydroxy pyrimidinyl moiety (2.5 percent TRR, 0.054 mg eq/kg). Enzymatic and acid hydrolysis released G-27550 and GS-31144 as well as three unidentified metabolites. These accounted for 8.5 percent TRR (0.185 mg eq/kg) with the individual levels not specified. A further conjugate of G-27550 accounted for 3 percent TRR (0.064 mg eq/kg).

In mature lettuce, the main fraction accounted for 16.7 percent TRR (0.125 mg eq/kg). This fraction contained conjugates of G-27550, GS-31144 and three unknown metabolites. The individual levels were not specified. The metabolites G-27550/unknown (4.7 percent TRR, 0.035 mg/ eq/kg), G-27550 (3.2 percent TRR, 0.024 mg/eq/kg), GS-31144 (7.8 percent TRR, 0.059 mg eq/kg) and JAK-III-57 (1.3 percent TRR, 0.01 mg eq/kg). Glucose conjugates of GS-31144 (3.6 percent TRR, 0.027 mg eq/kg), CL-XIX-29/ JAK-III-57 (10.1 percent TRR, 0.076 mg eq/kg) and of the trihydroxy pyrimidinyl moeity (4 percent TRR, 0.03 mg eq/kg) were also identified.

The PES, from the mature lettuce from the supplementary study were subject to further extraction with methanol: water (9:1), 1 percent NaCl, cellulase hydrolysis and protease hydrolysis. In the released radioactivity, low levels of diazinon, G-27550 and GS-31144 were identified along with a number of unidentified polar fractions, for which the number and levels of metabolites was not stated.

The PES accounted for approximately 80 percent TRR for tubers harvested -1 and 15 DALA (0.057 mg eq/kg for -1 DALA and 0.23 mg eq/kg for 15 DALA). Treatment of the PES from the tubers harvested 15 DALA with amyloglucosidase solubilized around 33 percent TRR. The identity of the solubilized radioactivity was not confirmed. Acid hydrolysis of the PES released around 72 percent TRR with 11.5 percent TRR identified as G-27550.

The TRR for foliage were 0.059 mg eq/kg and 1.930 mg eq/kg at -1 and 15 DALA respectively. The solvent extractabilities using methanol: water (9:1) were 32 percent TRR and 81.5 percent TRR for foliage harvested -1 DALA and 15 DALA respectively. Further characterization and identification was only undertaken on the foliage samples from 15 DALA. Diazinon was found in the potato foliage at 14.2 percent TRR (0.27 mg/kg). The highest contribution to the radioactivity, accounting for 20.8 percent TRR (0.4 mg eq/kg) was a glucose conjugate of JAK-111-57 and CL-XIX-29, with the individual levels not specified. A glucose conjugate of the trihydroxy pyrimidinyl moiety accounted for 14.1 percent TRR (0.27 mg eq/kg). Free CL-XIX-29 and a glucose conjugate of GS-31144 accounted for 11.5 percent TRR (0.22 mg eq/kg), with the individual levels not stated. The Total radioactivity identified was 68 percent TRR (1.31 mg eq/kg). The PES were 18.5 percent TRR (0.357 mg eq/kg).

Summary and conclusion of metabolism in crops

The metabolism of diazinon has been investigated in apples, beans with pods, sweet corn, lettuce and potatoes. All studies were undertaken with the application of [^{14}C]-pyrimidine-diazinon three times. The first application was either a pre-emergence application or a soil directed application followed by two foliar applications.

The Meeting noted that the time period over which the studies were conducted (15 months for apple, 21 and 68 months for beans with pods, 18 months for sweetcorn, 19 months and 69 months for lettuce and 16 months for potatoes) was longer than would be standard. HPLC profiles of crop fractions of lettuce and beans with pods extracted after 19 months of storage and extracted after 69 months of storage were comparable. The Meeting noted that stability data from fortified samples showed that G-24576 was unstable in a range of plant commodities and therefore the Meeting concluded that in the absence of data to support the storage interval from harvest to extraction, the plant metabolism data could not be relied on for an assessment of the residue definitions for risk assessment.

The data indicate that the metabolism in all 5 crops is qualitatively similar and proceeds with cleavage of the ester bond of diazinon leading to the loss of the diethylthiophosphate moiety and the formation of G-27550. Oxidation of the isopropyl moiety of G-27550 leads to the formation of GS-31144 and CL-XIX-29 while oxidation of the methyl moiety on the pyrimidine ring of G-27550 leads to the formation of JAK-III-57. Glucose conjugates of G-27550 and of the hydroxypyrimidine metabolites were identified.

However, the lower solvent extractability for beans with pods, sweet corn, lettuce and potatoes compared to apples indicates potential qualitative and quantitative differences in the metabolism. The radioactivity remaining in the PES, after solvent extraction, was high for beans with pods, sweet corns, lettuce and potatoes and subsequent analysis did not fully establish the identity of the released radioactivity but toxicological relevant metabolites were released such as diazinon, G-27550 and GS-31144.

The level of identification in beans with pods, sweet corn, lettuce and potatoes was low. For the crop fractions relevant to human consumption the level of identification was 24 percent TRR (0.109 mg eq/kg) for beans with pods, 1.1 percent TRR (0.005 mg eq/kg) for grain, 40 percent TRR (0.75 mg eq/kg) for immature lettuce leaves, 63.5 percent TRR (0.42 mg eq/kg) for mature lettuce leaves and 11.5 percent TRR (0.03 mg eq/kg) for potato tubers.

For apples, the radioactivity identified was 86 percent TRR (0.112 mg eq/kg) for pulp, 87 percent TRR (1.12 mg eq/kg) for whole apples and 89 percent TRR (3.01 mg eq/kg) for peel. The PES, following methanol: water extraction (9:1) accounted for 12 percent TRR (0.15 mg eq/kg) in whole apple.

For the plant metabolites identified, G-27550, GS-31144 and CL-XIX-29 were also identified in the rat.

Environmental fate

The Meeting received information on the environmental fate and behaviour of diazinon, including hydrolytic stability, aqueous photolysis, photochemical degradation in soil, and aerobic soil degradation studies.

Aqueous hydrolysis

Hydrolysis of diazinon was rapid at pH 5 (DT_{50} of 0.15 days at 70 °C and 12 days at 25 °C) and significantly slower under neutral conditions (DT_{50} of 0.4 days at 70 °C and 138 days at 25 °C) and basic conditions (0.2 days at 70 °C and 77 days at 25 °C).

Hydrolysis of diazinon also increased with temperature. The main degradation product identified was G-27550. At pH 5 and 25 °C, after 21 days diazinon accounted for 29 percent AR and G-27550 accounted for 67 percent AR.

The Meeting concluded that hydrolysis could be a significant degradation pathway under environmental conditions.

Aqueous photolysis

The photolysis of diazinon in aqueous solutions was investigated in two studies. Diazinon was found to be stable in deionised water at 20–25 °C when exposed to artificial light for 12 days. In a buffer solution at pH 7, temperatures 12–49 °C, diazinon was found to degrade when exposed to natural sunlight over 30 days. Only metabolite G-27550 was identified. The DT50 values calculated from the second study were 23 and 26 days.

The Meeting concluded that the degradation of diazinon, as a result of aqueous photolysis, was slow and was therefore unlikely to be a significant pathway under environmental conditions.

Soil photolysis

The photodegradation of diazinon in soil was investigated in two studies.

The DT50 for diazinon ranged from 17.3 to 37.4 hours under sunlight, while the DT_{50} values for diazinon in the dark control samples ranged from 15–39 days. The DT_{50}, calculated from study 1 only, for artificial light was 5.5 days.

The only degradation products identified were G-27550 and GS-31144. G-27550 accounted for up to 43 percent, 24 percent and 18 percent AR in artificially irradiated, natural sunlight and dark control samples, respectively after the exposure period. GS-31144 accounted for up to 3 percent AR after 21 hours of natural sunlight but was not found in the dark or artificial sunlight samples.

There were four unknown compounds which individually exceeded 10 percent of the AR at several time points.

The Meeting concluded that soil photolysis is a route of degradation for diazinon.

Aerobic soil degradation

Soil degradation studies were conducted in three soil types at application rates ranging from 1.2 to 10 mg ai/kg dry weight (dw) of soil. After 76 days of incubation at 20 °C in the dark, diazinon ranged from 1.8–9.1 percent AR. The main degradation products identified were GS-31144 and G-27550. G-27550 accounted for 48.1–65.8 percent AR after 76 days of incubation.

The mineralization of diazinon into CO2 accounted for up to a maximum 9.9 percent AR. Extractability from all samples declined with time, with 62.2–73 percent AR extracted at day 76.

The DT_{50} values calculated for diazinon were 8 days, 23 days and 9.9 days for sandy loam, loamy sand and clay loam soils respectively. The DT_{50} values calculated for G-27550 were 124 days, 131 days and 124 days for sandy loam, loamy sand and clay loam soils respectively

The Meeting concluded that under aerobic conditions diazinon was non-persistent in soil and that G-27550 was persistent in soil.

Residues in Succeeding or Rotational crops

Confined rotational crop studies

The Meeting received two confined rotational crop studies.

In the first confined rotational crop study a primary crop of maize was treated with [^{14}C]-pyrimidine-diazinon three times. The first application was made at a rate of 4.48 kg ai/ha (pre-emergence) followed by two foliar applications at a rate of 3.5 kg ai/ha (BBCH 30–39). The two foliar applications were made 50 and 74 days after sowing of the primary crop

Rotational crops of wheat, lettuce, sugar beet and soya bean were planted after harvest of the primary maize crop, which was 98 days after the last foliar application.

The TRR in immature lettuce and mature lettuce were 0.072 mg eq/kg and 0.039 mg eq/kg respectively. The solvent extractability for the mature lettuce using methanol: water (9:1, v/v) was 72 percent TRR. No further characterization/ identification of the residue was undertaken for the lettuce samples.

For sugar beet roots the TRR were 0.048 mg eq/kg and 0.016 mg eq/kg for immature roots and mature roots respectively. The TRR in the leaves were 0.061 mg eq/kg, 0.040 mg eq/kg and 0.16 mg eq/kg for 25 percent mature leaves, immature leaves and mature leaves respectively. Solvent extraction was only undertaken for the 50 percent mature leaves with 91 percent TRR extracted with methanol: water (9:1, v/v). No further characterization/ identification of the residue was undertaken for the sugar beet samples.

In the mature soya beans, the TRR was 0.19 mg eq/kg. The solvent extractability with methanol: water (9:1, v/v) was 16 percent TRR. For the mature pods, the TRR was 0.23 mg eq/kg and the solvent extractability with methanol: water (9:1, v/v) was 43 percent TRR. The TRR determined in the stalks were 0.12 mg eq/kg, 0.16 mg eq/kg and 0.19 mg eq/kg for 25 percent mature stalks, 50 percent mature stalks and 50 percent mature stalks respectively. The solvent extractability with methanol: water (9:1, v/v) ranged from 63–72 percent TRR. No further characterization/ identification of the extracted residue and unextracted residue was undertaken for the soya bean samples.

In wheat grain, the TRR was 0.24 mg eq/kg and the solvent extractability with methanol: water (9:1, v/v) was 11.6 percent TRR. In the extracted residue, a glucose conjugate of the trihydroxy pyrimidinyl moiety (0.8 percent TRR, 0.002 mg eq/kg), GS-31144 (0.4 percent TRR, < 0.001 mg eq/kg) and an unknown metabolite with G-27750 (0.8 percent TRR, 0.002 mg eq/kg) were identified.

For wheat hulls, stalks and foliage the TRR range from 0.14 mg eq/kg (foliage) to 0.62 mg eq/kg (wheat hulls). Solvent extractabilities with methanol water (9:1, v/v) ranged from 58 percent TRR (wheat hulls) to 70 percent TRR (stalks). In the extracted residue from these crop fractions a glucose conjugate of the trihydroxy pyrimidinyl moiety (0.012–0.055 mg eq/kg), CL-XIX-29 (0.004–0.023 mg eq/kg), JAK-111-57 (0.004–0.08 mg eq/kg), CL-XIX-29 with a glucose conjugate of JAK-III-57 (0.008 mg eq/kg) and GS-31144 (0.008–0.029 mg eq/kg) were identified at various levels. An unknown metabolite quantified with G-27750 was also identified (0.03–0.085 mg eq/kg. Diazinon was only found in the stalks at 0.045 mg/kg.

The TRR identified was 1.2 percent TRR (0.002 mg eq/kg) for wheat grain, 19 percent TRR (0.094 mg eq/kg) for wheat hulls, 21 percent TRR (0.133 mg eq/kg) for mature stalks and 24 percent TRR

(0.033 mg eq/kg) for 25 percent mature foliage. No further analysis of the unextracted residue was undertaken.

A second study focused on the uptake of residues in rotational crops with no identification of the radioactive residues undertaken. Primary crops of lettuce, beans with pods and potatoes were all treated with [^{14}C]-pyrimidine-diazinon at a rate of 4.48 kg ai/ha, applied pre-emergence, one day after sowing of the primary crop, followed by two foliar applications at 1.4 kg ai/ha. The foliar applications were made at intervals of 34 days and 41 days for lettuce, 34 days and 49 days for beans with pods, and 75 days and 82 days for potatoes, after the pre-emergence application. Rotational crops of wheat, lettuce, soya beans and sugar beet were planted 90 to 327 DALA after harvest of the primary crop.

The TRR in wheat grain, immature wheat stalks, immature and mature lettuce, and sugar beet roots and leaves were < 0.01 mg eq/kg. Residues above 0.01 mg/kg were obtained in mature wheat stalks (0.012 mg eq/kg), wheat hulls (0.011 mg eq/kg) and wheat full grazing leaves (0.014 mg eq/kg). Residues above 0.01 mg eq/kg were also obtained in various soya bean fractions; mature beans (0.015 mg eq/kg), mature pods (0.012 mg eq/kg), 0.011 mg eq/kg (50 percent mature stalks) and 0.027 mg eq/kg (mature stalks).

Solvent extraction was undertaken for a limited number of crop fractions using methanol: water (9:1, v/v). The solvent extractabilities were low and ranged from 16 percent TRR (mature soya beans) to 61 percent TRR (50 percent mature soya bean stalks).

Summary and conclusion of metabolism in rotational crops

The Meeting noted that the rotational crops did not investigate shorter PBI of 30 days and the rotational crops were planted after the harvest of treated primary crops, rather than being planted after applications to the bare soil. Although significant crop interception will have occurred for the foliar applications and the intervals (34–75 days) between the pre-emergence and first foliar application may have further reduced the total amount of residue available in the soil, residues above 0.01 mg/kg were identified in rotational crops. Based on the use patterns provided to the current Meeting residues above 0.01 mg/kg are expected in rotational crops. However, the identity of the residues in rotational crops has not been confirmed.

Owing to the limited information on the identity of the metabolites in rotated crops, the Meeting decided a comparison of the primary and rotational crop metabolism was not possible. The data available for wheat, indicates that the metabolite G-27550 is likely to be significant in rotational crops.

Field rotational crop study

A field rotational crop study was conducted using lettuce, turnips and wheat planted 30, 60 and 180 days after the last application to a primary crop. Primary crops of lettuce, squash, melons and tomatoes were treated with one pre-emergence application at 4.48 kg ai/ha followed by five foliar applications at 0.56 kg ai/ha and then harvested before planting of the rotational crops. The foliar applications to the primary crop were made at various growth stages and therefore the intervals between the applications and the amount of crop interception varied.

Residues of diazinon, G-24576 and CGA-14128 were < 0.01 mg/kg in all rotational crop samples from all plant back periods. The Meeting decided that a conclusion on the residue levels in rotational crops could not be drawn as the relevant residues for rotational crops could not be confirmed from the metabolism studies, noting that metabolite G-27550, which based on the available information may be significant in rotational crops, had not been included in the trials. The storage stability data on fortified samples showed that G-24576 rapidly degraded in a range of plant commodities and therefore the

reported results of < 0.01 mg/kg for this metabolite in rotational crops could not be relied on as the samples were stored for up to 15 months prior to analysis.

Animal metabolism

The meeting received information on metabolism of diazinon in ruminants (lactating goat) and poultry (laying hens).

Rat

Metabolism studies on laboratory animals including rats were reviewed in the framework of the toxicological evaluation by the WHO core Assessment Group of the 2016 JMPR.

Lactating goat

Two goats were orally dosed, by capsule, with [^{14}C]-pyrimidine-diazinon at a rate of 109–114 ppm for four consecutive days. Goats were slaughtered 24 hours after the last dose. The majority (72–77 percent) of the administered dose was excreted in urine (64 percent) and faeces (9–12 percent). The total radioactivity excreted in milk was 0.31 percent AD.

The TRR in milk over the 4 days ranged from 0.45–0.469 mg eq/kg, for animal 1 and 0.33 mg eq/kg–0.46 mg eq/kg for animal 2. A plateau was reached after 2-3 days. The solvent extractability for milk, using acetonitrile: water (1:1, v/v), was 91 percent TRR (0.627 mg eq/kg). The predominant residues were GS-31144 (37.3 percent TRR, 0.256 mg eq/kg) and G-27550 (39.3 percent TRR, 0.270 mg eq/kg). A total of 77 percent TRR was identified and 9 percent TRR (0.06 mg eq/kg) remained in the PES.

For liver the TRR was 1.57 mg eq/kg. The solvent extractability with methanol: water (9:1, v/v) was 79 percent TRR (1.24 mg eq/kg). The predominant metabolites identified were GS-31144 (19 percent TRR, 0.298 mg eq/kg) and G-27550 (19.2 percent TRR, 0.301 mg eq/kg). A total of 39 percent TRR was identified. The PES accounted for 21 percent TRR (0.33 mg eq/kg) and was not subject to any further analysis.

In kidney the TRR was 3.0 mg eq/kg. Solvent extraction with methanol: water (9:1 v/v) released 94 percent TRR (2.84 mg eq/kg). The predominant metabolites identified were GS-31144 (31 percent TRR, 0.92 mg eq/kg) and G-27550 (19.8 percent TRR, 0.60 mg eq/kg). A total of 52 percent TRR was identified. The PES accounted for 6 percent TRR (0.18 mg eq/kg).

The TRR determined in fat was around 0.36 mg eq/kg. Around 95 percent TRR was extracted with methanol: water (9:1, v/v). The predominant residue was diazinon (maximum 68 percent TRR, 0.25 mg/kg). The metabolite CGA-14128 accounted for around 12.8 percent TRR (0.047 mg eq/kg). The metabolites GS-31144, G-27550 and G-24576 were also identified. The total radioactivity identified exceeded 95 percent TRR for fat.

The TRR determined in muscle was around 0.40 mg eq/kg. Methanol: water (9:1, v/v) extracted ≥ 95 percent TRR The predominant residues were GS-31144 (maximum 40 percent TRR, 0.18 mg eq/kg) and G-27550 (maximum 35 percent TRR, 0.16 mg eq/kg). Over 76 percent TRR was identified in muscle and the PES was ≤ 5 percent TRR (0.02 mg eq/kg).

The solvent extracts from the tissue samples were subject to acid hydrolysis (6 M HCl at 85°C, left overnight). Increased levels of GS-31144, G27550, G-24576 and CGA-14128 indicate the potential presence of conjugates of these metabolites. The liver and kidney solvent extracts were also treated with b-glucuronidase (incubated at 37°C, left overnight). No details were reported but increased levels of the

metabolites GS-31144 and G-27550 were observed indicating the potential presence of glucuronic conjugates of these metabolites.

Laying hens

Laying hens were orally dosed, by capsule, with [^{14}C]-pyrimidine-diazinon at a rate of 25 ppm for seven consecutive days. Hens were slaughtered 24 hours after the last dose. The majority (79 percent) of the administered dose was excreted. The total radioactivity found in eggs was 0.3 percent of the administered dose. For tissues the total radioactivity, taken 24 hours after the last dose, accounted for 0.09 percent of the administered dose.

The TRR in egg yolk and egg white were 0.065 mg eq/kg and 0.066 mg eq/kg respectively. Solvent extractability with methanol: water (9,1, v/v) was 67 percent TRR (0.043 mg eq/kg) for egg yolk and 98 percent TRR (0.065 mg eq/kg) for egg white. The predominant residues identified were a mixture of glucuronic conjugates of G-27550, GS-31144 and CL-XIX-29 and CL-XIX-29 (25 percent TRR, 0.016 mg eq/kg in yolk and 41 percent TRR, 0.027 mg eq/kg in egg white). The metabolite G-31144 accounted for 19 percent TRR (0.012 mg eq/kg) in yolk and 33 percent TRR (0.022 mg eq/kg in egg white) and G-27550 accounted for 11 percent TRR (0.007 mg eq/kg) in yolk and 9.4 percent TRR (0.006 mg eq/kg) in egg white. For egg yolks 33 percent TRR (0.022 mg eq/kg) remained in the PES and a total of 63 percent TRR (0.04 mg eq/kg) was identified. For egg whites, 2 percent TRR (0.001 mg eq/kg) remained in the PES and 98 percent TRR (0.065 mg eq/kg) was identified.

In liver, the TRR was 0.11 mg eq/kg and 63 percent of the TRR was extracted with methanol: water (9:1, v/v). The predominant residue, accounting for 47 percent TRR (0.052 mg eq/kg) was a mixture of glucuronic acid conjugates of G-27550, GS-31144 and CL-XIX-29. The levels of the individual metabolites were not determined. The PES, following solvent extraction, accounted for 37 percent TRR (0.041 mg eq/kg). Following treatment with protease diazinon, CGA-14128, G-24576, G-27550, GS-31144 and CL-XIX-29 were identified. For liver, 63 percent TRR (0.69 mg eq/kg) was identified.

For kidney, the TRR was 0.15 mg eq/kg. The solvent extractability using methanol: water (9:1,v/v) was 76 percent TRR (0.11 mg eq/kg). The predominant residue (56 percent TRR, 0.083 mg eq/kg) was a mixture of glucuronic conjugates of G-27550, GS-31144, CL-XIX-29 with the individual levels not being reported. The PES accounted for 24 percent TRR and 76 percent TRR (0.11 mg eq/kg) was identified.

In muscle, the TRR was 0.025 mg eq/kg and 64 percent TRR (0.016 mg eq/kg) was extracted with methanol: water (9: 1, v/v). Glucuronic acid conjugates of G-27550, GS-31144, CL-XIX-29 and CL-XIX-29 accounted for 22 percent TRR (0.006 mg eq/kg) in the organosoluble fraction. Glucuronic acid conjugates of G-27550, GS-31144, G-27550, GS-31144 and CL-XIX-29 also accounted for a further 31 percent TRR (0.008 mg eq/kg) in the aqueous soluble fraction. In all cases, the individual levels of the metabolites were not reported. After solvent extraction, 36 percent TRR (0.009 mg eq/kg) remained in the PES. For muscle, 64 percent of the TRR (0.016 mg eq/kg) was identified.

The TRR in skin with fat and peritoneal fat were low at 0.018 mg eq/kg and 0.01 mg eq/kg respectively. The solvent extractability with methanol water (9:1, v/v) was 46 percent TRR (0.008 mg eq/kg) and 31 percent TRR (0.003 mg eq/kg) for skin with fat and peritoneal fat respectively. The PES were ≤ 0.01 mg eq/kg. No metabolites > 10 percent TRR or > 0.01 mg eq/kg were identified.

Treatment of the PES from eggs and tissues with protease released between 21 percent TRR (egg yolk) and 69 percent TRR (Peritoneal fat). Further analysis was only undertaken for the liver samples. It was reported that residues of diazinon, CGA-14128, G-24576, G-27550, GS-31144 and CL-XIX-29 were found, with the individual levels not specified.

Summary and conclusion of metabolism in livestock

The Meeting concluded that, in all species investigated (goats, hens and rats), the total administered radioactivity was predominantly eliminated in excreta. The data indicate that the metabolic profile was qualitatively similar in all three species and proceeds with cleavage of the ester bond of diazinon leading to the loss of the diethyl thiophosphate moiety and the formation of G-27550. Oxidation of the isopropyl moiety of diazinon leads to the formation of CGA-14128 while replacement of sulphur in the diethyl thiophosphate group of diazinon with oxygen leads to the formation of G-24576. Oxidation of the isopropyl moiety of G-27550 leads to the formation of GS-31144. Oxidation of the isopropyl moiety also leads to the formation of CL-XIX-29 (hens and rats only).

For poultry, based on the low levels of radioactivity observed in eggs and tissues, the Meeting decided the extractability and level of identification was sufficient. However, the predominant residues in eggs, liver, kidney and muscle were glucuronic acid conjugates of various aglycones for which the individual levels were not specified. In goat liver the PES was high and the level of identification in both liver and kidney was low (< 52 percent TRR). Owing to these deficiencies a quantitative consideration of the data for the assessment of the residue definitions for livestock was not possible.

The metabolite G-24576 (diazoxon) was identified in all tissues, milk and eggs. The Meeting noted that the storage stability data conducted with fortified samples, evaluated by the 1999 JMPR, demonstrated that G-24576 rapidly degrades in milk and tissues (except fat) within 0–4 months. As samples were stored for up to 5 months in the lactating goat study and up to 12 months in the poultry study the levels reported for this metabolite in the various animal matrices cannot be relied on. The Meeting considered that this metabolite is more toxic than diazinon.

Methods of analysis

The Meeting received information on analytical methods for diazinon in plant and animal matrices.

The Meeting received the description and validation data for the analysis of diazinon in pineapple. Residues were extracted with acetonitrile: water (9:1, v/v) with final determination by LC-MS/MS. The method was successfully validated with an LOQ of 0.01 mg/kg. This method was also successfully validated by an independent laboratory, demonstrating good reproducibility.

The Meeting also received the description and validation data for the analysis of diazinon, G-24576 and CGA-14128 in a range of plant commodities. Residues were extracted with acetone: water (9:1, v/v) followed by extraction with petroleum ether: dichloromethane: water (5:5:1, v/v/v) with final determination by GC-FPD. The Meeting noted that the individual recoveries were not available for each fortification level and for some commodities the mean recoveries were (up to 128 percent. However, the Meeting decided the method was suitable for the determination of diazinon, G-24576 and CGA-14128 in crops of a high water content, crops of a high acid content, crops of a high oil content, crops of a high starch content, crude and refined corn oil. The LOQ validated was 0.01 mg/kg for all three analytes. For hops the method was validated with an LOQ of 0.01 mg/kg for diazinon and CGA-14128. The Meeting decided that the method was not validated for the determination of G-24576 in hops owing to the low mean recovery of 54 percent.

For animal matrices, the Meeting received the description and validation data for the determination of diazinon in muscle, fat, liver, milks and egg. Residues were extracted from animal matrices, except fat, with acetone: water (9:1, v/v) followed by extraction with petroleum ether: dichloromethane: water (5:5:1, v/v/v). For fat, residues were extracted with acetonitrile. Final determination was by GC-FPD or GC-NPD (diazinon only). The Meeting noted that the individual recoveries were not available and the mean

recoveries for some commodities ranged from 68–125 percent. However, the Meeting decided the method was suitable for the determination of diazinon, G-24576 and CGA-14128 in animal commodities (muscle, fat, liver, milk and eggs) with an LOQ of 0.01 mg/kg for the GC-FPD method. Diazinon could also be determined in muscle, fat, liver, milk and eggs with an LOQ of 0.005 mg/kg for the GC-NPD method.

The Meeting was unable to conclude on the acceptability of method REM 4/81, used in the storage stability studies for animal matrices, as no validation data were provided.

The extraction efficiency was not investigated for any of the analytical methods, The meeting noted that the plant and animal metabolism data involved extraction with methanol: water (except milk which used acetonitrile: water) whereas the analytical methods used acetonitrile: water or acetone: water followed by petroleum ether: dichloromethane: water. Based on the high solubility of diazinon in a range of organic solvents the Meeting concluded that the methods are acceptable for the extraction of incurred residues of diazinon in plants and animals.

Stability of pesticide residues in stored analytical samples

The Meeting received freezer storage stability data for diazinon, CGA-14128, G-27550 and G-24576 in various homogenised plant matrices.

Diazinon was stable in pineapple pulp (high acid) and peel at ≤ -18 °C for at least 3 months of storage. Diazinon, CGA-14128 and G-27550 were stable in strawberries (high acid) at ≤ -20 °C for at least 56 days of storage. G-24576 was found to rapidly decline in strawberries, when stored at ≤ -20 °C, with a recovery of < 9 percent at the first time point of 1 month of storage. The strawberry samples, fortified with G-24576 only, were found to contain residues of G-27550, indicating that G-24576 may degrade to G-27550.

The Meeting also received storage stability data for diazinon, G-24576 and CGA-1412 in maize (high starch), tomato (high water), potato (high starch), apple (high water), strawberry (high acid), lettuce (high water) and various processed fractions.

The Meeting concluded that G-24576 was stable in refined corn oil for at least 26 month of storage at ≤ -12 °C. Significant degradation was observed in all other commodities.

For diazinon and CGA-14128 the data showed different rates of decline in the various commodities. The Meeting concluded on the following storage stability intervals, see Table 2 below.

Table 86 Storage stability of diazinon and CGA-14128 in different plant matrices

Matrices	Crop	Storage interval demonstrated (months) at ≤ -12 °C	
		Diazinon	CGA-14128
High water	Tomato	26	Up to 6
	Apple	Up to 26	Not stable
	Lettuce	26	26
High acid	Strawberries	2	2
	Pineapples	3	No data
High starch	Maize	26	26
	Potato	26	26
High oil	Soya beans	27	27
Processed commodities	Refined corn oil	27	27
	Tomato paste	16	4
	Sugar beet molasses	16	4

The Meeting agreed that the demonstrated storage stability covered the storage intervals for diazinon in the field trials for apple and pineapple.The Meeting also agreed that the data were sufficient to support the storage interval for the rotational crop field trial samples (lettuce, wheat and turnips).

For animal commodities, the Meeting decided that the data indicated that diazinon is stable in muscle, liver, kidney and fat for at least 8 months of freezer storage. However, the Meeting was unable to conclude on the acceptability of the data as no validation data were provided for the analytical method used to quantify residues.

The Meeting noted that the 1999 JMPR evaluated storage stability data for diazinon, CGA-14128 and G-24576 for milk and tissues in an iterim study. This study was not provided to the current Meeting. Diazinon and CGA-14128 were stable in milk and tissues for 9 months of storage when stored at ≤ -18 °C. G-24576 was stable in fat for 9 months of storage at ≤ -18 °C. However, in milk and muscle degradation of G-24576 was observed at the first time point of 4 months. For liver, G-24576 was found to have degraded in the time zero sample.

The Meeting concluded that the demonstrated storage stability for diazinon and CGA-14128 in animal commodities covered the storage interval in the feeding studies. The data do not support the storage interval in the feeding studies for G-24576.

Definition of the residue

Plant commodities

The nature of the diazinon residue was investigated in apple, bean with pods, sweet corn, lettuce and potatoes following one pre-emergence or soil directed application and two foliar applications. In the metabolism studies, the storage intervals between harvest and extraction were not supported, the individual levels of the metabolites were not always quantified in the extracted residues, the level of identification was low and the residue remaining in the PES was high and therefore the data is of limited value for a comparison of the metabolism for the assessment of the residue definition for risk assessment for plants.

The Meeting noted that diazinon was identified in various crop fractions in the metabolism studies and was found at levels above 0.01 mg/kg in the residue trials provided to the Meeting for apples, pears, pineapples and cabbage. The Meeting considered that diazinon was a suitable marker for the enforcement of MRLs. Suitable analytical methods are available to analyse diazinon in plants.

The nature of the residue on processing has not been investigated. The environmental hydrolysis studies included information on the effects of pH and temperature and indicate that diazinon is susceptible to hydrolysis and this is likely to be more prominent at higher temperatures. The potential for degradation products to be formed in processed commodities, particularly G-27550 cannot be excluded. Therefore, the Meeting could not confirm if diazinon would be a suitable marker for the enforcement of MRLs for processed commodities.

As a result of concerns relating to the lack of suitable quantitative information on the individual levels of metabolites in plants, a conclusion was unable to be reached on a residue definition for dietary risk assessment.

Animal commodities

The nature of the diazinon residue was investigated in lactating goats and laying hens following oral administration of the test substance. Owing to the low level of identification in some tissues, the lack of

suitable quantitative information on the individual levels of metabolites and the storage stability, the Meeting decided not to use the animal metabolism studies to establish residue definitions for livestock.

The Meeting recommended the following residue definitions for diazinon:

Definition of the residue for compliance with MRLs for plants: *Diazinon*

As for risk assessment, the Meeting was unable to reach a conclusion on a residue definition for plant commodities.

The Meeting was unable to conclude on the residue definition for compliance with MRLs and for risk assessment for animal commodities.

Results of supervised residue trials on crops

Supervised trials were available for apple, pears, pineapple, cabbage and wheat.

As a conclusion could not be reached on the residue definition for risk assessment, the Meeting withdrew all previous recommendations for maximum residue levels for diazinon, including the spice MRLs.

Pome fruits

The critical GAP for apples, pears and quince is for Chile which is 3 applications of 0.07 kg ai/hL applied at 15 day intervals with a PHI of 21 days.

Apple

A total of three independent trials conducted in the United States at 0.053 kg ai/hl–0.087 kg ai/hl, with a RTI of 14-15 days and a PHI of 21 days matched the GAP. Where replicate trials at different application rates were conducted at the trial site and both application regimes matched the GAP, the trial giving the highest residue was selected.

Residues of diazinon in independent trials approximating the critical GAP were (n= 3): 0.02, <u>0.04</u> (2) mg/kg.

Pears

A total of two independent trials conducted in the United States at 0.078 kg ai/hl, with a RTI of 14-15 days and a PHI of 21 days matched the GAP.

The Meeting agreed to apply the proportionality principle to two trials conducted at 0.18 kg ai/hl, noting that as residues were < 0.01 mg/kg a scaling factor was not required.

Residues of diazinon in independent trials approximating the critical GAP were (n= 4): < 0.01 (3) and 0.06 mg/kg.

As the median residues of diazinon for apples and pears are within a 5-fold range, the Meeting decided to make a recommendation for apples, pears and quince on the basis of the combined data sets for apples and pears.

Residues of diazinon on the basis of the combined data set were (n= 7): < 0.01 (3), 0.02, 0.04 (2) and 0.06 mg/kg

The Meeting estimated a maximum residue level of 0.15 mg/kg for apples, pears and quince.

Assorted tropical and sub-tropical fruits–inedible peel

Pineapples

The critical GAP is for the United States which is 2 applications of 1.12 kg ai/ha with retreatment intervals of 28 days and a PHI of 7 days.

As no trials matched the GAP the Meeting did not estimate a maximum residue level.

Brassica vegetables

Cabbag, Head

The critical GAP is for Costa Rica which is 3 applications of 0.75 kg ai/ha with a retreatment interval of 8 days and a PHI of 10 days.

As no trials matched the GAP the Meeting did not estimate a maximum residue level.

Cereals grains

Wheat

The critical GAP is for Russia which is 1 application of 1.08 kg ai/ha and a PHI of 60 days.

As no trials matched the GAP the Meeting did not estimate a maximum residue level.

Residues in animal feeds

Wheat straw

The critical GAP is for Russia which is 1 application of 1.08 kg ai/ha and a PHI of 60 days.

As no trials matched the GAP the Meeting did not estimate residue levels for use in the estimation of livestock dietary burdens.

Fate of residues during processing

High temperature hydrolysis

No information on the nature of the residue on processing was received by the Meeting. Based on the environmental hydrolysis study, that included the effects of temperature and pH, the Meeting concluded that diazinon is likely to be susceptible to hydrolysis and this is likely to be more prominent at higher temperatures. The formation of hydrolysis products such as G-27550 could not be excluded in processed foods.

Processing

The Meeting received information on the processing of apples and pears. Residues of diazinon, G-24576 and CGA-14128 were determined in the RAC and processed fractions. No details on the processing conditions employed were available.

Residues of G-24576 and CGA-14128 were all < 0.01 mg/kg. Residues of diazinon were also < 0.01 mg/kg in the RAC for pears. The Meeting decided that the storage interval of 9 months was supported for diazinon and CGA-14128. However, G-24576 would not be stable over the storage interval. No information about the level of the metabolite G-27550 was available.

The Meeting did not estimate processing factors, STMR-P and HR-P as a conclusion could not be reached on the residue definitions for processed commodities.

Residues in animal commodities

Farm animal feeding studies

Farm animal feeding studies in lactating cattle and laying hens were provided to the Meeting.

Lactating cattle

Groups of three lactating cows were fed diazinon in the diet once daily at a dose rate of 41, 124 and 414 ppm for 28–30 consecutive days. One animal served as a control.

Milk samples were taken twice daily and combined for each individual animal. Residues of CGA 14128 and G-24576 were < 0.01 mg/kg in all milk samples. At a feeding rate of 124 ppm, diazon in milk was ≤ 0.01 mg/kg. At a feeding rate of 414 ppm, the highest residue of diazinon was 0.08 mg/kg at 7 days. A plateau was reached in milk after 3 days and the mean residue over the plateau period was 0.03 mg/kg.

Tissue samples were collected at sacrifice which occurred within 24 hours of the last dose. The metabolite G-24576 was not found in any tissue samples. However, the storage stability data demonstrated that G-24576 would be unstable in all tissue samples, apart from fat, and therefore no conclusions can be drawn from the levels reported. The storage data evaluated by the 1999 JMPR indicated that diazinon and GCA 14128 would be stable over the 5 months of storage, but this study was not available to the current Meeting. The mean and highest residues in tissue samples are summarised below.

Table 87 Mean and highest residues of diazinon and GCA 14128 found in animal tissues

Tissue	Residue level at day 28, day 29 and day 30 (mg/kg)								
	41 ppm			121 ppm			414 ppm		
	Diazinon	G-24576	CGA-14128	Diazinon	G-24576	CGA-14128	Diazinon	G-24576†	CGA-14128
Liver									
HR	< 0.01	< 0.01	< 0.01	0.02	< 0.01	< 0.01	0.06	< 0.01	< 0.01
Mean	< 0.01	< 0.01	< 0.01	0.01	< 0.01	< 0.01	0.03	< 0.01	< 0.01
Kidney									
HR	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.01	< 0.01	< 0.01
Mean	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.01	< 0.01	< 0.01
Fat									
HR	0.04	< 0.01	< 0.01	0.10	< 0.01	< 0.01	0.84	< 0.01	0.06
Mean	0.03	< 0.01	< 0.01	0.08	< 0.01	< 0.01	0.56	< 0.01	0.05
Muscle									
HR	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.02	< 0.01	< 0.01
Mean	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.01	< 0.01	< 0.01

† G-24576 was found to be unstable in all animal commodities except fat

Laying hens

Three groups of 15 hens were fed diazinon in the diet once daily at a dose rate of 0.5, 1.5 and 5 ppm for 28 consecutive days. A further 15 hens served as control animals.

Egg samples were taken daily. Tissue samples were taken at sacrifice which ocurred 19–23 hours after the last dose.

Residues of diazinon, G-24576 and GCA 14128 were < 0.01 mg/kg in all samples. However, the Meeting noted that G-24576 would not be stable in the samples over the storage interval prior to analysis.

Farm animal dietary burden

As the Meeting was unable to estimate STMRs and HRs for plants, the dietary burdens of livestock could not be estimated.

Animal commodity maximum residue levels

As the dietary burdens of livestock could not be estimated, the Meeting was unable to estimate maximum residue levels for animal commodities.

RECOMMENDATIONS

The residue definition for compliance with the MRL for plant is: *diazinon*.

The Meeting was unable to conclude on a residue definition for risk assessment for plants.

The Meeting was unable to conclude on a residue definition for compliance with MRLs and for risk assessment for animal commodities.

Table 88 Recommendations for residues of diazinon from the 2022 JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
AM 0660	Almond hulls	W	5		
TN 0660	Almonds	W	0.05		
FB 0264	Blackberries	W	0.1		
FB 4079	Boysenberry	W	0.1		
VB 0400	Broccoli	W	0.5		
VB 0041	Cabbage, head	W	0.5		
VC 4199	Cantaloupe	W	0.2		
VR 0577	Carrot	W	0.5		
FS 0013	Cherries	W	1		
PE 0840	Chicken eggs	W	0.02*		
PM 0840	Chicken meat	W	0.02*		
PO 0840	Chicken, edible offal of	W	0.02*		
VL 0467	Chinese cabbage	W	0.05		
VP 0526	Common bean Pods and/or immature seeds)	W	0.2		
FB 0265	Cranberry	W	0.2		
VC 0424	Cucumber	W	0.1		
FB 0021	Currants, black, red and white	W	0.2		
VP 0529	Garden pea, shelled (succulent seed)	W	0.2		
MM 0814	Goat meat	W	2 (fat)†		
DH 1100	Hops, dry	W	0.5		
VL 0480	Kale (including collards, curly, scotch and thousand-headed kale; mot including marrow-stem kele)	W	0.05		
MO 0098	Kidney of cattle, goats, pigs and sheep	W	0.03†		
FI 0341	Kiwifruit	W	0.2		
VB 0405	Kohlrabi	W	0.2		
VL 0482	Lettuce, head	W	0.5		
VL 0483	Lettuce, leaf	W	0.5		

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
MO 0099	Liver of cattle, goat, pigs and sheep	W	0.03†		
GC 0646	Maize	W	0.02*		
MM 0097	Meat of cattle, pigs and sheep	W	2 (fat)†		
ML 0106	Milks	W	0.02		
VA 0385	Onion, bulb	W	0.05		
FS 0247	Peach	W	0.2		
HS 0444	Peppers chili, dried	W	0.5		
VO 0445	Peppers, sweet	W	0.05		
FI 0353	Pineapple	W	0.1		
FS 0014	Plums	W	1		
FP 0009	Pome fruits	W	0.3		
VR 0589	Potato	W	0.01*		
DF 0014	Prunes, dried	W	2		
VR 0494	Radish	W	0.1		
FB 0272	Raspberries, red, black	W	0.2		
HS 0191	Spices, fruit and berries	W	0.1*		
HS 0193	Spices, roots and rhizomes	W	0.5		
HS 0190	Spices, seeds	W	5		
VL 0502	Spinach	W	0.5		
VA 0389	Spring onion	W	1		
VC 0431	Squash, summer	W	0.05		
FB 0275	Strawberry	W	0.1		
VR 0596	Sugar beet	W	0.1		
VO 0447	Sweet corn (corn on the cob)	W	0.02		
VO 0448	Tomato	W	0.5		
TN 0578	Walnuts	W	0.01*		

Note:

† MRL accommodates external animal treatment

DIETARY RISK ASSESSMENT

As the Meeting was unable to recommend residue definitions for risk assessment for plants and animal commodities, long-term and acute dietary exposure assessments could not be conducted.

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R-259	Keller, A.	1981	Degradation of diazinon (Basudin®) in aerobic soil Report No. R-259 Non-GLP, Unpublished 30 Nov 1981

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1472	Krautter, M. S.	1994	Diazinon – Magnitude of the residue in meat and milk resulting from the feeding of three levels to dairy cattle, Part A: Biological Phase Report No. 1472 GLP, Unpublished 05 May 1994
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Study Reference	Author	Year	Study Title
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R-6500	Pesselman, R. L.	1992b	Solubility determination of diazinon, technical grade active ingredient Report No. R-6500 Study No. HWI-6413-107 GLP, Unpublished 30 Apr 1992
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R-6492	Pesselman, R. L.	1992d	Density determination of diazinon, manufacturing-use product Report No. R-6492 Study No. HWI 6413-101 GLP, Unpublished 30 Apr 1992
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10-88	Ross, J. A.	1992	Residue test report for field test number 0S-IR-602-91 Report No. 1 Project No. 302204 Unpublished 15 November 1989
2156 To 2166	Ross, J. A.	1992	Residue test report for field test number 0S-IR-602-91 Report No. 1 Project No. 302236 Unpublished 24 Sep 1992
2149 To 2155	Ross, J. A.	1992	Residue test report for field test number 02-IR-002-91 Report No. 1 Project No. 302193 Unpublished 21 Sep 1992
G24480/1802	Ross, J. A. and Gold, B.	1989	Residue Test Report OW-IR-618-88 Report No. G24480/1802 Unpublished 15 Nov 1989
R-4599	Schlesinger, H. M.	1987a	Diazinon – partition coefficient (n-octanol/water) Report No. R-4599 Study No. 424 Non-GLP, Unpublished 27 May 1987
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R-4628	Schlesinger, H. M.	1987c	Diazinon – solubility in organic solvents Report No. R-4628 Analyst Ltd. Report No. 432 Non-GLP, Unpublished 23 Jul 1987
SPR 19/81	Schnabel, D.	1981	Deep freeze storage stability study of residues in muscle, liver, kidney and fat of sheep Report No. SPR 19/81 Unpublished 11 Aug 1981
2374	Selman, F. B.	1992	Residue Test Report for 'Diazinon – Three level/28-day poultry study' (ABR-92083) Project No. 302997 Field Test No. IA, Report No. 1 GLP, Unpublished 16 Dec 1992

Study Reference	Author	Year	Study Title
2374	Selman, F. B.	1993	Residue Test Report for 'Diazinon – Three level/28-day poultry study' (ABR-92083) Project No. 302997 Field Test No. IA, Report No. 2 GLP, Unpublished 27 Jan 1993
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ABR-92021	Senzel, A. J. and Ross, J. A.	1992	Diazinon – Magnitude of residues in cabbage following application of D·z·n® Diazinon 14G, D·z·n® Diazinon 50W, and D·z·n® Diazinon AG500 Report No. ABR-92021 GLP, Unpublished 23 Sep 1992
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ABR-90065	Sobralske, M., Wong, A. and McFarland, J.	1990	Uptake and metabolism of 2Δ-^{14}C-diazinon in field rotational crops grown in soil which has been previously used for growing target crops in New York field plots Report No. ABR-90065 GLP, Unpublished 15 Aug 1990
G24480/0239	Spare, W. C.	1988a	Aqueous photolysis of ^{14}C-diazinon by natural sunlight Report No. G24480/0239 Study No. 12100-A GLP, Unpublished 17 Oct 1988
R-2376	Stamm, E.	1994	Rate estimation of the hydroxyl radical oxidation of diazinon G 24480 Report No. R-2376 Study No. 94SM07 Non-GLP, Unpublished 17 Aug 1994

Study Reference	Author	Year	Study Title
G24480/2514	Vincent, T. P. and Ediger, K.	1999	Diazinon – field accumulation in rotational crops Report No. G24480/2514 GLP, Unpublished 01 Mar 1999
2418	Walser, M.	1996	Validation of Analytical Method AG-550A (Determination of G 24480 residues in animal tissues using GC) at a LOQ of 0.005 mg/kg Report No. 2418 Ciba-Geigy Report No. 109/96 GLP, Unpublished 08 Jul 1996
R-16603	Wilson, A.	2004	Residue (at harvest) study with Diazol 60EC applied to pineapples in Costa Rica, 2004 Report No. R-16603 Makhteshim Study No. MAK/823/042630 GLP, Unpublished 25 Aug 2004
ABR-89058	Wong, A., Rezaaiyan, R. and McFarland, J.	1989	Uptake and metabolism of 2Δ-^{14}C-diazinon in field grown apples Report No. ABR-89058 GLP, Unpublished 29 Dec 1989
ABR-90040	Wong, A. and McFarland, J.	1990a	Uptake and metabolism of 2Δ-^{14}C-diazinon in field grown green beans Report No. ABR-90040 GLP, Unpublished 15 Aug 1990
ABR-90039	Wong, A. and McFarland, J.	1990b	Uptake and metabolism of 2Δ-^{14}C-diazinon in field grown lettuce Report No. ABR-90039 GLP, Unpublished 15 Aug 1990
ABR-89059	Wong, A., McDonald, J. and McFarland, J.	1989	Uptake and metabolism of 2Δ-^{14}C-diazinon in field grown potatoes Report No. ABR-89059 GLP, Unpublished 29 Dec 1989
R-10042	Not reported	1997	Report on residue studies with insecticide DIAZOL 60 EC produced by MAKHTESHIM-AGAN (Israel) in grain and straw of winter wheat variety “Donskaya Yubileinaya” in conditions of Rostov region in 1997 Report No. R-10042 Unpublished 1997
R-2251	Not specified	1993	Data sheet – G 24480 diazinon, Henry’s Law constant Ciba-Geigy Ltd. Unpublished 01 Apr 1993

DIFENOCONAZOLE (224)

First draft prepared by A Leahigh the Environmental Protection Agency, United States of America

EXPLANATION

Difenoconazole was evaluated by the JMPR for the first time in 2007 when an ADI of 0–0.01 mg/kg bw and an ARfD of 0.3 mg/kg bw were established. In 2007, 2010, 2013, 2015, 2017, and 2021 the JMPR evaluated for residues and recommended numerous maximum residue levels.

The physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, national registered use patterns, supervised residue trials, and processing in several plant commodities and residue of animal commodities were evaluated in previous JMPR evaluations.

The definition of the residue for compliance with MRL and for dietary intake for plant commodities is parent difenoconazole, while for animal commodities it is defined as sum of difenoconazole and 1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol (CGA205375), expressed as difenoconazole. The residue is fat-soluble.

Difenoconazole was scheduled at the Fifty-second Session of the CCPR for the evaluation of additional MRLs in 2022 JMPR. The current Meeting received additional information on analytical methods, storage stability, use pattern, and residue data of supervised trials in China on Goji Berry, Dried Goji Berry, Pencil Yam, Dried Pencil Yam, Ginger, Dried Ginger, Fresh Tea Leaves, Green Tea and Black Tea.

METHODS OF RESIDUE ANALYSIS

Goji berry (fresh and dried)

The analytical method (Study report number: R20002A, Yanmei Yang, 2021) for determining residues of difenoconazole in fresh and dried goji berry is as follows.

Goji berry samples were analysed for the residues of difenoconazole using UPLC-MS/MS. The validated limit of quantitation (LOQ) for residues of difenoconazole in goji berries (fresh and dried) is 0.01 mg/kg.

Briefly, 10 grams of fresh goji berry sample is mixed with 10 mL of acetonitrile and homogenized by a vortexer for 10 minutes at 2500 rpm. For dried goji berries, five grams was mixed with 8 mL of distilled water and 10 mL of acetonitrile and homogenized by a vortexer for 10 minutes at 2500 rpmn. Five grams of NaCl was added to the homogenized sample, which was then shaken by a vortexer for 5 minutes at 2500 rpm. Finally, the acetonitrile and water phases were completely separated by centrifugation for 5 minutes at 8000 rpm. A 1.5 mL aliquot was taken from the acetonitrile phase and transferred to a 2 mL centrifuge tube with 50 mg of primary secondary amine (PSA), 50 mg of octadecyl silica (C18), and 8 mg of Graphitised Carbon Black (GCB). After being homogenized by a vortexer for 5 minutes at 2500 rpm, the tube was centrifuged for 2 minutes at 5000 rpm. The upper layer was filtered through a 0.22 µm nylon syringe filter. Then 0.5 mL of the filtrate was diluted with 0.5 mL of water for analysis by UPLC-MS/MS. The quantitative and confirmation ions of difenoconazole were m/z 406.1/251.1 and 406.1/337.1, respectively. The residue levels were calculated with single point by direct comparison of the sample peak responses to those of external matrix-matched standards.

The method validation was performed by analysing fresh and dried goji berry for difenoconazole for each of 2 blank control specimens, 5 replicate specimens fortified at LOQ (0.01 mg/kg), 5 replicate

specimens fortified at 10× LOQ (0.1 mg/kg), and 5 replicate specimens fortified at 500× LOQ (5.0 mg/kg). Recoveries of difenoconazole in goji berry ranged from 86 to 105 percent (mean: 92–102 percent, n=6) and calculations of the relative standard deviation (RSD%) of the recoveries obtained in each spiking level presents RSDs ≤ 6 percent. The validated LOQ for residues of difenoconazole in both fresh and dried goji berry were 0.01 mg/kg. Table 1 lists all recoveries obtained during method validation.

Pencil yam (fresh and dried)

The analytical method (Study report number: HZ2020N001A, Xueyan Zhang, 2021) for determining residues of difenoconazole in fresh and dried pencil yam is as follows.

Briefly, 5.0 g of homogenized fresh pencil yam sample was weighed into a 50-mL PTFE centrifuge tube, to which 5-mL distilled water and 25-mL acetonitrile was added. For dried pencil yams 5.0 g of the pulverized dried pencil yam sample was weighed into a 50-mL PTFE centrifuge tube, to which 10-mL distilled water was added to soak for 10 minutes. After the 10 minute soaking period, 25 mL of acetonitrile was added. Then, the sample was homogenized thoroughly with the Multi-Tube Vortexer for 10 minutes, and shaken vigorously for 1 minute after the addition of 8 g NaCl. After being centrifuged for 5 minutes at 3000 rpm, 10-mL of the supernatant acetonitrile phase was transferred into a 150-mL round-bottomed flask. The extract was evaporated to near dryness by rotary evaporation at 40 °C, blow-dried and re-dissolved with 2.5 mL of acetonitrile/toluene (3:l, v/v) for the subsequent clean-up procedure. The extract was loaded onto a Sep-Pak Vac NH_2 SPE column (1 g, 6 mL) that had been preconditioned with 5-mL acetonitrile/toluene (3:l, v/v). Then, each of the three 2.5 mL portions of acetonitrile/toluene (3:l, v/v) was used to rinse the vessel and were successively loaded onto the column (the elution is approximately 10-mL in total). The effluent was collected into a 150-mL round bottomed flask, and concentrated to about 0.5 mL by rotary evaporators at 40 °C. Then it was blow dried and re-dissolved with acetonitrile. One mililitre of the extract was transferred into an HPLC sample vial after being filtered through 0.22-m syringe filter for UPLC/ESI-MS/MS analysis. The quantitative and confirmation ions of difenoconazole were *m/z* 406.1/251.0 and *m/z* 406.1/337.0, respectively. The residue levels were calculated with single point by direct comparison of the sample peak responses to those of external matrix-matched standards.

The method validation was performed by analysing fresh and dried pencil yam for difenoconazole for each of 2 blank control specimen, 5 replicate specimens fortified at LOQ (0.01 mg/kg), 5 replicate specimens fortified at 50× LOQ (0.5 mg/kg) and 5 replicate specimens fortified at 200× LOQ (2 mg/kg). Recoveries of difenoconazole in pencil yam ranged from 71 to 109 percent (mean: 85–103 percent, n=6) and calculations of the RSD of the recoveries obtained in each spiking level presents RSD ≤ 10 percent. Table 1 lists all recoveries obtained during method validation. The validated limit of quantitation (LOQ) for residual of difenoconazole in pencil yams (fresh and dried) is 0.01 mg/kg.

Ginger (fresh and dried)

The analytical method (Study report number: AR-2020PR20, Yizhi Feng, 2020) for difenoconazole in fresh ginger and dried ginger was submitted as follows.

Briefly, 10 mL of acetonitrile was mixed with 5 grams of fresh ginger or 2 grams of dried ginger. For dried ginger samples, 10 mL of water was added and the samples were shaken prior to adding the 10 mL of acetonitrile. Samples were shaken in a mechanical shaker for 5 minutes at approximately 2500 rpm. Approximately 5 g NaCl was added into the sample tubes. The samples were shaken in a mechanical shaker for 5 minutes at approximately 2500 rpm, and centrifuged for 5 minutes at 5000 rpm. A 1.0 mL aliquot of the acetonitrile phase (upper phase) was transferred into a centrifuge tube with, approximately, the following proportion of salts: Cl8 (100 mg) and PSA (100 mg). The centrifuge tube was shaken at approximately 2500 rpm shake for 30 seconds, followed by centrifugation at 5000 rpm for

1 minute. The extract was filtered with 0.22 µm organic filter membrane and submitted for analysis by LC-MS/MS. The quantitative and confirmation ions of difenoconazole were *m/z* 406.1/251.1 and *m/z* 406.1/336.9, respectively. The residue levels were calculated with single point by direct comparison of the sample peak responses to those of external matrix-matched standards.

The method was validated for analysis of ginger, hot-dried ginger, and freeze-dried ginger by adding known concentrations to control samples and analysing for difenoconazole. Fortification levels were LOQ (0.01 mg/kg), 10× LOQ (0.1 mg/kg), and 100× LOQ (1.0 mg/kg). Recoveries of difenoconazole in ginger, hot-dried ginger, and freeze-dried ginger were between 90 percent and 119 percent (mean: 93–115 percent, n=9), and the RSDs of the recoveries obtained in each spiking level present ≤ 5 percent. The validated LOQ for residues of difenoconazole in fresh and dried ginger were 0.01 mg/kg. The analytical method was considered to be suitable for the analysis of difenoconazole residues in the fresh ginger, hot-dried ginger, and freeze-dried ginger samples. Table 1 lists all recoveries obtained during method validation.

Fresh tea leaves, green tea, and black tea

The analytical method (Study report number: RM20004G20001, Yanjie Li, 2021) for difenoconazole in fresh green tea, green tea and black tea was submitted as follows.

Briefly, 20 mL of acetonitrile was mixed with 5 grams of fresh tea leaves or 2 grams of black and green tea. For black and green tea, 4 mL of water was added and the samples were shaken prior to adding the 20 mL of acetonitrile. Samples were shaken in a mechanical shaker for 10 minutes. Approximately 3 g NaCl was added to the sample tubes. The samples were shaken in a mechanical shaker for 5 minutes and centrifuged for 5 minutes at 4000 rpm. A 1.0 mL aliquot of the acetonitrile phase (upper phase) was transferred into round bottom flask and evaporated using a rotary evaporator at a bath temperature of 40 °C to near dryness. The samples were reconstituted to a final volume of 3 mL with acetonitrile/toluene (3:1, v/v). An SPE cartridge was conditioned with 5 mL of acetonitrile/toluene (3:1, v/v) and extracts were transferred into the SPE cartridges. The round bottom flask was rinsed twice with 2 mL portions of acetonitrile/toluene (3:1, v/v) and the rinsate was added to the SPE cartridge. The eluents were collected into clean round bottom flasks and evaporated using a rotary evaporator at a bath temperature of 40 °C to near dryness. The samples were reconstituted to a final volume of 2.5 mL with acetonitrile/toluene (3:1, v/v). The samples were transferred into an HPLC sample vial after being filtered through 0.22-m syringe filter for UPLC-MS/MS analysis. The quantitative and confirmation ions of difenoconazole were *m/z* 406.1/251.1 and *m/z* 406.1/337.05, respectively. The residue levels were calculated with single point by direct comparison of the sample peak responses to those of external matrix-matched standards.

For validation, untreated fresh tea leaves, green tea, and black tea were fortified with difenoconazole and analysed. The validation for each matrix typically consisted of at least , two untreated samples (control sample), five samples fortified at the LOQ (0.01 mg/kg), five samples fortified at 100× LOQ (1.0 mg/kg), and five samples fortified at 1000× LOQ (10 mg/kg). Fresh tea leaves were additionally fortified (n=5) at 2000× LOQ (20 mg/kg). Recoveries of difenoconazole in tea leaves, green tea, and black tea were between 71 percent and 105 percent (mean: 76–96 percent, n=10), and the RSDs of the recoveries obtained in each spiking level present ≤ 5 percent. The validated LOQ for residues of difenoconazole in tea leaves, green tea, and black tea were 0.01 mg/kg. The analytical method was considered to be suitable for the analysis of difenoconazole residues in fresh tea, green tea, and black tea. Table 1 lists all recoveries obtained during method validation.

Overall, the Meeting concluded that the presented methods were sufficiently validated and are suitable to measure difenoconazole in plant commodities. The demonstrated LOQ for all commodities evaluated was 0.01 mg/kg.

Table1 Summary of the method validation recovery data.

Matrix	Fortification (mg/kg)	n	Recovery (percent)	Mean recovery (percent)	RSD (percent)
Goji Berry[1]	0.01	5	94, 95(2), 97, 105	97	5
	0.1	5	93, 94, 96(2), 101	96	3
	5.0	5	98(2), 101, 102(2)	100	2
Dried Goji Berry[1]	0.01	5	86, 88, 94, 96, 98	92	6
	0.1	5	98, 99, 101, 103(2)	101	2
	5.0	5	92, 102, 104, 105(2)	102	5
Pencil Yam[2]	0.01	5	88, 90, 96, 102, 106	96	8
	0.5	5	95, 98, 99, 100, 101	99	2
	2.0	5	71, 87, 88, 90, 92	85	10
Dried Pencil Yam[2]	0.01	5	95(2), 96, 98, 99	97	2
	0.5	5	99(2), 104, 105, 109	103	4
	2.0	5	79, 82, 90(2), 93	87	7
Ginger[3]	0.01	5	109, 114, 115, 117, 119	115	3
	0.1	5	93, 102(2), 103(2)	101	5
	1	5	92, 101, 103(2), 105	101	5
Hot-dried Ginger[3]	0.01	5	90, 91, 93, 95, 96	93	3
	0.1	5	95, 96(2), 97(2)	96	1
	1	5	95, 96(2), 97(2)	96	1
Freeze-dried Ginger[3]	0.01	5	90, 93, 94, 96, 98	94	3
	0.1	5	90, 9592), 96, 97	94	3
	1	5	95(2), 96(3)	96	1
Fresh Tea Leaves[4]	0.01	5	77, 79, 83, 84, 92	83	7
	1.0	5	74, 75, 78(2), 79	77	3
	10	5	71, 74, 76, 80, 81	76	5
	20	5	81, 83, 88, 89, 98	88	8
Green Tea[4]	0.01	5	83, 87, 99, 100, 105	95	10
	1.0	5	88, 89, 90, 92, 93	90	2
	10	5	93, 94, 96, 97, 101	96	3
Black Tea[4]	0.01	5	77, 82, 86, 87, 90	84	6
	1.0	5	90(2), 92, 93, 95	92	3
	10	5	89, 91, 92, 96, 97	93	4

Notes:

[1] Study report number: R20002A, Yanmei Yang, 2021.

[2] Study report number: HZ2020N001A, Xueyan Zhang, 2021.

[3] Study report number: AR-2020PR20, Yizhi Feng, 2020.

[4] Study report number: RM20004G20001, Yanjie Li, 2021.

STABILITY OF PESTICIDES IN STORED ANALYTICAL SAMPLES

The freezer storage stability of difenoconazole has been assessed previously by the JMPR.

The current Meeting received additional information on storage stability of difenoconazole in goji berry, dried goji berry, pencil yam, dried pencil yam, ginger, dried ginger, fresh tea leaves, green tea, and black tea.

Goji berry (fresh and dried)

The Meeting received a study (study report number: R20002B, Yanli Qi, 2021) evaluating the stability of difenoconazole in goji berry and dried goji berry.

Briefly, the untreated control goji berry and dried goji berry samples were fortified separately with standard solutions containing difenoconazole at a level of 1.0 mg/kg and stored frozen (≤-18 °C) for durations of approximately 0, 1, 3, 6, and 12 months. Samples were analysed according to the method described in study report number R20002A.

Storage stability results of difenoconazole in goji berry and dried goji berry are summarised in Table 2. The mean concentrations of difenoconazole in fresh goji berry and dried goji berry were 81–99 percent and 85–111 percent, respectively. Therefore, the Meeting concluded that residues of difenoconazole were stable for at least 12 months in whole fresh goji berry and dried goji berry stored frozen at ≤-18 °C. Corresponding magnitude of the residue samples were stored frozen up to approximately 6 months prior to analysis for fresh and dried goji berry

Pencil yam (fresh and dried)

The Meeting received a study (study report number: HZ2020N001B, Xueyan Zhang, 2021) evaluating the stability of difenoconazole in fresh pencil yam and dried pencil yam.

Briefly, the untreated control fresh pencil yam and dried pencil yam samples were fortified separately with standard solutions containing difenoconazole at a level of 0.5 mg/kg and then stored at (≤-18 °C) for durations of approximately 0, 1, 3, 6, 9, and 14 months. Samples were analysed according to the method described in study report number HZ2020N001A.

Storage stability results of difenoconazole in fresh pencil yam and dried pencil yam are summarised in Table 2. Mean concentrations of difenoconazole from stored fortified fresh pencil yam and dried pencil yam were 80–97 percent and 80–100 percent for of at least 14 months of frozen storage. Therefore, the Meeting concluded that residues of difenoconazole were stable for at least 14 months in fresh pencil yam and dried pencil yam stored frozen at ≤-18 °C. Corresponding magnitude of the residue samples were stored frozen up to approximately 10 months for pencil yam and approximately 9 months for dried pencil yam.

Ginger (fresh and dried)

The Meeting received a study (Study report number: SR-2020SS01, Yizhi Feng, 2020) evaluating the stability of difenoconazole in fresh ginger, hot-dried ginger, and freeze-dried ginger under frozen storage.

Briefly, the untreated control fresh ginger, hot-dried ginger, and freeze-dried ginger were fortified separately with difenoconazole at 1.0 mg/kg and stored frozen (≤-18 °C) for approximately 0, 1, 3, 6, 9, and 12 months. Samples were analysed according to the method described in study report number AR-2020PR20.

Storage stability results of difenoconazole in fresh ginger, hot-dried ginger, and freeze-dried ginger are summarised in Table 2. Mean concentrations of difenoconazole from stored fortified fresh ginger, hot-dried ginger, and freeze-dried ginger samples were 86–89 percent after approximately 12 months of frozen storage. Corresponding magnitude of the residue samples were stored frozen up to approximately 3 months for fresh ginger and approximately 4 months hot- and freeze-dried ginger.

Fresh tea leaves, green tea, and black tea

The Meeting received a study (Study report number: RB20004G20001, Yanjie Li, 2021) evaluating the stability of difenoconazole in fresh tea leaves, green tea, and black tea under frozen storage.

Briefly, untreated samples of fresh tea leaves, green tea, and black tea were fortified separately with difenoconazole at 1.0 mg/kg and stored frozen (-27.1 °C to -19.0 °C) for durations of approximately 0, 1, 3, 7, 9, and 12 months. Samples were analysed according to the method described in study report number RM20004G20001.

Storage stability results of difenoconazole in fresh tea leaves, green tea, and black tea are summarised in Table 2. Mean concentrations of difenoconazole from stored fortified samples of fresh tea leaves, green tea, and black tea were 95–112 percent after approximately 12 months of frozen storage. Corresponding magnitude of the residue samples were stored frozen up to approximately 11 months for fresh tea leaves and black tea and approximately 12 months for green tea.

Table 2 Stability of difenoconazole residues in various plant matrices under frozen conditions

Analyte (Report)	Fortification (mg/kg)	Storage (days)	Concentration (mg/kg)	Concentration (percent)	Concurrent Recovery (percent)	Mean Concurrent Recovery (percent)
Goji berry						
Difenoconazole (R20002B)	1.0	0	0.99, 0.99	99	105, 99	102
		30	0.98, 0.92	95	96, 95	96
		92	0.91, 0.91	91	108, 105	106
		182	0.95, 0.89	92	110, 107	108
		365	0.82, 0.80	81	100, 100	100
Goji berry, dried						
Difenoconazole (R20002B)	1.0	0	0.97, 0.97	97	106, 105	106
		30	1.09, 1.12	111	108, 109	108
		90	0.95, 0.91	93	105, 104	104
		193	0.91, 0.84	88	103, 105	104
		365	0.85, 0.84	85	107, 107	107
Pencil yam						
Difenoconazole (HZ2020N001B)	0.53	0	0.508, 0.523	97	99, 106	103
		30	0.489, 0.530	96	102, 109	106
		108	0.453, 0.455	86	94, 88	91
		187	0.467, 0.444	86	102, 102	102
		290	0.442, 0.419	81	100, 94	97
		439	0.402, 0.444	80	91, 99	95
Dried pencil yam						
Difenoconazole (HZ2020N001B)	0.46	0	0.447, 0.456	98	100, 96	98
		30	0.450, 0.466	100	106, 98	102
		108	0.367, 0.368	80	87, 90	89
		187	0.383, 0.377	83	95, 98	97
		290	0.390, 0.384	84	98, 102	100
		439	0.391, 0.422	88	95, 83	89
Ginger						
Difenoconazole (SR-2020SS01)	1.0	0	1.0, 0.99, 1.0	100	103, 99, 104	102
		30	1.0, 1.0, 0.95	98	91, 102, 102	98

Analyte (Report)	Fortification (mg/kg)	Storage (days)	Concentration (mg/kg)	Concentration (percent)	Concurrent Recovery (percent)	Mean Concurrent Recovery (percent)
		90	1.1, 1.1, 0.99	106	91, 101, 103	98
		193	1.0, 1.1, 0.97	102	94, 102, 103	100
		272	1.0, 1.0, 1.0	100	102, 98, 105	102
		364	0.88, 0.88, 0.88	88	93, 93, 95	94
Hot-dried ginger						
Difenoconazole (SR-2020SS01)	1.0	0	0.91, 0.91, 0.91	91	92, 92, 93	92
		30	0.95, 0.94, 0.94	94	94, 94, 95	94
		90	0.94, 0.94, 0.95	94	94, 95, 95	95
		193	0.89, 0.89, 0.89	89	89, 89, 89	89
		272	0.91, 0.87, 0.90	89	91, 91, 88	90
		364	0.85, 0.86, 0.86	86	91, 92, 91	91
Freeze-dried ginger						
Difenoconazole (SR-2020SS01)	1.0	0	0.88, 0.87, 0.87	87	92, 91, 89	91
		30	0.93, 0.99, 0.90	94	95, 93, 93	94
		90	0.94, 0.94, 0.93	94	95, 93, 93	94
		193	0.93, 0.94, 0.92	93	95, 94, 92	94
		272	0.88, 0.87, 0.91	89	92, 90, 90	91
		364	0.90, 0.90, 0.87	89	96, 96, 97	96
Fresh tea leaves						
Difenoconazole (RB20004G20001)	1.0	0	0.896, 0.924	91	107, 101	104
		30	0.932, 0.946	94	99, 100	100
		95	0.805, 0.776	79	78, 79	78
		213	0.925, 0.816	87	87, 84	86
		275	0.976, 0.859	92	83, 84	84
		365	0.957, 0.936	95	103, 102	103
Green tea						
Difenoconazole (RB20004G20001)	1.0	0	0.940, 0.940	94	101, 100	101
		30	0.917, 0.911	91	97, 95	96
		95	0.865, 0.839	85	81, 85	83
		213	0.973, 0.965	97	100, 95	98
		275	1.10, 1.07	108	88, 87	88
		365	1.05, 0.965	101	83, 95	89
Black tea						
Difenoconazole (RB20004G20001)	1.0	0	0.990, 0.980	98	99, 98	98
		30	0.943, 0.913	93	99, 97	98
		95	0.822, 0.855	84	88, 93	91

Analyte (Report)	Fortification (mg/kg)	Storage (days)	Concentration (mg/kg)	Concentration (percent)	Concurrent Recovery (percent)	Mean Concurrent Recovery (percent)
		213	0.990, 0.988	99	102, 97	100
		275	1.06, 1.06	106	85, 91	88
		365	1.17, 1.15	116	101, 99	100

USE PATTERN

Difenoconazole is a systemic triazole fungicide globally registered in many countries for the control of a broad-spectrum of foliar, seed and soil-borne diseases caused by Ascomycetes, Basidiomycetes, and Deuteromycetes. In the following table, GAP information on all crops supported with residue trial data are summarised in Table 3.

Table 3 Approved uses of difenoconazole on goji berry, pencil yam, ginger, and tea in China.

Crop	Formulation percent	Method	Rate (kg ai/ha)	Spray Concentration (kg ai/hL)	Number of Applications	PHI (Days)
Goji berry	WG, 10 percent	Foliar spray		0.0050-0.0067	1	7
	EW, 4 percent	Foliar spray		0.0027-0.0040	3	3
	SC, 10 percent	Foliar spray		0.0050-0.010	3*	5
	WG, 37 percent	Foliar spray		0.0050-0.0067	1	7
Pencil yam	WG, 10 percent	Foliar spray	0.057-0.0795		3*	60
	WG, 10 percent	Foliar spray	0.045-0.0675		3*	60
Ginger	SC, 125 (g/L)	Foliar spray	0.075-0.1125		3*	14
	WP, 10 percent	Foliar spray	0.045-0.09		2	14
	WP, 60 percent	Foliar spray	0.0444-0.0888		2	14
	WP, 37 percent	Foliar spray	0.045-0.09		2	14
	WP, 30 percent	Foliar spray	0.045-0.09		2	14
Tea	WG, 10 percent	Foliar spray		0.0067-0.01	3*	14

Notes:

*RTI = Re-treatment interval of 7 days.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data on supervised field trials following foliar application of difenoconazole to goji berry (Study report Number: R20002, Weirong Wang, 2021), pencil yam (Study report Number: HZ2020N001, Wenxi Li, 2021), ginger (Study report Number: PR-2020PR20, Yizhi Feng, 2020) and tea (Study report Number: RA20004G20001, Yanjie Li, 2022).

The field trial reports included method validation data, as recoveries from spiked samples at levels reflecting those observed in the field trial samples; dates from critical events during the study, including application, harvest, storage, and analysis; as well as detailed information on the field site and

treatment parameters. Analytical reports were sufficiently detailed and included example chromatograms and example calculations.

Residues from the trials conducted according to Critical GAP have been used for the estimation of maximum residue levels and they are underlined. The higher mean result of replicate plots in each trial site is used for estimation of maximum residue levels and supervised trial median residues (STMRs). However, the highest result of all individual samples is used for highest Residues (HRs).

Goji berry

Four residue trials on goji berry conducted in China are presented below. Two replicate treated plots were set in each trial site and duplicate fresh goji berry samples were taken from each treated plot.

Fresh goji berry samples were collected immediately prior to the last application then 2 hours, 1, 3, 5, 7, and 10 days after the last application at all trials. Samples in untreated control plot were collected at the first sampling and the last sampling at each test site. Sampling started in the untreated control plot, followed by the treated plot. Two independent samples were collected at each plot. A minimum of 1kg fresh goji berry (minimum of 12 plants) was collected per sample. No diseased or damaged fruit was collected.

Samples were analysed according to the method described in study report number R20002A.

Table 4 Residues of difenoconazole in goji berry from supervised trials conducted in China.[1]

Trial No. Country Location Year	Form, percent	Spray Conc. (kg ai/hL)	Water Vol (L)	RTI (Days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2]	
cGAP in China	SC, 10 percent	0.010		7		5		Plot 1	Plot 2
R20002-01 China Taiyuan, Shanxi Province 2020	SC, 10 percent	0.010	Plot 1: 10.0 10.0 10.0 Plot 2: 10.0 10.0 10.0	7	3	0-	Goji Berry	1.7 (1.7, 1.7)	1.6 (1.5, 1.6)
						0		4.0 (3.9, 4.2)	3.5 (3.7, 3.3)
						1		3.4 (3.3, 3.5)	3.9 (4.0, 3.9)
						3		2.5 (2.8, 2.3)	2.1 (2.1, 2.1)
						5		<u>2.2</u> (2.4, 2.0)	2.2 (2.3, 2.0)
						7		2.1 (2.2, 2.1)	2.0 (2.1, 1.9)
						10		0.76 (0.74, 0.77)	0.83 (0.89, 0.76)
R20002-02 China Yinchuan, Ningxia Province 2020	SC, 10 percent	0.010	Plot 1: 5.9 6.7 7.3 Plot 2: 5.9 6.67	7	3	0-	Goji Berry	0.75 (0.70, 0.80)	0.51 (0.52, 0.50)

Trial No. Country Location Year	Form, percent	Spray Conc. (kg ai/hL)	Water Vol (L)	RTI (Days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2]	
			7.32						
						0		1.5 (1.5, 1.5)	1.4 (1.4, 1.4)
						1		1.4 (1.4, 1.3)	1.5 (1.4, 1.6)
						3		1.1 (1.1, 1.0)	0.92 (0.93, 0.90)
						5		0.70 (0.70, 0.69)	0.67 (0.66, 0.67)
						7		0.49 (0.51, 0.47)	0.46 (0.44, 0.48)
						10		0.31 (0.30, 0.31)	033 (0.33, 0.33)
R20002-03 China Bayannur, Inner Mongolia Province 2020	SC, 10 percent	0.010	Plot 1: 8.0 8.0 8.0 Plot 2: 8.0 8.0 8.0	7	3	0-	Goji Berry	0.37 (0.38, 0.37)	0.37 (0.37, 0.38)
						0		1.8 (1.7, 1.8)	1.8 (1.8, 1.8)
						1		0.81 (0.86, 0.75)	0.83 (0.91, 0.75)
						3		0.33 (0.29, 0.36)	0.37 (0.42, 0.31)
						5		0.016 (< 0.010, 0.023)	< 0.010 (< 0.010, < 0.010)
						7		0.24 (0.26, 0.23)	0.22 (0.22, 0.22)
						10		0.19 (0.19, 0.18)	0.18 (0.16, 0.19)
R20002-04 China Xingtai, Hebei Province 2020	SC, 10 percent	0.010	Plot 1: 8.47 8.28 8.48 Plot 2: 8.21 8.40 8.18	7	3	0-	Goji Berry	0.32 (0.31, 0.32)	0.32 (0.35, 0.30)
						0		0.59 (0.57, 0.62)	0.63 (0.65, 0.60)
						1		0.57 (0.54, 0.59)	0.64 (0.71, 0.58)
						3		0.37 (0.34, 0.40)	0.40 (0.39, 0.40)
						5		0.54 (0.53, 0.54)	0.53 (0.53, 0.53)
						7		0.59 (0.58, 0.61)	0.55 (0.55, 0.56)
						10		0.45	0.52

Trial No. Country Location Year	Form, percent	Spray Conc. (kg ai/hL)	Water Vol (L)	RTI (Days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2]	
								(0.45, 0.45)	(0.57, 0.48)

Notes:

[1] Reference: Study report Number: R20002, Weirong Wang, 2021

[2] Mean of replicate trial samples (individual values)

Pencil Yam

Four residue trials on pencil yam conducted in China are presented below. Two replicate treated plots were set in each trial site and duplicate fresh pencil yam samples were taken from a single plot.

Fresh pencil yam samples were taken from each field site. At least 500 g of samples of pencil yam roots with normal growth and no disease were collected from 12 random points in each plot. Fresh pencil yam samples were homogenized. All samples were stored in a freezer at -20 °C until analysis.

Samples were analysed according to the method described in study report number HZ2020N001A.

Table 5 Residues of difenoconazole in fresh pencil yam from supervised trials conducted in China[1]

Trial No. Country Location Year	Form (percent)	Rate (g ai/ha)	Water (L/ha)	RTI (days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2] Plot 1	Difenoconazole Residues (mg/kg)[2] Plot 2
cGAP in China	WD 10 percent	79.5	750	7	3	60			
HZ2020N001 71 China, Mile, Yunnan Province 2020	WD, 10 percent	Plot 1: 82.0 84.5 77.3 Plot 2: 81.6 85.5 78.8	Plot 1: 769 793 725 Plot 2: 765 800 738	7	3	0	Fresh pencil yam	0.028 (0.030,0.025)	0.044 (0.034,0.054)
						3		0.038 (0.040,0.036)	< 0.01 (< 0.01,< 0.01)
						7		0.017 (0.016,0.019)	0.042 (0.033,0.051)
						15		< 0.01 (< 0.01,< 0.01)	0.019 (0.019,0.019)
						30		0.018 (0.018,0.017)	0.020 (0.025,0.015)
						45		< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)
						60		< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)
						70		< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)
HZ2020N001 72 China, Zhanyi, Yunnan	WD, 10 percent	Plot 1: 78.2 80.7 81.2	Plot 1:733 756 763	7	3	0	Fresh pencil yam	0.026 (0.032,0.019)	0.13 (0.12,0.14)

Trial No. Country Location Year	Form (percent)	Rate (g ai/ha)	Water (L/ha)	RTI (days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2] Plot 1	Difenoconazole Residues (mg/kg)[2] Plot 2
Province 2020		Plot 2: 78.3 80.9 84.1	Plot 2: 733 756 788						
						3		0.12 (0.10,0.13)	0.12 (0.13,0.10)
						7		0.014 (0.012,0.017)	0.067 (0.078,0.057)
						15		0.017 (0.013,0.021)	0.024 (0.031,0.017)
						30		< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)
						45		< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)
						60		< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)
						70		< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)
HZ2020N001 74 China, Anning, Yunnan Province 2020	WD, 10 percent	Plot 1: 79.9 81.9 80.1 Plot 2: 81.9 82.3 81.7	Plot 1:749 768 751 Plot 2:767 772 766	7	3	45	Fresh pencil yam	0.012 (< 0.01,0.013);	0.014 (0.011,0.017)
						60		< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)
						70		0.01 (< 0.01, 0.01)	< 0.01 (< 0.01,< 0.01)
HZ2020N001 75 China, Shilin, Yunnan Province 2020	WD, 10 percent	Plot 1: 79.3 83.1 78.7 Plot 2: 80.0 82.9 80.1	Plot 1: 744 779 738 Plot 2: 750 778 750	7	3	45	Fresh pencil yam	< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)
						60		< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)
						70		< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)

Notes:

[1] Reference: Study report Number: HZ2020N001, Wenxi Li, 2021.

[2] Mean of replicate trial samples (individual values).

Ginger

Eight residue trials on ginger conducted in China are presented below. Three replicate treated plots were set at each trial site and duplicate fresh ginger root were taken from each plot. Ginger samples were randomly collected from at least 12 sites in one plot for each sample, and mature ginger samples with normal growth and no disease were collected with a sampling amount of no less than 10 kg.

Samples were analysed according to the method described in study report number AR-2020PR20.

Table 6 Difenoconazole residues in ginger from supervised trials in China[1]

Trial No. Country Location year	Form (g/L)	Rate (g ai/ha)	Water (L/ha)	RTI (days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2] Plot 1	Difenoconazole Residues (mg/kg)[2] Plot 2	Difenoconazole Residues (mg/kg)[2] Plot 3
cGAP in China		113		7	3	14				
PR-2020PR2001 China Xingtai, Hebei Province 2020	SC 125	Plot1: 113 113 113 Plot2: 113 115 115 Plot3: 111 111, 110	Plot1 747 748 747 Plot2: 754 769 768 Plot3: 743 744 732	7	3	7	Fresh Ginger	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						14		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						21		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
PR-2020PR2002 China Jinan, Shandong Province 2020	SC 125	Plot1: 113 110, 113 Plot2: 109 113 110 Plot3: 111 110 110	Plot1: 602 594 601 Plot2: 579 602 605 Plot3 591 589 589	7	3	0	Fresh Ginger	0.014 (0.014, 0.014)	0.027 (0.027, 0.027)	0.025 (0.025, 0.025)
						3		0.072 (0.071, 0.072)	0.084 (0.083, 0.084)	0.072 (0.072, 0.071)
						7		0.064 (0.063, 0.064)	0.060 (0.059, 0.060)	0.059 (0.059, 0.059)
						14		0.062 (0.062, 0.061)	0.040 (0.039, 0.040)	0.052 (0.052, 0.051)
						21		0.048	0.032	0.026

Trial No. Country Location year	Form (g/L)	Rate (g ai/ha)	Water (L/ha)	RTI (days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2] Plot 1	Difenoconazole Residues (mg/kg)[2] Plot 2	Difenoconazole Residues (mg/kg)[2] Plot 3
								(0.048, 0.049)	(0.032, 0.033)	(0.023, 0.028)
						28		0.036 (0.038, 0.035)	0.015 (0.015, 0.015)	0.020 (0.019, 0.020)
PR-2020PR2003 China Weifang, Shandong Province 2020	SC 125	Plot1: 114 113 111 Plot2: 113 110 114 Plot3: 114 111 113	Plot1: 605 599 592 Plot2: 597 588 603 Plot3: 607 591 599	7	3	7	Fresh Ginger	0.011 (0.011, 0.011)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						14		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						21		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
PR-2020PR2004 China Xinxiang, Henan Province 2020	SC 125	Plot1: 113 113 113 Plot2: 113 113 113 Plot3: 113 113 113	Plot1: 751 750 749 Plot2: 754 750 751 Plot3: 749 752 752	7	3	7	Fresh Ginger	0.034 (0.034, 0.034)	0.042 (0.042, 0.041)	0.034 (0.034, 0.033)
						14		0.033 (0.034, 0.032)	0.030 (0.034, 0.027)	0.032 (0.033, 0.032)
						21		0.022 (0.023, 0.022)	0.020 (0.022, 0.019)	0.022 (0.023, 0.022)
PR-2020PR2005 China Fuyang, Anhui Province 2020	SC 125	Plot1: 116 114 120 Plot2: 115 116 116 Plot3: 120	Plot1: 620 605 640 Plot2: 615 620 620 Plot3: 640	7	3	0	Fresh Ginger	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)

Trial No. Country Location year	Form (g/L)	Rate (g ai/ha)	Water (L/ha)	RTI (days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2] Plot 1	Difenoconazole Residues (mg/kg)[2] Plot 2	Difenoconazole Residues (mg/kg)[2] Plot 3
		110 118	590 625							
						3		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						7		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						14		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						21		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						28		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
PR-2020PR2006 China Changsha, Hunan Province 2020	SC 125	Plot1: 106 114 114 Plot2: 113 114 113 Plot3: 113 113 113	Plot1: 655 699 698 Plot2: 687 696 691 Plot3: 691 691 692	7	3	0	Fresh Ginger	0.048 (0.049, 0.048)	0.049 (0.049, 0.049)	0.052 (0.053, 0.052)
						3		0.13 (0.13, 0.13)	0.14 (0.14, 0.14)	0.14 (0.14, 0.15)
						7		0.15 (0.15, 0.15)	0.16 (0.16, 0.15)	0.1 (0.15, 0.15)
						14		0.035 (0.034, 0.036)	0.037 (0.037, 0.037)	0.038 (0.037, 0.038)
						21		0.017 (0.016, 0.018)	0.018 (0.017, 0.018)	0.018 (0.018, 0.019)
						28		< 0.01 (< 0.01, < 0.01)	< 0.01(< 0.01, < 0.01)	< 0.01(< 0.01, < 0.01)
PR-2020PR2007 China Deyang, Sichuan Province 2020	SC 125	Plot1: 114 113 115 Plot2: 113 113 118 Plot3: 114 113 104	Plot1: 604 597 612 Plot2: 600 598 623 Plot3 607 598 555	7	3	0	Fresh Ginger	0.10 (0.10, 0.10)	0.061 (0.061, 0.061)	0.14 (0.13, 0.14)
						3		0.24 (0.24, 0.24)	0.25 (0.25, 0.25)	0.17 (0.17, 0.17)
						7		0.086 (0.085, 0.086)	0.19 (0.19, 0.19)	0.13 (0.13, 0.13)
						14		0.068	0.059	0.10

Trial No. Country Location year	Form (g/L)	Rate (g ai/ha)	Water (L/ha)	RTI (days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2] Plot 1	Difenoconazole Residues (mg/kg)[2] Plot 2	Difenoconazole Residues (mg/kg)[2] Plot 3
								(0.067, 0.068)	(0.059, 0.059)	(0.10, 0.10)
						21		0.026 (0.026, 0.026)	0.037 (0.037, 0.037)	0.019 (0.019, 0.019)
						28		0.026 (0.027, 0.026)	0.038 (0.038, 0.038)	0.020 (0.020, 0.020)
PR-2020PR20 08 China Nanning Guangxi Zhuang Autonomous Region 2020	SC 125	Plot1: 116 113 115 Plot2: 110 115 114 Plot3: 111 110 109	Plot1: 617 606 614 Plot2: 589 612 607 Plot3: 591 589 579	7	3	7	Fresh Ginger	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						14		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						21		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)

Notes:

[1] Reference: Study report Number: PR-2020PR20, Yizhi Feng, 2020.

[2] Mean of replicate trial samples (individual values).

Tea

Eight residue trials on tea conducted in China are presented below. Two replicate treated plots were set in each trial site and duplicate fresh tea leaves samples were taken from a single plot

Fresh tea leaves sample were taken from each field site. A minimum of 1kg fresh tea leaves were collected from at least 12 separate points of the plot.

Samples were analysed according to the method described in study report number RM20004G20001.

Table 7 Residues of difenoconazole in fresh tea leaves from supervised trials conducted in China.[1]

Trial No. Country Location Year	Form (percent)	Rate[2] (g ai/hL)	Water (L/ha)	RTI (days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[3] Plot 1	Difenoconazole Residues (mg/kg)[3] Plot 2
GAP in China	WD 10 percent	75		7	3	14			
RA20004G20001-01 China	WD 10	Plot 1: 75.5	Plot 1: 755	7	3	0	Fresh tea leaves	12 (10, 13)	12 (12, 12)

Trial No. Country Location Year	Form (percent)	Rate[2] (g ai/hL)	Water (L/ha)	RTI (days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[3] Plot 1	Difenoconazole Residues (mg/kg)[3] Plot 2
Hangzhou, Zhejiang Province 2020	percent	75.2 75.4 Plot 2: 75.0 75.5 75.4	752 754 Plot 2: 750 755 754						
						1		10 (9.0, 12)	10 (9.9, 11)
						3		4.1 (3.9, 4.3)	4.3 (4.4, 4.2)
						5		0.47 (0.50, 0.44)	0.48 (0.48, 0.49)
						7		0.22 (0.22, 0.22)	0.17 (0.17, 0.17)
						14		< 0.010 (< 0.010, < 0.010)	< 0.010 (< 0.010, < 0.010)
						21		< 0.010 (< 0.010, < 0.010)	< 0.010 (< 0.010, < 0.010)
RA20004G20001-02 China Jiujiang, Jiangxi Province 2020	WD 10 percent	Plot 1: 81.5 87.5 77.5 Plot 2: 83.5 88.5 79.5	Plot 1: 815 875 775 Plot 2: 835 885 795	7	3	14	Fresh tea leaves	1.6 (1.4, 1.8)	1.4 (1.4, 1.5)
						21		1.2 (1.2, 1.3)	1.4 (1.2, 1.6)
RA20004G20001-03 China Changsha, Hunan Province 2020	WD 10 percent	Plot 1: 75.3 75.4 73.3 Plot 2: 74.9 76.1 73.4	Plot 1: 753 754 733 Plot 2: 749 761 733	7	3	0	Fresh tea leaves	9.3 (8.8, 9.9)	9.4 (9.6, 9.3)
						1		6.4 (5.5, 7.3)	6.1 (5.9, 6.2)
						3		4.2 (3.9, 4.6)	3.7 (3.8, 3.6)
						5		0.45 (0.39, 0.51)	0.39 (0.35, 0.44)
						7		0.35 (0.41, 0.29)	0.31 (0.31, 0.31)
						14		0.12 (0.11,0.13)	0.099 (0.092, 0.11)
						21		0.017 (0.016,0.018)	0.019 (0.019, 0.019)
RA20004G20001-04 China Chengdu, Sichuan Province 2020	WD 10 percent	Plot 1: 73.8 73.8 74.5 Plot 2: 74.8 75.5 74.1	Plot 1: 738 738 745 Plot 2: 748 755 741	7	3	14	Fresh tea leaves	0.038 (0.037, 0.040)	0.036 (0.040, 0.033)
						21		0.017 (0.017, 0.017)	0.021 (0.021, 0.022)
RA20004G20001-05 China Anshun, Guizhou Province	WD 10 percent	Plot 1: 74.6 75.8 73.9	Plot 1: 746 758 736	7	3	14	Fresh tea leaves	0.47 (0.46, 0.47)	0.41 (0.27, 0.55)

Trial No. Country Location Year	Form (percent)	Rate[2] (g ai/hL)	Water (L/ha)	RTI (days)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[3] Plot 1	Difenoconazole Residues (mg/kg)[3] Plot 2
2020		Plot 2: 74.4 74.8 74.6	Plot 2: 744 748 746						
						21		0.36 (0.42, 0.30)	0.36 (0.32, 0.40)
RA20004G20001-06 China Kunming, Yunnan Province 2020	WD 10 percent	Plot 1: 75.1 75.3 75.0 Plot 2: 75.5 74.7 75.9	Plot 1: 751 753 750 Plot 2: 755 747 759	7	3	0	Fresh tea leaves	10 (11, 8.6)	12 (9.8, 14)
						1		5.0 (4.9, 5.1)	4.5 (4.4, 4.7)
						3		1.7 (1.8, 1.7)	1.8 (1.7, 1.8)
						5		1.2 (1.2, 1.2)	1.2 (1.1, 1.3)
						7		0.66 (0.57, 0.74)	0.68 (0.72, 0.64)
						14		0.094 (0.083, 0.11)	0.049 (0.049, 0.050)
						21		< 0.010 (< 0.010, < 0.010)	< 0.010 (< 0.010, < 0.010)
RA20004G20001-07 China Fuan, Fujian Province 2020	WD 10 percent	Plot 1: 74.4 75.0 74.0 Plot 2: 74.3 74.2 75.5	Plot 1: 744 750 740 Plot 2: 743 742 755	7	3	0	Fresh tea leaves	8.0 (7.5, 8.6)	7.5 (6.5, 8.4)
						1		3.7 (3.2, 4.1)	3.7 (3.7, 3.7)
						3		2.3 (2.3, 2.4)	2.3 (2.1, 2.4)
						5		0.99 (0.98, 1.0)	1.0 (0.95, 1.1)
						7		0.40 (0.38, 0.42)	0.48 (0.43, 0.53)
						14		0.031 (0.031, 0.031)	0.084 (0.084, 0.084)
						21		0.013 (< 0.010, 0.019)	0.026 (0.022, 0.031)
RA20004G20001-08 China Qiongzhong, Hainan Province 2020	WD 10 percent	Plot 1: 73.4 72.3 73.5 Plot 2: 72.7 72.6 75.8	Plot 1: 734 723 735 Plot 2: 727 726 758	7	3	14	Fresh tea leaves	0.026 (0.016, 0.036)	0.037 (0.043, 0.030)
						21		< 0.010 (< 0.010, < 0.010)	< 0.010 (< 0.010, < 0.010)

Notes:

[1] Reference: Study report Number: RA20004G20001, Yanjie Li, 2022.

[2] Spray concentration in all trials was reported as 0.01 kg ai/hL.

[3] Mean of replicate trial samples (individual values).

NS = Not specified.

FATE OF RESIDUES IN PROCESSING

The Meeting received new information on the fate of difenoconazole residues during the processing in goji berry, pencil yam, ginger, and tea.

If residues in the RAC were below the LOQ, no processing factor could be derived. In case of residues below the LOQ, but above the LOD in the processed product, the numeric value of the LOQ was used for the calculation. If residues in the processed product were below the LOD, the numeric value of the LOQ was used for the calculation but the PF was expressed as "less than" (e.g. < 0.5).

Dried goji berry

Goji berry samples were processed by two drying methods (sun drying and hot-air drying) with the fresh goji berry samples collected 3 days and 5 days after the last application in all trials. The processing simulated industrial practice as closely as possible. The water content of dried Goji berry was less than 13 percent. Two kinds of processing procedures are presented as follows:

Sun drying: Fresh Goji berry were impregnated with 2 percent food-grade sodium carbonate solution for 5–10 seconds to break the epidermal waxy layer of fruits, and then spread on fruit stacks and dried naturally outside for 5-11 days.

Hot-air drying: Fresh Goji berry were impregnated with 2 percent food-grade sodium carbonate solution for 5–10 seconds to break the epidermal waxy layer of fruits, and then spread on fruit stacks and baked at 50 °C in an electric blast drying oven for 2–3 days.

Samples were analysed according to the method described in study report number R20002A.

Table 8 Residues of difenoconazole in dried goji berry from supervised trials conducted in China[1]

Trial No. Location Year	Form (percent)	Spray Conc. (kg ai/hL)	No.	Crop/ Variety	PHI (Days)	Method	Difenoconazole Residues RAC (mg/kg)	Difenoconazole Residues PF (mg/kg)	Processing Factor
R20002-01 Taiyuan, Shanxi Province 2020	SC 10 percent	0.010	3	Goji Berry/ Ningqi 7	3	Sun drying	2.3 (2.5, 2.1)	3.9 (3.8, 4.0)	1.7
						Hot-air drying	2.3 (2.5, 2.1)	4.6 (4.5,4.6)	2.0
					5	Sun drying	2.2 (2.2, 2.2)	3.4 (3.4, 3.4)	1.5
						Hot-air drying	2.2 (2.2, 2.2)	4.0 (4.0, 4.0)	1.8
R20002-02 Yinchuan, Ningxia Province 2020	SC 10 percent	0.010	3	Goji Berry/ Ningqi 7	3	Sun drying	1.0 (1.1, 0.92)	1.6 (1.6, 1.7)	1.6
						Hot-air drying	1.0 (1.1, 0.92)	1.3 (1.2, 1.4)	1.3
					5	Sun drying	0.68 (0.70, 0.67)	1.3 (1.3, 1.3)	1.9

Trial No. Location Year	Form (percent)	Spray Conc. (kg ai/hL)	No.	Crop/ Variety	PHI (Days)	Method	Difenoconazole Residues RAC (mg/kg)	Difenoconazole Residues PF (mg/kg)	Processing Factor
						Hot-air drying	0.68 (0.70, 0.67)	1.8 (1.7, 1.8)	2.6
R20002-03 Bayannur, Inner Mongolia Province 2020	SC 10 percent	0.010	3	Goji Berry/ Ningqi 25	3	Sun drying	0.35 (0.33, 0.37)	0.88 (0.87, 0.89)	2.5
						Hot-air drying	0.35 (0.33, 0.37)	0.88 (0.93, 0.83)	2.5
					5	Sun drying	0.013 (0.016, < 0.010)	0.37 (0.36, 0.38)	28
						Hot-air drying	0.013 (0.016, < 0.010)	0.34 (0.32, 0.35)	26
R20002-04 Xingtai, Hebei Province 2020	S, 10 percent	0.010	3	Goji Berry/ Hanqi	3	Sun drying	0.38 (0.37, 0.40)	1.6 (1.7, 1.6)	4.2
						Hot-air drying	0.38 (0.37, 0.40)	4.2 (4.5, 4.0)	11
					5	Sun drying	0.54 (0.54, 0.53)	1.7 (1.9, 1.5)	3.1
						Hot-air drying	0.54 (0.54, 0.53)	2.7 (2.4, 3.0)	5.0

Notes:

[1] Reference: Study report Number: R20002, Weirong Wang, 2021.

Dried pencil Yam

Fresh pencil yam samples were taken from each field site described above for the RAC and a portion was processed into dried pencil yam using simulated commercial practices: Yam tap roots were washed in cool water and dried, lateral roots were removed by scissors, the tap root was then cut into thin slices and baked under infrared light (50–60 °C, 2–3 days) until the moisture content was <13 percent.

Samples were analysed according to the method described in study report number HZ2020N001A.

Table 9 Residues of difenoconazole in dried pencil yam from supervised trials conducted in China[1]

Trial No. Country Location Year	Form (percent)	Rate (g ai/ha)	Water (L/ha)	No.	PHI (Days)	Sample	Difenoconazole Residues RAC (mg/kg)	Difenoconazole Residues PF (mg/kg)[2] Plot 1	Difenoconazole Residues PF (mg/kg)[2] Plot 2	Processing Factors
cGAP in China	WD 10 percent	79.5	750	3	60	Dried pencil yam				
HZ2020N001 71 China,	2020 WD 10 percent	77.3 Plot 2: 81.6 85.5	Plot 1: 769 793 725	800 738 3	45	Dried pencil yam	< 0.01 (< 0.01, < 0.01)	0.040 (0.041,0.039)	0.021 (0.023,0.019)	3.1

Trial No. Country Location Year	Form (percent)	Rate (g ai/ha)	Water (L/ha)	No.	PHI (Days)	Sample	Difenoconazole Residues RAC (mg/kg)	Difenoconazole Residues PF $(mg/kg)^2$ Plot 1	Difenoconazole Residues PF $(mg/kg)^2$ Plot 2	Processing Factors
Mile, Yunnan Province	Plot 1: 82.0 84.5	78.8	Plot 2: 765							
					60		< 0.01 (< 0.01, < 0.01)	0.017 (0.014,0.020)	0.036 (0.041,0.031)	2.7
					70		< 0.01 (< 0.01, < 0.01)	0.017 (0.017,0.018)	0.040 (0.036,0.044)	2.9
HZ2020N00172 China, Zhanyi, Yunnan Province	2020 WD 10 percent Plot 1: 78.2 80.7	81.2 Plot 2: 78.3 80.9 84.1	Plot 1:733 756 763 Plot 2: 733	756 788 3	45	Dried pencil yam	< 0.01 (< 0.01, < 0.01)	0.025 (0.028,0.022)	0.034 (0.035,0.033)	3.0
					60		< 0.01 (< 0.01, < 0.01)	0.019 (0.019,0.018)	0.018 (0.017,0.019)	1.9
					70		< 0.01 (< 0.01, < 0.01)	0.015 (0.014,0.015)	0.020 (0.023,0.016)	1.8
HZ2020N00174 China, Anning, Yunnan Province	2020 WD 10 percent Plot 1: 79.9 81.9	80.1 Plot 2: 81.9 82.3 81.7	Plot 1:749 768 751 Plot 2:767	772 766 3	45	Dried pencil yam	0.013 (0.012, 0.014)	0.024 (0.037,0.031)	0.035 (0.030,0.040)	2.3
					60		< 0.01 (< 0.01, < 0.01)	0.037 (0.039,0.036)	0.026 (0.036,0.016)	3.2
					70		0.01 (0.01, < 0.01)	0.031 (0.035,0.027)	0.036 (0.038,0.034)	3.4
HZ2020N00175 China,Shilin, Yunnan Province 2020	WD 10 percent Plot 1: 79.3 83.1 78.7	Plot 2: 80.0 82.9 80.1 Plot 1: 744	779 738 Plot 2: 750	778 750 3	45	Dried pencil yam	< 0.01 (< 0.01, < 0.01)	0.020 (0.021,0.019)	0.031 (0.031,0.032)	2.6
					60		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01,< 0.01)	0.022 (0.019,0.026)	1.6
					70		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01,< 0.01)	< 0.01 (< 0.01,< 0.01)	1.0

Notes:

[1] Reference: Study report Number: HZ2020N001, Wenxi Li, 2021.

[2] Mean of replicate trial samples (individual values).

Dried ginger

Fresh ginger samples were taken from each field site described above for the RAC and a portion was processed into dried ginger using hot-air and freeze-drying processes that simulate typical commercial processes. For both processes, samples of fresh ginger were washed under cold water to remove skin impurities, cut into small (approximately 1 mm) sections, and the sections quartered. For the hot-air process, the quarters were dried in a drying oven (90 °C, 12 hours). For the freeze-drying process, samples were placed in a vacuum freeze dryer for 24 hours. After drying samples were brought to room temperature and crushed with a grinder.

Samples were analysed according to the method described in study report number AR-2020PR20.

Table 10 Difenoconazole residues in dried ginger from supervised trials in China [1]

Trial No. Country Location year	Form (g/L)	Rate (g ai/ha)	Water (L/ha)	No.	PHI (Days)	Sample	Difenoconazole Residues RAC (mg/kg)	Difenoconazole Residues PF (mg/kg)^2 Plot 1	Difenoconazole Residues PF (mg/kg)^2 Plot 2	Difenoconazole Residues PF (mg/kg)^2 Plot 3	Processing Factor
cGAP in China		112.5		3	14						
PR-2020PR2001 China Xingtai, Hebei Province 2020	SC 125	Plot1: 113 113 113 Plot2: 113 115 115 Plot3: 111 111, 110	Plot1 747 748 747 Plot2: 754 769 768 Plot3: 743 744 732	3	7	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.056 (0.056, 0.057)	0.055 (0.054, 0.056)	0.056 (0.056, 0.056)	5.6
					7	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.037 (0.036, 0.038)	0.038 (0.038, 0.038)	0.036 (0.035, 0.037	3.7
					14	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.036 (0.036, 0.036)	0.034 (0.032, 0.035)	0.036 (0.037, 0.036)	3.5
					14	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.015 (0.015, 0.015)	0.014 (0.013, 0.016)	0.014 (0.013, 0.015)	1.4
					21	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.032 (0.032, 0.031)	0.031 (0.032, 0.030)	0.032 (0.032, 0.031)	3.2
					21	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.0
PR-2020PR2002 China Jinan, Shandong Province 2020	SC 125	Plot1: 113 110, 113 Plot2: 109 113 110 Plot3: 111 110 110	Plot1: 602 594 601 Plot2: 579 602 605 Plot3 591 589 589	3	7	Hot Dried	0.061 (0.064, 0.060, 0.059)	0.45 (0.45, 0.45)	0.46 (0.46, 0.46)	0.38 (0.37, 0.38)	7.0
					7	Freeze Dried	0.061 (0.064, 0.060, 0.059)	0.44 (0.44, 0.45)	0.46 (0.46, 0.45)	0.46 (0.45, 0.46)	7.4
					14	Hot Dried	0.051 (0.062, 0.040, 0.052)	0.36 (0.36, 0.36)	0.37 (0.37, 0.37)	0.50 (0.49, 0.50)	8.0
					14	Freeze Dried	0.051 (0.062, 0.040, 0.052)	0.34 (0.34, 0.35)	0.36 (0.36, 0.36)	0.35 (0.35, 0.35)	6.9
					21	Hot	0.035	0.26	0.27	0.27	7.6

Trial No. Country Location year	Form (g/L)	Rate (g ai/ha)	Water (L/ha)	No.	PHI (Days)	Sample	Difenoconazole Residues RAC (mg/kg)	Difenoconazole Residues PF (mg/kg)^2 Plot 1	Difenoconazole Residues PF (mg/kg)^2 Plot 2	Difenoconazole Residues PF (mg/kg)^2 Plot 3	Processing Factor
						Dried	(0.048, 0.032, 0.026)	(0.26, 0.25)	(0.28, 0.26)	(0.27, 0.27)	
					21	Freeze Dried	0.035 (0.048, 0.032, 0.026)	0.24 (0.25, 0.24)	0.24 (0.25, 0.23)	0.26 (0.26, 0.26)	7.0
PR-2020PR2003 China Weifang, Shandong Province 2020	SC 125	Plot1: 114 113 111 Plot2: 113 110 114 Plot3: 114 111 113	Plot1: 605 599 592 Plot2: 597 588 603 Plot3: 607 591 599	3	7	Hot Dried	0.01 (0.011, < 0.01, < 0.01)	0.042 (0.043, 0.042)	0.041 (0.041, 0.041)	0.042 (0.041, 0.042)	4.2
					7	Freeze Dried	0.01 (0.011, < 0.01, < 0.01)	0.044 (0.046, 0.043)	0.045 (0.045, 0.045)	0.044 (0.045, 0.044)	4.4
					14	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.024 (0.024, 0.024)	0.024 (0.025, 0.024)	0.024 (0.024, 0.023)	2.4
					14	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.024 (0.024, 0.023)	0.021 (0.022, 0.020)	0.024 (0.025, 0.024)	2.3
					21	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.017 (0.017, 0.017)	0.017 (0.017, 0.017)	0.016 (0.017, 0.016)	1.7
					21	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.0
PR-2020PR2004 China Xinxiang, Henan Province 2020	SC 125	Plot1: 113 113 113 Plot2: 113 113 113 Plot3: 113 113 113	Plot1: 751 750 749 Plot2: 754 750 751 Plot3: 749 752 752	3	7	Hot Dried	0.037 (0.034, 0.042, 0.034)	0.18 (0.18, 0.19)	0.20 (0.20, 0.19)	0.19 (0.19, 0.19)	5.1
					7	Freeze Dried	0.037 (0.034, 0.042, 0.034)	0.16 (0.16, 0.17)	0.16 (0.16, 0.16)	0.16 (0.16, 0.17)	4.3
					14	Hot Dried	0.032 (0.033, 0.030, 0.032)	0.16 (0.16, 0.16)	0.17 (0.17, 0.17)	0.16 (0.16, 0.16)	5.1
					14	Freeze Dried	0.032 (0.033, 0.030, 0.032)	0.11 (0.13, 0.094)	0.14 (0.14, 0.15)	0.14 (0.14, 0.14)	4.1

Trial No. Country Location year	Form (g/L)	Rate (g ai/ha)	Water (L/ha)	No.	PHI (Days)	Sample	Difenoconazole Residues RAC (mg/kg)	Difenoconazole Residues PF (mg/kg)^2 Plot 1	Difenoconazole Residues PF (mg/kg)^2 Plot 2	Difenoconazole Residues PF (mg/kg)^2 Plot 3	Processing Factor
					21	Hot Dried	0.021 (0.022, 0.020, 0.022)	0.12 (0.12, 0.12)	0.12 (0.12, 0.12)	0.12 (0.12, 0.12)	5.7
					21	Freeze Dried	0.021 (0.022, 0.020, 0.022)	0.11 (0.096, 0.13)	0.098 (0.098, 0.097)	0.094 (0.097, 0.092)	4.8
PR-2020PR2005 China Fuyang, Anhui Province 2020	SC 125	Plot1: 116 114 120 Plot2: 115 116 116 Plot3: 120 110 118	Plot1: 620 605 640 Plot2: 615 620 620 Plot3: 640 590 625	3	7	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.054 (0.055, 0.054)	0.054 (0.053, 0.054)	0.054 (0.0543, 0.055)	5.4
					7	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.036 (0.036, 0.037)	0.036 (0.036, 0.037)	0.035 (0.034, 0.036)	3.6
					14	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.020 (0.020, 0.020)	0.019 (0.019, 0.019)	0.019 (0.019, 0.019)	1.9
					14	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.018 (0.018, 0.018)	0.016 (0.016, 0.016)	0.020 (0.020, 0.020)	1.8
					21	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.0
					21	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.0
PR-2020PR2006 China Changsha, Hunan Province 2020	SC 125	Plot1: 106 114 114 Plot2: 113 114 113 Plot3: 113 113 113	Plot1: 655 699 698 Plot2: 687 696 691 Plot3: 691 691 692	3	7	Hot Dried	0.14 (0.15, 0.16, 0.10)	0.49 (0.49, 0.49)	0.48 (0.48, 0.47)	0.47 (0.47, 0.47)	3.4
					7	Freeze Dried	0.14 (0.15, 0.16, 0.10)	0.48 (0.47, 0.48)	0.42 (0.41, 0.42)	0.46 (0.45, 0.46	3.2
					14	Hot Dried	0.037 (0.035, 0.037, 0.038)	0.26 (0.26, 0.26)	0.27 (0.27, 0.27)	0.30 (0.31, 0.30)	7.5
					14	Freeze Dried	0.037 (0.035, 0.037, 0.038)	0.28 (0.28, 0.27)	0.27 (0.27, 0.27)	0.35 (0.35, 0.35)	8.1

Trial No. Country Location year	Form (g/L)	Rate (g ai/ha)	Water (L/ha)	No.	PHI (Days)	Sample	Difenoconazole Residues RAC (mg/kg)	Difenoconazole Residues PF $(mg/kg)^2$ Plot 1	Difenoconazole Residues PF $(mg/kg)^2$ Plot 2	Difenoconazole Residues PF $(mg/kg)^2$ Plot 3	Processing Factor
					21	Hot Dried	0.018 (0.017, 0.018, 0.018)	0.12 (0.12, 0.12)	0.13 (0.13, 0.13)	0.11 (0.11, 0.11)	6.7
					21	Freeze Dried	0.018 (0.017, 0.018, 0.018)	0.16 (0.15, 0.16)	0.086 (0.086, 0.085)	0.16 (0.16, 0.16)	7.5
PR-2020PR2007 China Deyang, Sichuan Province 2020	SC 125	Plot1: 114 113 115 Plot2: 113 113 118 Plot3: 114 113 104	Plot1: 604 597 612 Plot2: 600 598 623 Plot3 607 598 555	3	7	Hot Dried	0.14 (0.086, 0.19, 0.13)	0.36 (0.36, 0.36)	0.46 (0.47, 0.46)	0.52 (0.52, 0.51)	3.2
					7	Freeze Dried	0.14 (0.086, 0.19, 0.13)	0.44 (0.45, 0.43)	0.48 (0.47, 0.48)	0.46 (0.46, 0.46)	3.3
					14	Hot Dried	0.076 (0.068, 0.059, 0.10)	0.34 (0.34, 0.34)	0.36 (0.37, 0.36)	0.35 (0.35, 0.35)	4.6
					14	Freeze Dried	0.076 (0.068, 0.059, 0.10)	0.27 (0.27, 0.27)	0.30 (0.30, 0.29)	0.38 (0.38, 0.37)	4.2
					21	Hot Dried	0.027 (0.026, 0.037, 0.019)	0.21 (0.21, 0.21)	0.21 (0.21, 0.21)	0.21 (0.21, 0.21)	7.8
					21	Freeze Dried	0.027 (0.026, 0.037, 0.019)	0.093 (0.091, 0.095)	0.16 (0.16, 0.17)	0.16 (0.16, 0.17)	5.1
PR-2020PR2008 China Nanning Guangxi Zhuang Autonomous Region 2020	SC 125	Plot1: 116 113 115 Plot2: 110 115 114 Plot3: 111 110 109	Plot1: 617 606 614 Plot2: 589 612 607 Plot3: 591 589 579	3	7	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.040 (0.041, 0.038)	0.022 (0.012, 0.033)	0.044 (0.044, 0.045)	3.5
					7	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.028 (0.025, 0.031)	0.022 (0.026, 0.019)	0.029 (0.029, 0.029)	2.6
					14	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.028 (0.027, 0.029)	0.018 (0.019, 0.018)	0.023 (0.022, 0.024)	2.3
					14	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.020 (0.020, 0.019)	0.016 (0.015, 0.016)	0.016 (0.016, 0.016)	1.7

Trial No. Country Location year	Form (g/L)	Rate (g ai/ha)	Water (L/ha)	No.	PHI (Days)	Sample	Difenoconazole Residues RAC (mg/kg)	Difenoconazole Residues PF (mg/kg)[2] Plot 1	Difenoconazole Residues PF (mg/kg)[2] Plot 2	Difenoconazole Residues PF (mg/kg)[2] Plot 3	Processing Factor
					21	Hot Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	0.014 (0.013, 0.014)	0.010 (0.010, 0.011)	< 0.01 (< 0.01, < 0.01)	1.1
					21	Freeze Dried	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.0

Notes:

[1] Reference: Study report Number: PR-2020PR20, Yizhi Feng, 2020.

[2] Mean of replicate trial samples (individual values).

Tea

Fresh tea leaves sample were taken from each field site and a portion of which were processed into green tea and black tea with simulation of industrial practices as closely as possible. The processing procedure is presented as follows:

Green tea: fresh tea leaves were heated to inactivate the enzymes at 220 °C for 5 minutes (fixing), rolled at 25 °C for 30 minutess (rolling), and dried at 120 °C for 60 minutess (drying).

Black tea: fresh tea leaves were withered for 6 hours (withering), rolled at 25 °C for 30 minutes (rolling), fermented at 38 °C for 6 to 8 hours (fermentation), and dried at 120 °C for 60 minutes (drying).

Samples were analysed according to the method described in study report number RM20004G20001.

Table 11 Residues of difenoconazole in processed tea leaves from supervised trials conducted in China [1]

Trial No. Country Location Year	Form (percent)	Rate (g ai/ha)	Water (L/ha)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2] Plot 1	Difenoconazole Residues (mg/kg)[2] Plot 2
GAP in China	WD 10 percent	75		3	14			
RA20004G20001-01 China Hangzhou, Zhejiang Province 2020	WD 10 percent	Plot 1: 75.5 75.2 75.4 Plot 2: 75.0 75.5 75.4	Plot 1: 755 752 754 Plot 2: 750 755 754	3	14	Green Tea, Dried	0.019 (0.018, 0.020)	0.017 (0.019, 0.016)
					14	Black Tea, Dried	0.022 (0.023, 0.022)	0.023 (0.024,0.022)
					21	Green Tea, Dried	< 0.010 (< 0.010, < 0.010)	< 0.010 (< 0.010, < 0.010)
					21	Black Tea, Dried	< 0.010 (< 0.010, < 0.010)	< 0.010 (< 0.010, < 0.010)
RA20004G20001-02 China	WD 10	Plot 1: 81.5	Plot 1: 815	3	14	Green Tea, Dried	3.8 (3.9, 3.7)	4.2 (4.3, 4.1)

Trial No. Country Location Year	Form (percent)	Rate (g ai/ha)	Water (L/ha)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2] Plot 1	Difenoconazole Residues (mg/kg)[2] Plot 2
Jiujiang, Jiangxi Province 2020	percent	87.5 77.5 Plot 2: 83.5 88.5 79.5	875 775 Plot 2: 835 885 795					
					14	Black Tea, Dried	4.5 (4.7, 4.3)	2.2 (2.1, 2.3)
					21	Green Tea, Dried	2.9 (2.7, 3.1)	3.4 (4.0, 2.7)
					21	Black Tea, Dried	0.99 (1.1, 0.88)	0.89 (0.68, 1.1)
RA20004G20001-03 China Changsha, Hunan Province 2020	WD 10 percent	Plot 1: 75.3 75.4 73.3 Plot 2: 74.9 76.1 73.4	Plot 1: 753 754 733 Plot 2: 749 761 733	3	14	Green Tea, Dried	0.62 (0.65, 0.58)	0.27 (0.25, 0.28)
					14	Black Tea, Dried	0.26 (0.22, 0.25)	0.20 (0.19, 0.21)
					21	Green Tea, Dried	0.070 (0.096, 0.043)	0.053 (0.064, 0.041)
					21	Black Tea, Dried	0.077 (0.068, 0.085)	0.067 (0.048, 0.086)
RA20004G20001-04 China Chengdu, Sichuan Province 2020	WD 10 percent	Plot 1: 73.8 73.8 74.5 Plot 2: 74.8 75.5 74.1	Plot 1: 738 738 745 Plot 2: 748 755 741	3	14	Green Tea, Dried	0.20 (0.21, 0.20)	0.18 (0.18, 0.18)
					14	Black Tea, Dried	0.22 (0.24, 0.20)	0.17 (0.17, 0.18)
					21	Green Tea, Dried	0.011 (0.013, < 0.010)	0.059 (0.073, 0.045)
					21	Black Tea, Dried	0.011 (0.011, 0.012)	0.064 (0.082, 0.047)
RA20004G20001-05 China Anshun, Guizhou Province 2020	WD 10 percent	Plot 1: 74.6 75.8 73.9 Plot 2: 74.4 74.8 74.6	Plot 1: 746 758 736 Plot 2: 744 748 746	3	14	Green Tea, Dried	2.4 (2.2, 2.6)	2.3 (2.1, 2.4)
					14	Black Tea, Dried	1.7 (1.7, 1.7)	1.6 (1.7, 1.4)
					21	Green Tea, Dried	1.0 (0.98, 1.1)	0.92 (0.79, 1.1)
					21	Black Tea, Dried	1.4 (1.5, 1.3)	1.2 (1.1, 1.3)
RA20004G20001-06 China Kunming, Yunnan Province 2020	WD 10 percent	Plot 1: 75.1 75.3 75.0 Plot 2:	Plot 1: 751 753 750 Plot 2:	3	14	Green Tea, Dried	0.60 (0.47, 0.73)	0.77 (0.76, 0.77)

Trial No. Country Location Year	Form (percent)	Rate (g ai/ha)	Water (L/ha)	No.	PHI (Days)	Sample	Difenoconazole Residues (mg/kg)[2] Plot 1	Difenoconazole Residues (mg/kg)[2] Plot 2
		75.5 74.7 75.9	755 747 759					
					14	Black Tea, Dried	0.24 (0.23, 0.25)	0.39 (0.39, 0.40)
					21	Green Tea, Dried	0.015 (0.012, 0.018)	0.017 (0.016, 0.017)
					21	Black Tea, Dried	0.018 (0.013, 0.023)	0.031 (0.022, 0.040)
RA20004G20001-07 China Fuan, Fujian Province 2020	WD 10 percent	Plot 1: 74.4 75.0 74.0 Plot 2: 74.3 74.2 75.5	Plot 1: 744 750 740 Plot 2: 743 742 755	3	14	Green Tea, Dried	0.20 (0.20, 0.21)	0.35 (0.36, 0.34)
					14	Black Tea, Dried	0.14 (0.12, 0.16)	0.32 (0.31, 0.34)
					21	Green Tea, Dried	0.34 (0.31, 0.37)	0.16 (0.15, 0.17)
					21	Black Tea, Dried	0.13 (0.12, 0.14)	0.19 (0.18, 0.21)
RA20004G20001-08 China Qiongzhong, Hainan Province 2020	WD 10 percent	Plot 1: 73.4 72.3 73.5 Plot 2: 72.7 72.6 75.8	Plot 1: 734 723 735 Plot 2: 727 726 758	3	14	Green Tea, Dried	0.060 (0.062, 0.058)	0.056 (0.053, 0.060)
					14	Black Tea, Dried	0.061 (0.056, 0.066)	0.061 (0.055, 0.068)
					21	Green Tea, Dried	0.026 (0.025, 0.027)	0.022 (0.021, 0.023)
					21	Black Tea, Dried	0.015 (0.017, 0.013)	0.016 (0.016, 0.017)

Notes:

[1] Reference: Study report Number: RA20004G20001, Yanjie Li, 2022.

[2] Mean of replicate trial samples (individual value).

APPRAISAL

Difenoconazole was evaluated for the first time by the JMPR 2007 when an acceptable daily intake (ADI) of 0–0.01 mg/kg bw and an acute reference dose (ARfD) of 0.3 mg/kg bw were established. In 2007, 2010, 2013, 2015, and 2017 the JMPR evaluated the compound for residues and recommended a number of maximum residue levels.

The definition of the residue for compliance with MRL and for dietary risk assessment for plant commodities is parent *difenoconazole*, while for animal commodities it is defined as *sum of difenoconazole*

and 1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol (CGA205375), expressed as difenoconazole. The residue is fat-soluble.

Difenoconazole was scheduled at the Fifty-second Session of the CCPR for the evaluation of additional MRLs in 2022 JMPR. The current Meeting received additional analytical methods, storage stability data, GAP information and residue trial data from uses on goji berries, pencil yams, ginger, and tea as well as their processed commodities.

Methods of analysis

The Meeting received additional information on analytical methods for difenoconazole in goji berry, pencil yam, ginger, and tea commodities.

For all provided analytical methods, residues of difenoconazole were extracted with acetonitrile/water (1/1, v/v), cleaned up by SPE, and analysed by HPLC-MS/MS.

Recoveries and percentRSDs were within the acceptable range. The LOQ was 0.01 mg/kg for all commodities tested.

The Meeting concluded that the presented methods were sufficiently validated and are suitable to measure difenoconazole in plant commodities.

Stability of pesticides residues in stored analytical samples

The current Meeting received additional information on freezer storage stability of difenoconazole in goji berry, dried goji berry, pencil yam, dried pencil yam, ginger, dried ginger, fresh tea leaves, green tea, and black tea.

Residues of difenoconazole were stable for at least 12 months in fresh and dried goji berry, 14 months in fresh and dried pencil yam, 12 months in fresh ginger, hot-dried ginger, and freeze-dried ginger, and 12 months in fresh tea leaves, green tea, and black tea when stored frozen at ≤-18 °C.

The Meeting concluded that the storage stability data were sufficiently validated and are adequate to support the storage durations in the studies provided to the current Meeting.

Results of supervised residue trials on crops

Goji berry

The use of difenoconazole on goji berry is registered in the People's Republic of China for foliar spray applications. The Meeting determined that the Critical GAP consists of three treatments with a re-treatment interval (RTI) of 7 days at a target application concentration of 0.010 kg ai/hL with a PHI of 5 days.

In independent trials matching the cGAP, residues of difenoconazole in goji berries were (n=4): 0.24, 0.59, 0.70, and 2.2 mg/kg.

The Meeting estimated a maximum residue level of 5 mg/kg, an STMR of 0.65 mg/kg, and an HR of 2.4 mg/kg (from a single sample) for difenoconazole in goji berry.

Meeting withdrew its previous recommendation of 0.6 mg/kg for difenoconazole in fruiting vegetables other than cucurbits except dried chili pepper and recommended a new maximum residue level of 0.6 mg/kg in fruiting vegetables other than cucurbits except dried chili pepper and goji berry.

Pencil Yam

The use of difenoconazole on pencil yam is registered in the People's Republic of China for foliar spray applications. The Meeting determined that the Critical GAP consists of three treatments (RTI=7 days) at a target application rate of 0.080 kg ai/ha with a PHI of 60 days.

In independent trials matching the cGAP, residues of difenoconazole in pencil yams were (n=4): < 0.01 (3) and 0.010 mg/kg.

The Meeting estimated a maximum residue level of 0.02 mg/kg, an STMR of 0.010 mg/kg, and an HR of 0.010 mg/kg for difenoconazole in pencil yam.

Ginger

The use of difenoconazole on ginger is registered in the People's Republic of China for foliar spray applications. The Meeting determined that the Critical GAP consists of three treatments (RTI=7 days) at a target application rate of 0.11 kg ai/ha with a PHI of 14 days.

In independent trials matching the cGAP, residues of difenoconazole in ginger were (n=8): < 0.01 (4), 0.033, 0.038, 0.062, and 0.10 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg, an STMR of 0.022 mg/kg, and an HR of 0.10 mg/kg for difenoconazole in fresh ginger.

Tea

The Meeting received a GAP for tea from the People's Republic of China consisting of three treatments (RTI=7 days) at a target application concentration of 0.010 kg ai/hL with a PHI of 14 days.

In independent trials matching the GAP, residues of difenoconazole in green tea (dry) were (n=8): 0.019, 0.060, 0.20, 0.35, 0.62, 0.77, 2.4, and 4.2 mg/kg.

Residues in black tea (fermented and dry) derived from the green tea (dry) samples were (n=8): 0.023, 0.061, 0.22, 0.26, 0.32, 0.39, 1.7, and 4.5 mg/kg.

The 2021 Extra Meeting recommended a maximum residue level of 20 mg/kg and an STMR of 4.85 mg/kg for residues of difenoconazole in tea, green, black (black fermented and dried). The current Meeting confirmed its previous recommendation, which accommodates the residues listed above.

Fate of residues during processing

The Meeting received new information on the fate of difenoconazole residues during processing in goji berry, pencil yam, and ginger.

Table 12 Estimated processing factors for the commodities considered at this Meeting

RAC	Processed Commodity	Processing Factor	Median Processing Factor	STMR RAC (mg/kg)	HR RAC (mg/kg)	STMR-P (mg/kg)	HR-P (mg/kg)
Goji berry	Dried goji berry	1.3, 1.5, 1.6, 1.7, 1.8, 1.9, 2.0, 2.5(2), 2.6, 3.1, 4.2, 5.0, 11, 26, 28	2.5	0.65	2.4	1.6	5.5
Pencil Yam	Dried Pencil Yam	2.3, 3.4	2.9	0.010	0.010	0.029	0.029
Ginger	Hot Dried [a]	3.2, 3.4, 4.6, 5.1(2), 5.7, 6.7, 7.0, 7.5, 7.6, 7.8, 8.0	6.0	0.022	-	0.13	-

Notes:

[a] Hot dried ginger processing factors are higher than freeze dried ginger processing factors. Dried ginger was grinded to a powder prior to analysis.

Using the estimated maximum residue level of 5 mg/kg for goji berry and applying the processing factor of 2.5, the Meeting estimated a maximum residue level of 15 mg/kg for difenoconazole in goji berry, dried.

Using the estimated maximum residue level of 0.02 mg/kg for pencil yam and applying the processing factor of 2.9, the Meeting estimated a maximum residue level of 0.07 mg/kg for difenoconazole in pencil yam, dried.

Using the estimated maximum residue level of 0.2 mg/kg for ginger and applying the processing factor of 6.0, the Meeting estimated a maximum residue level of 1.5 mg/kg for difenoconazole in ginger, dried.

Residues in animal commodities

Estimated maximum and mean dietary burdens of livestock and animal commodities maximum residue levels

No animal feeds are associated with the uses considered by the current Meeting. The Meeting confirmed its previous recommendations.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with the MRL and for dietary risk assessment for plant commodities: *difenoconazole*.

Definition of the residue for compliance with the MRL and for dietary risk assessment for animal commodities: *sum of difenoconazole and 1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol), expressed as difenoconazole*.

The residue is fat-soluble.

Table 13 Residue levels suitable for establishing maximum residue limits and for IEDI and IESTI estimations

Commodity		Recommended Maximum Residue Level (mg/kg)		STMR or STMR-P (mg/kg)	HR or HR-P (mg/kg)
CCN	Name	New	Previous		
VO 2704	Goji berry	5	-	0.65	2.4
DV 2704	Goji berry, dried	15	-	1.6	5.5
VO 0050	Group of fruiting vegetables other than cucurbits (except peppers, chili)	W	0.6	0.14	0.39
VO 0050	Group of fruiting vegetables other than cucurbits (except goji berry and pepper, chili)	0.6	-	0.14	0.39
VR 2950	Pencil yam	0.02	-	0.010	0.010
	Pencil yam, dried	0.07	-	0.029	0.029

Commodity		Recommended Maximum Residue Level (mg/kg)		STMR or STMR-P (mg/kg)	HR or HR-P (mg/kg)
CCN	Name	New	Previous		
HS 0784	Ginger, rhizome	0.2	-	0.022	0.10
DV 0784	Ginger rhizome, dried	1.5	-	0.13	-

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for difenoconazole is 0–0.01 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for difenoconazole were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the previous and present JMPR. The results are shown in Annex 3 of the 2022 JMPR Report. The IEDIs ranged 10–80 percent of the maximum ADI.

The Meeting concluded that the long-term dietary exposure to residues of difenoconazole from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2007 JMPR established an ARfD of 0.3 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for difenoconazole were calculated for the food commodities for which STMRs or HRs were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report. The IESTIs varied from 0–3 percent of the ARfD for children and 0 percent for the general population.

The Meeting concluded that the acute dietary exposure to residues of difenoconazole from other uses that have been considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Study Report	Author	Year	Citation
R20002A	Yanmei Yang	2021	Method Performance Validation for the Determination of Residue of Difenoconazole in Goji Berry by UPLC-MS/MS
HZ2020N001A	Xueyan Zhang	2021	Method Performance Validation for the Determination of Residues of Difenoconazole in Pencil Yam by UPLC-MS/MS
AR-2020PR20	Aijuan Zhang	2022	Method Performance Validation for the Determination of Residues of Difenoconazole in Fresh Ginger and Dried Ginger by LC-MS/MS
RM20004G20001	Yanjie Li	2021	Method Performance Validation for the Determination of Residues of Difenoconazole in Tea by UPLC-MS/MS
R20002B	Yanli Qi	2021	Storage Stability of Difenoconazole in Goji Berry Frozen Storage for up to 12 Months
HZ2020N001B	Xueyan Zhang	2021	Storage Stability of Difenoconazole in Pencil Yam Frozen Storage for up to 439 Days
SR-2020SS01	Yizhi Feng	2022	Storage Stability of Difenoconazole in Ginger for up to 12 Months of Frozen Storage
RB20004G20001	Yanjie Li	2021	Storage Stability of Difenoconazole in Tea Matrices for up to 12

Study Report	Author	Year	Citation
			Months of Frozen Storage
R20002	Weirong Wang	2021	Magnitude of the Residue of Difenoconazole in Goji Berry and Dried Goji Berry
HZ2020N001	Wenxi Li	2021	Magnitude of the Residue of Difenoconazole in Pencil Yam and Processed Commodity
PR-2020PR20	Yizhi Feng	2022	Magnitude of the Residue of Difenoconazole in Giner
RA20004G20001	Yanjie Li	2022	Magnitude of the Residue of Difenoconazole on/in Tea

DIMETHOATE (027)/OMETHOATE (055)

First draft prepared by D.W. Lunn, Ministry for Primary Industries, New Zealand

APPRAISAL

Dimethoate is an organophosphate insecticide which acts through acetylcholinesterase inhibition. It has been evaluated on numerous occasions by the JMPR since 1963, with the last periodic review conducted in 1996 (toxicology) and 1998 (residues), a subsequent evaluation for toxicology and residues in 2003 to establish an acute reference dose and further evaluations for additional uses in 2006 and 2008.

Dimethoate was scheduled by the Fiftieth Session of the CCPR (2018) for periodic review and the 2019 JMPR considered information supplied by the sponsor on identity, physicochemical properties, metabolism and environmental fate, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials, fate of residues in processing, and animal feeding studies, together with additional supervised residue trial data supplied by Australia for mandarin, oranges, avocados, mangoes, capsicum and pulses, and by Thailand for yard-long bean.

The 2019 JMPR established a revised ADI of 0–0.001 mg/kg bw and reaffirmed the ARfD of 0.02 mg/kg bw for dimethoate, but was unable to complete the assessment of omethoate, a metabolite of dimethoate and also used as a pesticide, with respect to its mutagenic potential.

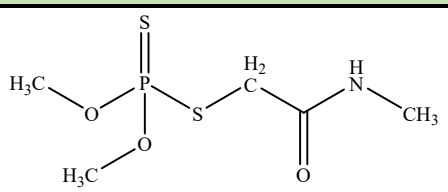
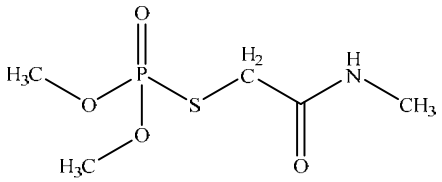
The 2019 JMPR also recommended a residue definition of dimethoate and omethoate (measured and reported seperately) for MRL-compliance in plant and animal commodities and concluded that the residue is not fat-soluble.

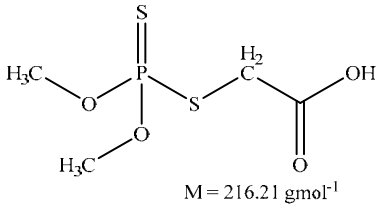
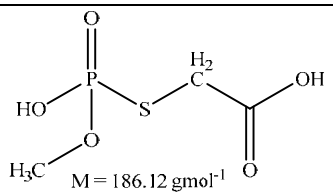
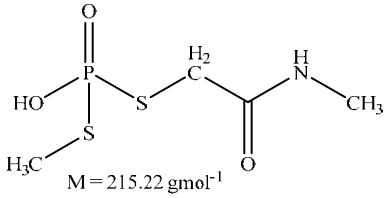
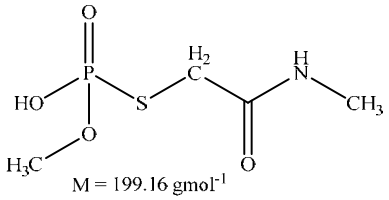
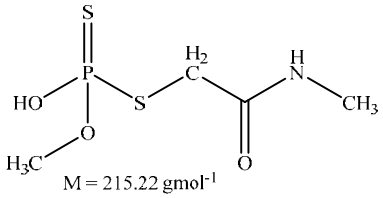
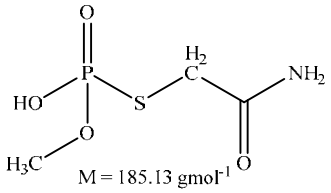
However, the Meeting was unable to recommend residue definitions for dietary risk assessment because of concerns relating to the genotoxicity of omethoate and other related metabolites.

Evaluation of the metabolism studies in rats was carried out by the WHO Core Assessment Group in 2019 and a further assessment of omethoate and its metabolites was conducted by the current Meeting. Residue components observed in the dimethoate rat metabolism study were dimethoate, omethoate, dimethoate carboxylic acid, dimethyl dithiophosphate, dimethyl thiophosphate, and dimethyl phosphate.

The previously-submitted residue information evaluated by the 2019 JMPR was re-evaluated in light of this new toxicological information and new information on current dimethoate GAP.

Table 1 Major metabolites discussed in this Appraisal

Component name	Structure	Origin
Dimethoate		Parent compound
Omethoate (XI)		Potato, olives, wheat, rat, goat, hen

Component name	Structure	Origin
Dimethoate carboxylic acid (III)		Potato, olives, wheat, rat, goat, hen
O-desmethyl omethoate carboxylic acid (XX)		Potato, wheat
O-desmethyl isodimethoate (XII)		Potato, olives, wheat
O-desmethyl omethoate (XI)		Potato, olives, wheat
Desmethyl dimethoate (X)		Potato, hydrolysis, soil (minor component)
O-desmethyl N-desmethyl omethoate (XXIII)		Potato, olives, wheat

Residue definition

Plant commodities

For MRL-compliance, based on the metabolism studies and field trials, the 2019 JMPR concluded that dimethoate and omethoate were good marker compounds particularly for shorter pre-harvest intervals, and in directly treated commodities such as leafy vegetables and fruits and noted that suitable validated methods were available for dimethoate and omethoate in an extensive range of plant commodities.

The 2019 JMPR further noted that in residue trials for some commodities, for example cherries and olives, omethoate was present at higher levels than dimethoate, particularly around harvest. Therefore,

inclusion of both dimethoate and omethoate in the definition for compliance with MRLs was warranted and since omethoate is itself a pesticide, it should therefore be measured separately from dimethoate.

The 2019 JMPR considered that a suitable residue definition for compliance with MRLs in plant commodities was *dimethoate and omethoate, measured and reported separately*.

The 2019 JMPR also considered that dimethoate and omethoate were expected to have similar bioavailability to livestock, and determined that the *sum of dimethoate and omethoate* would be used to estimate median and highest residues in feed commodities for estimation of livestock dietary burden.

For dietary risk assessment, the 2019 JMPR reviewed metabolism studies in olives, potatoes and wheat, supervised field trials where a number of these metabolites were analysed and a number of older studies containing summarized metabolism data on lemons, sugar beet, maize, cotton, peas, potatoes and beans.

The 2019 JMPR concluded that in the olive, wheat and potato studies, the major components of the residue in matrices treated directly with dimethoate at shorter sampling intervals (0–14 days) were dimethoate and omethoate.

In matrices to which residues are translocated, and at longer intervals after application, the metabolite pattern is different and dimethoate and omethoate are present at lower levels and the major residues are metabolites O-desmethyl N-desmethyl omethoate (XXIII), O-desmethyl isodimethoate (XII), O-desmethyl omethoate carboxylic acid (XX) and O-desmethyl omethoate (XI).

In a number of residue trials in wheat, olives and sugar beet, dimethoate, omethoate and six metabolites were analysed. No residues of any of the components were found above 0.01 mg/kg in wheat grain, and in sugar beet roots all components other than desmethyl dimethoate (X) were also below the LOQ. In olives, dimethoate and omethoate were the most significant residue components, with dimethoate carboxylic acid (III) found in olive flesh (0.02 mg/kg) and desmethyl dimethoate (X) found in sugar beet roots (0.03 mg/kg).

The current Meeting, in deciding which metabolites should be included in the residue definition for plant commodities, considered the likely occurrence and toxicological relevance of the compounds present at more than 10 percent of total identified residues in the metabolism studies.

Compounds considered were omethoate, O-desmethyl-N-desmethyl omethoate, O-desmethyl-isodimethoate, desmethyl dimethoate, O-desmethyl omethoate carboxylic acid, O-desmethyl-omethoate and O,O-dimethyl phosphonic acid.

The Meeting noted that O-desmethyl-N-desmethyl omethoate, O-desmethyl-isodimethoate, desmethyl dimethoate, O-desmethyl omethoate carboxylic acid, O-desmethyl-omethoate and O,O-dimethyl phosphonic acid were of no toxicological relevance and these metabolites were not discussed further.

Omethoate was a significant residue in most directly treated plant matrices, both in the plant metabolism studies and supervised field trials, and present in some processed olive and orange commodities. It was also found in the rat metabolism study, has a lower ADI (0.0004 mg/kg bw/day) and a lower ARfD (0.002 mg/kg bw) than dimethoate. The Meeting considered that omethoate should be included in the residue definition.

Based on the above, the current Meeting considered that for dietary intake risk assessment for plant commodities, the residue definition should be: *Dimethoate plus 2.5× omethoate, expressed as dimethoate for long-term dietary exposure and dimethoate plus 10× omethoate for acute dietary exposure*.

Animal commodities

Based on the goat and laying hen metabolism studies, the 2019 JMPR concluded that the major component of the residue was incorporated into phosphorylated natural products. Dimethoate residues were not detected in any matrix, indicating rapid metabolism.

Omethoate residues were found in cattle liver (0.12 mg eq/kg), poultry liver (0.081 mg eq/kg) and egg white (0.004 mg eq/kg).

Residues of dimethoate carboxylic acid (III) made up 16 percent TRR (0.13 mg eq/kg) in poultry liver, 2.5 percent TRR (0.031 mg eq/kg) in goat liver, 8.3 percent TRR (0.019 mg eq/kg) in goats milk and 3.9 percent TRR (0.005 mg eq/kg) in egg white.

In the lactating cattle feeding study, no residues of dimethoate were found above the LOQ in milk, muscle, liver, or kidney, while low levels of omethoate were detected in milk, kidney, and muscle for the highest dose group, in liver for the highest and second highest dose groups, while low levels of dimethoate were detected in fat at all doses, without any clear correlation between dose and residue level. Omethoate was detected in fat at higher dose levels.

In the laying hen feeding study, no residues of dimethoate or omethoate were detected in tissues or eggs at any dose level.

The 2019 JMPR considered that a suitable residue definition for compliance with MRLs in animal commodities was *dimethoate and omethoate, measured and reported separately* and that residues of dimethoate and omethoate are not fat-soluble.

For dietary risk assessment, in deciding which metabolites should be included in the residue definition for animal commodities, the current Meeting noted that the only metabolites found in animal commodities were considered omethoate and dimethoate carboxylic acid. The Meeting noted that dimethoate carboxylic acid was of no toxicological relevance and decided it need not be included in the residue definition.

Omethoate was found in at low levels in cattle milk (< 0.02 mg/kg) and most cattle tissues (< 0.005 mg/kg) but in the goat and poultry metabolism studies, was only found in goat liver (9.8 percent TRR, 0.12 mg eq/kg), poultry liver (16 percent TRR, 0.081 mg eq/kg) and egg white (3.9 percent TRR, 0.005 mg eq/kg).

It was also found in the rat metabolism study and is more toxic than dimethoate. The Meeting considered that omethoate should be included in the residue definition for risk assessment.

Based on the above, the current Meeting considered that for dietary intake risk assessment for animal commodities, the residue definition should be: *Dimethoate plus 2.5× omethoate, expressed as dimethoate for long-term dietary exposure and dimethoate plus 10× omethoate for acute dietary exposure*.

Results of supervised residue trials on crops

The 2019 JMPR evaluated supervised trials on the use of dimethoate on mandarins, oranges, cherries, olives, avocados, mangoes, bulb onions, brassica vegetables, melons, sweet peppers, tomatoes, leaf lettuce, legume vegetables, pulses, root and tuber vegetables, barley, wheat and rape seed.

Product labels provided to the 2019 Meeting were from Australia, Brazil, Thailand, the United States and a number of European Union member states. The current Meeting noted that since 2019, all European Union dimethoate authorisations have been withdrawn and that of the proposed MRLs recommended by the 2019 JMPR, only those for citrus, avocados, tomatoes, dried beans, rape seed

(Australian GAPs) and yard-long beans (Thailand GAP) were still valid. New GAP information was provided for Brussels sprouts (Canada) and a new GAP was identified for wheat.

For acute dietary exposure estimation, the highest individual total residue values from the trials have been used to derive the highest residues.

The residue trial tables include values for the sum of dimethoate and omethoate for use in the livestock dietary burden calculation where applicable.

Where residues were reported below the LOQ, the following conventions were adopted for summing residues (using an LOQ of 0.01 mg/kg as an example):

Table 2 Convention adopted for summing of residues

Dimethoate (mg/kg)	Omethoate (mg/kg)	Sum of dimethoate and omethoate (mg/kg)
0.30	0.04	0.34
0.30	< 0.01	0.31
< 0.01	< 0.01	< 0.02

For dietary intake estimation it is necessary to account for the residues of both dimethoate and omethoate. In order to estimate STMR and HR values for use in the dietary intake calculations, the relative toxicity of the two compounds must be taken into account. Since dimethoate and omethoate share a common toxicological mode of action, in line with the approach taken by previous Meetings, the toxicologically significant residues were estimated by adding the dimethoate and omethoate residues after scaling the omethoate residues to dimethoate toxicity equivalents - based on the ratio of the dimethoate to omethoate maximum ADIs for STMR estimation and acute RfDs for HR estimation.

For long-term dietary exposure estimation, toxic equivalent residues (mg teq/kg) = dimethoate + 2.5×omethoate.

For short-term dietary exposure estimation, toxic equivalent residues (mg teq/kg) = dimethoate + 10×omethoate.

Where residues were reported as < 0.01 mg/kg, a value of 0.01 mg/kg was used when calculating total residues for dietary exposure estimation.

Citrus fruits

The 2019 JMPR concluded that the critical GAP for citrus fruit was in Australia, a post-harvest dip or flood application of 0.04 kg ai/100 L (40 ppm), with no withholding period required.

In mandarin trials supporting this GAP, dimethoate residues in whole fruit were: 0.58, 0.70, 0.71 and 0.82 mg/kg (n=4) and omethoate residues were: < 0.01 (4) mg/kg (n=4).

In orange trials supporting this GAP, dimethoate residues in whole fruit were: 0.51, 0.59, 0.60, 0.63, 0.66 and 0.67 mg/kg (n=6) and omethoate residues were: 0.003 (3), 0.004 (2) and 0.005 mg/kg (n=6).

The 2019 Meeting also agreed to combine the residue data sets for oranges and mandarins to estimate maximum residue levels for the subgroups of mandarins and oranges and noted that these levels would accommodate the foliar application GAPs in Australia and Brazil.

The combined mandarin and orange (whole fruit) dataset for dimethoate was: 0.51, 0.58, 0.59, 0.60, 0.63, 0.65, 0.67, 0.70, 0.71, and 0.82 mg/kg (n=10).

The combined mandarin and orange (whole fruit) dataset for omethoate was: 0.003 (3), 0.004 (2), 0.005, and < 0.01 (4) mg/kg (n=10).

The Meeting confirmed the 2019 JMPR estimated maximum residue levels of 2 mg/kg for dimethoate and 0.02 mg/kg for omethoate in the subgroup of mandarins and the subgroup of oranges.

Residues for livestock dietary burden estimation (sum of dimethoate and omethoate) in citrus (whole fruit) were: 0.51, 0.59 (2), 0.6, 0.63, 0.66, 0.675, 0.71, 0.72 and 0.83 mg/kg (n=10). The median residue was 0.645 mg/kg.

Mandarin

For dietary exposure estimation, in the Australian trials matching the critical GAP, dimethoate residues in mandarin flesh were: 0.014, 0.056, 0.0.056 and 0.076 mg/kg (n=4) with a highest value of 0.089 mg/kg and omethoate residues were: < 0.01 (4) mg/kg (n=4).

For assessing long-term dietary exposure, the toxic equivalent residues in mandarin flesh (dimethoate + 2.5× omethoate residues) were: 0.039, 0.081 0.081 and 0.1 mg teq/kg.

For assessing short-term dietary exposure, the toxic equivalent residues in mandarin flesh were: 0.11, 0.16, 0.16 and 0.18 mg teq/kg and the highest individual value was 0.19 mg teq/kg.

The Meeting estimated an $STMR_{chronic}$ of 0.081 mg teq/kg, an HR of 0.19 mg teq/kg and a $STMR_{(acute)}$ residue of 0.16 mg teq/kg for dimethoate in the subgroup of mandarins.

Orange

For dietary exposure estimation, in the Australian trials matching the critical GAP, dimethoate residues in orange flesh were: 0.19, 0.26, 0.275, 0.34, 0.37 and 0.38 mg/kg (n=6) and omethoate residues were: 0.001 (2), 0.002 (3) and 0.003 mg/kg (n=6).

For assessing long-term dietary exposure, the toxic equivalent residues in orange flesh were: 0.19, 0.265, 0.28, 0.345, 0.38 and 0.385 mg teq/kg.

For assessing short-term dietary exposure from residues in oranges, the toxic equivalent residues in flesh were: 0.2, 0.28, 0.285, 0.36, 0.4 and 0.4 mg teq/kg.

The Meeting estimated an $STMR_{chronic}$ of 0.31 mg teq/kg, an HR of 0.4 mg teq/kg and a $STMR_{(acute)}$ residue of 0.32 mg teq/kg for dimethoate in the subgroup of oranges.

The Meeting noted that an acute dietary exposure assessment showed that residues in the *subgroup of oranges exceed the ARfD of 0.02 mg/kg bw, at 120 percent for peeled oranges for Australian children.* No alternative GAP was available.

Avocados

The 2019 JMPR concluded that the critical GAP for avocados was in Australia, for dilute foliar applications of 0.03 kg ai/100 L as required (with a 7-day PHI) followed by a 1-minute post-harvest dip using 0.04 kg ai/100 L, with no withholding period.

In avocado trials supporting this GAP, dimethoate residues in (whole fruit) were: 0.41, 0.44, 0.71, and 0.75 mg/kg (n=4) and omethoate residues were: 0.016, 0.025, 0.042, and 0.067 mg/kg (n=4).

The Meeting confirmed the 2019 JMPR estimated maximum residue level of 2 mg/kg for dimethoate and 0.15 mg/kg for omethoate in avocado.

For dietary exposure estimation, in the Australian trials matching the critical GAP, dimethoate residues in flesh were: 0.062, 0.062, 0.11 and 0.17 mg/kg (n=4) and omethoate residues were: < 0.01, < 0.01, 0.01 and 0.032 mg/kg (n=4).

For assessing long-term dietary exposure from residues in avocados, the toxic equivalent residues in flesh were: 0.087, 0.087, 0.135 and 0.25 mg teq/kg.

For assessing short-term dietary exposure from residues in avocados, the toxic equivalent residues in flesh were: 0.16, 0.16, 0.21 and 0.49 mg teq/kg.

The Meeting estimated an $STMR_{chronic}$ of 0.11 mg teq/kg, an HR of 0.49 mg teq/kg and a $STMR_{(acute)}$ residue of 0.37 mg teq/kg for dimethoate in avocados.

Brussels sprouts

The 2019 JMPR estimated a maximum residue level for Brussels sprouts based on the GAP in the Czech Republic. As this GAP no longer exists, the current Meeting re-evaluated the available data based on a newly provided Canadian GAP.

The critical GAP in Canada for brussels sprouts is for 2 foliar applications of 0.48 kg ai/ha, with a 7-day minimum retreatment interval and a PHI of 21 days.

In trials matching this GAP, but with a lower application rate (0.24–0.25 kg ai/ha) dimethoate residues in Brussels sprouts were: < 0.01 (3), 0.01, 0.02,(3) and 0.03 (2) mg/kg (n=9) and omethoate residues were: < 0.01 (8) and 0.01 mg/kg (n=9).

When proportionally adjusted to the Canadian application rate (scaling factor of 1.9), dimethoate residues were: < 0.019 (3), 0.019, 0.038 (3) and 0.058 (2) mg/kg (n=9) and omethoate residues were: < 0.019 (8) and 0.019 mg/kg (n=9).

The current Meeting estimated a maximum residue level of 0.1 mg/kg for dimethoate and 0.03 mg/kg for for omethoate in Brussels sprouts, to replace the 2019 JMPR estimations.

For assessing long-term dietary exposure from residues in Brussels sprouts, the toxic equivalent residues were: < 0.067 (3), 0.067, 0.086 (3) and 0.11 (2) mg teq/kg (n=9).

For assessing short-term dietary exposure from residues in Brussels sprouts, the toxic equivalent residues were: < 0.21 (3), 0.21, 0.23 (3) and 0.25 (2) mg teq/kg (n=9).

The Meeting estimated an $STMR_{chronic}$ of 0.086 mg teq/kg, an HR of 0.25 mg teq/kg and a $STMR_{(acute)}$ residue of 0.23 mg teq/kg for dimethoate in Brussels sprouts.

Tomato

The 2019 JMPR concluded that the critical GAP for tomatoes was in Australia, for 2 × 0.3 kg ai/ha foliar applications with a minimum 14-day retreatment interval and a 21-day PHI. Scaled dimethoate residues in trials from Europe supporting this GAP were: < 0.005 mg/kg (n=8) and omethoate residues were: < 0.005 (6), 0.005 and 0.005 mg/kg (n=8).

The Meeting confirmed the 2019 JMPR estimated maximum residue level of 0.01(*) mg/kg for dimethoate and 0.01 mg/kg for for omethoate in tomato.

Residues for livestock dietary burden estimation (sum of dimethoate and omethoate) in tomato were: < 0.01 (6) and 0.01 (2) mg/kg (n=8). The median residue was 0.01 mg/kg.

For assessing long-term dietary exposure from residues in tomatoes, the toxic equivalent residues were: < 0.0175 (6) and 0.0175 (2) mg teq/kg.

For assessing short-term dietary exposure from residues in tomatoes, the toxic equivalent residues were: < 0.055 (6) and 0.055 (2) mg teq/kg.

The Meeting estimated an $STMR_{chronic}$ of 0.0175 mg teq/kg, an HR of 0.055 mg teq/kg and a $STMR_{(acute)}$ residue of 0.055 mg teq/kg for dimethoate in tomatoes.

Yard-long bean (pods)

The 2019 JMPR concluded that the critical GAP for yard-long beans was in Thailand, for 4 × 0.6 kg ai/ha foliar applications with a 7-day PHI.

Dimethoate residues in trials in supporting this GAP were: < 0.05 (5) and 0.05 mg/kg (n=6) and omethoate residues were: < 0.05 (6) mg/kg (n=6).

The Meeting confirmed the 2019 JMPR estimated maximum residue level of 0.07 mg/kg for dimethoate and 0.05 mg/kg for for omethoate in yard long bean.

For assessing long-term dietary exposure from residues in yard-long bean, the toxic equivalent residues were: < 0.175 (5) and 0.175 mg teq/kg.

For assessing short-term dietary exposure from residues in yard-long bean, the toxic equivalent residues were: < 0.55 (5) and 0.55 mg teq/kg.

The Meeting estimated an $STMR_{chronic}$ of 0.175 mg teq/kg, an HR of 0.55 mg teq/kg and a $STMR_{(acute)}$ residue of 0.55 mg teq/kg for dimethoate in yard-long bean.

Beans (dry)

The 2019 JMPR concluded that the critical GAP for dry beans (except soya beans) was in Australia, for foliar applications of 0.32 kg ai/ha, with a minimum 14-day retreatment interval and a 14 day PHI for both grazing and harvest.

Dimethoate residues in dry beans from trials supporting this GAP were: < 0.05 (4), 0.066, and 0.4 mg/kg (n=6) and omethoate residues were: < 0.05 (5), and 0.064 mg/kg (n=6).

Residues for livestock dietary burden estimation (sum of dimethoate and omethoate) in dry beans were: < 0.10 (4), 0.12, and 0.46 mg/kg (n=6). The median residue was 0.1 mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg for dimethoate and 0.08 mg/kg for omethoate in the subgroup of dry beans (except soya bean) to replace the previous estimation for the subgroup of dry beans.

For assessing long-term dietary exposure from residues in dry beans, the toxic equivalent residues were: < 0.175 (4), 0.19 and 0.56 mg teq/kg.

For assessing short-term dietary exposure from residues in dry beans, the toxic equivalent residues were: < 0.55 (4), 0.57 and 1.0 mg teq/kg.

The Meeting estimated an $STMR_{chronic}$ of 0.175 mg teq/kg and a $STMR_{(acute)}$ residue of 0.38 mg/kg for dimethoate in dry beans (subgroup) except soya bean.

Wheat

The 2019 JMPR estimated a maximum residue level for wheat based on the GAP in the Czech Republic. As this GAP no longer exists, the current Meeting re-evaluated the available data based on the current GAP in the United States.

The critical GAP in the United States for wheat is for a single foliar application of 0.56 kg ai/ha, with a 35-day PHI and a 14-day grazing interval.

In trials matching this GAP, but at a lower application rate of 0.2–0.21 kg ai/ha, dimethoate residues in wheat grain were: < 0.001 (9), 0.002 (2), 0.005, < 0.01 (5) and 0.01 mg/kg (n=18) and omethoate residues were: < 0.0012 (12) and < 0.01 (6) mg/kg (n=18).

When proportionally adjusted to the United States application rate (scaling factor of 2.66), dimethoate residues were: < 0.0027 (9), 0.0053 (2), 0.013, < 0.027 (5) and 0.027 mg/kg (n=18) and omethoate residues were: < 0.0027 (12) and < 0.027 (6) mg/kg (n=18).

Scaled residues for livestock dietary burden estimation (sum of dimethoate and omethoate) in wheat were: < 0.0053 (9), < 0.008 (2), < 0.016 and < 0.053 (6) mg/kg (n=18). The median residue was 0.008 mg/kg.

The Meeting estimated a maximum residue level of 0.06 mg/kg for dimethoate and 0.03 mg/kg for omethoate in wheat to replace the 2019 JMPR estimations.

For assessing long-term dietary exposure from wheat, the toxic equivalent residues were: < 0.0093 (9), 0.012 (2), 0.02, < 0.093 (5) and 0.09 mg teq/kg (n=18).

For assessing short-term dietary exposure from wheat, the toxic equivalent residues were: < 0.029 (9), 0.032 (2), 0.04, < 0.29 (5) and 0.29 mg teq/kg (n=18).

The Meeting estimated an $STMR_{chronic}$ of 0.011 mg teq/kg and a $STMR_{(acute)}$ residue of 0.032 mg/kg for dimethoate in wheat.

Rape seed (canola)

The 2019 JMPR concluded that the critical GAP for rape seed (canola) was in Australia, for a single foliar application of 0.14 kg ai/ha, with a 7 day PHI for both grazing and harvest.

In rape seed trials supporting this GAP, dimethoate residues were: < 0.02, 0.02, 0.026, 0.027, 0.028, 0.051, 0.066 and 0.084 mg/kg (n=8) and omethoate residues were: < 0.02 (7) and 0.02 mg/kg (n=8).

The Meeting confirmed the 2019 JMPR estimated maximum residue level of 0.15 mg/kg for dimethoate and 0.03 mg/kg for omethoate in rape seed.

For assessing long-term dietary exposure from rape seed, the toxic equivalent residues were: < 0.07 0.07, 0.076, 0.077, 0.078, 0.1, 0.12 and 0.13 mg teq/kg (n=8).

For assessing short-term dietary exposure from rape seed, the toxic equivalent residues were: < 0.22, 0.22, 0.23 (3), 0.25, 0.27 and 0.28 mg teq/kg.

The Meeting estimated an $STMR_{chronic}$ of 0.0775 mg teq/kg and a $STMR_{(acute)}$ residue of 0.23 mg/kg for dimethoate in rape seed.

Residues in animal feeds

Bean forage

The 2019 JMPR concluded that in the Australian residue trials on dry beans, the data for forage did not match GAP, as samples were only collected at intervals of 0 and 7 days after application and no median or highest residues could be estimated for bean forage.

Wheat forage

The current Meeting reviewed the available data on wheat forage (whole plants) in light of the US GAP and the 14-day pre-grazing interval. In trials matching this GAP, but at a lower application rate of 0.2–0.21 kg ai/ha, total residues (sum of dimethoate and omethoate) were: < 0.02, 0.03, 0.03, 0.05, 0.08, 0.28, 0.29, 0.44, 0.61, 0.75, 1.5 and 1.65 mg/kg as received (n=12).

When proportionally adjusted to the United States application rate (scaling factor of 2.66), total residues in wheat forage (for livestock dietary burden estimation) were: < 0.053, 0.08, 0.08, 0.13, 0.21, 0.75, 0.77, 1.2, 1.6, 2.0, 4.0 and 4.4 mg/kg as received (n=12).

The Meeting estimated a median total residue of 0.76 mg/kg fw and a highest total residue of 4.4 mg/kg fw for the sum of omethoate and dimethoate in wheat forage (for livestock dietary burden estimation).

Wheat straw

The 2019 JMPR estimated a maximum residue level for wheat straw and fodder, dry based on the GAP in the Czech Republic. As this GAP no longer exists, the current Meeting re-evaluated the available data based on the GAP in the United States.

The critical GAP in United States for wheat is for a single foliar application of 0.56 kg ai/ha, with a 35-day PHI and a 14-day grazing interval.

In trials matching this GAP, but at a lower application rate of 0.2–0.21 kg ai/ha, residues of dimethoate in wheat straw were: < 0.01 (8), 0.01 (2), 0.05 (3), 0.08, 0.19, 0.68, 0.76 and 0.83 mg/kg as received (n=18).

When proportionally adjusted to the United States application rate (scaling factor of 2.66), dimethoate residues in straw were: < 0.027 (8), 0.027 (2), 0.13 (3), 0.21, 0.51, 1.8, 2.0 and 2.2 mg/kg as received (n=18).

After adjustment for dry weight using the default dry matter content of 88 percent from the OECD livestock feed table, dry weight residues in straw were: < 0.03 (8), 0.03 (2), 0.15 (3), 0.24, 0.58, 2.1, 2.3 and 2.5 mg/kg dry weight.

In these trials, residues of omethoate were: < 0.01 (13), 0.01, 0.02, 0.05 (2) and 0.074 mg/kg as received (n=18).

When proportionally adjusted to the United States application rate (scaling factor of 2.66), omethoate residues in straw were: < 0.027 (13), 0.027, 0.053, 0.13 (2) and 0.2 mg/kg as received (n=18).

After adjustment for dry weight using the default dry matter content of 88 percent from the OECD livestock feed table, dry weight residues in straw were: < 0.03 (13), 0.03, 0.06, 0.15 (2) and 0.22 mg/kg dry weight.

The Meeting estimated a maximum residue level of 4 mg/kg (dw) for dimethoate and 0.3 mg/kg (dw) for omethoate in wheat straw and/or hay to replace the 2019 JMPR estimations.

For estimation of the livestock dietary burden, total (sum of dimethoate and omethoate) residues in straw were: < 0.02 (8), 0.02 (2), 0.06 (3), 0.09, 0.24, 0.7, 0.81 and 0.9 mg/kg (as received) (n=18).

When proportionally adjusted to the United States application rate (scaling factor of 2.66), total residues in straw were: < 0.053 (8), 0.053 (2), 0.16 (3), 0.24, 0.64, 1.9, 2.2 and 2.4 mg/kg as received (n=18). The median total residue was 0.053 mg/kg (0.06 mg/kg dry weight) and the highest total residue was 2.4 mg/kg (2.7 mg/kg dry weight).

Fate of residues during processing

The 2019 JMPR evaluated an hydrolysis study (simulating high temperature processing conditions) and concluded that both dimethoate and omethoate were hydrolysed to their desmethyl metabolites under simulated baking/boiling/brewing conditions (28/36 percent AR) and after sterilization (60/63 percent) respectively and that no conversion from dimethoate to omethoate was observed under any of the conditions.

Residues in processed commodities

Processing factors were calculated by the 2019 JMPR for dimethoate and omethoate (for maximum residue level estimation, for calculating livestock dietary burdens and for risk assessment.

Table 3 Processing factors for citrus and cereal commodities

Processed commodity	Dimethoate	Omethoate
ORANGE		
Juice	0.14	0.20
Dry pulp	2.1	1.6
Molasses	5.8	5.9
Orange oil	0.20	< 0.07
WHEAT		
Wholemeal flour	0.66	0.5
White flour	0.21	0.5
Bran	4.4	3.5
Wheat germ	2.9	2.0

Maximum residue levels in processed commodities

Where residues concentrated in the processed food commodities, maximum residue levels were estimated using the estimated maximum residue levels for the raw commodities and applying the calculated mean processing factors.

Table 4 Estimated dimethoate and omethoate maximum residue levels for processed commodities.

Commodity	Processing factors [a]		Maximum Residue Level (mg/kg)	
	Dimethoate	Omethoate	Dimethoate	Omethoate
Orange			MRL=2.0	0.02
Orange dried pulp	2.1	1.6	4.2	0.032
Wheat			MRL=0.06	0.03
Wheat bran	4.4	3.5	0.26	0.105
Wheat germ	2.9	2.0	0.17	0.06

Notes:

[a] The ratio of the residues in the processed item divided by the residue in the Raw Agricultural Commodity.

The Meeting estimated a maximum residue level of 5 mg/kg for dimethoate and 0.04 mg/kg for omethoate in citrus pulp, dry to replace the previous estimations.

The Meeting estimated a maximum residue level of 0.3 mg/kg for dimethoate and 0.15 mg/kg for omethoate in wheat bran to replace the previous estimations.

The Meeting estimated a maximum residue level of 0.2 mg/kg for dimethoate and 0.06 mg/kg for omethoate in wheat germ to replace the previous estimations.

Residues in processed food commodities

For estimating dietary exposure from toxic equivalent residues in processed food commodities, the Meeting applied the processing factors for dimethoate and omethoate to the levels of dimethoate and omethoate in the individual trials then scaled the individual omethoate residues for "potency" (based on the ratio of the dimethoate to omethoate HBGVs) and summed the resulting values from each trial to obtain a data set of toxic equivalent residues for estimating STMR-P and HR-P values.

Table 5 Calculated dimethoate toxic equivalent residue STMR-Ps. $STMR_{acute}$ and HR-Ps for processed food commodities

RAC	Processing factors [a]		Residues (mg/kg) [b]		Toxic equivalent residues [c] (mg teq/kg)	
	Dimethoate	Omethoate	Dimethoate Median-P	Omethoate Median-P	$STMR\text{-}P_{chronic}$	$STMR\text{-}P_{acute}$
Orange	median=0.615	median=0.0035				
Juice	0.14	0.2	0.086	0.0007	0.088	0.093
Oil	0.2	0.07	0.12	0.000245	0.12	0.13
Molasses	5.8	5.9	3.6	0.021	3.6	3.8
Wheat grain	median=0.004	median=0.0027				
Wheat bran	4.4	3.5	0.018	0.009	0.041	0.11
Wheat germ	2.9	2.0	0.012	0.0053	0.025	0.065
Wholemeal flour	0.66	0.5	0.0026	0.0013	0.006	0.016
White flour	0.21	0.5	0.00084	0.0013	0.0042	0.014

Notes:

[a] The ratios of the residue in the processed item divided by the residue in the Raw Agricultural Commodity.

[b] Sum of calculated [dimethoate+10×omethoate] residues in the processed commodity.

[c] Sum of calculated [dimethoate+2.5×omethoate] residues in the processed commodity.

Residues in processed feed commodities

For estimating residues in processed feed commodities, the Meeting applied the processing factors for dimethoate and omethoate to the levels of dimethoate and omethoate in the individual trials and summed the resulting values to obtain a data set of total residues for estimating median-P and highest-P values.

Table 6 Calculated median-Ps for total residues (dimethoate+omethoate) in processed feed commodities

RAC	Processing factors [a]		Total residues (mg/kg)		
	Dimethoate	Omethoate	Dimethoate [b]	Omethoate [b]	Median-P
Orange + mandarin (whole fruit)	0.645	0.0045			
Citrus pulp, dry	2.1	1.6	1.35	0.0072	1.36

Notes:

[a] The ratios of the residue in the processed item divided by the residue in the Raw Agricultural Commodity.

[b] Each value is the sum of calculated [dimethoate+omethoate] residues in the processed commodity.

Residues in animal commodities

Farm animal dietary burden

Farm animal feeding studies in lactating cattle and laying hens were evaluated by the 2019 JMPR. In the lactating cow study, no residues of dimethoate were found above the LOQ (0.001 mg/kg) in milk, muscle, liver or kidney from any dose group ((1, 3.4, 10 and 33 ppm in the diet).

Low levels of omethoate were measured in milk and cattle tissues from the 33 ppm dose group and in cattle liver from the 10 ppm dose group. No residues of omethoate were found above the LOQ (0.001 mg/kg) in any of the egg or tissue samples from any dose group.

For fat, there were some low level residues of dimethoate and to a lesser extent of omethoate, without a consistent relationship between dose and residue level. The depuration data showed that clearance was rapid, with no detections above the LOQ.

Table 7 Residues of dimethoate and omethoate in milk and tissues from lactating cows dosed with the equivalent of 1, 3.4, 10.1 or 33.2 ppm dimethoate daily for 28 days

Matrix	Dose (ppm)	Dimethoate residues (mg/kg)			Omethoate residues (mg/kg)		
		Values [a]	mean	Max [b]	Values [a]	mean	Max [b]
Milk (28d)	3.4	< 0.001 (3)	< 0.001	< 0.001	< 0.001 (3)	< 0.001	< 0.001
	10.1	< 0.001 (3)	< 0.001	< 0.001	< 0.001 (3)	< 0.001	< 0.001
	33.2	< 0.001 (3)	< 0.001	< 0.001	0.0188, 0.001465, 0.0054, 0.0125, 0.013, 0.01355	0.011	0.0189
Muscle	3.4	< 0.001 (9)	< 0.001	< 0.001	< 0.001 (9)	< 0.001	< 0.001
	33.2	< 0.001 (9)	< 0.001	< 0.001	Loin: 0.00495, 0.0021, < 0.001 Flank: 0.005, 0.002, < 0.001 Round: 0.00485, 0.00195, < 0.001	0.0025	0.0051
Liver	3.4	< 0.001 (3)	< 0.001	< 0.001	< 0.001 (3)	< 0.001	< 0.001
	10.1	< 0.001 (3)	< 0.001	< 0.001	0.0018, 0.00135, 0.00105	0.0014	0.0018
	33.2	< 0.001 (3)	< 0.001	< 0.001	0.004, 0.0056, 0.0039	0.00455	0.0059
Kidney	3.4	< 0.001 (3)	< 0.001	< 0.001	< 0.001 (3)	< 0.001	< 0.001
	10.1	< 0.001 (3)	< 0.001	< 0.001	< 0.001 (3)	< 0.001	< 0.001
	33.2	< 0.001 (3)	< 0.001	< 0.001	0.0047, < 0.001, < 0.001	0.0019	0.0047
Fat	1.0	O: 0.0023, < 0.001, < 0.001 P: 0.0269, 0.0046, < 0.001 S: 0.00255, < 0.001, < 0.001	0.0014(3)	0.0026(3)	O: < 0.001 (3) P: < 0.001 (3) S: < 0.001 (3)	< 0.001	< 0.001
	3.4	O: 0.00255, < 0.001, < 0.001 P: 0.0014, < 0.001, < 0.001 S: 0.0019, 0.00185, < 0.001	0.0014	0.0026	O: < 0.001 (3) P: < 0.001 (3) S: < 0.001 (3)	< 0.001	< 0.001
	10.1	O: 0.0175, < 0.001, < 0.001 P: 0.00275, < 0.001, < 0.001 S: 0.0099, < 0.001, < 0.001	0.00125(3)	0.0028(3)	O: < 0.001 (3) P: < 0.001 (3) S: < 0.001 (3)	< 0.001	< 0.001
	33.2	O: 0.0054, 0.0016, < 0.001 P: 0.004, 0.0019, < 0.001 S: 0.0019, < 0.001, < 0.001	0.002	0.0055	O: 0.00125, < 0.001, < 0.001 P: 0.00215, < 0.001, < 0.001 S: 0.004, < 0.001, < 0.001	0.001	0.004

Notes:

P = perirenal, S = subcutaneous, O = omental.

[a] Mean values from duplicate analyses.

[b] Highest individual result.

In the laying hen study, the 2019 JMPR reported that no residues of dimethoate or omethoate were found above the LOQ in any of the egg or tissue samples from animals in any dose group (0.15, 0.4, 1.2 and 4 ppm in the diet).

Livestock dietary burden

Dietary burden calculations for cattle and poultry are provided below. The dietary burdens were estimated using the 2018 OECD Feed diets listed in Appendix XIV Electronic attachments to the 2016 edition of the FAO manual.

Table 8 Summary of livestock dietary burden (ppm dimethoate+omethoate)

	United States-Canada		European Unions		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.42	0.16	3.6	0.71	17.6❶	3.0❷	0.0022	0.0022
Dairy cattle	3.7	0.76	3.8	0.93	11.0❸	2.3❹	0.0009	0.0009
Broiler hens	0.007	0.01	0.029	0.029	0.082	0.082	0.0009	0.0009
Laying hens	0.007	0.0067	1.8❺	0.33❻	0.082	0.082	-	-

❶ Highest maximum dietary burden for beef cattle suitable for estimation of MRLs for mammalian meat and offal.

❷ Highest mean dietary burden for beef cattle suitable for estimation of STMRs for mammalian meat and offal.

❸ Highest maximum dietary burden for dairy cattle suitable for estimation of MRLs for milk.

❹Highest mean dietary burden for dairy cattle suitable for estimation of STMRs for milk.

❺ Highest maximum dietary burden for broiler and layer poultry suitable for estimation of MRLs for poultry meat, offal and eggs.

❻ Highest mean dietary burden for broiler and layer poultry suitable for estimation of STMRs for poultry meat, offal and eggs.

Animal commodity maximum residue levels

Cattle

For estimating maximum residue levels in mammalian commodities, the maximum dietary burden for beef cattle was 17.6 ppm and for dairy cattle was 11 ppm. Mean dietary burdens were 3.0 ppm for beef cattle and 2.3 ppm for dairy cattle.

For dimethoate, the Meeting noted that in the cattle feeding study, residues of dimethoate were all < 0.001 mg/kg in milk, muscle, liver and kidney from animals in all dose groups (up to 33 ppm, about 2–3 times the maximum dietary burdens), and the Meeting estimated maximum residue levels of 0.001(*) mg/kg for dimethoate in mammalian meat, edible offal mammalian and milks.

For mammalian fat, there was no clear relationship between the administered dose and measured residues of dimethoate in fat. As a conservative estimate, the Meeting agreed to use the highest residue found in the feeding study (0.027 mg/kg) and the overall mean residue (0.003 mg/kg) to estimate a maximum residue level and assess dietary exposure.

The Meeting estimated a maximum residue level of 0.03 mg/kg for dimethoate in mammalian fat

Since dimethoate residues in muscle, liver, kidney and milk were all < 0.001 mg/kg in all dose groups (up to 33 ppm, about 2–3 times the maximum dietary burdens), the Meeting estimated dimethoate STMRs and HRs of 0 mg/kg for mammalian meat, liver, kidney and milk and for mammalian fat, the Meeting estimated an HR of 0.027 mg/kg and an STMR of 0.003 mg/kg.

For omethoate, the Meeting agreed to interpolate the results from the 10.1 ppm and 33.2 ppm feed levels to estimate maximum residue levels and HRs in tissues (at a maximum dietary burden of 17.6 ppm) and in milk (at a maximum dietary burden of 11 ppm) and median residues were extrapolated from the 3.4 ppm feed level.

Table 9 Omethoate highest and median residues in mammalian commodities

	Feed level (ppm) for milk	Residues (m/kg) in milk	Feed level (ppm) for tissues	Residues (mg/kg) in			
				Muscle	Liver	Kidney	Fat
HR and MRL (beef or dairy cattle)							
Feeding study	10.1 33.2	< 0.001 0.011	10.1 33.2	< 0.001 [a] 0.0051	< 0.001 0.0059	< 0.001 0.0047	- -
Dietary burden and highest residue	11	0.0014	17.6	0.003	0.0031	0.0022	0.027 [b]
Median residue (beef or dairy cattle)							
Feeding study	3.4	< 0.001	3.4	< 0.001	< 0.001	< 0.001	-
Dietary burden and highest residue	2.3	< 0.001	3.0	< 0.001	< 0.001	< 0.001	0.003 [b]

Notes:

[a] Residue is in muscle from animals in the 3.4 ppm dose group.

[b] The highest and overall mean values from all dose groups in the feeding study.

Highest residues of omethoate were 0.0014 mg/kg in milk, 0.0031 mg/kg in liver, 0.0022 mg/kg in kidney, 0.003 mg/kg in muscle and 0.002 mg/kg in fat.

The Meeting estimated maximum residue levels of 0.0015 mg/kg for omethoate in milk, 0.005 mg/kg in mammalian meat and edible offal (based on residues in liver) and 0.003 mg/kg in mammalian fat.

The Meeting estimated median omethoate residues of 0 mg/kg in kidney and fat (based on residues < 0.001 mg/kg in dose groups 3-fold higher than the mean dietary burden) and 0.001 mg/kg in milk, muscle and liver.

For assessing long-term dietary exposure from animal commodities, the toxic equivalent $median_{chronic}$ residues were 0.0025 mg teq/kg for milk, liver and mammalian meat, 0.003 mg teq/kg for mammalian fat and 0 mg teq/kg for kidney.

For assessing short-term dietary exposure from animal commodities, the toxic equivalent highest residues were 0.03 mg teq/kg for mammalian meat, 0.031 mg teq/kg for liver, 0.022 mg teq/kg for kidney and 0.047 mg teq/kg for mammalian fat and the toxic equivalent median residue for milk is 0.01 mg/kg.

Poultry

The maximum dietary burden for poultry for both meat and egg production was 1.8 ppm, while the highest mean dietary burden was 0.33 ppm.

In a poultry feeding study, when hens were fed dimethoate daily for 28 days at 0.15, 0.4, 1.2 or 4 ppm in the diet, the JMPR 2019 reported that no residues of dimethoate or omethoate were found above the LOQ (0.001 mg/kg) in any of the egg or tissue samples from any dose group.

The Meeting therefore estimated maximum residue levels of 0.001(*) mg/kg, HRs and STMRs of 0 mg/kg for both dimethoate and omethoate in poulty meat, poultry fats, poultry, edible offal of, and eggs

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL for plant and animal commodities: *Dimethoate and omethoate (measured and reported separately)*

Definition of the residue for dietary risk assessment for plant and animal commodities: Sum of dimethoate plus 2.5× omethoate for long-term dietary exposure and the sum of dimethoate plus 10× omethoate for acute dietary exposure.

The residue is not fat-soluble.

Table 10 Maximum residue levels and dietary intake–Dimethoate (see also omethoate)

CCN	Commodity	Recommended Maximum residue level (mg/kg)		$STMR_{chronic}$ or $STMR\text{-}P_{chronic}$ mg/kg	$STMR_{acute}$ or $STMR\text{-}P_{acute}$ mg/kg	HR or HR-P mg/kg
		New	Previous			
FC 0003	Mandarins (subgroup)	2		0.081	0.16	0.19
FC 0004	Oranges (subgroup)	2#		0.31	0.32	0.4
FI 0236	Avocado	2		0.11	0.37	0.49
VB 0402	Brussels sprouts	0.1		0.086	0.23	0.25
VO 0448	Tomato	0.01(*)		0.0175	0.055	0.055
VP 0544	Yard-long bean (pods)	0.07		0.175	0.55	0.55
VD 2065	Dry beans (subgroup) except soya bean	0.7		0.175	0.55	
SO 0495	Rape seed	0.15		0.0775	0.23	
GC 0654	Wheat	0.06		0.011	0.032	
CF 0654	Wheat bran, processed	0.26		0.041	0.11	
CF 1210	Wheat germ	0.17		0.025	0.065	
MO 0105	Edible offal (Mammalian)	0.001(*)		0.0025 (liver)		0.031 (liver)
MF 0100	Mammalian fats (except milk fats)	0.03		0.003		0.047
MM 0095	Meat (from mammals other than marine mammals)	0.001(*)		0.0025 (muscle) 0.003 (fat		0.03 (muscle 0.047 (fat)
ML 0106	Milks	0.001(*)		0.0025	0.01	
PE 0112	Eggs	0.001(*)		0		0
PF 0111	Poultry fats	0.001(*)		0		0
PM 0110	Poultry meat	0.001(*)		0		0
PO 0111	Poultry, Edible offal of	0.001(*)		0		0
	Orange juice			0.088	0.093	
	Orange oil			0.12	0.12	
	Orange molasses			3.6	3.8	
	Wheat Wholemeal flour			0.006	0.016	
	Wheat White flour			0.0042	0.14	
AS 0654	Wheat, hay and/or straw (straw)	4 dw		0.06 dw		2.7 dw
AB 0001	Citrus pulp, dry [FEED]	4.2		1.36		
	Wheat grain (feed)			0.008 median		
	Dry beans (feed)			0.1 median		
	Tomato pomace (feed)			0.01 median		
	Wheat forage			0.76 fw median		4.4 fw

Notes:

STMR(-P)chronic	Expressed as toxic equivalent residues (dimethoate + 2.5×omethoate)
STMR(-P)acute	Expressed as toxic equivalent residues (dimethoate + 10×omethoate)
HR	Expressed as toxic equivalent residues (dimethoate + 10×omethoate)
Median	median total residue (sum of dimethoate and omethoate) for livestock dietary burden estimation
fw	fresh weight
dw	dry weight

On the basis of the information provided to the JMPR it was concluded that the estimated acute dietary exposure to residues of dimethoate and omethoate for the consumption of commodities in the subgroup of oranges may present a public health concern

Table 11 Maximum residue levels and dietary intake_Omethoate (from the use of dimethoate)

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous	See Dimethoate	See Dimethoate
FC 0003	Mandarins (subgroup	0.02			
FC 0004	Oranges (subgroup)	0.02			
FI 0236	Avocado	0.15			
VB 0402	Brussels sprouts	0.03			
VO 0448	Tomato	0.01			
VP 0544	Yard-long bean (pods)	0.05			
VD 2065	Dry beans (subgroup) except soya bean	0.08			
SO 0495	Rape seed	0.03			
GC 0654	Wheat	0.03			
CF 0654	Wheat bran, processed	0.105			
CF 1210	Wheat germ	0.06			
AS 0654	Wheat hay and/or straw (straw)	0.3 dw			
AB 0001	Citrus pulp, dry	0.032			
MO 0105	Edible offal (Mammalian)	0.005			
MF 0100	Mammalian fats (except milk fats)	0.003			
MM 0095	Meat (from mammals other than marine mammals)	0.005			
ML 0106	Milks	0.0015			
ML 0106	Milks	0.001(*)			
PE 0112	Eggs	0.001(*)			
PF 0111	Poultry fats	0.001(*)			
PM 0110	Poultry meat	0.001(*)			
PO 0111	Poultry, Edible offal of	0.001(*)			

DIETARY RISK ASSESSMENT

The Meeting considered how to best approach the dietary risk assessment of mixed residues of dimethoate and omethoate and decided that an appropriately conservative approach would be to sum the dimethoate and omethoate residues after first scaling the omethoate residues to account for the differences in toxicity. The relevant factors for chronic and short-term intake were derived from the ratios of the dimethoate and omethoate maximum ADI and acute RfD values and are 2.5 and 10 respectively. Dietary intake estimates for the combined adjusted residues were compared with the dimethoate maximum ADI and ARfD.

Long-term dietary exposure

The ADI for dimethoate is 0–0.001 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for dimethoate (including omethoate) were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 10–100 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of dimethoate (including omethoate) from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for dimethoate is 0.02 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for dimethoate (including omethoate) were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs varied from 0–120 percent of the ARfD for children and 0–70 percent of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of dimethoate (including omethoate) from uses considered by the present Meeting is unlikely to present a public health concern except oranges (120 percent for Australian children).

EMAMECTIN BENZOATE (247)

First draft prepared by D Poflotski, Australian Pesticides and Veterinary Medicines Authority, Armidale, Australia

EXPLANATION

Emamectin benzoate is a foliar insecticide derivative of abamectin, which is isolated from fermentation of Streptomyces avermitilis, a naturally occurring soil actinomycete. It acts by stimulating the release of γ-aminobutyric acid, an inhibitory neurotransmitter, thus causing insect paralysis within hours of ingestion, and subsequent insect death 2–4 days later. It is also registered for use as a veterinary drug in the treatment of sea lice (Siphonostomatoida) infestations in salmon and trout in several countries.

Emamectin benzoate was considered for the first time for toxicology and residues by the 2011 JMPR. An ADI of 0–0.0005 mg/kg bw and ARfD of 0.02 mg/kg bw have been established.

The residue definition for emamectin benzoate is:

Definition of the residue for compliance with the MRL or for estimation of the dietary intake for plant and animal commodities: emamectin B1a benzoate.

The residue not fat soluble.

Emamectin benzoate was scheduled at the Fifty-second Session of the CCPR for the evaluation of additional MRLs in the 2022 JMPR.

The current Meeting received information on analytical methodology, storage stability and additional supervised residues trials on basil, chives, coffee, flowerhead brassica vegetables, leafy vegetables (including brassica leafy vegetables), soya bean and tea.

RESIDUE ANALYSIS

Analytical methods

The Meeting received details of analytical methods including validation data for the determination of emamectin B1a benzoate (B1a, NOA426007), emamectin B1b benzoate (B1b, NOA422390) and the isomers/metabolites; 8,9-ZMa (8,9-ZMa, NOA438376), L'649 (AB1a, NOA438309), L'599 (MFB1a, NOA415692) and L'831 (FAB1a, NOA415693)) in foodstuffs of plant and animal origin. In addition, the Meeting received information on analytical methods for the determination of emamectin B1a benzoate, emamectin B1b benzoate and its avermectin-like metabolites in foodstuffs of plant and animal origin as used in the various study reports (supervised residue trials and storage stability). The analytical methods are summarised below.

Table 1 Summary of the analytical methods for emamectin B1a benzoate, emamectin B1b benzoate and the avermectin-like metabolites

Author, Year, Report ID *(Method ID)*	Matrix	Analytes	Extraction	Clean-up	Separation Analysis/LOQ [a)]
Crook, 2006a, MK244/0484, (*Method RAM* 465/01) (Considered by the 2011 JMPR)	Almonds Almond hulls Apples, Beans with pods, Bean vines (fresh), Broccoli, Cauliflower, Cabbage,	Emamectin B1a benzoate (B1a), Emamectin B1b benzoate (B1b), 8,9-ZMa, AB1a, MFB1a, and FAB1a	Methanol	SPE clean-up for FAB1a	HPLC-MS/MS (internal standard quantification) LOQ: 0.001 mg/kg

Author, Year, Report ID *(Method ID)*	Matrix	Analytes	Extraction	Clean-up	Separation Analysis/LOQ [a)]
	Cucumbers Lettuce leaves, Melons, Oil seed rape seeds, Peaches/nectarines, Pears, Pecan, Potatoes, Sugar snap peas with pods (fresh), Sweet peppers, Tomatoes, Winter wheat grain, and wheat straw.				
Alves, 2020b, S19-23176, (*Method RAM 465/02 -Validation*)	Coffee beans, Grapes, Potatoes, Tomatoes	Emamectin B1a benzoate (B1a), Emamectin B1b benzoate (B1b), Coffee beans only 8,9-ZMa, AB1a, MFB1a, and FAB1a	Methanol	SPE clean-up for FAB1a	HPLC-MS/MS (internal standard quantification) LOQ: 0.001 mg/kg
Melquiades, 2021, S21-06372, (*Method RAM 465/02–Validation*)	Coffee beans, Roasted coffee, Instant coffee				
Tessier and Braid, 2013, (*Method No. GRM004.06A*)	Broad beans (dry), Lettuce, Oranges, Tobacco (dry leaves), Walnuts, Wheat (grain)	Emamectin B1a benzoate (B1a), Emamectin B1b benzoate (B1b), 8,9-ZMa, AB1a, MFB1a, and FAB1a	Acetonitrile + QuE citrate extraction mixture	-	HPLC-MS/MS (internal standard quantification) LOQ: 0.001 mg/kg
Sayed, 2020, S20-05346, (*Method GRM004.06A–Validation*)	Apricot, Cotton, Melon, Zucchini				
Garrigue, 2019, BPL 19-0017, (*QuEChERS Method (Plant) EN 15662:2009-2 Validation*)	Broad beans (dry), Orange (whole fruit), Lettuce, Tea (black, dry), Tobacco (dry leaves), Walnut, Wheat (grain)	Emamectin B1a benzoate (B1a), Emamectin B1b benzoate (B1b)	Acetonitrile + QuEChERS extraction mixture	SPE clean-up	HPLC-MS/MS (internal standard quantification) LOQ: 0.001 mg/kg
Homazava, 2019, 20190183, (*QuEChERS Method (Plant) EN 15662:2008–Validation (ILV)*)	Lettuce, Orange, Tea, Tobacco (dry leaves)				
Mechelke, 2021, 20200259, (*QuEChERS Method (Animal) DIN EN 15662:2018 Validation*)	Muscle (cattle), Liver (cattle), Kidney (cattle), Fat (cattle), Milk (cattle) Eggs (poultry)	Emamectin B1a benzoate (B1a), Emamectin B1b benzoate (B1b)	Acetonitrile + QuEChERS extraction mixture	SPE clean-up	HPLC-MS/MS (internal standard quantification) LOQ: 0.001 mg/kg
Baumy, 2021, RNB20-00065, *(QuEChERS Method (Animal) DIN EN 15662:2018 Validation ILV)*)	Liver (cattle), Fat (cattle)				

Notes:

[a] Defined by the lowest limit of method validation.

Method RAM 465/01

The analytical method is suitable for the determination of emamectin benzoate and its avermectin-like residues in apples, beans with pods and bean vines (fresh), broccoli, cauliflower, cabbage, cucumbers, lettuce leaves, melons, oil seed rape seeds, peaches/nectarines, pears, potatoes, sugar snap peas with pods (fresh), sweet peppers, tomatoes, winter wheat grain, and wheat straw. A modification also allows for analysis in almond and pecan nutmeat and almond hulls. Description and validation of RAM 465/01 and its modifications was previously considered by the JMPR 2011 evaluation.

The method involves extraction of residues of emamectin benzoate from crops by homogenisation with methanol. Extracts are centrifuged and aliquots diluted with ultra-pure water. Sample clean-up for FAB1a is by solid phase extraction (SPE) using OasisTM HLB cartridges. Final determination of all analytes is by high performance liquid chromatography (HPLC) using a two-column switching method with triple quadrupole mass spectrometric detection (LC-LC-MS/MS, positive ion spray). The LOQ is 0.001 mg/kg for each analyte.

Additional validation results were available from the 2008 supervised trials in basil and chives (Samoil, 2017, IR-4 PR No. 07137) demonstrating that RAM 465/01 is suitable for the determination of residues of emamectin benzoate (B1a and B1b) and the metabolites 8,9-ZMa, AB1a, MFB1a and FAB1a in basil and chives fresh and dried samples.

The working method was validated in fresh basil at 0.001 mg/kg for B1a, B1b, 8,9-ZMa, AB1a and MFB1a and at 0.002 mg/kg for FAB1a (lowest level of method validation, LLMV). In dry chive samples the working method was validated at 0.001 mg/kg residue level for B1a, B1b, 8,9-ZMa, AB1a and MFB1a and at 0.010 mg/kg for FAB1a (lowest level of method validation, LLMV). Additional higher validation levels for fresh and dry samples ranged from 0.005 mg/kg to 0.300 mg/kg, depending on the analyte.

Table 2 Validation recoveries of emamectin benzoate and its metabolites in basil (fresh) and chives (dry)

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Range [percent]	Mean	RSD [percent]
Basil (fresh leaves and stems)	B1a	3	0.001	99-113	104	8
		3	0.005	91-96	94	3
		3	0.150	102-112	105	4
		3	0.300	103-124	113	9
	B1b	3	0.001	102-117	108	7
		3	0.005	94-105	101	6
		3	0.050	101-128	111	14
	8,9-ZMa	3	0.001	92-95	94	2
		3	0.005	106-113	110	3
		3	0.150	99-112	104	7
	AB1a	3	0.001	83-88	86	3
		3	0.005	83-104	92	12
		3	0.150	100-105	102	3
	MFB1a	4	0.001	87-127	99	19
		3	0.005	94-128	109	16
		3	0.150	94-102	99	4
	FAB1a	3	0.002	71-75	73	3
		3	0.005	78-97	87	11
		3	0.150	84-95	89	6
Chives (dry leaves)	B1a	3	0.001	94-96	95	1
		3	0.005	86-102	94	9
		3	0.150	87-95	91	4
	B1b	3	0.001	92-103	96	7

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Range [percent]	Mean	RSD [percent]
		3	0.005	89-95	92	3
		3	0.050	76-85	79	6
	8,9-ZMa	3	0.001	92-98	96	3
		3	0.005	75-96	88	13
		3	0.150	77-86	83	6
	AB1a	3	0.001	83-90	87	4
		3	0.005	71-74	72	2
		3	0.150	86-99	93	7
	MFB1a	3	0.001	91-94	88	8
		3	0.005	73-74	74	1
		3	0.150	81-94	90	8
	FAB1a	3	0.010	63-77	71	10
		3	0.050	71-89	79	12
		3	0.150	91-98	95	4

Overall validation recoveries fortified at levels between 0.001 to 0.300 mg/kg averaged; 104 percent (n=12) for B1a, 107 percent (n=9) for B1b, 103 percent (n=9) for 8,9-ZMa, 93 percent (n=9) for AB1a, 83 percent (n=9) for FAB1a 102 percent (n=10) for MFB1a, from control fresh basil and 93 percent (n=9) for B1a, 89 percent (n=9) for B1b, 89 percent (n=9) for 8,9-ZMa, 84 percent (n=9) for AB1a, and 82 percent (n=9) for FAB1a and 84 percent (n=9) for MFB1a from control dry chives samples with relative standard deviations <20 percent for both matrices.

Method 465/01 is considered suitable for determination of emamectin benzoate (B1a and B1b) and its metabolites in fresh and dried basil and chives.

The supervised trials on tea (Ogiyama, 2019a, JP2018C324, Ogiyama, 2019b, JP2018C081 and Morita, 2020, JP2019C109) were analysed with a method similar to RAM 465/01. For dried tea leaves, the method involved extraction with methanol, clean-up with C_{18}, NH_2, PRS, florisil and graphite mini columns. Tea infusion samples were directly purified using C18 and PRS mini columns. Quantification was by LC-MS/MS with the LOQs at 0.001 mg/kg for each analyte.

Matrix effects were not significant (< 20 percent) for emamectin B1a benzoate in both dried green tea leaf samples and tea infusion samples, therefore matrix matched standards were not used.

For studies JP2018C324 and JP2018C081, method linearity was validated over the range of 0.125 to 5 pg/mL for B1a, B1b and 8,9-ZMa, 0.115 to 4.6 pg/mL for AB1a, 0.222 to 8.88 pg/mL for FAB1a and 0.225 to 9 pg/mL for MFB1a. Correlation coefficients (r) were >0.99 for all analytes and transitions. For study JP2019C109, method linearity was validated over the range of 0.0625 to 5 pg/mL for B1a, B1b and 8,9-ZMa, 0.0575 to 4.61 pg/mL for AB1a, 0.111 to 8.88 pg/mL for FAB1a and 0.1125 to 9 pg/mL for MFB1a. Correlation coefficients (r) were > 0.99 for all analytes and transitions.

The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified at concentrations of 0.0009–0.001 (LOQ), 0.009–0.01, 0.02 and 0.09–0.1 mg/kg. Mean recoveries per concentration level were in a range of 79–108 percent, with acceptable RSD values within the range of 0.4–11 percent.

Additional validation recoveries from the supervised trials in tea demonstrated that the method is suitable for the determination of residues of emamectin benzoate (B1a and B1b) and the metabolites 8,9-ZMa, AB1a, MFB1a and FAB1a in dried tea leaves and tea infusion samples.

Table 3 Validation recoveries of emamectin benzoate in tea samples

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory			Study Reference
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]	
Dried green tea leaves[1]	B1a	-	-	Transition ***m/z*** 886.5 → 158.1			Transition ***m/z*** 886.5 → 81.5			JP2018C324 and JP2018C081
		5	0.001	103-108	105	1.9	105-108	106	1.4	
		5	0.01	104-105	104	0.4	103-105	104	0.7	
		5	0.02	100-101	101	0.4	100-102	101	1.0	
	B1b	-	-	Transition ***m/z*** 872.5 → 158.1			Transition ***m/z*** 872.4 → 81.6			
		5	0.001	97-103	100	2.4	98-105	101	2.9	
		5	0.01	99-103	101	1.5	100-102	101	0.8	
	8,9-ZMa	-	-	Transition ***m/z*** 886.6 → 157.6			Transition ***m/z*** 886.4 → 82.2			
		5	0.001	97-106	103	4.5	94-105	100	4.4	
		5	0.01	99-102	101	1.3	101-103	102	0.7	
	AB1a	-	-	Transition ***m/z*** 872.7 → 144.2			Transition ***m/z*** 872.4 → 67.8			
		5	0.0009	97-102	99	2.4	95-113	102	6.7	
		5	0.009	97-99	98	0.9	97-100	99	1.2	
	MFB1a	-	-	Transition ***m/z*** 914.5 → 186.1			Transition ***m/z*** 914.5 → 113.2			
		5	0.0009	84-88	86	1.9	89-100	94	4.4	
		5	0.009	80-85	83	2.6	80-85	83	2.2	
	FAB1a	-	-	Transition ***m/z*** 900.5 → 172.0			Transition ***m/z*** 900.5 → 140.0			
		5	0.0009	82-94	91	5.6	85-94	91	4.0	
		5	0.009	86-93	91	3.1	87-96	93	4.1	
Tea infusion	B1a	-	-	Transition ***m/z*** 886.5 → 158.1			Transition ***m/z*** 886.5 → 81.5			
		5	0.001	98-102	100	1.8	98-103	100	1.8	
		5	0.01	97-101	98	1.8	97-100	98	1.3	
	B1b	-	-	Transition ***m/z*** 872.5 → 158.1			Transition ***m/z*** 872.4 → 81.6			
		5	0.001	92-96	95	1.8	95-98	96	1.5	
		5	0.01	93-96	95	1.2	94-96	95	1.1	
	8,9-ZMa	-	-	Transition ***m/z*** 886.6 → 157.6			Transition ***m/z*** 886.4 → 82.2			
		5	0.001	90-94	93	1.8	88-92	90	1.8	
		5	0.01	90-93	91	1.2	90-92	91	0.9	
	AB1a	-	-	Transition ***m/z*** 872.7 → 144.2			Transition ***m/z*** 872.4 → 67.8			
		5	0.0009	88-94	91	2.6	90-100	95	5.1	
		5	0.009	90-91	91	0.6	89-94	91	2.1	
	MFB1a	-	-	Transition ***m/z*** 914.5 → 186.1			Transition ***m/z*** 914.5 → 113.2			
		5	0.0009	100-106	103	2.3	106-113	108	2.8	
		5	0.009	93-99	95	2.7	95-101	97	2.6	
	FAB1a	-	-	Transition ***m/z*** 900.5 → 172.0			Transition ***m/z*** 900.5 → 140.0			
		5	0.0009	94-106	100	5.9	99-101	100	0.8	
		5	0.009	98-101	100	1.3	100-103	102	1.5	
Tea (dry leaves)[2]	B1a	-	-	Transition ***m/z*** 886.5 → 158.1			Transition ***m/z*** 886.5 → 81.5			JP2019C109
		5	0.001	92-102	96	3.9	93-101	95	3.5	
		5	0.01	92-94	93	0.9	91-94	93	1.2	
		5	0.1	101-103	93	0.9	101-106	103	1.9	
	B1b	-	-	Transition ***m/z*** 872.5 → 158.1			Transition ***m/z*** 872.4 → 81.6			
		5	0.001	96-106	99	3.9	92-104	97	4.7	
		5	0.01	94-97	96	1.1	93-97	95	1.7	
	8,9-ZMa	-	-	Transition ***m/z*** 886.6 → 157.6			Transition ***m/z*** 886.4 → 82.2			
		5	0.001	93-102	96	3.7	94-101	96	3.2	
		5	0.01	90-93	92	1.8	91-95	92	1.6	
		5	0.1	100-103	101	1.2	100-102	101	1.1	
	AB1a	-	-	Transition ***m/z*** 872.7 → 144.2			Transition ***m/z*** 872.4 → 67.8			
		5	0.0009	91-97	93	2.5	86-99	92	5.4	
		5	0.009	91-94	92	1.5	91-95	92	1.8	
	MFB1a	-	-	Transition ***m/z*** 914.5 → 186.1			Transition ***m/z*** 914.5 → 113.2			
		5	0.0009	75-82	79	4.2	77-86	81	5.0	
		5	0.009	80-85	83	2.6	80-85	82	2.8	
		5	0.09	87-92	90	2.3	89-92	90	1.3	
	FAB1a	-	-	Transition ***m/z*** 900.5 → 172.0			Transition ***m/z*** 900.5 → 140.0			
		5	0.0009	83-103	95	8.0	86-114	101	11.2	

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory			Study Reference
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]	
		5	0.009	88-99	92	4.9	88-95	90	3.1	

Notes:

[1] Tea leaves were sampled and steamed at the conditions of 1 m/45 seconds and 60 kg/h for about 45 seconds using a conveyor belt steaming machine and dried at 80°C for 120 minutes to produce dried green tea leaves.

[2] Commercial tea sample

The method is considered suitable for determination of emamectin benzoate (B1a and B1b) and its metabolites in tea.

Method RAM 465/02

The method is suitable for determination of emamectin benzoate (B1a and B1b) in grape (fruits), potato (tubers) and tomato (fruits) and emamectin benzoate (B1a and B1b) and its avermectin-like metabolites in coffee (bean).

The analytical method involves extraction of emamectin benzoate from crops by homogenisation with methanol. Extracts are centrifuged and filtered via PTFE filter. The method was modified to include sample clean-up for FAB1a by SPE using OasisTM HLB cartridges prior to analysis. Final determination of all analytes is by HPLC with triple quadrupole mass spectrometric detection (LC-MS/MS, positive ion spray).

Table 4 Ion transitions for emamectin benzoate and its metabolites for method RAM 465/02

Analyte	Ion Transition (***m/z***)	
	Primary	Confirmatory
Emamectin B1a benzoate (B1a, NOA426007)	886.4 → 158.2	886.4 → 82.1
Emamectin B1b benzoate (B1b, NOA422390)	872.4 → 158.1	872.4 → 144.1
Emamectin isomer 8, 9-Z (8,9-ZMa, NOA438376)	886.4 → 158.1	886.4 → 82.1
Emamectin metabolite L'649 (AB1a, NOA438309)	872.4 → 158.2	872.4 → 144.1
Emamectin metabolite L'599 (MFB1a, NOA415692)	914.4 → 186.1	914.4 → 113.0
Emamectin metabolite L'831 (FAB1a, NOA415693)	900.4 → 172.1	900.4 → 140.1

Method RAM 465/02 was validated for determination of B1a and B1b in grape, tomato and potato and for B1a, B1b, 8,9-ZMa, AB1a, MFB1a and FAB1a in coffee bean (Alves, 2020b, S19-23176).

Matrix effects were evaluated during the validation. Calibration standards with matrix effect were used for the quantification of B1a and B1b in grape and tomato fruits, and potato tubers and for emamectin benzoate (B1a and B1b), 8,9-ZMa and AB1a in coffee beans. For MFB1a and FAB1a in coffee beans, standards without a matrix effect were used for quantification.

The final injection extracts showed stability at a range of 1 °C to 10 °C for a period of 10 days for tomato, 14 days for coffee bean and 9 days for grape and potato.

Method linearity was validated over the range of 0.05 to 10 ng/mL (matrix-matched calibration solutions). Correlation coefficients (r) were >0.99 for each transition.

The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified at concentrations of 0.001 mg/kg (LOQ) and 0.10 mg/kg (100× LOQ). Mean recoveries per concentration level were in a range of 74–106 percent, with acceptable RSD values within the range of 2–16 percent.

The limit of quantification (LOQ) for each analyte was demonstrated to be 0.001 mg/kg in each matrix. The limit of detection (LOD) was calculated for B1a and B1b in grape (fruit), tomato (fruit), and potato (tuber) and for all analytes in coffee beans for both the primary and confirmatory transitions were found to be 0.0002 mg/kg (<30 percent of the LOQ).

Table 5 RAM 465/02: Validation recoveries of emamectin B1a benzoate and emamectin B1b benzoate in coffee, grape, potato and tomato and their metabolites in coffee

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
Coffee (bean)	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	80.5-103	93.5	9.95	79.5-109	95.6	11.3
		5	0.100	92.4-108	98.4	7.46	90.4-108	96.0	7.93
	B1b	-	-	Transition ***m/z*** 872.4 → 158.1			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	79.5-106	93.5	10.5	82.5-110	96.8	10.9
		5	0.100	90.5-107	100	6.16	77.2-87.8	83.5	5.33
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.1			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	71.0-110	97.9	16.2	71.0-110	97.4	15.9
		5	0.100	93.2-109	100	7.21	92.7-108	99.8	6.79
	AB1a	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	80.0-108	96.1	10.7	86.0-107	97.9	9.38
		5	0.100	93.2-107	102	5.29	79.6-89.2	84.2	4.55
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 113.0		
		5	0.001	81.5-101	89.9	8.28	87.5-93.5	90.2	3.08
		5	0.100	98.9-109	106	4.37	94.5-115	103	9.01
	FAB1a	-	-	Transition ***m/z*** 900.4 → 172.1			Transition ***m/z*** 900.4 → 140.1		
		5	0.001	77.0-97.0	84.1	10.5	72.0-99.0	81.7	14.3
		5	0.100	83.8-108	94.2	9.12	83.1-95.0	89.2	5.37
Grape (fruits)	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	85.0-98.5	92.2	6.42	86.5-100	94.0	6.18
		5	0.100	98.1-108	103	5.81	94.6-112	102	6.56
	B1b	-	-	Transition ***m/z*** 872.4 → 158.1			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	98.0-106	101	3.35	94.5-102	98.5	2.61
		5	0.100	92.3-106	97.3	6.41	82.0-91.4	88.4	4.28
Potato (tubers)	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	68.5-78.5	74.0	4.97	71.5-80.5	76.6	4.29
		5	0.100	100-106	103	2.50	101-107	103	2.16
	B1b	-	-	Transition ***m/z*** 872.4 → 158.1			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	78.5-85.0	81.5	2.91	110-120	115	3.27
		5	0.100	98.1-105	101	2.51	86.2-92.2	89.1	3.03
Tomato (fruits)	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	79.5-86.0	82.4	3.11	76.0-85.0	81.9	4.37
		5	0.100	82.8-101	95.0	7.42	83.6-102	96.2	7.79
	B1b	-	-	Transition ***m/z*** 872.4 → 158.1			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	84.0-91.5	85.8	3.72	85.0-96.0	91.2	4.48
		5	0.100	85.0-101	94.3	6.34	70.2-80.8	76.3	5.44

Method RAM 465/02 was also validated for the determination of B1a, B1b, 8,9-ZMa, AB1a, MFB1a and FAB1a in coffee beans, roasted coffee and instant coffee (Melquiades, 2021, S21-06372).

The magnitude of matrix effects was considered significant (≥20 percent suppression or enhancement) for all analytes in coffee beans, roasted coffee and instant coffee.

Final matrix extracts stored in vials at a temperature of approximately 1 °C and 10 °C showed stability for the analysis of residues of B1a, B1b, 8,9-ZMa, AB1a, MFB1a and FAB1a for a storage period of 7 days for coffee beans, 9 or 10 days for roasted coffee and 9 days for instant coffee.

Method linearity was validated over the range of 0.05 to 10 ng/mL. Correlation coefficients were >0.99 for each transition.

The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified at concentrations of 0.001 mg/kg (LOQ) and 0.01 mg/kg (10× LOQ). Mean recoveries per concentration level were in a range of 83.2–116 percent, with acceptable RSD values within the range of 2–14 percent.

The LOQ for each analyte was demonstrated to be 0.001 mg/kg in each matrix. For coffee beans, roasted coffee and instant coffee the calculated LODs were 0.0003 mg/kg for each analyte and both transitions (30 percent of the LOQ).

Table 6 RAM 465/02: Recoveries of emamectin B1a benzoate and emamectin B1b benzoate and their metabolites in coffee beans, roasted coffee and instant coffee

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
Coffee beans	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	90.0-105	95.9	6.40	90.5-104	96.9	6.14
		5	0.010	96.6-107	99.8	4.16	94.4-106	100	4.45
	B1b	-	-	Transition ***m/z*** 872.4 → 158.1			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	90.5-108	99.6	6.48	78.0-108	95.9	13.1
		5	0.010	96.7-107	104	4.14	76.2-101	89.9	11.3
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.1			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	91.6-114	102	9.09	94.7-116	105	8.95
		5	0.010	92.8-105	99.4	4.39	92.7-105	100	4.63
	AB1a	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	89.5-107	100	7.05	77.0-98.5	89.4	9.98
		5	0.010	101-109	106	2.92	75.3-96.6	88.3	9.50
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 113.0		
		5	0.001	105-115	108	3.79	100-114	107	5.20
		5	0.010	109-118	116	3.27	113-119	116	2.17
	FAB1a	-	-	Transition ***m/z*** 900.4 → 172.1			Transition ***m/z*** 900.4 → 140.1		
		5	0.001	101-112	107	4.34	96.0-116	105	7.28
		5	0.010	107-119	113	5.18	104-120	113	5.11
Roasted coffee	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	92.0-101	97.6	3.58	91.5-100	97.1	3.65
		5	0.010	93.2-102	97.5	3.49	93.3-103	97.5	3.92
	B1b	-	-	Transition ***m/z*** 872.4 → 158.1			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	92.5-103	96.6	3.91	86.5-101	95.3	5.58
		5	0.010	94.7-100	96.9	2.14	83.3-94.9	90.1	5.30
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.1			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	97.2-105	99.9	3.27	98.2-104	101	2.37
		5	0.010	96.2-105	101	3.39	98.2-105	101	2.73
	AB1a	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	94.0-101	97.4	2.84	87.0-102	94.8	6.81
		5	0.010	93.9-99.8	96.7	2.78	87.8-94.7	91.1	3.06
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 113.0		
		5	0.001	83.5-102	90.8	8.20	85.0-105	94.6	7.58
		5	0.010	101-115	109	4.69	89.5-116	106	11.6
	FAB1a	-	-	Transition ***m/z*** 900.4 → 172.1			Transition ***m/z*** 900.4 → 140.1		
		5	0.001	71.5-94.0	83.2	12.9	73.0-104	85.7	14.0
		5	0.010	87.6-106	98.6	8.45	77.2-112	98.9	13.9
Instant coffee	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	89.5-105	97.3	6.02	90.5-100	95.9	3.87
		5	0.010	92.3-102	96.6	3.95	92.4-101	96.6	3.66
	B1b	-	-	Transition ***m/z*** 872.4 → 158.1			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	88.0-94.5	90.1	2.87	96.5-107	101	4.55
		5	0.010	97.3-104	100	2.71	87.0-95.9	91.7	3.79

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.1			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	92.6-98.2	95.3	2.19	92.6-100	95.2	3.46
		5	0.010	92.8-104	98.7	4.10	97.3-106	101	4.17
	AB1a	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 144.1		
		5	0.001	90.5-98.5	95.4	3.28	87.5-96.5	90.8	4.22
		5	0.010	95.8-106	101	3.69	90.0-97.6	93.0	4.40
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 113.0		
		5	0.001	92.5-110	98.4	7.11	80.5-103	95.9	9.25
		5	0.010	101-118	106	6.72	108-115	110	9.41
	FAB1a	-	-	Transition ***m/z*** 900.4 → 172.1			Transition ***m/z*** 900.4 → 140.1		
		5	0.001	82.5-110	97.3	12.1	78.5-105	89.8	13.1
		5	0.010	98.9-115	109	8.12	94.9-117	108	10.9

Method 465/02 is considered suitable for determination of emamectin benzoate (B1a and B1b) in grape (fruits), potato (tubers) and tomato (fruits) and emamectin benzoate (B1a and B1b) and its avermectin-like metabolites in coffee (bean), roasted coffee and instant coffee.

Method GRM004.06A

The method is suitable for the determination of B1a, B1b, 8,9-ZMa, AB1a, MFB1a and FAB1a in broad bean (dry), lettuce, orange, tobacco (dry leaves), walnut, and wheat (grain) (Tessier and Braid, 2013).

The analytical method involves extraction of emamectin benzoate from crops with acetonitrile by shaking with QuEChERS salts. The extract was centrifuged, and an aliquot diluted with ultra-pure water (50/50, v/v).

For dry crops such as tobacco (dry leaves), walnuts and dried broad beans, a sub sample is wetted with ultra-pure water and allowed to soak before extraction. The extract is centrifuged, and an aliquot evaporated to dryness. The extract is re-suspended in acetonitrile and diluted with ultra-pure water (50/50, v/v).

Final determination is by HPLC with triple quadrupole mass spectrometric detection (LC-MS/MS).

Table 7 Ion transitions for emamectin benzoate and its metabolites for method GRM004.06A

Analyte	Ion Transition (***m/z***)	
	Primary	Confirmatory
Emamectin B1a benzoate (B1a, NOA426007)	886.4 → 158.2	886.4 → 82.1
Emamectin B1b benzoate (B1b, NOA422390)	872.4 → 158.2	872.4 → 82.0
Emamectin isomer 8, 9-Z (8,9-ZMa, NOA438376)	886.4 → 158.1	886.4 → 82.1
Emamectin metabolite L'649 (AB1a, NOA438309)	872.4 → 144.1	872.4 → 68.1
Emamectin metabolite L'599 (MFB1a, NOA415692)	914.4 → 186.1	914.4 → 154.1
Emamectin metabolite L'831 (FAB1a, NOA415693)	900.4 → 140.0	900.4 → 112.1

Method GRM006.06A was validated for determination of B1a, B1b, 8,9-ZMa, AB1a, MFB1a and FAB1a in broad bean (dry), lettuce, orange, tobacco (dry leaves), walnut, and wheat (grain). Summary validation data from Tessier, 2013, S12-03679 was provided within the analytical method report.

The magnitude of the matrix effects was insignificant (<20 percent suppression or enhancement) in lettuce, orange, wheat grain and dried broad beans and non-matrix matched standards should be used for calibration. In walnuts, the magnitude of the matrix effects was insignificant for B1a and AB1a but was

significant (>20 percent suppression or enhancement) for B1b, 8,9-ZMa, MFB1a and FAB1a and matrix-matched standards should normally be used for calibration if analysing for these analytes. The magnitude of the matrix effects was considered significant for all analytes in tobacco and matrix-matched standards should normally be used for calibration.

Method linearity was validated over the range of 0.025 to 5 ng/mL for lettuce and 0.05 to 10 ng/mL for broad bean (dry), orange, tobacco (dry leaves), walnut, and wheat (grain). Correlation coefficients (r) were >0.99 for all matrices and transitions.

The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified at concentrations of 0.001 mg/kg (LOQ) and 1 mg/kg (1000× LOQ). Mean recoveries per concentration level were in a range of 70–100 percent, with acceptable RSD values within the range of < 0.5–19 percent.

The LOQ for each analyte was demonstrated to be 0.001 mg/kg in each matrix. The calculated LODs (3× background noise) for all analytes in all validated commodities were less than or equivalent to 0.0003 mg/kg (≤ 30 percent of the LOQ) for both transitions.

Table 8 GRM004.06A: Recoveries of emamectin B1a benzoate and emamectin B1b benzoate and their metabolites in broad bean (dry), lettuce, orange, tobacco (dry leaves), walnuts and wheat (grain)

Matrix	Analyte	No. of tests	Spiking level	Primary			Confirmatory		
			(mg/kg)	Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
Broad bean (dry)	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	69-78	73	5	67-74	71	4
		5	0.01	77-83	80	3	78-85	81	3
	B1b	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 82.0		
		5	0.001	69-77	72	5	63-74	70	6
		5	0.01	79-86	83	3	79-86	83	4
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	65-73	70	4	81-92	87	5
		5	0.01	75-84	80	5	76-86	81	5
	AB1a	-	-	Transition ***m/z*** 872.4 → 144.1			Transition ***m/z*** 872.4 → 68.1		
		5	0.001	82-95	88	7	79-88	84	5
		5	0.01	75-84	79	6	75-82	80	4
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 154.1		
		5	0.001	66-80	72	8	66-75	71	5
		5	0.01	72-81	77	4	70-80	75	5
	FAB1a	-	-	Transition ***m/z*** 900.4 → 140.0			Transition ***m/z*** 900.4 → 112.1		
		5	0.001	69-82	75	8	88-112	98	9
		5	0.01	83-89	87	3	78-82	80	2
Lettuce	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	90-96	93	3	93-99	96	3
		5	0.01	86-90	88	2	85-91	89	3
	B1b	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 82.0		
		5	0.001	95-101	97	2	92-100	96	3
		5	0.01	88-94	90	3	90-94	92	2
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	93-101	97	3	93-100	97	3
		5	0.01	86-91	88	3	87-91	89	2
	AB1a	-	-	Transition ***m/z*** 872.4 → 144.1			Transition ***m/z*** 872.4 → 68.1		
		5	0.001	91-102	97	4	83-101	94	7
		5	0.01	85-94	89	4	86-91	87	2
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 154.1		
		5	0.001	93-101	97	4	86-100	93	6
		5	0.01	82-90	85	4	80-89	85	4
	FAB1a	-	-	Transition ***m/z*** 900.4 → 140.0			Transition ***m/z*** 900.4 → 112.1		
		5	0.001	92-120	100	11	73-107	91	14

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
		5	0.01	93-107	98	6	84-102	94	7
Orange	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	84-91	88	3	85-93	89	3
		5	0.01	93-94	93	0	91-95	93	2
	B1b	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 82.0		
		5	0.001	84-92	88	4	84-90	88	3
		5	0.01	89-93	91	2	90-95	92	2
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	84-93	89	4	82-90	88	4
		5	0.01	89-95	92	3	91-97	94	2
	AB1a	-	-	Transition ***m/z*** 872.4 → 144.1			Transition ***m/z*** 872.4 → 68.1		
		5	0.001	77-99	92	10	87-96	90	4
		5	0.01	93-99	95	2	93-97	95	2
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 1541		
		5	0.001	84-98	91	6	89-98	93	4
		5	0.01	85-91	89	3	84-91	89	3
	FAB1a	-	-	Transition ***m/z*** 900.4 → 140.0			Transition ***m/z*** 900.4 → 112.1		
		5	0.001	81-114	96	13	79-103	91	10
		5	0.01	97-104	100	3	92-106	99	7
Tobacco (dry leaves)	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	66-101	88	15	73-98	91	11
		5	0.01	93-101	98	3	96-100	98	2
	B1b	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 82.0		
		5	0.001	76-99	92	10	76-101	93	11
		5	0.01	92-102	96	4	94-100	98	2
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	72-100	90	12	76-100	94	11
		5	0.01	93-100	98	3	90-101	97	4
	AB1a	-	-	Transition ***m/z*** 872.4 → 144.1			Transition ***m/z*** 872.4 → 68.1		
		5	0.001	65-106	90	17	76-104	94	11
		5	0.01	93-103	98	4	93-102	98	4
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 154.1		
		5	0.001	77-107	94	14	72-93	85	11
		5	0.01	93-103	99	4	94-105	97	5
	FAB1a	-	-	Transition ***m/z*** 900.4 → 140.0			Transition ***m/z*** 900.4 → 112.1		
		5	0.001	79-99	85	10	78-96	86	10
		5	0.01	79-96	88	9	92-101	95	5
Walnut	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	86-94	90	3	90-94	92	2
		5	0.01	88-90	89	1	89-93	91	2
	B1b	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 82.0		
		5	0.001	93-99	96	3	93-100	97	3
		5	0.01	91-94	93	2	91-94	92	2
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	89-97	92	3	90-96	92	3
		5	0.01	88-92	90	2	87-91	89	2
	AB1a	-	-	Transition ***m/z*** 872.4 → 144.1			Transition ***m/z*** 872.4 → 68.1		
		5	0.001	79-93	85	6	82-91	87	4
		5	0.01	88-89	89	1	86-90	88	2
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 154.1		
		5	0.001	77-88	81	6	84-100	92	7
		5	0.01	77-82	78	3	78-82	80	2
	FAB1a	-	-	Transition ***m/z*** 900.4 → 140.0			Transition ***m/z*** 900.4 → 112.1		
		5	0.001	65-89	75	13	55-91	72	19
		5	0.01	81.87	84	3	75-87	81	6
Wheat (grain)	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	87-92	90	2	85-91	88	3
		5	0.01	87-94	91	3	89-95	93	2

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
	B1b	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 82.0		
		5	0.001	84-93	91	4	89-95	92	2
		5	0.01	88-93	91	3	88-93	90	2
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	90-93	91	1	86-92	90	3
		5	0.01	88-94	92	3	90-97	93	3
	AB1a	-	-	Transition ***m/z*** 872.4 → 144.1			Transition ***m/z*** 872.4 → 68.1		
		5	0.001	82-90	86	3	86-93	89	3
		5	0.01	87-94	90	3	88-93	91	2
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 154.1		
		5	0.001	81-93	88	6	86-93	89	3
		5	0.01	89-95	91	3	90-97	92	3
	FAB1a	-	-	Transition ***m/z*** 900.4 → 140.0			Transition ***m/z*** 900.4 → 112.1		
		5	0.001	74-97	85	11	65-106	91	17
		5	0.01	72-98	86	12	78-94	84	8

Method GRM004.06A was also validated for the determination of B1a, B1b, 8,9-ZMa, AB1a, MFB1a and FAB1a in apricot, cotton, melon and zucchini (Sayed, 2020, S20-05346).

Matrix effects on detector response caused by apricot (for B1a, B1b, AB1a and MFB1a), melon (for B1a, AB1a and FAB1a) and zucchini (for B1b, AB1a and 8,9-ZMa) were considered insignificant, nevertheless matrix matched standards were used for quantification. Matrix effects caused by apricot (for 8,9-ZMa and FAB1a), melon (for B1b, 8,9-ZMa and MFB1a), zucchini (for B1a, MFB1a and FAB1a) and cotton (for all analytes) were considered significant and therefore matrix matched standards were used for quantification.

The final injection extracts of emamectin benzoate (B1a and B1b), 8,9-ZMa, AB1a, MFB1a and FAB1a showed stability at 4°C for a period of 11 days for apricots and zucchini and 9 days for melons and cotton.

Method linearity was validated over the range of 0.05 to 5 ng/mL for apricot, melon and zucchini and 0.05 to 10 ng/mL for cotton (matrix-matched calibration solutions). Correlation coefficients were >0.99 for each transition.

The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified at concentrations of 0.001 mg/kg (LOQ) and 0.01 mg/kg (10× LOQ). Mean recoveries per concentration level were in a range of 70–111 percent, with acceptable RSD values within the range of 1–20 percent.

The LOQ for each analyte was demonstrated to be 0.001 mg/kg in each matrix. The calculated LODs (3× background noise) for all analytes in all validated commodities were less than 0.0003 mg/kg (<30 percent of the LOQ) for both transitions.

Table 9 RAM 465/02: Validation recoveries of emamectin B1a benzoate and emamectin B1b benzoate in apricot, melon, zucchini and cotton

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
Apricot	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	86-95	91	4	88-96	90	4
		5	0.01	85-89	86	2	82-90	87	4

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
	B1b	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 82.0		
		5	0.001	87-93	91	3	88-95	91	3
		5	0.01	85-90	87	2	88-92	89	2
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	81-85	84	2	85-90	87	2
		5	0.01	76-80	79	2	80-83	81	1
	AB1a	-	-	Transition ***m/z*** 872.4 → 144.1			Transition ***m/z*** 872.4 → 68.1		
		5	0.001	87-101	94	5	92-102	97	4
		5	0.01	86-91	89	2	86-94	91	3
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 154.1		
		5	0.001	77-98	87	10	79-93	84	7
		5	0.01	64-92	81	14	72-120	101	20
	FAB1a	-	-	Transition ***m/z*** 900.4 → 140.0			Transition ***m/z*** 900.4 → 112.1		
		5	0.001	74-110	92	16	84-117	96	16
		5	0.×	70-82	76	7	66-79	74	7
Melon	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	98-105	101	3	100-105	102	2
		5	0.01	104-113	108	4	108-116	111	3
	B1b	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 82.0		
		5	0.001	90-102	96	5	95-105	100	4
		5	0.01	100-104	102	2	103-108	106	2
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	95-102	99	3	94-104	101	3
		5	0.01	98-106	103	3	103-115	109	4
	AB1a	-	-	Transition ***m/z*** 872.4 → 144.1			Transition ***m/z*** 872.4 → 68.1		
		5	0.001	101-117	107	6	103-112	107	4
		5	0.01	98-116	105	7	97-112	104	6
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 154.1		
		5	0.001	86-95	91	4	89-102	95	5
		5	0.01	97-107	102	4	93-108	102	6
	FAB1a	-	-	Transition ***m/z*** 900.4 → 140.0			Transition ***m/z*** 900.4 → 112.1		
		5	0.001	78-113	96	13	70-75	73	3
		5	0.01	90-114	107	9	76-87	80	6
Zucchini	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	71-87	82	8	72-91	84	9
		5	0.01	81-90	86	4	83-92	87	4
	B1b	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 82.0		
		5	0.001	67-84	80	9	68-83	80	8
		5	0.01	80-87	83	4	80-87	83	4
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	70-88	82	9	68-88	82	10
		5	0.01	77-91	84	7	78-89	83	5
	AB1a	-	-	Transition ***m/z*** 872.4 → 144.1			Transition ***m/z*** 872.4 → 68.1		
		5	0.001	73-88	83	7	73-85	81	6
		5	0.01	81-90	85	5	81-90	86	4
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 154.1		
		5	0.001	62-84	72	11	60-105	84	19
		5	0.01	67-103	81	17	80-98	89	9
	FAB1a	-	-	Transition ***m/z*** 900.4 → 140.0			Transition ***m/z*** 900.4 → 112.1		
		5	0.001	77-107	90	15	90-117	104	11
		5	0.01	83-120	107	14	67-100	80	18
Cotton	B1a	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		
		5	0.001	89-97	94	3	91-98	93	3
		5	0.01	90-102	96	5	93-104	99	5
	B1b	-	-	Transition ***m/z*** 872.4 → 158.2			Transition ***m/z*** 872.4 → 82.0		
		5	0.001	88-94	91	3	90-93	92	1
		5	0.01	95-100	97	2	93-97	95	2
	8,9-ZMa	-	-	Transition ***m/z*** 886.4 → 158.2			Transition ***m/z*** 886.4 → 82.1		

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
		5	0.001	90-98	94	4	89-93	91	2
		5	0.01	94-103	98	4	95-98	96	1
	AB1a	-	-	Transition ***m/z*** 872.4 → 144.1			Transition ***m/z*** 872.4 → 68.1		
		5	0.001	88-107	96	9	86-104	93	8
		5	0.01	91-107	100	6	91-108	98	7
	MFB1a	-	-	Transition ***m/z*** 914.4 → 186.1			Transition ***m/z*** 914.4 → 154.1		
		5	0.001	85-91	88	3	88-99	95	4
		5	0.01	95-103	100	3	91-103	96	5
	FAB1a	-	-	Transition ***m/z*** 900.4 → 140.0			Transition ***m/z*** 900.4 → 112.1		
		5	0.001	79-112	92	14	76-99	86	10
		5	0.01	93-108	99	6	76-102	88	12

Method GRM004.06A is suitable for the determination emamectin benzoate and its metabolites in apricot, broad bean (dry), cotton, lettuce, melon, orange, tobacco (dry leaves), walnut, and wheat (grain) and zucchini.

QuEChERS method (foods of plant origin) (enforcement)

The multi-residue QuEChERS method (EN 15662:2009-2) was validated for the determination of residues of emamectin benzoate (B1a and B1b) in broad beans (dry), orange (whole fruit), lettuce, tea (black, dry), tobacco (dried leaves), walnut, and wheat (grain) (Garrigue, 2019, BPL19-0017).

Samples are extracted by agitation with ultra-pure water and acetonitrile (50/50, v/v) followed by addition of magnesium sulphate, sodium chloride, sodium citrate dibasic sesquihydrate and sodium citrate tribasic dihydrate and further agitation. After centrifugation, an aliquot is purified via SPE clean-up. After further centrifugation, an aliquot is diluted with ultra-pure water (50/50, v/v) prior to quantification by HPLC with tandem mass spectrometric detection (LC-MS/MS, positive ion spray).

Table 10 Ion transitions for emamectin benzoate (B1a and B1b) for QuEChERS method

Analyte	Ion Transition (***m/z***)	
	Primary	Confirmatory
Emamectin B1a benzoate (B1a, NOA426007)	886.5 → 158	886.4 → 82
Emamectin B1b benzoate (B1b, NOA422390)	872.4 → 158	872.4 → 82

Matrix effects on detector response caused by broad beans (dry), lettuce, walnut and wheat (grain) matrices for emamectin B1a and emamectin B1b were considered insignificant (< 20 percent) and therefore solvent standards were used for quantification. Matrix effects on detector response caused by orange (whole fruit), black tea and tobacco matrices for emamectin B1a and emamectin B1b were considered significant and therefore matrix matched standards were used for quantification.

The final injection extracts of B1a and B1b showed stability at a range of 2–14 °C for a period of 14 days in broad bean (dry) and wheat (grain), 11 days for tobacco (dry leaves), 10 days for lettuce, 9 days for tea (black, dried) and 8 days in orange (whole fruit) except for B1b in walnut and dried broad bean where the mean recoveries were below 70 percent (69 percent in walnut and 66 percent in dried broad bean) although the difference from the original analysis was within ± 20 percent of the initial values. The report recommended that final extracts for B1b in walnut and broad bean (dry) are analysed within 24 hours after extraction.

The linearity of the detector response was confirmed by injecting at least five solvent or matrix matched standards covering the working range of 0.025 to 2 ng/mL. The lower margin of the linearity test was 25 percent of the LOQ and the upper margin was at least 20 percent above the highest concentration in the final extracts. Correlation coefficients were > 0.99 for each transition.

The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified at concentrations of 0.001 mg/kg (LOQ) and 0.01 mg/kg (10× LOQ). Mean recoveries per concentration level were in a range of 75–110 percent, with acceptable RSD values within the range of 1–10 percent.

The LOQ was found to be 0.001 mg/kg for both analytes in all validated matrices. The LOD for both analytes was calculated (3× background) for both the primary and confirmatory transitions in all validated matrices and was less than 0.0003 mg/kg (< 30 percent of the LOQ).

Table 11 QuEChERS method: Recoveries of emamectin B1a benzoate and emamectin B1b benzoate in broad bean (dry), lettuce, orange, tea (black, dried), tobacco (dry leaves), walnuts and wheat (grain)

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
Broad bean (dry)	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	73-90	78	9.0	73-91	78	9.5
		5	0.01	82-84	83	1.3	79-82	81	1.6
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		
		5	0.001	71-87	76	8.3	67-86	75	9.6
		5	0.01	78-81	79	1.8	76-79	77	1.8
Lettuce	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	79-84	81	2.7	78-83	80	2.7
		5	0.01	83-90	85	3.7	83-89	85	3.2
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		
		5	0.001	82-87	85	1.8	83-89	86	2.5
		5	0.01	86-94	89	3.6	87-92	89	2.7
Orange (whole fruit)	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	80-101	87	9.5	81-100	87	9.6
		5	0.01	79-85	84	2.8	79-84	83	2.8
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		
		5	0.001	85-97	90	5.1	84-91	89	3.3
		5	0.01	82-90	88	3.9	82-91	88	4.1
Tea (black, dried)	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	92-96	94	1.6	85-92	88	3.6
		5	0.01	95-111	101	6.8	90-108	100	6.9
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		
		5	0.001	99-108	104	3.6	103-117	110	4.7
		5	0.01	100-120	110	7.9	101-120	108	6.9
Tobacco (dry leaves)	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	90-97	93	3.0	81-98	88	7.8
		5	0.01	99-103	101	1.7	98-107	101	3.5
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		
		5	0.001	97-103	101	2.4	87-99	95	4.8
		5	0.01	105-111	106	2.5	99-109	104	3.6
Walnut	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	81-85	83	2.3	79-86	81	3.2
		5	0.01	81-85	83	1.7	80-86	83	2.6
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		
		5	0.001	82-88	84	2.8	82-88	85	3.4
		5	0.01	80-85	83	2.5	80-86	83	2.5
Wheat (grain)	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	76-79	78	1.9	77-81	78	1.9
		5	0.01	92-97	95	2.1	92-97	95	2.2

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		
		5	0.001	77-82	81	2.7	82-88	84	3.1
		5	0.01	94-100	97	2.4	95-97	96	1.0

An independent laboratory validation (ILV) for the QuEChERS method (EN 15662:2008) was conducted in lettuce, orange, tea and tobacco crop matrices (Homazava, 2019, 20190183).

Significant matrix effects on the LC-MS/MS detector response were observed in tobacco (for B1a and B1b) and tea (for B1b only). Therefore, all sample extracts were evaluated with multi-point calibrations based on matrix-matched calibration standards for both analytes in these two matrices. For lettuce and orange, the matrix effects were found to be insignificant and therefore all sample extracts were evaluated with multi-point calibrations based on solvent calibration standards for both analytes in these two matrices.

The linearity of the LC-MS/MS detector response was confirmed by injecting eight solvent or matrix matched standard solutions, covering a working range of 0.05 ng/mL to 10 ng/mL (equivalent to 0.00025–0.05 mg/kg). The lower margin of the linear range was at least 30 percent of the LOQ and the upper margin was higher by at least 20 percent above the highest concentrations in the final extracts. Correlation coefficients were > 0.99 for each transition.

The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified at concentrations of 0.001 mg/kg (LOQ) for lettuce, orange, tea and tobacco and 0.02 mg/kg (20× LOQ) for tea (black, dried), 0.05 mg/kg (50× LOQ) for orange (whole fruit) and 1.0 mg/kg (1000× LOQ) for lettuce and tobacco (dry leaves). Mean recoveries per concentration level were in a range of 83–107 percent, with acceptable RSD values within the range of 0.4–4 percent.

The LOQ was found to be 0.001 mg/kg for both analytes in all validated matrices. The LOD for both analytes was calculated (3× background) for both the primary and confirmatory transitions in all validated matrices and was less than 0.0003 mg/kg (<30 percent of the LOQ).

Table 12 QuEChERS method: ILV recoveries of emamectin B1a benzoate and emamectin B1b benzoate in lettuce, orange, tea (black, dried) and tobacco (dry leaves)

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
Lettuce	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	97-101	99	1.4	98-100	99	1.1
		5	1.0	89-99	98	0.6	98-100	98	0.9
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		
		5	0.001	97-99	98	0.8	97-98	97	0.4
		5	1.0	99-100	99	0.4	98-101	99	1.2
Orange (whole fruit)	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	105-109	107	1.4	103-108	106	1.9
		5	0.05	107-110	107	1.1	105-109	107	1.4
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		
		5	0.001	100-103	101	1.5	98-102	100	1.4
		5	0.05	107-109	108	0.9	106-109	107	1.0
Tea (black, dried)	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	83-88	86	1.9	84-92	87	3.3
		5	0.02	83-84	83	0.4	83-84	83	0.5
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
		5	0.001	80-86	83	3.2	81-85	83	2.3
		5	0.02	83-85	84	1.1	83-84	84	0.8
Tobacco (dry leaves)	B1a	-	-	Transition ***m/z*** 886.5 → 158			Transition ***m/z*** 886.5 → 82		
		5	0.001	89-94	91	2.4	91-99	94	3.8
		5	1.0	92-97	94	2.1	92-96	94	1.8
	B1b	-	-	Transition ***m/z*** 872.4 → 158			Transition ***m/z*** 872.4 → 82		
		5	0.001	91-96	93	2.1	89-98	92	3.8
		5	1.0	92-97	94	2.0	91-98	94	2.3

The QuEChERS enforcement method for plant commodities is considered suitable for determination of emamectin (B1a and B1b).

QuEChERS method (foods of animal origin) (enforcement)

The multi-residue QuEChERS method (DIN EN 15662:2018), first developed for foods of plant origin, was validated for the determination of residues of emamectin benzoate (B1a and B1b) in animal matrices muscle (cattle), liver (cattle), kidney (cattle), fat (cattle), milk (cattle) and eggs (poultry)) (Mechelke, 2021, 20200259).

Samples are extracted by agitation with ultra-pure water and acetonitrile (50/50, v/v) followed by addition of magnesium sulfate, sodium chloride, trisodium citrate dihydrate and disodium hydrogen citrate and further agitation. After centrifugation, an aliquot of the upper acetonitrile layer is purified via SPE clean-up (primary and secondary amine + magnesium sulphate). After further agitation and centrifugation, an aliquot transferred to an auto sampler vial. A small amount of 5 percent formic acid in acetonitrile is added. An aliquot thereof is diluted in water (50/50, v/v) prior to quantification by HPLC with triple quadrupole mass spectrometric detection (LC-MS/MS, positive ion spray).

Table 13 Ion transitions for emamectin benzoate (B1a and B1b) for QuEChERS method

Analyte	Ion Transition (***m/z***)	
	Primary	Confirmatory
Emamectin B1a benzoate (B1a, NOA426007)	886 → 158	886 → 82
Emamectin B1b benzoate (B1b, NOA422390)	872 → 158	872 → 82

Significant matrix effects on the LC-MS/MS detector response were observed in muscle (for B1a and B1b) and milk and fat (for B1b only). Therefore, all sample extracts were evaluated with multi-point calibrations based on matrix-matched calibration standards for both analytes in these three matrices. For liver and kidney (cattle) matrix effects were found to be insignificant. Nevertheless, all sample extracts were evaluated with multi-point calibrations based on matrix-matched calibration standards for both analytes in these two matrices. For eggs, matrix effects were found to be insignificant (<20 percent suppression or enhancement). Therefore, all sample extracts were evaluated with multi-point calibrations based on solvent calibration standards for both analytes in eggs.

The final injection extracts of B1a and B1b showed stability at a range of 2–8 °C for a period of 7–8 days for all validated matrices.

The linearity of the detector response was confirmed by injecting at least five solvent or matrix matched standards covering the working range of 0.03 to 5 ng/mL in milk (cattle) and 0.03 to 2.5 ng/mL in muscle (cattle), liver (cattle), kidney (cattle), fat (cattle) and eggs. The lower margin of the linearity test

was 30 percent of the LOQ and the upper margin was at least 20 percent above the highest concentration in the final extracts. Correlation coefficients were > 0.99 for each transition.

The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified at concentrations of 0.001 mg/kg (LOQ) and 0.01 mg/kg (10× LOQ). Mean recoveries per concentration level were in a range of 77–102 percent, with acceptable RSD values within the range of 1–9 percent.

The LOQ was found to be 0.001 mg/kg for both analytes in all validated matrices. The LOD for both analytes was calculated (3× background) for both the primary and confirmatory transitions in all validated matrices and was less than 0.0002 mg/kg (< 30 percent of the LOQ).

Table 14 QuEChERS method: Recoveries of emamectin B1a benzoate and emamectin B1b benzoate in muscle (cattle), liver (cattle), kidney (cattle), fat (cattle), milk (cattle) and eggs

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
Muscle (cattle)	B1a	-	-	Transition ***m/z*** 886 → 158			Transition ***m/z*** 886 → 82		
		5	0.001	91-100	95	3.5	86-94	88	3.7
		5	0.01	85-94	89	3.6	85-91	89	2.6
	B1b	-	-	Transition ***m/z*** 872 → 158			Transition ***m/z*** 872 → 82		
		5	0.001	86-103	93	8.3	90-102	98	5.2
		5	0.01	84-90	86	2.9	85-95	89	4.2
Liver (cattle)	B1a	-	-	Transition ***m/z*** 886 → 158			Transition ***m/z*** 886 → 82		
		5	0.001	83-94	90	5.8	88-99	92	5.3
		5	0.01	86-91	89	2.2	82-92	87	4.4
	B1b	-	-	Transition ***m/z*** 872 → 158			Transition ***m/z*** 872 → 82		
		5	0.001	95-102	99	2.7	81-100	93	8.9
		5	0.01	90-93	92	1.0	86-91	90	2.3
Kidney (cattle)	B1a	-	-	Transition ***m/z*** 886 → 158			Transition ***m/z*** 886 → 82		
		5	0.001	85-94	90	4.6	78-90	85	5.5
		5	0.01	96-103	100	3.0	94-102	99	3.1
	B1b	-	-	Transition ***m/z*** 872 → 158			Transition ***m/z*** 872 → 82		
		5	0.001	83-97	88	6.3	86-97	92	5.3
		5	0.01	97-105	102	3.1	97-104	101	2.7
Fat (cattle)	B1a	-	-	Transition ***m/z*** 886 → 158			Transition ***m/z*** 886 → 82		
		5	0.001	91-104	99	5.0	85-94	90	4.4
		5	0.01	92-99	95	3.5	92-100	95	3.6
	B1b	-	-	Transition ***m/z*** 872 → 158			Transition ***m/z*** 872 → 82		
		5	0.001	90-103	95	5.1	89-97	95	3.5
		5	0.01	90-99	94	4.6	91-101	95	4.5
Milk (cattle)	B1a	-	-	Transition ***m/z*** 886 → 158			Transition ***m/z*** 886 → 82		
		5	0.001	93-107	99	6.0	92-108	101	8.1
		5	0.01	97-105	102	3.6	96-106	102	4.4
	B1b	-	-	Transition ***m/z*** 872 → 158			Transition ***m/z*** 872 → 82		
		5	0.001	92-111	101	7.0	94-108	100	5.7
		5	0.01	97-103	99	2.8	96-105	101	3.5
Eggs	B1a	-	-	Transition ***m/z*** 886 → 158			Transition ***m/z*** 886 → 82		
		5	0.001	92-110	99	8.0	86-102	93	6.8
		5	0.01	88-96	93	3.6	89-95	93	2.7
	B1b	-	-	Transition ***m/z*** 872 → 158			Transition ***m/z*** 872 → 82		
		5	0.001	69-85	77	7.5	68-85	79	8.6
		5	0.01	73-79	77	3.5	77-83	78	3.0

An independent laboratory validation (ILV) for the QuEChERS method was conducted in cattle fat and liver (Baumy, 2021, RNB20-00065).

Significant matrix effects on detector response caused by liver (for B1a only) were observed, therefore matrix matched standards were used for quantification. Matrix effects on detector response caused by liver (for B1b only) and fat (for B1a and B1b) were considered insignificant, nevertheless matrix matched standards were used for quantification.

The linearity of the LC-MS/MS detector response was confirmed by injecting seven matrix matched standard solutions, covering a working range of 0.03 ng/mL to 5 ng/mL (equivalent to 0.0003–0.05 mg/kg). The lower margin of the linear range was at least 30 percent of the LOQ and the upper margin was higher by at least 20 percent above the highest concentrations in the final extracts. Correlation coefficients were > 0.99 for each transition.

The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified at concentrations of 0.001 mg/kg (LOQ) in both matrices and 0.2 mg/kg (200× LOQ) for fat and 0.8 mg/kg (800× LOQ) for liver. Mean recoveries per concentration level were in a range of 77–104 percent, with acceptable RSD values within the range of 1–14 percent.

The LOQ was found to be 0.001 mg/kg for both analytes in both validated matrices. The LOD for both analytes was calculated (3× background) for both the primary and confirmatory transitions in both validated matrices and was less than 0.0003 mg/kg (< 30 percent of the LOQ).

Table 15 QuEChERS method: ILV recoveries of emamectin B1a benzoate and emamectin B1b benzoate in cattle fat and liver

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Primary			Confirmatory		
				Range [percent]	Mean	RSD [percent]	Range [percent]	Mean	RSD [percent]
Liver (cattle)	B1a	-	-	Transition ***m/z*** 886.0 → 158.0			Transition ***m/z*** 886.0 → 82.0		
		5	0.001	95-115	104	8.1	87-101	94	7.3
		5	0.8	74-90	79	7.7	74-88	79	7.0
	B1b	-	-	Transition ***m/z*** 872.0 → 158.0			Transition ***m/z*** 872.0 → 82.0		
		5	0.001	84-104	92	7.7	80-107	93	13.5
		5	0.8	75-88	80	6.4	73-85	77	6.0
Fat (cattle)	B1a	-	-	Transition ***m/z*** 886.0 → 158.0			Transition ***m/z*** 886.0 → 82.0		
		5	0.001	83-90	88	2.8	85-93	88	3.1
		5	0.2	87-83	90	2.4	88-93	90	2.0
	B1b	-	-	Transition ***m/z*** 872.0 → 158.0			Transition ***m/z*** 872.0 → 82.0		
		5	0.001	89-97	93	3.4	94-100	97	3.1
		5	0.2	96-100	98	1.4	97-100	98	1.2

The QuEChERs enforcement method for animal commodities is considered suitable for determination of emamectin (B1a and B1b).

Stability of pesticide residues in stored analytical samples

The 2011 JMPR considered storage stability in numerous crops and concluded that emamectin B1a benzoate and emamectin B1b benzoate were stable when stored at -20 °C or lower for at least 27 months (804 days) in plant commodities with high water content (tomatoes and green beans with pods), at least 18 months (545 days) in plant commodities with high starch content (potatoes), and at least 9 months (273 days) in plant commodities with high oil content (cottonseed), and special plant commodities (cotton gin trash). Avermectin-like metabolites 8,9-ZMa, AB1a, MFB1a, and FAB1a were stable when stored frozen at -20 ˚C or lower for at least 18 months in plant commodities with high water content (tomatoes and green beans with pods), at least 18 months commodities with high starch content (potatoes), while 8,9-

ZMa was stable for at least 6 months in commodities with high oil content (cottonseed), and special commodities (cotton gin trash).

A new study was conducted to evaluate the freezer storage stability of the residues of emamectin benzoate and its metabolites in frozen orange (whole fruit) (Ford, 2018, CEMR-7499). Homogenised samples of whole oranges were fortified with 0.1 mg/kg of standard solution containing B1a, B1b, 8,9-ZMa, AB1a, MFB1a or FAB1a and stored at -18 °C for 3, 6, 9, 12, 18 and 24 months until analysis. The samples were analysed using procedures described in residue analytical method GRM004.06A. Residues of emamectin benzoate and its metabolites are stable in whole orange when stored deep frozen at <-18 °C for up to 24 months with no significant decrease (> 30 percent as compared to the zero-time value) in the observed residue levels after frozen storage for up to 24 months.

Table 16 Stability of emamectin benzoate in orange (whole fruit) following storage at <-18 °C

Matrix	Analyte	Fortification level (mg/kg)	Days of storage	Percent concurrent recovery (mean)	Percent stored recovery (mean)	Percent remaining (mean)
Orange (whole fruit)	B1a	0.10	0	89.6, 87.8, 89.5, 89.5, 86.8 (88.6)	-	-
			98	101, 102 (102)	85.6, 90.1 (87.8)	99
			190	96.0, 95.5 (95.8)	85.2, 82.6 (83.9)	95
			278	99.8, 96.3 (98.0)	82.6, 85.2 (83.9)	95
			371	95.1, 97.5 (96.3)	79.9, 83.2 (81.6)	92
			560	104, 105 (104)	108, 96.2 (102)	115
			756	85.0, 78.8 (81.9)	79.5, 73.7 (76.6)	86
	B1b	0.10	0	90.2, 88.0, 84.5, 90.2, 80.9 (86.8)	-	-
			98	98.0, 98.5 (98.3)	83.4, 80.0 (81.7)	94
			190	83.7, 85.9 (84.8)	85.1, 81.3 (83.2)	96
			278	97.9, 94.4 (96.1)	90.8, 95.6 (93.2)	107
			370	90.0, 89.9 (89.9)	84.8, 77.8 (81.3)	94
			550	93.9, 109 (102)	79.9, 85.1 (82.5)	95
			756	77.7, 80.2 (79.0)	92.8, 95.1 (63.9)	74
	8,9-ZMa	0.10	0	101, 102, 102, 96.8, 98.7 (100)	-	-
			80	98.4, 97.5 (97.9)	87.6, 87.4 (87.5)	87
			168	92.6, 95.0 (93.8)	82.7, 80.8 (81.7)	82
			266	104, 102 (103)	91.5, 94.2 (92.9)	93
			360	107, 111 (108)	95.3, 90.2 (92.7)	93
			553	94.5, (95.2 (94.9)	87.6, 88.5 (88.0)	88
			730	81.6, 86.0 (83.8)	86.7, 84.5 (85.6)	86
	AB1a	0.10	0	104, 97.8, 108, 110, 108 (106)	-	-
			98	98.8, 99.2 (99.0)	81.6, 89.4 (85.5)	81
			196	104, 103 (104)	90.7, 91.8 (91.2)	86
			277	96.5, 104 (100)	91.5, 86.4 (88.9)	84
			369	99.1, 99.8 (99.5)	91.2, 97.1 (94.1)	89
			549	89.2, 88.9 (89.0)	83.3, 77.2 (80.3)	76
			756	86.0, 86.7 (86.3)	71.6, 75.9 (73.8)	70
	MFB1a	0.10	0	90.7, 88.5, 86.6, 86.0, 80.0 (86.4)	-	-
			98	90.5, 96.7 (93.6)	79.1, 76.6 (77.9)	90
			196	92.3, 89.7 (91.0)	78.7, 76.7 (77.7)	90
			279	92.6, 94.0 (93.3)	85.0, 80.0 (82.5)	95
			370	90.2, 90.0 (90.1)	93.0, 91.9 (92.5)	107
			549	94.1, 101 (97.7)	78.2, 78.0 (78.1)	90
			756	87.8, 89.5 (88.7)	74.7, 80.2 (77.4)	90
	FAB1a	0.10	0	83.9, 84.7, 87.2, 87.0, 78.8 (84.3)	87.2, 87.0, 78.8 (84.3)	-
			98	95.6, 89.4 (92.5)	80.7, 81.2 (80.9)	96
			189	99.9, 101 (100)	89.8, 86.1 (88.0)	104
			279	97.3, 89.9 (93.6)	86.8, 82.4 (84.6)	100
			370	94.8, 91.9 (93.4)	90.2, 82.7 (86.4)	102
			549	85.4, 73.5 (79.4)	83.5, 71.3 (77.4)	92

Matrix	Analyte	Fortification level (mg/kg)	Days of storage	Percent concurrent recovery (mean)	Percent stored recovery (mean)	Percent remaining (mean)
			760	73.1, 70.6 (71.9)	70.7, 68.8 (69.7)	83

Notes:

Percent remaining = (Mean stored recovery/Mean 0 day recovery) × 100

Storage stability data were included in the magnitude of residue study conducted for emamectin benzoate on basil and chives (Samoil, 2017, IR-4 PR NO. 07137). The concurrent storage stabilities were found to be acceptable for emamectin B1a benzoate, emamectin B1b benzoate, 8,9-ZMa, FAB1a and MFB1a when stored at <-15 °C for approximately 56 months (1670–1672 days) for fresh basil and approximately 52 months (1548–1551 days) for dry chives. This represented approximately 102 percent of the storage interval for fresh basil trial samples (1637 days) and approximately 92 percent of the storage interval for dry chive trial samples (1659 days for B1a). The mean storage stability recoveries for AB1a (fresh and dry) < 60 percent as summarised below.

Table 17 Summary of storage stability recoveries of emamectin benzoate and its metabolites in basil and chives following storage at <-15 °C

Matrix	Analyte	Approx. Spike level (mg/kg)	Days of storage	Sample size (n)	Observed residue (mg/kg)	Percent stored spike	Percent Mean store spike
Basil (fresh leaves and stems)	B1a	0.120	1670	3	0.081, 0.093, 0.084	67, 78, 70	72
	B1b	0.005	1670	3	0.004, 0.003, 0.003	61, 61, 84	69
	8,9-ZMa	0.050	1671	4	0.042, 0.039, 0.041, 0.047	84, 79, 83, 94	85
	AB1a	0.120	1672	3	0.058, 0.081, 0.053	48, 67, 44	53
	FAB1a	0.005	1671	6	0.004, 0.003, 0.003, 0.003, 0.004, 0.004	71, 62, 63, 64, 79, 71	68
	MFB1a	0.005	1672	3	0.004, 0.005, 0.005	90, 98, 106	98
Chives (dry leaves)	B1a	0.120	1548	4	0.094, 0.085, 0.093, 0.093	78, 71, 77, 77	76
	B1b	0.005	1551	3	0.004, 0.003, 0.003	71, 70, 64	68
	8,9-ZMa	0.050	1552	3	0.039, 0.040, 0.036	78, 81, 73	77
	AB1a	0.120	1551	3	0.075, 0.060, 0.053	62, 50, 44	52
	FAB1a	0.005	1552	3	0.004, 0.002, 0.003	83, 52, 54	63
	MFB1a	0.005	1551	1	0.004, 0.004, 0.003	70, 75, 59	68

Storage stability data were included in the magnitude of residue studies conducted for emamectin benzoate on tea (Ogiyama, 2019a, JP2018C324, Ogiyama, 2019b, JP2018C081 and Morita, 2020, JP2019C109). The data demonstrated the stability of emamectin B1a benzoate in dried tea leaves stored at <-20 °C for a time period that accommodates the storage period in the residue study.

Samples of dried green tea leaves were fortified at a nominal rate of 0.01 mg/kg and stored frozen at *ca.* -20 °C. Two stored samples were analysed following 19, 24, 35 days (Ogiyama, 2019b, JP2018C081), 38 days (Ogiyama, 2019a, JP2018C324) and 96 days (Morita, 2020, JP2019C109). At each timepoint, an untreated control and a concurrent recovery were also analysed. Residues of emamectin B1a benzoate were determined using the validated analytical tea method with an LOQ 0.001 mg/kg.

Table 18 Stability of emamectin benzoate in tea leaves following storage at <-20 °C

Matrix	Analyte	Fortification level (mg/kg)	Days of storage	percent concurrent recovery (mean)	percent stored recovery (mean)	percent remaining (mean)	Study Reference
Dried green tea leaves	B1a	0.01	0	104, 104, 105, 104, 104 (104)	-	-	Ogiyama, 2019a, JP2018C324 and Ogiyama, 2019b, JP2018C081
			19	97	101, 99 (100)	96	
			24	102	99, 97 (98)	94	
			35	102	99, 100 (100)	96	

Matrix	Analyte	Fortification level (mg/kg)	Days of storage	percent concurrent recovery (mean)	percent stored recovery (mean)	percent remaining (mean)	Study Reference
			38	102	102, 102 (102)	98	
	B1b	0.01	0	99, 101, 100, 103, 101 (101)	-	-	
			19	100	98, 99 (98)	97	
			24	102	98, 98 (98)	97	
			35	102	97, 97 (97)	96	
			38	102	98, 100 (99)	95	
	8,9-ZMa	0.01	0	99, 102, 102, 101, 100 (101)	-	-	
			19	99	102, 101 (102)	101	
			24	100	100, 100 (100)	99	
			35	101	99, 101 (100)	99	
			38	99	104, 105 (104)	103	
	AB1a	0.009	0	97, 99, 97, 98, 97 (98)		-	
			19	93	94, 96 (95)	97	
			24	96	98, 100 (99)	101	
			35	97	97, 96 (96)	98	
			38	94	97, 98 (98)	100	
	FAB1a	0.009	0	93, 86, 92, 92, 90 (91)	-	-	
			19	94	87, 89 (88)	97	
			24	100	85, 83 (84)	92	
			35	87	84, 83 (84)	92	
			38	88	81, 82 (82)	90	
	MFB1a	0.009	0	85, 82, 85, 83, 80 (83)	-	-	
			19	77	75, 78 (76)	92	
			24	86	80, 82 (81)	98	
			35	78	76, 77 (76)	92	
			38	77	71, 70 (70)	84	
Dried green tea leaves	B1a	0.01	0	93, 94, 93 (93)	-	-	Morita, 2020, JP2019C109
			96	98, 97 (98)	98, 99 (98)	105	
	B1b	0.01	0	96, 96, 96 (96)	-	-	
			96	98, 97 (98)	97, 98 (98)	105	
	8,9-ZMa	0.01	0	90, 93, 93 (92)	-	-	
			96	98, 97 (98)	99, 100 (100)	108	
	AB1a	0.009	0	91, 91, 93 (92)	-	-	
			96	96, 92 (94)	93, 94 (94)	101	
	FAB1a	0.009	0	89, 94, 99 (94)	-	-	
			96	95, 92 (94)	89, 91 (90)	97	
	MFB1a	0.009	0	85, 84, 84 (84)	-	-	
			96	83, 86 (84)	83, 81, 81 (82)	88	

Notes:

Percent remaining = (Mean stored recovery/Mean concurrent 0 day recovery) × 100.

Storage stability data were included in the magnitude of residue trials conducted for emamectin benzoate on Chinese broccoli (Thongsam, 2017, APSRDD trials 01-06). The concurrent storage stabilities were found to be acceptable for emamectin B1a benzoate when stored at <-18 °C for approximately 9 months (270 days) as summarised below.

Table 19 Summary of storage stability recoveries of emamectin B1a benzoate in Chinese broccoli following storage at <-18 °C

Matrix	Analyte	Fortification level (mg/kg)	Days of storage	percent concurrent recovery (mean)	percent stored recoveries (mean)	percent remaining (mean)	Study Reference
Chinese broccoli (Whole commodity)	B1a	0.05	0	(83)	88, 78 (83)	-	Thongsam, 2017, APSRDD trials 01-06
			7	(95)	96, 94 (94)	113	
			14	(88)	84, 78 (82)	99	
			30	(82)	80, 84 (82)	99	

Matrix	Analyte	Fortification level (mg/kg)	Days of storage	percent concurrent recovery (mean)	percent stored recoveries (mean)	percent remaining (mean)	Study Reference
			60	(80)	80, 82 (81)	98	
			90	(95)	94, 96 (95)	114	
			180	(80)	78, 82 (80)	96	
			270	(96)	90, 80 (85)	102	

Notes:

Percent remaining = (0 day percent stored Mean stored recovery/Mean 0 day stored recovery) × 100.

Concurrent recoveries from supervised residue trials

Concurrent recoveries for each study were generally acceptable as summarised below.

Table 20 Summary of concurrent recoveries of emamectin benzoate and its metabolites from the supervised trials

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Range [percent]	Mean	RSD [percent]	Report and analytical method reference
Basil (fresh leaves and stems)	B1a	7	0.001	81-134	103	17	Samoil, 2017, IR-4 PR No. 07137 RAM 465/01
		5	0.005	72-114	95	17	
		2	0.150	114	114	NA	
	B1b	7	0.001	76-112	92	16	
		6	0.005	75-99	86	11	
	8,9-ZMa	8	0.001	79-111	98	13	
		5	0.005	87-111	95	11	
		2	0.150	106-113	110	5	
	AB1a	7	0.001	70-102	78	14	
		5	0.005	64-85	75	11	
		2	0.150	97-107	102	7	
	FAB1a	6	0.002	60-116	98	20	
		3	0.005	84-119	96	21	
	MFB1a	6	0.001	87-129	112	16	
		3	0.005	103-109	107	2	
Basil (dry leaves and stems)	B1a	4	0.001	99-117	110	7	
		1	0.005	92	NA	NA	
		3	0.300	87-96	92	5	
	B1b	4	0.001	84-110	99	11	
		1	0.005	86	NA	NA	
	8,9-ZMa	4	0.001	101-110	106	5	
		2	0.005	86-95	91	7	
	AB1a	4	0.001	102-122	109	8	
		1	0.005	97	NA	NA	
	FAB1a	1	0.010	112	NA	NA	
	MFB1a	1	0.001	63	NA	NA	
Chives (fresh leaves)	B1a	4	0.001	79-132	107	25	
		4	0.005	87-107	94	10	
	B1b	4	0.001	74-83	79	6	
		4	0.005	73-87	78	8	
	8,9-ZMa	4	0.001	88-120	103	13	
		4	0.005	83-95	90	7	
	AB1a	4	0.001	82-94	89	6	
		4	0.005	65-82	73	10	
	FAB1a	4	0.002	67-101	88	17	
		2	0.005	70-107	89	30	
	MFB1a	4	0.001	77-135	98	27	
		2	0.005	93-105	99	9	
Chives	B1a	3	0.002	75-87	81	7	

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Range [percent]	Mean	RSD [percent]	Report and analytical method reference
(dry leaves)		3	0.005	86-102	94	9	
	B1b	3	0.002	75-100	97	9	
	8,9-ZMa	3	0.002	85-91	87	4	
	AB1a	3	0.002	79-83	81	3	
	FAB1a	3	0.010	69-75	71	5	
	MFB1a	3	0.001	89-118	105	14	
Chinese broccoli (whole commodity)	B1a	18	0.005	68-106	83	13	Thongsam, 2017, trials 01-06 QuEChERS (EN 15662:2008)
		18	0.050	79-105	87	6.7	
		14	0.40	89-103	97	4.6	
Coffee beans (green)	B1a	1	0.001	97.4	NA	NA	Evangelista, 2020, S19-23395
		1	0.100	95.9	NA	NA	
	B1b	1	0.001	98.4	NA	NA	
		1	0.100	101	NA	NA	RAM 465/02
	8,9-ZMa	1	0.001	96.9	NA	NA	Alves, 2020a, S19-23631-L1
		1	0.100	95.8	NA	NA	
	AB1a	1	0.001	99.4	NA	NA	
		1	0.100	101	NA	NA	RAM 465/02
	FAB1a	1	0.001	99.4	NA	NA	
		1	0.100	74.7	NA	NA	
	MFB1a	1	0.001	106	NA	NA	
		1	0.100	85.1	NA	NA	
Coffee beans (green)	B1a	1	0.001	100	NA	NA	Delongui, 2022, LBS20033
		1	0.010	84.8	NA	NA	
	B1b	1	0.001	101	NA	NA	
		1	0.010	87.1	NA	NA	RAM 465/02
	8,9-ZMa	1	0.001	107	NA	NA	
		1	0.010	90.9	NA	NA	
	AB1a	1	0.001	96.5	NA	NA	
		1	0.010	85.7	NA	NA	
	FAB1a	1	0.001	119	NA	NA	
		1	0.010	118	NA	NA	
	MFB1a	1	0.001	116	NA	NA	
		1	0.010	116	NA	NA	
Roasted coffee	B1a	1	0.001	103	NA	NA	
		1	0.010	103	NA	NA	
	B1b	1	0.001	108	NA	NA	
		1	0.010	101	NA	NA	
	8,9-ZMa	1	0.001	110	NA	NA	
		1	0.010	103	NA	NA	
	AB1a	1	0.001	106	NA	NA	
		1	0.010	106	NA	NA	
	FAB1a	1	0.001	111	NA	NA	
		1	0.010	98.7	NA	NA	
	MFB1a	1	0.001	107	NA	NA	
		1	0.010	96.5	NA	NA	
Instant coffee	B1a	1	0.001	97.3	NA	NA	
		1	0.010	101	NA	NA	
	B1b	1	0.001	89.4	NA	NA	
		1	0.010	100	NA	NA	
	8,9-ZMa	1	0.001	92.2	NA	NA	
		1	0.010	107	NA	NA	
	AB1a	1	0.001	90.9	NA	NA	
		1	0.010	98.9	NA	NA	
	FAB1a	1	0.001	109	NA	NA	
		1	0.010	109	NA	NA	
	MFB1a	1	0.001	103	NA	NA	
		1	0.010	100	NA	NA	

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Range [percent]	Mean	RSD [percent]	Report and analytical method reference
Leaf lettuce	B1a	4	0.005	60-103	77	25	Vincent, 1998, ABR-98047
		7	0.050	82-107	95	9.1	
		6	0.500	87-104	96	7.4	
		4	1.00	94-115	105	9.6	ARM 344-92-3
	B1b	4	0.027	91-107	101	7.1	
		4	0.053	102-122	111	8.8	
	AB1a	1	0.005	118	NA	NA	
		1	0.050	86	NA	NA	
	FAB1a + MFB1a	4	0.005	48-58	52	8.5	
		4	0.020	54-66	58	9.8	
		8	0.050	51-81	71	17	
Soya bean seed (dry)	B1a	7	0.001	91-107	99	5.9	Bledsoe, 2019, TK0347414
		7	0.010	93-108	101	5.2	
	B1b	7	0.001	91-102	97	4.3	
		7	0.010	94-105	99	3.7	RAM 465/02
	8,9-ZMa	7	0.001	92-105	98	4.4	
		7	0.010	93-107	99	5.5	
	AB1a	7	0.001	92-106	98	6.1	
		7	0.010	93-118	100	8.6	
	FAB1a	7	0.001	86-104	96	8.8	
		7	0.010	80-112	98	13	
	MFB1a	7	0.001	90-115	102	7.8	
		7	0.010	79-109	93	10	
Spinach (leaves)	B1a	3	0.005	66-70	68	3.0	Vincent, 1998, ABR-98047
		7	0.050	72-84	77	6.8	
		3	0.500	62-72	66	8.0	
		2	1.000	70-76	73	NA	244-92-3
	B1b	1	0.005	61	NA	NA	
		3	0.027	61-77	69	12	
		2	0.053	76-80	78	NA	
	AB1a	1	0.005	40	NA	NA	
		1	0.050	50	NA	NA	
	FAB1a + MFB1a	3	0.005	42-55	47	15	
		6	0.020	46-55	50	8.1	
		6	0.050	45-64	52	14	
Spinach (leaves)	B1a	6	0.005	72-90	82	7.4	Ediger and Oakes, 2005, T002301-03
		1	0.010	85	NA	NA	
		3	0.050	81-89	85	4.8	
		1	0.100	75	NA	NA	
		1	0.200	82	NA	NA	244-92-3 (modified)
	B1b	6	0.005	72-85	78	7.2	
		1	0.01	85	NA	NA	
		3	0.050	83-88	86	3.1	
		1	0.100	76	NA	NA	
		1	0.200	83	NA	NA	
	8,9-ZMa	6	0.005	70-102	83	15	
		1	0.01	79	NA	NA	
		3	0.050	76-122	95	25	
		1	0.100	73	NA	NA	
		1	0.200	76	NA	NA	
	AB1a	6	0.005	77-97	88	8.0	
		1	0.01	94	NA	NA	
		3	0.050	72-93	82	13	

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Range [percent]	Mean	RSD [percent]	Report and analytical method reference
		1	0.100	70	NA	NA	
		1	0.200	73	NA	NA	
	FAB1a	10	0.005	67-80	72	5.7	
		1	0.01	73	NA	NA	
		5	0.050	71-89	79	8.5	
		1	0.100	70	NA	NA	
		3	0.200	62-67	66	3.2	
	MFB1a	10	0.005	64-85	73	10	
		1	0.01	74	NA	NA	
		5	0.050	65-89	77	7.1	
		1	0.100	70	NA	NA	
		3	0.200	67-74	71	NA	
Tea (Dried green leaves)	B1a	5	0.001	98-102	100	1.8	Ogiyama, 2019a, JP2018C324 Tea method (similar to RAM 465/01)
		5	0.010	97-101	98	1.8	
	B1b	5	0.001	92-96	95	1.8	
		5	0.010	93-96	95	1.2	
	8,9-ZMa	5	0.001	90-94	93	1.8	
		5	0.010	90-93	91	1.2	
	AB1a	5	0.0009	88-94	91	2.6	
		5	0.009	90-91	91	0.6	
	FAB1a	5	0.0009	94-106	100	5.9	
		5	0.009	98-101	100	1.3	
	MFB1a	5	0.0009	100-106	103	2.3	
		5	0.009	93-99	95	1.7	
Tea (Dried green leaves)	B1a	5	0.001	103-108	105	1.9	Ogiyama 2019b JP2018C081 Tea method (similar to RAM 465/01)
		5	0.010	104-105	104	0.4	
		5	0.020	100-101	101	0.4	
	B1b	5	0.001	97-103	100	2.4	
		5	0.010	99-103	101	1.5	
	8,9-ZMa	5	0.001	97-107	103	4.5	
		5	0.010	99-102	101	1.3	
	AB1a	5	0.0009	96-102	99	2.4	
		5	0.009	97-99	98	0.9	
	FAB1a	5	0.0009	82-94	91	5.6	
		5	0.009	86-93	91	3.1	
	MFB1a	5	0.0009	84-88	86	1.9	
		5	0.009	80-85	83	2.6	
Tea (Dried leaves)	B1a	5	0.001	92-102	96	3.9	Morita, 2020, JP2019C109 Tea method (similar to RAM 465/01)
		5	0.010	92-94	93	0.9	
		5	0.100	101-103	102	0.9	
	B1b	5	0.001	96-106	99	3.9	
		5	0.010	94-97	96	1.1	
	8,9-ZMa	5	0.001	93-102	96	3.7	
		5	0.010	90-93	92	1.8	
		5	0.100	100-103	101	1.2	
	AB1a	5	0.0009	91-97	93	2.5	
		5	0.009	91-94	92	1.5	

Matrix	Analyte	No. of tests	Spiking level (mg/kg)	Range [percent]	Mean	RSD [percent]	Report and analytical method reference
		10	Overall	91-97	93	2.0	
	FAB1a	5	0.0009	83-103	95	8.0	
		5	0.009	88-99	92	4.9	
	MFB1a	5	0.0009	75-82	79	4.2	
		5	0.009	80-85	83	2.6	
		5	0.090	87-92	90	2.3	

USE PATTERN

Emamectin benzoate is registered for use in several countries for control of insects on several commodities as is indicated in the JMPR 2011 and 2014 evaluations. Additional label information on brassica head and stem vegetables, brassica leafy vegetables, herbs, leafy vegetables, soya bean and tea has been submitted to the present Meeting. Registered uses made available to this Meeting are presented below.

Table 21 Registered uses of emamectin benzoate, relevant to the present evaluation

Crop	Country	Formulation		Application					WHP / PHI (days)	Notes
		Active substance content	Type	Method	Rate (g ai/ha)	Max No.	Spray volume (L/ha)	Spray interval (days)		
Brassica Head and Stem Vegetables	United States	5 percent	SG	Foliar spray	8.4-16.8 Max. 101 g ai/ha/season	-	Ground min. 94 Aerial: 47-187	7	7	Field or greenhouse – not stated. DO NOT graze
Brassica Head and Stem Vegetables	United States	5 percent	SG	Foliar spray	8.4-16.8 Max. 67.3 g ai/ha/year	-	Ground min. 94 Aerial: 47-187	7	7	Field grown only. DO NOT graze
Brassica Leafy Greens	United States	5 percent	SG	Foliar spray	16.8 Max. 101g ai/ha/year	-	Ground min. 94 Aerial: 47-187	7	14	Field or greenhouse – not stated. DO NOT graze
Brassica Leafy Greens	United States	5 percent	SG	Foliar spray	16.8 Max. 67.3 g ai/ha/year	-	Ground min. 94 Aerial: 47-187	7	14	Field grown only. DO NOT graze
Broccoli and Cauliflower	Italy	0.95 percent	SG	Foliar spray	14.2	3	1000	7-14	3	Field grown only.
Chinese broccoli	Thailand	1.92 percent	EC	Foliar spray	14.4	2	750	7	3	-
Herbs	United States	5 percent	SG	Foliar spray	16.8 Max. 50.4 g ai/ha/year	-	Ground min. 94 Aerial: 47-187	7	7	Field grown only. DO NOT graze
Leafy vegetables (except brassica)	United States	5 percent	SG	Foliar spray	16.8 Max. 101 g ai/ha/year	-	Ground min. 94 Aerial: 47-187	7	7	Field or greenhouse – not stated. DO NOT graze
Leafy Greens	United States	5 percent	SG	Foliar spray	16.8 Max. 50.4 g ai/ha/year	-	Ground min. 94 Aerial: 47-187	7	7	-
Soya bean	United States	2.15 percent (19 g/L)	EC	Foliar spray	16.8 Max. 50.4 g	-	Min. 47	7	28	Use prohibits grazing or harvesting for

Crop	Country	Formulation		Application					WHP / PHI (days)	Notes
		Active substance content	Type	Method	Rate (g ai/ha)	Max No.	Spray volume (L/ha)	Spray interval (days)		
					ai/ha/year					livestock feed
Tea	Japan	1 percent	EC	Foliar spray	(0.5-1 g ai/hL)	1	200-400 L/10a	n/a	7 days before plucking	Dilution rate ×1000-2000

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received supervised residue trials on basil, broccoli, cauliflower, Chinese broccoli, coffee, lettuce leaf, mustard greens, soya bean, spinach, and tea.

Crop group	Subgroup	Commodity (Code)	Table No.
Bulb vegetables (009)	Green Onions (009B)	Chives (VA 2605)	Table 22
Brassica vegetables (except brassica Leafy vegetables) (010)	Flowerhead brassicas (010A, VB 0042)	Broccoli (VB 0400)	Table 23 and 24
		Cauliflower (VB 0404)	Table 25 and 26
Leafy vegetables (including brassica leafy vegetables) (013)	Leafy greens (013A)	Lettuce, Leaf (VL 0482)	Table 27
		Spinach (VL 0502)	Table 27 and 28
	Brassica Leafy vegetables (013B)	Chinese broccoli (VL 0401)	Table 29
		Mustard greens (VL 0485)	Table 30
Pulses (015)	Dry beans (015A)	Soya bean (dry) (VD 0541)	Table 31
Seed for beverages and sweets (024)	-	Coffee (SB 0716)	Table 32 and 33
Herbs (027)	Herbs (herbaceous plants) (027A)	Basil (HH 0722)	Table 34
Teas (066)	Teas from *Camellia sinensis* (066A)	Tea, green, black (fermented and dried) (DT 1114)	Table 35

All trials were well documented with field and analytical reports. The studies included method validation including recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Mean concurrent recoveries were acceptable for all studies. Dates of analysis and/or duration of sample storage were also provided. Samples were collected and stored frozen immediately or soon after sampling. Although trials included control plots, no control data are recorded in the Tables because, unless noted, residues in control samples did not exceed the LOQ. Residues are not adjusted for recoveries.

Chives

The Meeting received information on supervised residues trials for the use of emamectin benzoate on chives (Samoil, 2017, IR-4 PR No. 07137).

Four supervised residue trials on chives (4 harvest trials) were conducted in the United States in 2008. At each location, emamectin benzoate (5 percent SG) was applied as a foliar directed application to chives six times at 7(±1) day intervals.

At all sites, duplicate untreated control and treated basil samples were harvested by hand at commercial maturity, 7 days after the last treatment. A minimum of ~0.5 kg for fresh leaves were harvested at all sites.

All fresh samples were placed into a freezer <3 hours after sampling and stored frozen at <-18°C until analysis.

All samples were analysed in accordance with analytical method RAM 465/01. The LOQ was 0.001 mg/kg for B1a, B1b, 8,9-ZMa, AB1a and MFB1a and at 0.002 mg/kg for FAB1a in fresh chives and was 0.001 mg/kg for B1a, B1b, 8,9-ZMa, AB1a and MFB1a and at 0.01 mg/kg for FAB1a in dry chives. The LOD was 0.0001 mg/kg for Bla, B1b, 8,9-ZMa and AB1a, 0.0002 mg/kg for MFB1a and 0.0005 mg/kg for FAB1a. In dry chives, due to high ion suppression LOD for FAB1a was 0.002 mg/kg and the LOD for MFB1a at 0.0005 mg/kg.

Table 22 Residues trials on chives after foliar spray with an SG formulation

Trial No., Location, Year, Crop (Variety)	Application			Sample	DALA (days)	Residues (mg/kg)					
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)			B1a	B1b	8, 9-ZMa	AB1a	FAB1a	MFB1a
GAP, United States, Chives, Foliar	- (7)	16.8	GND min. 94 AE 47-187	-	7	-	-	-	-	-	-
07137.08-MD04, Salisbury, Maryland, United States, 2008, Chives (Fancy)	6 (7, 6, 8, 6, 6)	16.8 17.0 16.9 17.1 16.7 16.6	331 335 334 340[a] 329 328	Mature leaves	7	0.004, 0.006 (0.005)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	ND, < 0.001 (< 0.001	ND, ND (ND)	ND, < 0.001 (< 0.001)
07137.08-SC*02, Charleston, South Carolina, United States, 2008, Chives (Green chives)	6 (7, 7, 7, 7, 7)	16.5 17.1 16.6 16.9 16.4 16.5	332[b] 372 342 345 354 340	Mature leaves	7	0.002, 0.001 (0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)
07137.08-GA*03, Tifton, Georgia, United States, 2008, Chives (Staro)	6 (7, 7, 8, 6, 6)	17.1 17.1 17.0 16.9 17.1 17.1	305[c] 305 304 302 304 305	Mature leaves	6	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	0.002, ND (0.001)
07137.08-CA*107, Salinas, California, United States, 2008, Chives (Purely)	6 (8, 8, 6, 6, 8)	16.8 16.7 16.7 16.6 17.1 16.9	405[d] 669 636 725 826 891	Mature leaves	6	0.001, 0.001 (0.001)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)

Notes:

Mean values are in the parenthesis and calculated from unrounded results.

[a] Adjuvant: Silwet, 0.25 percent included in last three applications.

[b] Adjuvant: Activator 90, 0.25 percent included in all applications.

[c] Adjuvant: NIS Surfactant 80/20, 0.25 percent included in all applications.

[d] Adjuvant: R-11, 0.25 percent included in all applications.

Flowerhead brassicas

Broccoli

The Meeting received information on supervised residues trials for the use of emamectin benzoate on broccoli and sprouting broccoli conducted in France (2005), Germany, Spain and the United Kingdom (2008). Both studies (Oliver-Kang, 2006a, CEMR-2654 and Marshall, 2009b, T009258-07-REG) were considered by the 2011 JMPR.

Table 23 Residue results from supervised field trials on broccoli and sprouting broccoli (inflorescence) after foliar spray with an SG formulation (9.5 g ai/kg) without adjuvant, trials reported by the 2011 JMPR

Trial No., Location, Year, Crop (Variety)	Application				Sample	DALA (days)	Residues (mg/kg)					
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)	Growth stage (BBCH)			B1a	B1b	8,9-ZMa	AB1a	FAB1a	MFB1a
GAP, United States, Broccoli, Foliar	- (7)	16.8	GND min. 94 AE 47-187	-	-	7	-	-	-	-	-	-
AF/8596/SY/01, Canals, 82170, Southern France, 2005, Broccoli, sprouting (Chevalier), CEMR-2654	3 (7)	15	200	42	Inflorescence	-0	0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		15	199	44		0	0.028	0.002	0.002	< 0.001	0.002	< 0.001
		15	201	47		1	0.007	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						3	0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						7	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						10	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						14	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
AF/8596/SY/02, Cortes, 31530, Spain, 2005, Broccoli, sprouting (Maraton), CEMR-2654	3 (7)	15	198	44	Inflorescence	-0	0.002	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		15	200	45-46		0	0.031	0.002	< 0.001	< 0.001	< 0.001	< 0.001
		15	200	47-48		1	0.011	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						3	0.002	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						7	0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						10	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						14	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
S08-00671-02, Holstein, 25348, Germany, 2008, Broccoli, regular (Ironman) T009258-07-REG	3 (7)	16	209	39	Inflorescence	0	0.059	0.004	0.001	< 0.001	< 0.001	< 0.001
		15	202	41		1	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		16	209	43		3	0.002	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						7	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						14	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
S08-00671-03, Kent, ME3 9LU, United Kingdom, 2008, Broccoli, sprouting (Bordeaux) T009258-07-REG	3 (6-7)	15	196	51	Inflorescence	0[1]	0.079	0.005	0.005	< 0.001	< 0.001	< 0.001
		15	201	50-61		1[1]	0.005	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		15	198	51-60		3[1]	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						7[1]	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						10[1]	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
S08-00671-05, Lincolnshire, PE22 0JQ, United Kingdom, 2008, Broccoli, sprouting (Summer sprouting purple) T009258-07-REG	3 (7)	15	198	41	Inflorescence	0[2]	0.059	0.003	0.001	< 0.001	< 0.001	< 0.001
		15	202	41-43		1[2]	0.006	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
		15	207	41-43		3[2]	0.004	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						7[2]	0.003	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
						10	0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001

Notes:

[1] Samples were harvested outside the harvestable period (BBCH 51–60, S-08-00671-03). Samples are not considered representative for MRL setting and results cannot be selected.

[2] Samples size too low (0.5 kg for trial: S-08-00671-05 at 0, 1, 3 and 7 DALA samplings). Samples are not considered representative for MRL setting and results cannot be selected.

Other trials considered relevant to the use on broccoli have been extracted from the 2011 JMPR as presented below.

Table 24 Residue results from supervised field trials on broccoli (inflorescence) after foliar spray with an SG formulation (9.5 g ai/kg for the European Union and 50 g ai/kg for the United States), trials reported by the 2011 JMPR

Location, country year, Crop (variety)	Number, (interval) soil type, adjuvant	g ai/ha	g ai/hL	Last appl date, growth stage	PHI	MAB1a (mg/kg)	MAB1b (mg/kg)	Sum1a (mg/kg) [1]	Trial, Report
Fully, VS, Switzerland, 2006, (Broccoli: Lucky F1)	3, (7, 7) sandy loam, without adjuvant	16 16 16	3.0 3.0 3.0	19 Jun; BBCH 47–49	-0 0 3	< 0.001 0.006 < 0.001	< 0.001 < 0.001 < 0.001	< 0.001 0.006 < 0.001	Trial: CH-IR-06-0138 Report: CEMR-3019
Fully, VS, Switzerland, 2006 (regular broccoli: Ironman)	3, (6, 8) loamy sand, without adjuvant	16 16 16	3.0 3.0 3.0	30 Aug; BBCH 43–46	-0 0 3	0.004 0.048 0.004	< 0.001 0.003 < 0.001	0.004 0.055 0.006	Trial: CH-IR-06-0139 Report: CEMR-3019

Notes:

[1] Sum1a, expressed as MAB1a = sum of MAB1a plus its avermectin-like metabolites, corrected for molecular weight (MAB1a + 1.000 × 8,9-ZMa + 1.016 × AB1a + 0.9693 × MFB1a + 0.9844 FAB1a). Metabolites < LOQ were assumed not to be present.

Cauliflower

The Meeting received information on supervised residues trials on cauliflower (Bell, 2010, CEMR-4421, Eversfield, 2007, CEMR-3026; Marshall, 2009a, T009254-07-REG and Oliver-Kang, 2006b, CEMR-2655) that were considered by the 2011 JMPR.

Table 25 Residue results from supervised field trials on cauliflower (inflorescence) after foliar spray with an SG formulation (9.5 g ai/kg) without adjuvant, trials reported by the 2011 JMPR

Trial No., Location, Year, Crop (Variety), Report No.	Application				Sample	DALA	Residues (mg/kg)					
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)	Growth stage (BBCH)		(days)	B1a	B1b	8,9-ZMa	AB1a	FAB1a	MFB1a
GAP, United States, Cauliflower, Foliar	- (7)	16.8	GND min. 94 AE 47-187	-	-	7	-	-	-	-	-	-
AF/8597/SY/02, Blagnac, 31700, Southern France, 2005, Cauliflower (Aviso), CEMR-2655	3 (7)	15 15 15	201 197 199	43 45 45	Inflorescence	-0 0 1 3	0.002 0.065 0.040 < 0.001	< 0.001 0.005 0.003 < 0.001	< 0.001 < 0.001 0.006 < 0.001	< 0.001 < 0.001 0.001 < 0.001	< 0.001 < 0.001 0.003 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001
AF/8597/SY/03, Saint Caprias, 31330, Southern France, Cauliflower (Fridon), CEMR-2655	3 (7)	15 15 15	201 200 198	43 45 45	Inflorescence	-0 0 1 3 7 10 14	0.002 0.116 0.026 0.001 < 0.001 < 0.001 < 0.001	< 0.001 0.008 0.002 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 0.005 0.003 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 0.001 0.002 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 0.003 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001
AF/10362/SY/1, Blagnac, 31700, Southern France, 2006, Cauliflower (Aviso), CEMR-3026	3 (7)	15 15 15	200 198 196	41 41-43 45	Inflorescence	-0 0 1 3 7 10 14	< 0.001 0.003 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001
AF/10362/SY/2, Saint Caprias, Southern France,	3 (7)	15 15 15	194 196 193	41 43 47	Inflorescence	0 3	0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001	< 0.001 < 0.001

Trial No., Location, Year, Crop (Variety), Report No.	Application				Sample	DALA (days)	Residues (mg/kg)					
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)	Growth stage (BBCH)			B1a	B1b	8,9-ZMa	AB1a	FAB1a	MFB1a
2006, Cauliflower (Kintore), CEMR-3026												
S08-00670-01,	3 (7)	16	210	43	Inflorescence	0	0.007	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Innenheim, Alsace,		16	214	45		1	0.005	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
67880, Northern		16	210	48		3	0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
France, 2008,						7	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Cauliflower (Lecanu), T009254-07-REG						10	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
S08-00670-02,	3 (7)	16	209	41	Inflorescence	0	0.003	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Holstein, 25348,		15	205	43		1	0.002	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Germany, 2008,		15	204	48		3	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Cauliflower						7	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
(Clapton), T009254-07-REG						10	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
S08-00670-03,	3 (6-7)	15	203	45	Inflorescence	0	0.007	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Chester, Cheshire,		15	202	45		1	0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
United Kingdom, 2008,		15	199	47		3	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Cauliflower						7	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
(Glacier), T009254-07-REG						10	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
S08-00670-04,	3 (7)	15	200	45	Inflorescence	0	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Lincolnshire, PE20		15	194	43-49		1	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
1TW, United Kingdom, 2008,		15	203	49		3	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Cauliflower						7	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
(Triumphant), T009254-07-REG						10	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
S09-01553-02,	3 (7)	16	214	41-43	Inflorescence	-0	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Lincolnshire, PE20		15	197	41-43		0	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
1TW, United Kingdom, 2009,		15	206	49		1	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Cauliflower						3	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
(Belot F1),						7	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
CEMR-4421						13	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
S09-01553-03,	3 (7)	15	201	43-43	Inflorescence	-0	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Innenheim, Alsace,		16	208	44-45		0	0.004	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
67880, Northern		15	203	47-49		1	0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
France, 2009,						3	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Cauliflower						7	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
(Korlanu), CEMR-4421						10	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001

Other trials considered relevant to the use on cauliflower have been extracted from the 2011 JMPR as presented below.

Table 26 Residue results from supervised field trials on cauliflower (inflorescence) after foliar spray with an SG formulation (9.5 g ai/kg for European Union and 50 g ai/kg for the United States), trials reported by the 2011 JMPR

Location, country year, Crop (variety)	Number, (interval) soil type, adjuvant	g ai/ha	g ai/hL	Last appl date, growth stage	PHI	MAB1a (mg/kg)	MAB1b (mg/kg)	Sum1a (mg/kg) [1]	Trial, Report
Gosberton Clough,	3, (7–7),	16	7.5	29 Aug	0	0.001	< 0.001	0.001	Trial:
Lincolnshire, United Kingdom,	silty clay loam,	16	7.5	BBCH	3	< 0.001	< 0.001	< 0.001	AF/10361/SY/1
2006, Cauliflower (Valtross)	without adjuvant	14	7.5	45-47					Report: CEMR-3025
Fosdyke,	3, (8–6),	16	7.5	29 Sept	0	0.003	< 0.001	0.004	Trial:

Location, country year, Crop (variety)	Number, (interval) soil type, adjuvant	g ai/ha	g ai/hL	Last appl date, growth stage	PHI	MAB1a (mg/kg)	MAB1b (mg/kg)	Sum1a (mg/kg) [1]	Trial, Report
Lincolnshire, United Kingdom, 2006, Cauliflower (Cornell)	sandy clay loam, without adjuvant	16 16	7.5 7.5	BBCH 45	3	0.001	< 0.001	0.001	AF/10361/SY/2 Report: CEMR-3025

Notes:

[1] Sum1a, expressed as MAB1a = sum of MAB1a plus its avermectin-like metabolites, corrected for molecular weight (MAB1a + 1.000 × 8,9-ZMa + 1.016 × AB1a + 0.9693 × MFB1a + 0.9844 FAB1a). Metabolites < LOQ were assumed not to be present.

Leafy vegetables

Spinach

The Meeting received information on supervised residue trials on spinach and leaf lettuce.

Ten supervised residue trials with emamectin benzoate on spinach (6) and leaf lettuce (4) were conducted in the United States between 1997 and 2005 (Vincent, 1998, ABR-98047 and Ediger and Oakes, 2005, T002301-03). All trials were conducted under field conditions. In all trials, emamectin benzoate was applied as a SG formulation containing 5 percent emamectin benzoate. The test substance was applied four to six times as a foliar application at a nominal rate of 16.8 g ai/ha, with a nominal application interval of 7 ± 1 day and a PHI of 7 days. A non-ionic surfactant was added as an adjuvant to all applications at a rate of 0.1 percent.

Samples of leaf lettuce and spinach were collected from all trials at 7 days after the final application. Decline trials also sampled at 0, 3 and 7 days, and 10 or 14 days after the final application. Samples were collected and stored frozen prior to analysis. Samples were stored for a maximum of 9.6 months prior to analysis. Data previously evaluated by the JMPR on high water content commodities demonstrated storage stability for at least over 27 months when stored at ≤-18 °C.

For the trials conducted in 1997, samples were analysed for residues of emamectin benzoate and photodegradates using the validated analytical method ARM 244-92-3 (rev. 1) with an LOQ of 0.005 mg/kg. The HPLC Fluorescence method was considered by the 2011 JMPR. Since the 8,9-ZMa/b isomers cannot be distinguished from the parent by the analytical method used in these trials, residue levels for B1a and B1b include residues of its 8,9- ZMa/b isomers. Additionally, all samples were also analysed for the photodegradates AB1a, and FAB1a + MFB1a as presented below.

Table 27 Residues trials on leaf lettuce and spinach after foliar spray with an SG formulation

Trial No., Location, Year, Crop (Variety) Report No.	Application				Sample	DALA (days)	Residues (mg/kg)			
	No. (RTI, days)	Rate (g ai/ha	Spray Volume (L/ha)	Growth stage			B1a[1]	B1b[1]	AB1a	FAB1a + MFB1a
GAP, United States, Spinach, Foliar	- (7)	16.8	GND min. 94 AE 47-187	-	-	7	-	-	-	-
01-IR-024-97, Hunterdon, New Jersey, United States, 1997, Leaf lettuce (Grand Rapids Two Star)	5 (6-8)	16.8 16.8 16.8 16.8 16.8	702 711 711 711 711	5-8 cm 8-10 cm 10-15 cm 10-15 cm 15-25 cm	Leaves	0	0.494, 0.503 (0.499)	0.037, 0.038 (0.038)	0.007, 0.008 (0.008)	< 0.005, < 0.005 (< 0.005)
						3	0.032, 0.032 (0.032)	< 0.005, < 0.005 (< 0.005)	ND, ND (ND)	ND, ND (ND)
						7	0.013, 0.013 (0.013)	< 0.005, < 0.005 (< 0.005)	< 0.005, ND (< 0.005)	ND, ND (ND)
						14	ND, < 0.005 (< 0.005)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
01-IR-025-97, Palm Beach,	4 (7)	16.8 16.8	281 290	Open type 25-30 cm	Leaves	0	0.084, 0.094 (0.089)	0.006, 0.007 (0.006)	< 0.005, < 0.005 (< 0.005)	ND, ND (ND)

Trial No., Location, Year, Crop (Variety) Report No.	Application				Sample	DALA (days)	Residues (mg/kg)			
	No. (RTI, days)	Rate (g ai/ha	Spray Volume (L/ha)	Growth stage			B1a[1]	B1b[1]	AB1a	FAB1a + MFB1a
Florida, United States, 1997, Leaf lettuce (Romaine)		16.8 16.8	281 281	Open head Mature		3	0.008, 0.006 (0.007)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
						7	< 0.005, < 0.005 (< 0.005)	ND, ND (ND)	ND, < 0.005 (< 0.005)	ND, ND (ND)
						14	< 0.005, < 0.005 (< 0.005)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
01-IR-026-97, San Luis Obispo, California, United States, 1997, Leaf lettuce (Dark Green Boston MI)	6 (6-8)	16.8 16.8 16.8 16.8 16.8 16.8	159 178 187 196 187 178	Seedlings Immature Immature Immature Almost mature Mature	Leaves	0	0.102, 0.091 (0.096)	0.008, 0.007 (0.008)	0.011, 0.013 (0.012)	< 0.005, < 0.005 (< 0.005)
						3	0.024, 0.029 (0.026)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, ND (< 0.005)
						7	0.006, 0.009 (0.008)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
						14	< 0.005, ND (< 0.005)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
01-IR-027-97, Santa Cruz, California, United States, 1997, Leaf lettuce (Romaine)	6 (7)	16.8 16.8 16.8 16.8 16.8 16.8	281 281 281 281 281 281	0.6-1.3 cm 2.5-5 cm 5-10 cm 20 cm 20 cm Mature	Leaves	0	0.245, 0.258 (0.252)	0.019, 0.022 (0.020)	0.014, 0.014 (0.014)	0.007, 0.006 (0.006)
						3	0.076, 0.064 (0.070)	0.006, < 0.005 (0.006)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
						7	0.017, 0.019 (0.018)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	ND, ND (ND)
						14	< 0.005, 0.005 (0.005)	ND, ND (ND)	ND, < 0.005 (< 0.005)	ND, ND (ND)
01-IR-028-97, Wayne, New York, United States, 1997, Spinach (Tyee F1)	6 (7)	16.8 16.8 16.8 16.8 16.8 16.8	187 187 187 187 187 187	2.5 cm 8-10 cm 8-10 cm 10-15 cm 10-15 cm 10-15 cm	Leaves	0	0.281, 0.287 (0.284)	0.022, 0.023 (0.022)	0.028, 0.028 (0.028)	0.017, 0.017 (0.017)
						3	0.012, 0.014 0.032, 0.033 (0.023)	< 0.005, < 0.005 (< 0.005)	ND, ND (ND)	ND, ND (ND)
						7	0.006, < 0.005 (0.006)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
						14	ND, < 0.005 (< 0.005)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
01-IR-029-97, Rio Grande, Colorado, United States, 1997, Spinach (Unipac 151)	5 (7)	16.8 16.8 16.8 16.8 16.8	374 374 374 374 374	2.5 cm 5 cm 5 cm 8 cm 15-18 cm	Leaves	0	0.261, 0.299 (0.280)	0.020, 0.023 (0.022)	0.024, 0.025 (0.024)	0.027, 0.026 (0.026)
						3	0.025, 0.022 (0.024)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
						7	0.006, 0.006 (0.006)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
						14	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
01-IR-030-97, Santa Cruz, California, United States, 1997, Spinach (Mazurka)	6 (7)	16.8 16.8 16.8 16.8 16.8 16.8	468 468 468 468 468 468	0.6 cm 2.5-5 cm 2.5-8cm 8-10 cm 10-15 cm 20-23 cm	Leaves	0	0.043, 0.173 (0.108)	< 0.005, 0.012 (0.008)	0.007, 0.023 (0.015)	< 0.005, 0.013 (0.009)
						3	0.009, 0.014 (0.012)	ND, < 0.005 (< 0.005)	ND, < 0.005 (< 0.005)	ND, ND (ND)
						7	< 0.005, 0.009 (0.007)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
						14	ND, < 0.005 (< 0.005)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)

Notes:

Mean values are in the parenthesis and calculated from unrounded results.

Adjuvant: Non-ionic surfactant, 0.1 percent included in all applications.

The trials conducted in 2005, samples were analysed for residues of emamectin benzoate and its metabolites using the validated analytical method ARM 244-92-3 (rev. 1) with modifications to the clean-up and instrumental analysis with the LOQ remaining at 0.005 mg/kg. Chromatographic separation was achieved using contemporary columns with analysis by high performance liquid chromatography with triple quadrupole mass spectrometer detection (LC-MS/MS) with column switching. MS/MS detections of

emamectin benzoate (B1a, B1b, 8,9-ZMa, AB1a, FAB1a and MFB1a) was accomplished by monitoring the transitions of the molecular ions to the daughter ions using electrospray ionization in the positive mode similar to the instrumentation and transitions used in the other newer methods discuss is this evaluation. The results of this analysis are presented below.

Table 28 Residues trials on spinach after foliar spray with an SG formulation (50 g ai/kg)

Trial No., Location, Year, Crop (Variety)	Application				DALA (days)	Sample	Residues (mg/kg)					
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)	Growth stage (BBCH)			B1a	B1b	8,9-ZMa	AB1a	FAB1a	MFB1a
GAP, United States, Spinach, Foliar	3 (7)	16.8	GND min. 94 AE 47-187	-	-	7	-	-	-	-	-	-
SA-IR-04-5516, Wharton, Texas, United States, 2004-5, Spinach (Hybrid No. 7)	6 (7)	16.8 16.8 16.8 16.8 16.8 16.8	188 178 195 182 194 197	13-14 14-18 14-28 18-40 18-35 24-40	7	Leaves	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005, < 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005, < 0.005, < 0.005 (< 0.005)
WB-IR-04-5517, Aromas, California, United States, 2004-5, Spinach, (Avenger)	6 (7)	16.8 16.8 16.8 16.8 16.8 16.8	181 185 190 190 189 187	13 13 19 19 19 47	7	Leaves	< 0.005, 0.008 (0.007)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005, < 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005, < 0.005, < 0.005 (< 0.005)
SJ-IR-04-5518, Rose Hill, California, United States 2004-5, Spinach (Skookum)	6 (7)	16.8 16.8 16.8 16.8 16.8 16.8	126 133 146 144 147 144	2-3 leaf 6-8 leaf 6-8 leaf 8-10 leaf 8-10 leaf 8-10 leaf	0	Leaves	0.611, 0.673 (0.642)	0.038, 0.044 (0.041)	0.078, 0.081 (0.080)	0.032, 0.037 (0.034)	0.025, 0.032, 0.031, 0.042, 0.039, 0.040 (0.035)	< 0.005, < 0.005, 0.006, 0.009, 0.008, 0.010 (0.007)
					3	Leaves	0.091, 0.074 (0.083)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
					7	Leaves	0.036, 0.013 (0.024)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
					10	Leaves	0.013, 0.008 (0.010)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005

Notes:

Adjuvant: Non-ionic surfactant, 0.1 percent included in all applications.

Mean values are in the parenthesis and calculated from unrounded results.

Brassica leafy vegetables

The Meeting received information on supervised residues trials for the use of emamectin benzoate on brassica leafy vegetables (Chinese broccoli and Mustard greens).

Chinese broccoli

Six non-GLP field trials on Chinese broccoli were conducted in Thailand in 2017 to 19. In all trials, emamectin benzoate was applied as an EC formulation containing 1.92 percent emamectin benzoate. The test substance was applied twice as a foliar application at a nominal rate of 14.5 g ai/ha with a retreatment interval of 7 days.

At all sites, duplicate untreated control and treated samples of Chinese broccoli (> 2 kg) were collected at 0, 1, 3, 5, 7, 10 and 14 days after last application. All samples were received by the laboratory on the day of sampling, stored frozen at -18 °C, and analysed with ~1 month of sampling.

Samples were analysed for emamectin B1a benzoate in accordance with QuEChERS method EN 15662.2008. The LOQ for the method was 0.005 mg/kg.

Table 29 Residues trials on field Chinese broccoli after foliar spray with an EC formulation (19.2 g ai/kg)

Trial No., Location, Year, Crop (Variety)	Application				Sample	DALA (days)	Residues (mg/kg)
	No. (RTI, days)	Rate (g ai/ha)	Spray volume (L/ha)	Growth stage			B1a
GAP, Thailand, Chinese broccoli, Foliar	2 (7)	14.5	750	Stem elongation of rosette growth	-	3	-
061.17-01, Chaloem Phra Kiat, Saraburi, Thailand, 2017, Chinese broccoli	2 (7)	14.5 14.5	755 755	Last application at stem elongation of rosette growth 1st Appl. 28/2/17 2nd Appl. 7/3/17	Whole commodity	0	0.05, 0.05 (0.05)
						1	< 0.005, < 0.005 (< 0.005)
						3	< 0.005, < 0.005 (< 0.005)
						5	< 0.005, < 0.005 (< 0.005)
						7	< 0.005, < 0.005 (< 0.005)
						10	< 0.005, < 0.005 (< 0.005)
						14	< 0.005, < 0.005 (< 0.005)
061.17-02, Mueang Nakhon Pathom, Thailand, 2017, Chinese broccoli	2 (7)	14.7 14.7	766 766	Last application at stem elongation of rosette growth 1st Appl. 6/7/17 2nd Appl. 13/7/17	Whole commodity	0	0.05, 0.06 (0.06)
						1	0.02, 0.02 (0.02)
						3	< 0.005, < 0.005 (< 0.005)
						5	< 0.005, < 0.005 (< 0.005)
						7	< 0.005, < 0.005 (< 0.005)
						10	< 0.005, < 0.005 (< 0.005)
						14	< 0.005, < 0.005 (< 0.005)
061.17-03, Mueang Nakhon Pathom, Thailand, 2017, Chinese broccoli	2 (7)	14.7 14.7	766 766	Last application at stem elongation of rosette growth 1st Appl. 6/3/17 2nd Appl. 13/3/17	Whole commodity	0	0.31, 0.30 (0.30)
						1	0.03, 0.03 (0.03)
						3	< 0.005, < 0.005 (< 0.005)
						5	< 0.005, < 0.005 (< 0.005)
						7	< 0.005, < 0.005 (< 0.005)
						10	< 0.005, < 0.005 (< 0.005)
						14	< 0.005, < 0.005 (< 0.005)
061.17-04, U Thong, Suphan Buri, Thailand, 2017, Chinese broccoli	2 (7)	14.9 14.9	776 776	Last application at stem elongation of rosette growth 1st Appl. 4/4/17 2nd Appl. 11/4/17	Whole commodity	0	0.54, 0.34 (0.44)
						1	0.21, 0.19 (0.20)
						3	0.14, 0.10 (0.12)
						5	< 0.005, < 0.005 (< 0.005)
						7	< 0.005, < 0.005 (< 0.005)
						10	< 0.005, < 0.005 (< 0.005)
						14	< 0.005, < 0.005 (< 0.005)
061.19-05, Mueang Nakhon Pathom, Thailand, 2018/19, Chinese broccoli	2 (7)	14.6 14.6	760 760	Last application at stem elongation of rosette growth 1st Appl. 11/12/18 2nd Appl. 18/12/18	Whole commodity	0	0.08, 0.06 (0.07)
						1	0.01, 0.01 (0.01)
						3	< 0.005, < 0.005 (< 0.005)
						5	< 0.005, < 0.005 (< 0.005)
						7	< 0.005, < 0.005 (< 0.005)
						10	< 0.005, < 0.005 (< 0.005)
						14	< 0.005, < 0.005 (< 0.005)
061.19-06, Mueang Kanchanaburi, Thailand, 2019, Chinese broccoli	2 (7)	14.8 14.8	771 771	Last application at stem elongation of rosette growth 1st Appl. 11/12/18 2nd Appl. 18/12/18	Whole commodity	0	0.17, 0.16 (0.16)
						1	0.03, 0.03 (0.03)
						3	< 0.005, < 0.005 (< 0.005)
						5	< 0.005, < 0.005 (< 0.005)
						7	< 0.005, < 0.005 (< 0.005)
						10	< 0.005, < 0.005 (< 0.005)
						14	< 0.005, < 0.005 (< 0.005)

Notes:

Mean values are in the parenthesis and calculated from unrounded results.

Mustard greens

Six supervised trials on mustard greens were conducted in the United States in 1998 (Ediger, 1999, 136-98). These trials were considered by the 2011 JMPR.

Table 30 Residues trials on mustard after foliar spray with an SG formulation (50 g ai/kg) evaluated by the 2011 JMPR

Trial No., Location, Year, Crop (Variety) Report No.	Application				Sample	DALA (days)	Residues (mg/kg)			
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)	Growth stage			B1a[1]	B1b[1]	AB1a	FAB1a + MFB1a
GAP, United States, Brassica leafy vegetables, Foliar	6 (7)	16.8	GND min. 94 AE 47-187	-	-	14	-	-	-	-
0S-IR-308-98, Hidalgo County, Texas, United States, 1998, Mustard greens (Savannah)	6 (7)	16.8 16.8 16.8 16.8 16.8 16.8	150[a] 140 140 140 140 149	Vegetative Vegetative/early mature Vegetative/mature Late vegetative/mature Mature Mature	Leaves	7	0.034, 0.048 (0.041)	< 0.005, < 0.005, < 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
						14	0.011, 0.011 (0.011)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
0S-IR-618-98, Sampson County, North Carolina, United States, 1998, Mustard greens (Southern Giant Curled)	6 (7)	16.8 16.8 16.8 16.8 16.8 16.8	94[b] 94 94 94 94 94	1-2 true leaves 3-4 true leaves 4-6 true leaves 8-14 true leaves 10-15 true leaves 14-20 true leaves	Leaves	7	0.006, 0.007 (0.006)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005, < 0.005 (< 0.005)
						14	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
0S-IR-833-98, Mitchell County, Georgia, United States, 1998, Mustard greens (Florida Broadleaf)	6 (7)	16.8 16.8 16.8 16.8 16.8 16.8	187[c] 187 187 187 187 47	3-5 leaves 4-6 leaves 6-8 leaves 6-8 leaves 8-10 leaves 8-10 leaves	Leaves	7	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
						14	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005, < 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
0W-IR-428-98, San Joaquin County, California, United States, 1998, Mustard greens (Florida Broadleaf)	6 (5-8)	16.8 16.8 16.8 16.8 16.8 16.8	187[d] 187 187 187 187 187	First set of true leaves Immature/vegetative Immature/vegetative Vegetative/bolting Vegetative/bolting Bolting/mature	Leaves	0	0.233, 0.174 (0.204)	0.015, 0.011 (0.013)	0.017, 0.016 (0.016)	0.008, 0.009 (0.009)
						3	0.244, 0.167, < 0.005, 0.113 (0.132)	0.016, 0.010, < 0.005, 0.008 (0.010)	0.008, 0.006, < 0.005, < 0.005 (0.006)	< 0.005, < 0.005 (< 0.005)
						7	0.064, 0.045 (0.054)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
						14	< 0.005, 0.005, 0.201, 0.219 (0.108)	< 0.005, < 0.005, 0.012, 0.013 (0.009)	< 0.005, < 0.005, < 0.005, 0.005 (0.005)	< 0.005, < 0.005 (< 0.005)
						21	0.005, < 0.005 (0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)
0W-IR-441-98, Madera County, California, United States, 1998, Mustard greens (SLB Champion seed)	6 (7)	16.8 16.8 16.8 16.8 16.8 16.8	281[e] 281 281 281 281 281	First true cut 2 true leaves 3-4 true leaves 6-8 true leaves Mature Mature	Leaves	7	0.044, 0.080 (0.062)	< 0.005, 0.007 (0.006)	< 0.005, 0.006 (0.006)	< 0.005, < 0.005 (< 0.005)
						14	0.016, 0.012 (0.014)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)	< 0.005, < 0.005 (< 0.005)

Trial No., Location, Year, Crop (Variety) Report No.	Application				Sample	DALA (days)	Residues (mg/kg)			
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)	Growth stage			B1a[1]	B1b[1]	AB1a	FAB1a + MFB1a
FL-IR-012-98,	6 (7)	16.8	47[f]	2 leaves	Leaves	7	0.010,	< 0.005,	< 0.005,	< 0.005,
Seminole County,		16.8	47	4 leaves			0.013,	< 0.005,	< 0.005,	< 0.005,
Florida, United States, 1988,		16.8	47	6 leaves			< 0.005,	< 0.005,	< 0.005,	< 0.005,
Mustard greens		16.8	47	6 leaves			0.013	< 0.005	< 0.005	< 0.005
(Florida Broadleaf)		16.8	47	8 leaves			(0.010)	(< 0.005)	(< 0.005)	(< 0.005)
		16.8	47	10 leaves		14	0.012,	< 0.005,	< 0.005,	< 0.005,
							< 0.005	< 0.005	< 0.005	< 0.005
							(0.008)	(< 0.005)	(< 0.005)	(< 0.005)

Notes:

Mean values are in the parenthesis and calculated from unrounded results.

[a] Adjuvant: Dyne-Amic, 0.5 percent included in all applications.

[b] Adjuvant: Induce, 0.25 percent included in all applications.

[c] Adjuvant: NIS Surfactant 80/20, 0.01 percent included in all applications.

[d] Adjuvant: Latron B-1956, 0.1 percent included in all applications.

[e] Adjuvant: Agri-dex, 0.5 percent included in all applications.

[f] Adjuvant: Diamond R Activator, 0.06 percent included in all applications.

Soya bean (dry)

The Meeting received information on supervised residues trials for the use of emamectin benzoate on soya beans.

Twenty supervised trials involving the use of emamectin benzoate on soya beans were conducted in the United States during 2018 (Bledsoe, 2019, TK0347414) to determine the magnitude of residues of emamectin benzoate (B1a and B1b) and its metabolites 8,9-ZMa, AB1a, MFB1a and FAB1a. The test substance was an emulsifiable concentrate (EC) formulation (A10325A) with a nominal composition of 19.2 g ai/L. All trial sites established one control and one treated plot of soya bean. Treatment plots received three broadcast foliar spray applications of the test substance at the nominal rate of 17 g ai/ha with a retreatment interval of 7±1 days. Applications were targeted at 42±3 days before harvest (DBH), 35±2 DBH (7±1 days after first application; DAT1), and 28±1 DBH (7±1 days after second application; DAT2).

Control samples and duplicate treated samples were collected normal commercial harvest (28±1 days after last application (DALA). Additional duplicate treated (P2) soya bean seed samples were collected at three sites targeting 21±1, 25±1, 32±1, and 35±1 DALA to determine residue decline.

Samples of soya bean seed were stored frozen between -10 °C to -25 °C until analysis. The maximum duration between sampling an analysis was 10.1 months. Data previously evaluated by the JMPR on high oil content commodities demonstrated emamectin B1a benzoate is stable for at least 9 months of frozen storage.

All samples were analysed in accordance with an analytical method RAM 465/02. The LOQ was 0.001 mg/kg for all analytes.

Table 31 Residues of emamectin benzoate (B1a, B1b, 8,9-Z, AB1a, FAB1a and MFB1a) in soya bean seed after foliar applications with an EC formulation (19.2 g ai/L)

Trial No., Location, Year, Crop (Variety)	Application				DALA (days)	Sample	Residues (mg/kg)					
	No.	Rate (g ai/ha)	Volume (L/ha)	Growth stage (BBCH)			B1a	B1b	8,9-ZMa	AB1a	FAB1a	MFB1a
GAP, Japan, Soya bean, Foliar	3 (7)	16.8	-	-	28	-	-	-	-	-	-	-
TK0347414-01, Chula, Georgia, United States, 2018, Soya bean (AG7535)	3 (7)	16.7 16.8 16.7	196 196 196	79 81-82 83-84	27	Seed	ND, < 0.001 (< 0.001)	< 0.001, ND (< 0.001)	< 0.001, ND (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-02, Cheneyville, Louisiana, United States, 2018, Soya bean (AG46X6)	3 (7)	16.6 17.1 17.1	140 140 140	72-73 75-77 77-79	29	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-03, Fisk, Missouri, United States, 2018, Soya bean (S120090)	3 (6-7)	16.8 16.9 16.7	140 140 140	76-77 79 79	21	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					25	Seed	ND, ND (ND)	< 0.001, ND (< 0.001)	< 0.001, ND (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					28	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					32	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					36	Seed	ND, ND (ND)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-04, New Providence, Iowa, United States, 2018, Soya bean (AG2203)	3 (7-8)	16.9 17.6 17.0	253 271 262	72-74 75-76 77-78	30	Seed	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)
TK0347414-05, Cresco, Iowa, United States, 2018, Soya bean (AG2035)	3 (7-8)	16.6 16.9 16.8	234 234 234	73 74 77	30	Seed	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)
TK0347414-06, Richland, Iowa, United States, 2018, Soya bean (P31A22X)	3 (6-7)	16.8 16.8 16.8	178 178 196	78-79 78-79 78-79	29	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-07R, Stewardson, Illinois, United States, 2018, Soya bean (394L4)	3 (6-7)	17.0 16.6 17.0	140 131 131	77-79 79 79-81	28	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-08, Carlyle, Illinois, United States, 2018, Soya bean (H45L17)	3 (7)	17.1 16.6 16.7	140 112 159	77 77 78-79	28	Seed	< 0.001, < 0.001 (< 0.001)	ND, < 0.001 (< 0.001)	ND, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-09, Manilla, Indiana, United States, 2018, Soya bean	3 (7)	16.8 17.0 16.4	150 150 140	77 79 79	28	Seed	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)

Trial No., Location, Year, Crop (Variety)	Application				DALA (days)	Sample	Residues (mg/kg)					
	No.	Rate (g ai/ha)	Volume (L/ha)	Growth stage (BBCH)			B1a	B1b	8,9-ZMa	AB1a	FAB1a	MFB1a
(P40A47X)												
TK0347414-10, Stilwell, Kansas, United States, 2018, Soya bean (425-2R)	3 (7)	17.2 17.0 16.7	234 234 196	71-73 73-75 77-79	28	Seed	ND, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-11, Lawrence, Kansas, United States, 2018, Soya bean (MG 4247NXS)	3 (7)	16.8 17.2 16.8	150 150 140	77-78 78-79 79-80	28	Seed	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)
TK0347414-12, Stafford, Kansas, United States, 2018, Soya bean (P37T32XSU28)	3 (6-8)	17.5 15.9 16.6	178 159 168	75 77 79	29	Seed	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-13, St. Cloud, Minnesota, United States, 2018, Soya bean (P14T70R2)	3 (7)	16.8 17.1 16.7	187 187 187	78-79 80 81	28	Seed	ND, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	< 0.001, ND (< 0.001)	ND, ND (ND)	ND, ND (ND)
TK0347414-14, Aquila, Missouri, United States, 2018, Soya bean (456L4)	3 (6-8)	16.8 16.7 16.8	140 140 140	76-77 77-78 79-81	28	Seed	ND, ND (ND)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-15R, Anabel, Missouri, United States, 2018, Soya bean (P40T84X)	3 (6)	17.4 16.4 17.1	150 140 150	77-79 78-79 79	27	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-16, Northwood, North Dakota, United States, 2018, Soya bean (AG03X7)	3 (6-8)	16.9 16.7 16.9	187 187 187	79 79 81	20	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					27	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					29	Seed	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					32	Seed	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					35	Seed	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-17, Tolna, North Dakota, United States, 2018, Soya bean (AG03X7)	3 (6-8)	16.8 17.0 16.8	187 187 187	79 79 81-82	35	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)
TK0347414-18, Louisville, Nebraska, United States, 2018, Soya bean (AG28X8)	3 (6-8)	16.8 17.0 16.4	131 131 131	77-79 79 80-81	27	Seed	< 0.001, < 0.001 (< 0.001)	< 0.001, ND (< 0.001)	< 0.001, ND (< 0.001)	< 0.001, ND (< 0.001)	ND, ND (ND)	ND, ND (ND)
TK0347414-19, Brunswick,	3 (7)	16.9 16.6	122 122	76-77 78-79	21	Seed	< 0.001, < 0.001	ND, ND	< 0.001, < 0.001	ND, ND	ND, ND	ND, ND

Trial No., Location, Year, Crop (Variety)	Application				DALA (days)	Sample	Residues (mg/kg)					
	No.	Rate (g ai/ha)	Volume (L/ha)	Growth stage (BBCH)			B1a	B1b	8,9-ZMa	AB1a	FAB1a	MFB1a
Nebraska, United States, 2018, Soya bean (AG24X7)		16.8	122	84-85			(< 0.001)	(ND)	(< 0.001)	(ND)	(ND)	(ND)
					26	Seed	ND, ND (ND)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					28	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					31	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
					36	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)
TK0347414-20, York, Nebraska, United States, 2018, Soya bean (GH2981X)	3 (6-7)	16.8 16.8 16.9	187 178 187	79 79 81	29	Seed	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)	ND, ND (ND)

Coffee

The Meeting received information on supervised residues trials for the use of emamectin benzoate on coffee.

Four supervised residue trials on coffee were conducted in Brazil in 2020 (Evangelista, 2020, S19-23395 and Alves, 2020a, S19-23631-L1). At all locations, the treatment plots received two foliar applications of emamectin benzoate (50 g/kg WG) applied nominally at 25 g ai/ha at 51 days before harvest (DBH) and 30 days later (21 DBH) at a spray volume of 400 L/ha. The applications were applied in a spray volume of 410-438 L/ha of water and included addition of 0.1 percent adjuvant.

At all sites, duplicate untreated control and treated coffee (cherry) samples (3.7–10.5 kg) were collected at 21, 30 and 45 days after last application (DALA). After collection, the coffee cherries were left in the field for drying in sunshine for 10–37 days prior to processing. Following processing, at least 1 kg of coffee beans were transferred to a freezer within 24 hours of processing and stored frozen at <-20 °C. All samples were analysed within 135 days of collection.

All samples were analysed in accordance with analytical method RAM 465/02. The LOQ was 0.001 mg/kg for all analytes. The LOD was 0.0003 mg/kg for all analytes. Analysis results for B1a and B1b were reported in Evangelista, 2020, S19-23395 with results for 8,9-Z, AB1a, FAB1a and MFB1a reported in Alves, 2020a, S19-23631-L1 as presented below.

Table 32 Residues of emamectin benzoate (B1a, B1b, 8,9-Z, AB1a, FAB1a and MFB1a) in coffee beans after foliar spray with a WG formulation

Trial No., Location, Year, Crop (Variety)	Application				Sample	DALA (days)	Residues (mg/kg)					
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)	Growth stage (BBCH			B1a	B1b	8,9-Z	AB1a	FAB1a	MFB1a
GAP, Brazil, Coffee, Foliar	2 (30)	25	GND 400 AE min. 20	-	-	21	-	-	-	-	-	-
S19-23395-01, Indianópolis, Minas Gerias,	2 (30)	25.6 27	410 432	77 82	Coffee bean	21	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)

Trial No., Location, Year, Crop (Variety)	Application				Sample	DALA (days)	Residues (mg/kg)					
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)	Growth stage (BBCH			B1a	B1b	8,9-Z	AB1a	FAB1a	MFB1a
Brazil, 2020, Coffee (Mundo Novo)						30	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
						45	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
S19-23395-02, Araguari, Minas Gerias, Brazil, 2020, Coffee (Mundo Novo)	2 (30)	26 27.1	415 433	77 82	Coffee bean	21	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
						30	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
						45	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
S19-23395-03, Sooretama, Espírito Santo, Brazil, 2020, Coffee (Conilon)	2 (30)	27.4 26.7	438 427	75 79	Coffee bean	21	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
						30	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
						45	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
S19-23395-04, Dois Córregos, São Paulo, Brazil, 2020, Coffee (Topázio)	2 (30)	25.9 26.5	414 424	81 85	Coffee bean	21	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
						30	0.001, 0.001 -0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
						45	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)

Notes:

Mean values are in the parenthesis and calculated from unrounded results.

Adjuvant: Ochima non-ionic surfactant, 0.25 percent included for all applications.

A further study was received which determined the magnitude of residues of emamectin benzoate B1a, emamectin benzoate B1b and its metabolites 8,9-ZMa, AB1a, MFB1a and FAB1a in coffee; dry green beans, roasted coffee and instant coffee (Delongui, 2022, LBS20033).

Four supervised residue trials on were conducted in Brazil in 2021. At all locations, the treatment plots received two foliar applications of emamectin benzoate (50 g/kg WG) applied at 25 g ai/ha at 51 days before harvest (DBH) and 30 days later (21 DBH) at a spray volume of 400 L/ha. The applications were applied in a spray volume of 400–411 L/ha of water and included addition of 0.1 percent adjuvant. The two processing trials involved a further treatment plot with applications at 125 g ai/ha (5×).

At all sites, duplicate untreated control and treated coffee (cherry) samples (6.2–11.6 kg) were collected at 10, 15, 21, 25 and 30 days after last application (DALA). After collection, the coffee cherries were dried by placing inside plastic raffia bags were placed on a clean plastic tarp for 13–17 days prior to processing. Following processing, at least 1 kg of green coffee beans were transferred to a freezer within 24 hours of processing and stored frozen at ≤-20 °C. All samples were analysed within 135 days of collection.

All samples were analysed in accordance with analytical method RAM 465/02. The LOQ was 0.001 mg/kg for all analytes. The LOD was 0.0003 mg/kg for all analytes. Analysis results for B1a and B1b were reported in Evangelista, 2020, S19-23395 with results for 8,9-Z, AB1a, FAB1a and MFB1a reported in Alves, 2020a, S19-23631-L1 as presented below.

Table 33 Residues of emamectin benzoate (B1a, B1b, 8,9-Z, AB1a, FAB1a and MFB1a) in coffee beans after foliar spray with a WG formulation

Trial No., Location, Year, Crop (Variety)	Application				Sample	DALA (days)	Residues (mg/kg)					
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)	Growth stage (BBCH)			B1a	B1b	8,9-Z	AB1a	FAB1a	MFB1a
GAP, Brazil, Coffee, Foliar	2 (30)	25	GND 400 AE min. 20	-	-	21	-	-	-	-	-	-
LBS20033-01, Indianópolis, Minas Gerias, Brazil, 2021, Coffee (Mundo Novo Ponta Roxa)	2 (30)	25.0 25.0	400 400	85-87 85-87	Coffee bean	10	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
						15	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
						21	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
						25	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
						30	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
LBS20033-02, Nova Viçosa, Bahia, Brazil, 2021, Coffee (Conilon Clone A1)	2 (30)	25.0 25.0	400 400	75-79 76-82	Coffee bean	10	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
						15	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
						21	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
						25	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
						30	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
LBS20033-03, Caconde, São Paulo, Brazil, 2021, Coffee (Catuaí Vermelho)	2 (30)	25.0 25.0	400 400	78-81 79-85	Coffee bean	21	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)
LBS20033-04, Aracruz, Espírito Santo, Brazil, 2021, Coffee (Conilon Clone A1)	2 (30)	25.0 25.0	400 400	74-79 75-81	Coffee bean	21	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001	< 0.001, < 0.001 (< 0.001)

Notes:

Mean values are in the parenthesis and calculated from unrounded results.

Adjuvant: Ochima non-ionic surfactant, 0.1 percent included for all applications.

Basil

The Meeting received information on supervised residues trials for the use of emamectin benzoate on basil (Samoil, 2017, IR-4 PR No. 07137).

Four supervised residue trials on basil were conducted in the United States in 2008. At each location, emamectin benzoate (0.05 lb ai SG) was applied as a foliar directed application to basil six times at 7 day intervals. The individual application rates ranged from 16.4–17.5 g ai/ha and totalled 100.4–103.1 g ai/ha per season. The first application was applied at 39–41 days before normal harvest of the basil. The applications were applied in a spray volume of 242–390 L/ha of water and included a non-ionic surfactant or crop oil type additive.

At all sites, duplicate untreated control and treated basil samples were harvested by hand at commercial maturity, 7 days after the last treatment. A minimum of approximately 0.5 kg for fresh leaves and stems were harvested at all sites and a minimum of approximately 0.25 kg of dried leaves and stems also sampled at the New Mexico trial site. Additionally, at the North Carolina decline trial site, duplicate fresh basil samples were also taken from the treated plot at 0, 3 and 9 days after treatment.

All fresh samples were placed into a freezer, <4 hours after sampling and generally stored frozen at <-18 °C until analysis.

All samples were analysed in accordance with analytical method RAM 465/01. The LOQ was 0.001 mg/kg for B1a, B1b, 8,9-Z, AB1a and MFB1a in fresh and dry basil and, for FAB1a, at 0.002 mg/kg in fresh basil and at 0.01 mg/kg in dry basil. The LOD was 0.0001 mg/kg for Bla, B1b, 8,9-ZMA and AB1a, 0.0002 mg/kg for MFB1a and 0.0005 mg/kg for FAB1a.

Table 34 Residues trials on basil after foliar spray with an SG formulation

Trial No., Location, Year, Crop (Variety)	Application			Sample	DALA (days)	Residues (mg/kg)					
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)			B1a	B1b	8, 9-ZMa	AB1a	FAB1a	MFB1a
GAP, United States, Basil, Foliar	- (7)	16.8	GND min. 94 AE 47-187	-	7	-	-	-	-	-	-
07137.08-NY11, Freeville, New York. United States, 2008. Basil (Martina)	6 (6, 6, 6, 8, 8)	16.9 16.9 17.0 16.6 16.4 16.6	322[a] 322 324 314 311 315	Fresh leaves and stems	7	0.005, 0.002 (0.004)	< 0.001, ND (< 0.001)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)
07137.08-FL05, Citra, Florida, United States, 2008, Basil (Genova)	6 (7, 7, 7, 7, 7)	17.0 17.3 17.1 17.1 17.5 17.0	379 386[b] 382 384 390 380	Fresh leaves and stems	7	0.005, 0.006 (0.005)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, ND (< 0.001)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)
07137.08-NM03, Las Cruces, New Mexico, United States, 2008, Basil (Genovese)	6 (6, 7, 6, 6, 7)	16.9 17.4 17.4 16.7 16.9 17.5	282 299[c] 329 326 339 389	Fresh leaves and stems	7	0.025, 0.032 (0.028)	0.003, 0.003 (0.003)	0.006, 0.007 (0.007)	< 0.001, < 0.001 (< 0.001)	< 0.002, < 0.002 (< 0.002)	0.003, 0.003 (0.003)
07137.08-NC23, Clinton, North Carolina, United States, 2008, Basil (Genovese)	6 (6, 6, 7, 6, 7)	16.8 16.7 16.8 17.0 16.8 16.9	243[a] 242 242 246 242 244	Fresh leaves and stems	0	0.203, 0.183 (0.193)	< 0.001, < 0.001 (< 0.001)	0.005, 0.005 (0.005)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	0.024, 0.017 (0.021)
					3	0.003, 0.004 (0.003)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)
					7	0.001, 0.002 (0.001)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)

Trial No., Location, Year, Crop (Variety)	Application			Sample	DALA (days)	Residues (mg/kg)					
	No. (RTI, days)	Rate (g ai/ha)	Spray Volume (L/ha)			B1a	B1b	8, 9-ZMa	AB1a	FAB1a	MFB1a
					9	0.002, 0.001 (0.001)	ND, ND (ND)	< 0.001, < 0.001 (< 0.001)	ND, ND (ND)	ND, ND (ND)	ND, < 0.001 (< 0.001)

Notes:

[a] Adjuvant: Induce, 0.25 percent included in all applications.

[b] Adjuvant: Chem-nut, 0.25 percent included in all applications except the initial application.

[c] Adjuvant: Agridex, 1.0 percent included in all applications except the initial application.

Tea

The Meeting received information on supervised residues trials for the use of emamectin benzoate on tea.

Eight supervised trials involving the use of emamectin benzoate on tea were conducted in Japan during 2018-2019 (Ogiyama, 2019a, JP2018C324, Ogiyama 2019b JP2018C081 and Morita, 2020, JP2019C109) to determine the magnitude of residues of emamectin benzoate B1a, emamectin benzoate B1b and its metabolites 8,9-ZMa, AB1a, MFB1a and FAB1a. At all locations, the treatment plots received a single foliar application of 1 percent emamectin benzoate (10 g/L EC) applied at the leaf ages between sprouting and 6 leaf stage at 1000-fold dilution and spray volumes of 3000–4000 L/ha.

Separate plots in each trial were sampled at 7, 14 and 21 days after application. Tea leaves (picked at leaf ages between 2–6 leaf) were steamed using a conveyor belt steaming machine at the conditions of 1 metre of tea leaves/45 seconds (60 kg of tea leaves/hour) or using an autoclave for 60 seconds. Samples were then dried at 80 °C for 120–135 minutes using an air-permeable drying machine or a convection oven to produce dried green tea leaves. At the laboratory, each sample was cut to pieces (about 1 cm square), mixed well, pulverized using a mill, sealed, and stored at about -20 °C for a maximum of 82 days until analysis.

All samples were analysed in accordance with an analytical method for tea similar to RAM 465/01. The LOQ was 0.001 mg/kg for all analytes. The LOD was 0.0005 mg/kg for all analytes.

Table 35 Residues of emamectin benzoate (B1a, B1b, 8,9-Z, AB1a, FAB1a and MFB1a) in dried green tea leaves and tea infusions after foliar spray with an EC formulation (10 g/L emamectin)

Trial No., Location, Year, Crop (Variety)	Application			DALA (days)	Sample	Residues (mg/kg)					
	No.	Conc. (g ai/hL)	Volume (L/ha)			B1a	B1b	8,9-Z	AB1a	FAB1a	MFB1a
GAP, Japan, Tea, Foliar	1	1	-	7	-	-	-	-	-	-	-
JP2018C324A, Kagoshima, Japan, 2018, Tea, (Yamatomidori)	1	1	4000	7	Dried green tea leaves	0.011, 0.011 (0.011)	< 0.001, < 0.001 (< 0.001)	0.003, 0.002 (0.002)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	0.007, 0.007 (0.007)
	1	1	4000	14	Dried green tea leaves	0.001, < 0.001 (0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
	1	1	4000	21	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
JP2018C081A, Ibraki, Japan, 2018, Tea (Yabukita)	1	1	3020	7	Dried green tea leaves	0.003, 0.003 (0.003)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	0.002, 0.002 (0.002)
	1	1	3020	14	Dried green tea leaves	0.001, 0.001 (0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)

Trial No., Location, Year, Crop (Variety)	Application			DALA (days)	Sample	Residues (mg/kg)					
	No.	Conc. (g ai/hL)	Volume (L/ha)			B1a	B1b	8,9-Z	AB1a	FAB1a	MFB1a
	1	1	3020	21	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
JP2018C081B, Kochi, Japan, 2018, Tea (Yabukita)	1	1	3830	7	Dried green tea leaves	0.004, 0.004 (0.004)	< 0.001, < 0.001 (< 0.001)	0.002, 0.002 (0.002)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	0.002, 0.002 (0.002)
	1	1	3830	14	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
	1	1	3830	21	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
JP2018C081C, Miyazaki, Japan, 2018, Tea (Yamatomidori)	1	1	3330	7	Dried green tea leaves	0.008, 0.008 (0.008)	< 0.001, < 0.001 (< 0.001)	0.003, 0.003 (0.003)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	0.004, 0.004 (0.004)
	1	1	3330	14	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
	1	1	3330	21	Dried green tea leaves	0.001, 0.001 (0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
JP2019C109A, Ibaraki, Japan, 2019, Tea (Yabukita)	1	1	3060	7	Dried green tea leaves	0.011, 0.010 (0.010)	< 0.001, < 0.001 (< 0.001)	0.003, 0.003 (0.003)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	0.006, 0.006 (0.006)
	1	1	3060	14	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
	1	1	3060	21	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
JP2019C109B, Saitama, Japan, 2019, Tea (Fukumidori)	1	1	3330	7	Dried green tea leaves	0.068, 0.065 (0.066)	0.002, 0.002 (0.002)	0.016, 0.015 (0.016)	0.001, 0.001 (0.001)	0.001, 0.001 (0.001)	0.011, 0.010 (0.010)
	1	1	3330	14	Dried green tea leaves	0.014, 0.014 (0.014)	< 0.001, < 0.001 (< 0.001)	0.001, 0.001 (0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	0.002, 0.002 (0.002)
	1	1	3330	21	Dried green tea leaves	0.004, 0.004 (0.004)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
JP2019C109C, Kochi, Japan, 2019, Tea (Yabukita)	1	1	3830	7	Dried green tea leaves	0.007, 0.007 (0.007)	< 0.001, < 0.001 (< 0.001)	0.003, 0.003 (0.003)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	0.003, 0.003 (0.003)
	1	1	3830	14	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
	1	1	3830	21	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
JP2019C109D, Miyazaki, Japan, 2019, Tea (Yabukita)	1	1	3330	7	Dried green tea leaves	0.022, 0.022 (0.022)	< 0.001, < 0.001 (< 0.001)	0.012, 0.012 (0.012)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	0.017, 0.017 (0.017)
	1	1	3330	14	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)
	1	1	3330	21	Dried green tea leaves	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)	< 0.001, < 0.001 (< 0.001)

FATE OF RESIDUES IN STORAGE AND PROCESSING

Residues after processing

The fate of emamectin benzoate residues during processing of raw agricultural commodities was investigated in basil, chives, coffee, tea and soya beans.

Soya bean

Two processing trials were conducted in the United States in 2018 to demonstrate the effect of processing on residues of emamectin benzoate in soya beans. Treatment plots for the processing trials received three broadcast foliar spray applications at an exaggerated (5×) nominal rate of 84 g ai/ha. Soya bean seeds sampled from these trials were processed into pollards, soy sauce, miso, flour (defatted) and aspirated grain fractions. As residues in soya bean seed samples were all <LOQ only the aspirated grain fractions were analysed.

All samples were analysed in accordance with analytical method RAM 465/02. The limit of quantitation (LOQ) was 0.001 mg/kg for all analytes.

Residues of emamectin B1a benzoate concentrated when soya bean seeds were processed to aspirated grain fractions. Actual processing factors cannot be quantified noting that all residues in soya bean seed prior to processing were below the LOQ (< 0.001 mg/kg).

Table 38 Summary of residues of emamectin benzoate (B1a, B1b, 8,9-ZMa, AB1a, FAB1a and MFB1a) in soya beans and the aspirated grain fractions at 28-29 DALA after foliar spray with an EC formulation (19.2 g/L emamectin)

Trial No., Location, Year, Crop (Variety)	Analyte	Commodity		Mean processing factor[1] (PF)
		Soya bean seed prior to processing (RAC)	Aspirated grain fractions	
		Residues (mg/kg)		
TK0347414-06, Richland, Iowa, United States, 2018, Soya bean (P31A22X)	B1a	< 0.001, < 0.001, < 0.001 (< 0.001)	0.005	NC (observed to concentrate)[2]
	B1b	< 0.001, < 0.001, ND (< 0.001)	< 0.001	NC[2]
	8,9-ZMa	ND, ND, ND (ND)	< 0.001	NC (observed to concentrate)[2]
	AB1a	< 0.001, ND, ND (< 0.001)	< 0.001	NC[2]
	FAB1a	ND, ND, ND (ND)	0.0055	NC (observed to concentrate)[2]
	MFB1a	ND, ND, ND (ND)	< 0.001	NC (observed to concentrate)[2]
TK0347414-08, Carlyle, Illinois, United States, 2018, Soya bean (H45L17)	B1a	< 0.001, < 0.001, < 0.001 (< 0.001)	0.021	NC (observed to concentrate)[2]
	B1b	< 0.001, ND, < 0.001 (< 0.001)	< 0.001	NC[2]
	8,9-ZMa	ND, ND, ND (ND)	0.0023	NC (observed to concentrate)[2]
	AB1a	ND, ND, < 0.001 (< 0.001)	0.0035	NC (observed to concentrate)[2]
	FAB1a	ND, ND, ND (ND)	0.014	NC (observed to concentrate)[2]
	MFB1a	ND, ND, ND (ND)	0.0020	NC (observed to concentrate)[2]

Notes:

Mean results are reported in the parenthesis.

[1] Processing factor = Mean residue in the processed commodity / Mean residue in the RAC. For censored data (<LOQ), the finite LOQ value for the relevant analyte has been used for calculations.

[2] Where residues in the unprocessed commodity (RAC) were <LOQ no processing factor can be quantified and are reported as NC (Not Calculated). If residues were observed at <LOQ in the RAC and ≥LOQ in the processed commodity they are reported as NC (observed to concentrate).

Coffee

Two processing trials were conducted in Brazil in 2021 to demonstrate the effect of processing on residues of emamectin benzoate in coffee. Samples of treated coffee (the cherry) (129–355 kg) were collected at 21 days after last application (DALA). After collection, the coffee cherries were dried by

placing inside plastic raffia bags placed on a clean plastic tarp for 16 days prior to processing. After drying and processing, green coffee beans (45–75 kg) were transferred to a freezer within 24 hours (stored at -20 °C). Green coffee beans were processed by roasting the dry coffee beans to create roasted coffee and then the remaining coffee was milled and extracted with water and then lyophilized, producing instant coffee. At least 1kg of roasted and instant coffee were collected and frozen at -20 °C until analysis.

All samples were analysed in accordance with analytical method RAM 465/02. The limit of quantitation (LOQ) was 0.001 mg/kg for all analytes.

Residues of emamectin and its metabolites decreased in roasted or instant coffee (PF=<1). No residues were observed above the LOQ in roasted or instant coffee.

Table 37 Residues of emamectin benzoate (B1a, B1b, 8,9-ZMa, AB1a, FAB1a and MFB1a) in coffee beans, roasted coffee and instant coffee at 21 DALA after foliar spray with a WG formulation (50 g ai/kg)

Trial No., Location, Year, Crop (Variety)	Analyte	Commodity			Mean processing factor[1] (PF)
		Coffee dry green bean (RAC)	Roasted coffee	Instant coffee	
		Residues (mg/kg)			
LBS20033-03, Caconde, São Paulo, Brazil, 2021, Coffee (Catuaí Vermelho)	B1a	0.001, 0.001, 0.001 (0.001)	< 0.001, < 0.001	< 0.001, < 0.001	<1
	B1b	< 0.001, < 0.001, < 0.001	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	8,9-ZMa	< 0.001, < 0.001, < 0.001	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	AB1a	< 0.001, < 0.001, < 0.001	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	FAB1a	< 0.001, < 0.001, < 0.001	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	MFB1a	< 0.001, < 0.001, < 0.001	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
LBS20033-04, Aracruz, Espírito Santo, Brazil, 2021, Coffee (Conilon Clone A1)	B1a	0.003, 0.004, 0.007 (0.005)	< 0.001, < 0.001	< 0.001, < 0.001	< 0.2
	B1b	< 0.001, < 0.001, < 0.001	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	8,9-ZMa	< 0.001, < 0.001, < 0.001	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	AB1a	< 0.001, < 0.001, < 0.001	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	FAB1a	< 0.001, < 0.001, < 0.001	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	MFB1a	< 0.001, < 0.001, < 0.001	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]

Notes:

Mean results are reported in the parenthesis.

[1] Processing factor = Mean residue in the processed commodity / Mean residue in the RAC. For censored data (<LOQ), the finite LOQ value for the relevant analyte has been used for calculations.

[2] Where residues in the unprocessed commodity (RAC) were <LOQ no processing factor can be quantified and are reported as NC (Not Calculated). Where residues are >LOQ in the RAC and <LOQ in the processed commodity the processing factor is reported as less than (<).

Basil and Chives

Two trials were conducted in the United States in 2008 to assess residues in dried basil and chives. A minimum of approximately 0.5 kg of fresh basil (leaves and stems) or fresh chives (leaves) was harvested at 6–7 days after 6 foliar applications of emamectin benzoate (5 percent SG) and stored frozen (≤-15 °C) until processing and analysis. For dried samples, the samples were dried in an oven for 24 hours at 47 °C (basil) and 54 °C (chives). After cooling, at least 0.25 kg of dried samples were transferred to a freezer and stored frozen (≤-15 °C) until analysis.

All samples were analysed in accordance with analytical method RAM 465/01. The LOQ was 0.001 mg/kg for B1a, B1b, 8,9-ZMa, AB1a and MFB1a in fresh and dry basil and chives, 0.002 mg/kg for FAB1a in fresh basil and chives and 0.01 mg/kg in dry basil and chives. The LOD was 0.0001 mg/kg for Bla, B1b, 8,9-ZMa and AB1a, 0.0002 mg/kg for MFB1a in fresh and dried basil and chives and, for FAB1a, at 0.0005 mg/kg in dried basil and 0.002 mg/kg in dried chives.

Residues of emamectin benzoate were observed to concentrate in dried basil and chives with processing factors for B1a of 6.4 for dried basil and 5.0 for dried chives as presented below.

Table 36 Residues of emamectin benzoate (B1a, B1b, 8,9-ZMa, AB1a, FAB1a and MFB1a) in fresh and dry basil and chives at 6-7 DALA after foliar spray with an SG formulation (50 g ai/kg)

Trial No., Location, Year, Crop (Variety)	Analyte	Commodity		Mean processing factor[1] (PF)
		Fresh sample (RAC)	Dry sample	
		Residues (mg/kg)		
07137.08-NM03, Las Cruces, New Mexico, United States, 2008, Basil (Genovese)	B1a	0.025, 0.032 (0.028)	0.179	6.4
	B1b	0.003, 0.003 (0.003)	0.009, 0.010 (0.009)	3.0
	8,9-ZMa	0.006, 0.007 (0.007)	0.028	4.0
	AB1a	< 0.001, < 0.001 (< 0.001)	0.003	NC (observed to concentrate)[2]
	FAB1a	< 0.002, < 0.002 (< 0.002)	< 0.01	NC[2]
	MFB1a	0.003, 0.003 (0.003)	ND (LOQ = < 0.001)	< 0.33
07137.08-GA*03, Tifton, Georgia, United States, 2008, Chives (Staro)	B1a	0.001, 0.001 (0.001)	0.007, 0.004 (0.005)	5.0
	B1b	ND, ND (ND) (LOQ = < 0.001)	< 0.001, < 0.001 (< 0.001)	NC[2]
	8,9-ZMa	< 0.001, < 0.001 (< 0.001)	< 0.001, 0.001 (0.001)	NC (observed to concentrate)[2]
	AB1a	ND, ND (ND) (LOQ = < 0.001)	< 0.001, < 0.001 (< 0.001)	NC (observed to concentrate)[2]
	FAB1a	ND, ND (ND) (LOQ = < 0.002)	ND, ND (ND) (LOQ = < 0.01)	NC[2]
	MFB1a	< 0.001, < 0.001 (< 0.001)	0.001, < 0.001 (0.001)	NC (observed to concentrate)[2]

Notes:

Mean results are reported in the parenthesis.

[1] Processing factor = Mean residue in the processed commodity / Mean residue in the RAC. For censored data (<LOQ), the finite LOQ value for the relevant analyte has been used for calculations.

[2] Where residues in the unprocessed commodity (RAC) were <LOQ no processing factor can be quantified and are reported as NC (Not Calculated). If residues were observed at <LOQ in the RAC and ≥LOQ in the processed commodity they are reported as NC (observed to concentrate).

Where residues are >LOQ in the RAC and <LOQ in the processed commodity the processing factor is reported as less than (<).

Tea

Eight processing trials were conducted in Japan in 2018/19 to demonstrate the effect of processing on residues of emamectin benzoate in tea leaves. Samples of dried green tea leaves (>200 g) were collected at 7, 14 and 21 days after application (DAA). At the laboratory, each sample was cut to pieces (about 1 cm square), mixed well, sealed, and stored at about -20 °C for a maximum of 82 days until analysis. Tea infusions were prepared from the unpulverized tea leaves (9 grams) by addition of boiling water (540 mL), which was allowed to stand for 5 minutes and then filtered. Only the 7 DAA samples have been summarized below.

All samples were analysed in accordance with an analytical method for tea similar to RAM 465/01. The LOQ was 0.001 mg/kg for all analytes.

Residues of emamectin and its metabolites decreased in tea infusions (Mean PF=< 0.01). No residues were observed above the LOQ in tea infusions except for one result for B1a at 0.002 mg/kg (Mean PF = < 0.002).

Table 39 Summary of residues of emamectin benzoate (B1a, B1b, 8,9-ZMa, AB1a, FAB1a and MFB1a) in dried green tea and tea infusions at 7 DAA after foliar spray with an EC formulation (10 g/L emamectin)

Trial No., Location, Year, Crop (Variety)	Analyte	Commodity		Mean processing factor[1] (PF)
		Dried green tea leaves (RAC)	Tea infusions	
		Residues (mg/kg)		
JP2018C324A, Kagoshima, Japan, 2018, Tea, (Yamatomidori)	B1a	0.011, 0.011 (0.011)	< 0.001, < 0.001	< 0.0015
	B1b	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	8,9-ZMa	0.003, 0.002 (0.002)	< 0.001, < 0.001	< 0.00830

Trial No., Location, Year, Crop (Variety)	Analyte	Commodity		Mean processing factor[1] (PF)
		Dried green tea leaves (RAC)	Tea infusions	
		Residues (mg/kg)		
	AB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	FAB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	MFB1a	0.007, 0.007 (0.007)	< 0.001, < 0.001	< 0.0023
JP2018C081A, Ibraki, Japan, 2018, Tea (Yabukita)	B1a	0.003, 0.003 (0.003)	< 0.001, < 0.001	< 0.0055
	B1b	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	8,9-ZMa	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	AB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	FAB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	MFB1a	0.002, 0.002 (0.002)	< 0.001, < 0.001	< 0.00830
JP2018C081B, Kochi, Japan, 2018, Tea (Yabukita)	B1a	0.004, 0.004 (0.004)	< 0.001, < 0.001	< 0.0041
	B1b	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	8,9-ZMa	0.002, 0.002 (0.002)	< 0.001, < 0.001	< 0.0083
	AB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	FAB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	MFB1a	0.002, 0.002 (0.002)	< 0.001, < 0.001	< 0.0083
JP2018C081C, Miyazaki, Japan, 2018, Tea (Yamatomidori)	B1a	0.008, 0.008 (0.008)	< 0.001, < 0.001	< 0.0022
	B1b	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	8,9-ZMa	0.003, 0.003 (0.003)	< 0.001, < 0.001	< 0.0055
	AB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	FAB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	MFB1a	0.004, 0.004 (0.004)	< 0.001, < 0.001	< 0.0042
JP2019C109A, Ibaraki, Japan, 2019, Tea (Yabukita)	B1a	0.011, 0.010 (0.010)	< 0.001, < 0.001	< 0.0017
	B1b	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	8,9-ZMa	0.003, 0.003 (0.003)	< 0.001, < 0.001	< 0.0055
	AB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	FAB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	MFB1a	0.006, 0.006 (0.006)	< 0.001, < 0.001	< 0.0028
JP2019C109B, Saitama, Japan, 2019, Tea (Fukumidori)	B1a	0.068, 0.065 (0.066)	0.002, 0.002 (0.002)	0.0005
	B1b	0.002, 0.002 (0.002)	< 0.001, < 0.001	< 0.0083
	8,9-ZMa	0.016, 0.015 (0.016)	< 0.001, < 0.001	< 0.0012
	AB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	FAB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	MFB1a	0.011, 0.010 (0.010)	< 0.001, < 0.001	< 0.0017
JP2019C109C, Kochi, Japan, 2019, Tea (Yabukita)	B1a	0.007, 0.007 (0.007)	< 0.001, < 0.001	< 0.0023
	B1b	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	8,9-ZMa	0.003, 0.003 (0.003)	< 0.001, < 0.001	< 0.0055
	AB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	FAB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	MFB1a	0.003, 0.003 (0.003)	< 0.001, < 0.001	< 0.0055
JP2019C109D, Miyazaki, Japan, 2019, Tea (Yabukita)	B1a	0.022, 0.022 (0.022)	< 0.001, < 0.001	< 0.0008
	B1b	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	8,9-ZMa	0.012, 0.012 (0.012)	< 0.001, < 0.001	< 0.0013
	AB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	FAB1a	< 0.001, < 0.001	< 0.001, < 0.001	NC[2]
	MFB1a	0.017, 0.017 (0.017)	< 0.001, < 0.001	< 0.0010

Notes:

Mean results are reported in the parenthesis.

[1]Processing factor = (Mean residue in the processed commodity/60) / Mean residue in the RAC. For censored data (<LOQ), the finite LOQ value for the relevant analyte has been used for calculations.

[2]Where residues in the unprocessed commodity (RAC) were <LOQ no processing factor can be quantified and are reported as NC (Not Calculated).

Where residues are >LOQ in the RAC and <LOQ in the processed commodity the processing factor is reported as less than (<).

APPRAISAL

Emamectin benzoate is a foliar insecticide derivative of abamectin, which is isolated from fermentation of *Streptomyces avermitilis*, a naturally occurring soil actinomycete. It acts by stimulating the release of γ-aminobutyric acid, an inhibitory neurotransmitter, thus causing insect paralysis within hours of ingestion, and subsequent insect death 2–4 days later. It is also registered for use as a veterinary drug in the treatment of sea lice infestations in salmon and trout in several countries.

Emamectin benzoate was considered for the first time for toxicology and residues by the 2011 JMPR and for new uses by the 2014 JMPR. An ADI of 0–0.0005 mg/kg bw and ARfD of 0.02 mg/kg bw were established.

The Definition of the residue for compliance with the MRL and for the estimation of the dietary exposure for plant and animal commodities*: emamectin B1a benzoate.*

The residue is not fat soluble.

The Meeting received new information on analytical methodology, storage stability and additional supervised residues trials on basil, chives, coffee, flowerhead brassica vegetables, leafy vegetables (including brassica leafy vegetables), soya bean and tea.

Methods of analysis

The Meeting received description and validation data for QuEChERS analytical methods for determination of emamectin B1a benzoate and emamectin B1b benzoate in plant and animal commodities for enforcement as well as a QuEChERs method for determination of emamectin B1a benzoate, emamectin B1b benzoate, and its metabolites 8,9-ZMa, AB1a, MFB1a, and FAB1a in plant commodities. New description and validation data was also received for method RAM 465 in plant commodities.

Method RAM 465/01 was evaluated by the 2011 JMPR and is considered sufficiently validated for emamectin B1a benzoate, emamectin B1b benzoate and the avermectin-like metabolites 8,9-ZMa, AB1a, MFB1a, and FAB1a in high water, high starch, high acid, and high oil matrices. New validation data was provided for RAM 465/02 demonstrating the modified method is valid for determination of emamectin B1a benzoate, emamectin B1b benzoate and the avermectin-like metabolites 8,9-ZMa, AB1a, MFB1a, and FAB1a in coffee commodities (green beans, roasted and instant coffee), grapes, potatoes and tomatoes. The LOQ for this method is 0.001 mg/kg for each matrix and analyte.

Method GRM004.06A involves acetonitrile extraction with QuEChERs salts and final determination by HPLC with triple quadrupole mass spectrometric detection (LC-MS/MS). The method was sufficiently validated for emamectin B1a benzoate, emamectin B1b benzoate and the avermectin-like metabolites 8,9-ZMa, AB1a, MFB1a, and FAB1a in apricots, broad beans (dry), cotton, lettuces, melons, oranges, tobacco (dry leaves), walnuts, wheat (grain) and zucchinis. The LOQ was 0.001 mg/kg for each matrix and analyte.

QuEChERs multi-residues methods for enforcement were validated for the determination of parent emamectin benzoate (B1a and B1b) in both plant (EN 15662:2009-2) and animal (DIN EN 15662:2018) commodities. Both methods involve extraction in acetonitrile/water with QuEChERS salts followed by SPE clean-up, dilution, and quantification by HPLC with triple quadrupole mass spectrometric detection (LC/MS-MS, positive ion spray). The plant commodity method was validated in broad beans (dry), lettuces, oranges, tea, tobacco, walnuts and wheat grain. The animal commodity method was validated in muscle (cattle), liver (cattle), kidney (cattle), fat (cattle) and eggs. The LOQ was 0.001 mg/kg for each matrix and analyte.

The Meeting concluded that methods RAM465/02 and GRM004.06A are suitable analytical methods to measure emamectin B1a benzoate, emamectin B1b benzoate and the avermectin-like metabolites 8,9-ZMa, AB1a, MFB1a, and FAB1a for plant commodities and that QuEChERS multi-residues methods are now available for analysis of emamectin B1a benzoate and emamectin B1b benzoate in both plant and animal commodities.

Stability of pesticide residues in stored analytical samples

The 2011 JMPR found that emamectin B1a benzoate and emamectin B1b benzoate were stable when stored at -20 °C or lower for at least 27 months (804 days) in plant commodities with high water content (tomatoes and green beans with pods), at least 18 months (545 days) in plant commodities with high starch content (potatoes), and at least 9 months in plant commodities with high oil content (cottonseed), and special plant commodities (cotton gin trash) and recommended that storage stability information on a high acid commodity would be desirable.

A storage stability study on a high acid commodity (orange) was provided to the current Meeting demonstrating residues of emamectin benzoate and its metabolites are stable for up to 24 months in whole orange when stored deep frozen.

In storage stability studies conducted concurrently with the supervised residues trials residues of emamectin benzoate and its metabolites were observed to be stable for at least 56 months in fresh basil and 52 months in dried chives, at least 3 months in dried tea leaves and at least 9 months in Chinese broccoli when stored frozen. These durations generally covered the longest period of storage for all samples obtained from supervised residue trials with only minor exceptions for dried chives (56 months stored, demonstrated stability 52 months) and soya beans (10 months stored, demonstrated stability of 9 months in high oil matrices). The Meeting considered any potential losses would be minor and the data for dried chives and soya beans remains valid.

Results of supervised residue trials on crops

The Meeting received supervised residue trials for emamectin benzoate on basil, chives, coffee, broccoli, cauliflower, Chinese broccoli, lettuce leaf, mustard greens, spinach and tea.

Chives

In the United States, the critical GAP for herbs (including chives) is three foliar applications of emamectin benzoate at up to 16.8 g ai/ha (maximum of 50.4 g ai/ha per year) with a retreatment interval of 7 days and a harvest withholding period of 7 days.

The Meeting noted the cGAP for chives is for three applications whilst six applications were applied in the trials. Given the rapid decline of emamectin B1a benzoate and the RTI of 7 days it was concluded that the earlier applications would not contribute significantly to the final residue and the trials were considered suitable for maximum residue level estimation.

In independent trials approximating the critical GAP, residues of emamectin B1a benzoate in fresh chive leaves were (n=4): < 0.001, 0.001 (2) and 0.005 mg/kg (n=4).

The Meeting estimated a maximum residue level of 0.01 mg/kg, an STMR of 0.001 mg/kg and an HR of 0.006 (from a single sample) mg/kg for emamectin B1a benzoate in chives.

Flowerhead brassicas

In Italy, the critical GAP for broccoli and cauliflower is a maximum three foliar applications at 14.2 g ai/ha with a minimum retreatment interval of 7 days and a harvest withholding period of 3 days.

Broccoli

In the independent trials approximating cGAP in Italy, residues of emamectin B1a benzoate in broccoli at 3 DALA were (n=5): < 0.001, 0.001, 0.002, 0.002, and 0.004 mg/kg.

Cauliflower

In the independent trials approximating cGAP in Italy, residues of emamectin B1a benzoate in cauliflower were (n=12): < 0.001 (9) and 0.001 (3) mg/kg.

For the Italian GAP, the five trials on broccoli were considered sufficient to make a recommendation for broccoli individually. Based on the dataset for cauliflower the maximum residue level would be 0.002 mg/kg with an STMR at 0.001 mg/kg and a HR at 0.001 mg/kg.

The Meeting agreed to estimate the maximum residue level for the subgroup of flowerhead brassicas based on the dataset for broccoli. The Meeting estimated a maximum residue level of 0.007 mg/kg, an STMR of 0.002 mg/kg and an HR of 0.004 mg/kg for emamectin B1a benzoate in flowerhead brassicas, subgroup of.

Spinach

In the United States, the critical GAP for leafy vegetables (including spinach) is six foliar applications of emamectin benzoate at 16.8 g ai/ha (maximum of 101 g ai/ha per season) with a retreatment interval of 7 days and a harvest withholding period of 7 days.

In the independent trials approximating cGAP in the United States, residues of emamectin B1a benzoate in spinach were (n=6): < 0.005 (2), 0.006 (2), 0.007 and 0.024 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg, an STMR of 0.006 mg/kg and an HR of 0.036 (from a single sample) mg/kg for emamectin B1a benzoate in spinach.

Brassica leafy vegetables

Chinese broccoli

In Thailand, the GAP for Chinese broccoli is two foliar applications of emamectin benzoate at 14.4 g ai/ha with a retreatment interval of 7 days and a harvest withholding period of 3 days.

In the independent trials matching cGAP in Thailand, residues of emamectin B1a benzoate in Chinese broccoli were (n=6): < 0.005 (5) and 0.12 mg/kg. The HR was 0.14 mg/kg (from a single sample).

Mustard greens

In the United States, the critical GAP for brassica leafy vegetables is six foliar applications of emamectin benzoate at 16.8 g ai/ha (maximum of 101 g ai/ha per season) with a retreatment interval of 7 days and a harvest withholding period of 14 days.

In the independent trials matching cGAP in the United States, residues of emamectin B1a benzoate in mustard greens were (n=6): < 0.005 (2), 0.008, 0.011, 0.014 and 0.108 mg/kg (STMR = 0.01 mg/kg).

The Meeting noted that Mustard greens are a representative crop for the subgroup and concluded the dataset for mustard greens was sufficient for estimation of a maximum residue level for the subgroup of brassica leafy vegetables.

The Meeting estimated a maximum residues level of 0.2 mg/kg, an STMR of 0.01 mg/kg and a HR of 0.219 (from a single sample) mg/kg for the subgroup of brassica leafy vegetables. This maximum residue level will also cover the expected residues of emamectin benzoate for the Thailand GAP for Chinese broccoli.

Soya bean (dry)

In the United States, the critical GAP for soya beans is three foliar applications of emamectin benzoate at 16.8 g ai/ha (maximum of 50.4 g ai/ha per year) with a retreatment interval of 7 days and a harvest withholding period of 28 days.

In the independent trials matching cGAP in the United States residues of emamectin B1a benzoate in soya beans (dry) were (n=19): < 0.001 (19) mg/kg.

The Meeting estimated a maximum residue level of 0.001(*) mg/kg for soya beans (dry). The Meeting noted that residues of emamectin B1a benzoate were also < 0.001 mg/kg (LOQ) following application at 5× rate and concluded an STMR of 0 mg/kg for soya beans (dry) was appropriate.

Basil

In the United States, the critical GAP for herbs is three foliar applications of emamectin benzoate at up to 16.8 g ai/ha (maximum of 50.4 g ai/ha per year) with a retreatment interval of 7 days and a harvest withholding period of 7 days.

The Meeting noted that the cGAP for basil is for three applications whilst six applications were applied in the trials. Given the rapid decline of emamectin B1a benzoate and the RTI of 7 days it was concluded that the earlier applications would not contribute significantly to the final residue and the trials were considered suitable for maximum residue level estimation.

In independent trials approximating the critical GAP, residues of emamectin B1a benzoate in fresh basil leaves and stems were (n=4): 0.001, 0.004, 0.005, and 0.028 mg/kg.

The Meeting estimated a maximum residue level of 0.06 mg/kg, an STMR of 0.0045 mg/kg and an HR of 0.032 (from a single sample) mg/kg for emamectin B1a benzoate in basil leaves.

Tea

In Japan, the critical GAP for tea is one foliar application of emamectin benzoate at 1000-fold dilution (1 g ai/100L) applied at 7 days before plucking.

In the independent trials matching cGAP in Japan, residues of emamectin B1a benzoate in dried green tea leaves were (n=8): 0.003, 0.004, 0.007, 0.008, 0.010, 0.011, 0.022 and 0.066 mg/kg.

The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.009 mg/kg for emamectin B1a benzoate in Tea, Black, Green dried and fermented.

Fate of residues during processing

The fate of emamectin benzoate residues during processing of raw agricultural commodities was investigated in basil, chives, tea and soya beans.

The studies showed that emamectin B1a benzoate concentrated in dried basil (PF=6.4) and dried chives (PF=5.0) but did not concentrate in tea infusions (PF=0.002). Emamectin B1a benzoate was also observed to concentrate in soya bean aspirated grain fractions with finite results of 0.005 and 0.021 mg/kg observed however no finite residues were observed in the RAC therefore a processing factor

cannot be estimated. The Meeting noted residues in the soya bean aspirated grain fractions at 5× cGAP and agreed that expected residues at cGAP would be low and not contribute significantly to the livestock dietary burdens.

Table 40 Processing factors, STMR-Ps and HR-Ps for emamectin B1a benzoate, used for dietary risk assessment and MRL estimation.

Raw commodity	Processed commodity	Processing factor	STMR-P = $STMR_{RAC}$ × PF (mg/kg)	HR-P = HR_{RAC} × PF (mg/kg)	MRL_{RAC} × PF (mg/kg)	Recommended MRL (mg/kg)
Basil	Dried basil	6.4	0.0045 × 6.4 = 0.029	0.032 × 6.4 = 0.205	0.06 × 6.4 = 0.4	0.4
Chives	Dried chives	5.0	0.001 × 5.0 = 0.005	0.005 × 5.0 = 0.025	0.01 × 5.0 = 0.05	0.05
Tea	Tea infusion	0.002	0.009 × 0.002 = 0.000018	-	-	-

Based on the estimated maximum residue level of 0.06 mg/kg, the HR and STMR in basil at 0.032 mg/kg and 0.0045 mg/kg respectively, and the processing factor (6.4) for dried basil, the Meeting estimated a maximum residue level in dried basil (DH 0722) of 0.4 mg/kg (HR-P = 0.205 mg/kg, STMR-P = 0.029 mg/kg).

Based on the estimated maximum residue level of 0.01 mg/kg, the HR and STMR in chives at 0.005 mg/kg and 0.001 mg/kg respectively, and the processing factor (5.0) for dried chives, the Meeting estimated a maximum residue level in dried chives (DH 0727) of 0.05 mg/kg (HR-P = 0.025 mg/kg, STMR-P = 0.005 mg/kg).

Residues in animal commodities

Cattle

Based on the uses considered, kale and turnip tops (brassica leafy vegetables subgroup), soya bean seed and soya bean aspirated grain fractions may be part of the diet. It is noted that the soya bean GAP does not allow grazing or cutting for stock feed.

Estimation of livestock dietary burden

Dietary burdens were calculated for beef cattle and dairy cattle based on feed items presented above. The dietary burdens estimated using the most recent version of the OECD livestock dietary burden calculator diets are presented in Annex 6 and summarised below.

Table 41 Estimated maximum and mean dietary burdens of cattle (emamectin B1a benzoate).

	Animal dietary burden: emamectin B1a benzoate, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	max	mean	max	Mean	max	mean	max	mean
Beef cattle	0.0002#	0.0002#	0.2262❶	0.0302❸	0.1734#	0.0276#	-	-
Dairy cattle	0.1153	0.0155	0.2025❷	0.0232	0.1948#	0.0248❹#	-	-

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues.

❷ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk.

❸ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

The use of emamectin in Australia for brassica leafy vegetables and root and tuber vegetables prohibits the grazing, waste crop or produce for stock food being fed to livestock therefore kale leaves and turnip tops have not been included in the dietary burden calculations for Australia.

The maximum dietary burdens for beef cattle and dairy cattle are estimated to be 0.2302 ppm in the feed (dry weight) and 0.2051 ppm in the feed (dry weight) respectively.

Poultry

From all uses of emamectin benzoate considered by the JMPR, soya bean seed is the only significant feed item consumed by poultry. Given finite residues are not observed in soya bean seed the poultry burden is expected to be insignificant.

Animal commodity maximum residue levels

Cattle

A ruminant feeding study was evaluated by the 2011 JMPR. Lactating Holstein-Friesian cows were dosed daily with emamectin benzoate at 0, 0.03, 0.09 and 0.30 mg/kg dry matter in feed for 28 consecutive days. As noted by the 2011 JMPR, the analytical method could not discriminate between emamectin B1a benzoate and 8,9-ZMa, therefore residues are the sum of both. Since metabolism studies showed that 8,9-ZMa is not formed in livestock, values reported represent the mean and highest residues of emamectin B1a benzoate only. Maximum emamectin B1a benzoate residues at the highest dose were 0.12 mg/kg in liver, 0.042 mg/kg in kidney, 0.015 mg/kg in fat and 0.0061 mg/kg in muscle. Mean residues of emamectin B1a benzoate at the highest dose were 0.097 mg/kg in liver, 0.037 mg/kg in kidney, 0.013 mg/kg in fat, 0.0058 mg/kg in muscle and 0.0032 mg/kg in milk.

The calculations used to estimate highest total residues for use in estimating maximum residue levels, STMR and HR values are shown below.

For maximum residue level estimation, the high residues in the tissues and milk were calculated by interpolating the maximum dietary burden (0.19 ppm) between the relevant feeding levels (0.09 and 0.30 ppm) from the dairy cow feeding study and using the highest tissue concentrations from individual animals within those feeding groups and using the mean milk concentration from those feeding groups.

The STMR values for the tissues and milk were calculated by interpolating the mean dietary burden (0.018 ppm) between the relevant feeding levels (0 and 0.03 ppm) from the dairy cow feeding study and using the mean tissue and milk concentrations from those feeding groups.

Table 42 Residues of emamectin B1a benzoate in animal commodities

	Feed Level (ppm) for milk residues	Emamectin B1a benzoate residues (mg/kg) in milk	Feed Level (ppm) for tissue residues	Emamectin B1a benzoate (mg/kg)			
				Muscle	Liver	Kidney	Fat
HR Determination (beef or dairy cattle)							
Feeding Study	0.09 0.30	0.008 0.0032	0.09 0.30	0.0020 0.0061	0.029 0.12	0.013 0.042	0.0066 0.015
Dietary burden and estimate of highest residue	0.2025	0.0021	0.2262	0.0046	0.088	0.032	0.012
STMR Determination (beef or dairy cattle)							
Feeding Study	0	0	0	0	0	0	0

	Feed Level (ppm) for milk residues	Emamectin B1a benzoate residues (mg/kg) in milk	Feed Level (ppm) for tissue residues	Emamectin B1a benzoate (mg/kg)			
				Muscle	Liver	Kidney	Fat
	0.03	< 0.0005	0.03	< 0.002	0.0086	0.0037	0.0021
Dietary burden and estimate of highest residue	0.0248	< 0.0005	0.0302	< 0.002	0.0071	0.0031	< 0.002

The Meeting estimated a maximum residue level for emamectin B1a benzoate of 0.003 mg/kg in milks, 0.005 mg/kg in meat from mammals other than marine mammals, 0.1 mg/kg in mammalian offal to replace the previous recommendations for milks, meat and offal and confirmed the previous recommendation of 0.02 mg/kg in mammalian fat. The residue in animal commodities is not considered fat soluble.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue for compliance with the MRL and for dietary risk assessment for plant and animal commodities: *emamectin B1a benzoate. The Meeting considers the residue not fat soluble.*

Summary of recommendations are presented below.

Table 43 Residue levels suitable for establishing maximum residue limits and for IEDI and IESTI estimations

CCN	Commodity name	Recommended maximum residue level (mg/kg)		STMR or STMR-P (mg/kg)	HR or HR-P (mg/kg)
		New	Previous		
HH 0722	Basil, leaves	0.06	-	0.0045	0.032
DH 0722	Basil leaves, dry	0.4	-	0.029	0.205
VL 0054	Brassica leafy vegetables, subgroup of	0.2	-	0.010	0.219
VA 2605	Chives	0.01	-	0.001	0.006
DH 2605	Chive, dried	0.05	-	0.005	0.025
VB 0042	Flowerhead brassicas, subgroup of	0.007	-	0.002	0.004
MF 0100	Mammalian fats (except milk fats)	0.02	0.02	0.002	0.012
MM 0095	Meat (from mammals other than marine mammals)	0.005	0.004	0.002	0.0046
ML 0106	Milks	0.003	0.002	0.0005	-
MO 0105	Edible offal (mammalian)	0.1	0.08	0.0071	0.088
VL 0502	Spinach	0.05	-	0.006	0.036
VD 0541	Soya bean (dry)	0.001*	-	0	-
DT 1114	Tea, Black, Green, dried and fermented	0.1	-	0.009	-
	For dietary risk assessment and/or dietary burden calculations				
	Tea infusion	-	-	0.000018	-

DIETARY RISK ASSESSMENT

Emamectin benzoate is also registered for use as a veterinary drug in salmon and trout in several countries with Joint FAO/WHO Expert Committee on Food Additives (JECFA) recommending maximum residues levels for muscle and fillet (muscle + skin) at 0.1 mg/kg. The median residue (0.037 mg/kg) and MRL level reported by JECFA Meeting 78 (2013) have been used for dietary exposure estimations.

Long-term dietary exposure

The International Estimated Daily Intake (IEDI) for emamectin B1a benzoate was calculated for the food commodities for which STMRs were estimated and for which consumption data were available.

The International Estimated Daily Intakes of emamectin B1a benzoate for the 17 GEMS/Food cluster diets, based on estimated STMRs were 2–20 percent of the maximum ADI of 0.0005 mg/kg bw.

The Meeting concluded that the long-term dietary exposure to residues of emamectin B1a benzoate from uses that have been considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for emamectin B1a benzoate is 0.02 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for emamectin B1a benzoate were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The IESTIs varied from 0–70 percent of the ARfD for children and 0–60 percent for the general population.

The Meeting concluded that acute dietary exposure to residues of emamectin B1a benzoate from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Report reference
VV-880043	Alves, M. C.	2020a	Determination of NOA438376, NOA438309, NOA415693 and NOA415692 residues after spraying of A10324 in Coffee (grains) in 5 trials in Brazil. Report No. S19-23631-L1, Date: 11/11/2020, GLP unpublished
VV-880047	Alves, M. C.	2020b	Emamectin Benzoate (B1a + B1b) and its Metabolites-Validation of the Analytical method RAM 465/02 for determination of Emamectin Benzoate (B1a + B1b) in Tomato (fruits), Coffee (grain), Grape (fruits) e Potato (tubers) and its metabolites in Coffee (grain). Report No. S19-23176, Date 11/11/2020, GLP unpublished
VV-921552	Baumy, G.	2021	Emamectin-Independent Laboratory Validation of the QuEChERS Method for the Determination of Residues of Emamectin in Animal Matrices by LC-MS/MS. Report No. RNB20-00065, Date: 30/09/2021, GLP unpublished
VV-393840	Bell, A.	2010	Emamectin Benzoate–Residues Study on Cauliflower in Northern France and the United Kingdom in 2009. Report No. CEMR-4421, Date: 30/07/2010, GLP unpublished
VV-733458	Bledsoe, S.	2019	Emamectin (A10325A)–Magnitude of Residues in or on Soybeans Following Foliar Application, United States 2018. Report No. TK0347414, Date 09/12/2019, GLP unpublished
VV-936356	Delongui, R.	2022	A10324–Magnitude of Emamectin benzoate (Emamectin B1a, Emamectin B1b, and its metabolites NOA438376, NOA438309, NOA415692 and NOA415693) in coffee dry green beans, roasted coffee and instant coffee–Brazil, 2020-2021. Report No. LBS20033, Date: 14/01/2022, GLP unpublished

Code	Author	Year	Title, Report reference
VV-309040	Ediger, K.	1999	Emamectin–Magnitude of the Residues In or On Representative Commodities of Crop Group 5: Brassica (Cole) Leafy Vegetables. Study ID No. 136-98, Date 30/06/1999, GLP unpublished
VV484328	Ediger, K. and Oakes, T.	2005	Emamectin–Magnitude of the Residues in or on Crop Group 4: Leafy Vegetables, Except *Brassica*. Report No. T002301-03, Date: 17/06/2005, GLP unpublished
VV-880049	Evangelista N.	2020	Determination of residues of Emamectin Benzoate after spraying of A10324 in Coffee (grain) in 5 trials in Brazil. Report No. S19-23395, Date: 11/11/2020, GLP unpublished
VV-337967	Eversfield, S.	2007	Emamectin Benzoate (MK244): Residue study on cauliflower in France (South) in 2006. Report No. CEMR-3026, Date: 03/04/2007, GLP unpublished
VV-466838	Ford, K.	2018	Emamectin Benzoate (MK244)-Storage Stability of Residues of Emamectin Benzoate (MK244) in High Acid Commodities (Oranges) Stored Frozen for up to Two Years. Report No. CEMR-7499, Date 07/09/2018, GLP unpublished
VV-696903	Garrigue, P.	2019	Emamectin–Validation of the QuEChERS Method for the Determination of Residues of Emamectin in Crop Matrices by LC-MS/MS. Report No. BPL19-0017 Amendment 01, Date: 15/10/2019, GLP unpublished
VV-733157	Homazava, N.	2019	Emamectin-Independent Laboratory Validation of the QuEChERS Method for the Determination of Residues of Emamectin in Crop Matrices by LC-MS/MS. Report No. 20190183 Amendment 1, Date: 03/12/2019, GLP unpublished
VV-384867	Marshall, L.	2009a	Emamectin Benzoate–Residues Study on cauliflower in France (North), Germany and the United Kingdom in 2008.–2009. Report No. T009254-07-REG, Date: 25/06/2009, GLP unpublished
VV-384805	Marshall, L.	2009b	Emamectin Benzoate–Residues Study on Sprouting Broccoli in Germany and the United Kingdom in 2008. Report No. T009258-07-REG, Date: 13/08/2009, GLP unpublished
VV-888290	Mechelke, J.	2021	Emamectin-Validation of the QuEChERS Method for the Determination of Residues of Emamectin in Animal Matrices by LC-MS/MS. Report No. 20200259 Amendment 1, Date 14/01/2021, GLP unpublished
VV-939261	Melquiades, M. I.	2021	Emamectin Benzoate and Metabolites–Analytical method Validation of the RAM 465/02 for the determination of residues of Emamectin B1a Benzoate, Emamectin B1b Benzoate, NOA438376, NOA438309, NOA415693 and NOA415692 in Coffee beans, roasted coffee and instant coffee by LC-MS/MS. Report No. S21-06372, Date: 05/11/2021, unpublished
VV-884024	Morita, H.	2020	Crop Residue Study on Tea with Affirm Emulsifiable Concentrate. Study No. JP2019C109, Date: 22/06/2020, GLP unpublished
VV-471985	Ogiyama, K.	2019a	Crop Residue Study on Tea with Affirm Emulsifiable Concentrate. Study No. JP2018C324, Date: 18/01/2019, GLP unpublished
VV-472107	Ogiyama, K.	2019b	Crop Residue Study on Tea with Affirm Emulsifiable Concentrate. Study No. JP2018C081, Date: 29/01/2019, GLP unpublished
VV-335824	Oliver-Kang, J.	2006a	Emamectin Benzoate (MK244): Residue study on Broccoli in Southern France and Spain. Study No. CEMS-2654, Date: 23/08/2006, GLP unpublished
VV-335825	Oliver-Kang, J.	2006b	Emamectin Benzoate (MK244): Residue study in Cauliflower in Southern France and Spain. Study No. CEMS-2655, Date: 23/08/2006, GLP unpublished
VV-513125	Samoil, K. S.	2017	Emamectin Benzoate: Magnitude of the Residues on Basil and Chives. Report No. IR-4 PR No. 07137, Date: 06/07//2017, unpublished
VV-887489	Sayed, S.	2020	Emamectin–Validation of the Syngenta Method GRM004.06A for the Determination of Residues of Emamectin and its metabolites in various crops by LC-MS/MS. Study No. S20-05346, Date: 27/11/2020, GLP unpublished

Code	Author	Year	Title, Report reference
VV-128026	Tessier, V. and Braid, S.	2013	Emamectin Benzoate–Analytical Method GRM004.06A for the Determination of NOA426007, NOA438376, NOA422390, NOA415692 and NOA438309 in Crops. Report No. GRM004.06A. Date: 26/02/2013. Non-GLP unpublished
-	Thongsam, C.	2017	Reports on pesticide residue trials 01-06. Agricultural Toxic Substances Division, APSRDD Department of Agriculture, Ministry of Agriculture and Cooperatives, Chatuchak, Bangkok 10900, Thailand, Non-GLP unpublished
VV-500489	Vincent, T. P.	1998	Determination of the magnitude of residues of MK-0244 and its metabolites in/on the raw agricultural commodities, leaf lettuce and spinach, from MK-0244 applied with a non-ionic surfactant by ground equipment. Report No. ABR-98047, Date: 04/08/1998, GLP unpublished

FAMOXADONE (208)

First draft prepared by Dr M Doherty, the Environmental Protection Agency, United States of America

EXPLANATION

Famoxadone was evaluated for the first time by JMPR 2003 when an acceptable daily intake (ADI) of 0–0.006 mg/kg bw and an acute reference dose (ARfD) of 0.6 mg/kg bw were established.

The definition of the residue for compliance with MRLs and for dietary assessment is famoxadone. The residue is fat-soluble.

The current Meeting received information on analytical methods, storage stability, and field trials to support new MRLs in commodities of caneberry, bulb onion, green onion, cucurbits, fruiting vegetables, and hops.

METHODS OF RESIDUE ANALYSIS

AMR 2801-93

This method was evaluated by the 2003 JMPR and found to be suitable for analysis of famoxadone residues in various plant matrices, including cereal grains, straws, and forages, with LOQs of 0.02 mg/kg for foods and 0.05 mg/kg for feeds. This method was used for analysis of residues in Winter wheat and Winter barley. In wheat matrices at the lowest fortification level (0.1 mg/kg), recoveries were 101±11 percent (n=6) in forage, 97±15 percent (n=3) in grain, and 96–101 percent (n=2) in straw.

AMR 3705-95 RV2

This method (DeMario, D., *et al*., 1998, Report ARM 3705-95 RV2) was not previously reviewed by the JMPR. Residues are extracted with acetonitrile/water followed by salting out to separate the solvents. The acetonitrile phase is partitioned against hexane to remove fats and other non-polar interferents. Famoxadone residues are cleaned-up using Florisil chromatography and the analysed by gas chromatography with a nitrogen-phosphorus detector (GC-NP). Method ARM 3705-95 RV2 was used for the analysis of residues in caneberry, cucurbit vegetables, fruiting vegetables, and hops (samples in Study 07796). Mean recoveries across all matrices ranged from 81 to 117 percent, with relative standard deviations of ≤ 20 percent (Table 2). The method was validated for these matrices with an LOQ of 0.02 mg/kg (0.4 mg/kg in hops).

Samples of dry bulb onion, green onion, and hops (samples in Study A7796) were analysed for residues using this method with modifications (Table 1). The method with modifications gave mean recoveries 74 to 105 percent (RSD ≤ 7 percent) for hops and 79–90 percent (RSD ≤ 7 percent) for green onion. The modified method was validated with LOQs of 0.05 mg/kg in hops and 0.5 mg/kg in green onion (while recoveries were satisfactory at 0.02 mg/kg in green onion, there were too few replicates (n=3) to validate that level as the LOQ).

Table 1 Summary of analytical methods

		Method AMR 3705-95 RV2	Method AMR 3705-95 RV2 with modifications
Extraction and clean-up	Analytes	Famoxadone	Famoxadone
	Matrix	Caneberry, cucurbit vegetables, fruiting vegetables, hops	Dry bulb onion, green onion, hops
	Extraction	Add water followed by acetonitrile and homogenize for 3 minutes. Filter into a mixing cylinder containing 50 g NaCl. After the solvents separate, collect 100 mL of the acetonitrile layer and partition against 20 to 100 mL hexane, depending on the sample matrix. Retain and concentrate	

		Method AMR 3705-95 RV2	Method AMR 3705-95 RV2 with modifications
		the acetonitrile phase to 1-2- mL	
	Column	Florisil	Tandem polymeric + NH_2
	Eluent	Ethyl acetate:hexane (1:4, v/v)	Acetonitrile
Chromatography	Type	GC	LC
	Analytical column	DB-5ms	C18 at 35 °C
	Dimensions	15 m × 0.53 mm id	50 × 3 mm
	Sorbent size	1.5 µm film thickness	2.7 µm particles
	Parameters	Injector: 280 °C Column: 260 °C for 1 min, 3 °C/min to 275 °C, hold for 1 min. Detector: 280 °C	Time, min / 10 mM ammonium acetate / Methanol 0.0 / 70 / 30 0.2 / 70 / 30 0.5 / 2 / 98 4.0 / 2 / 98 4.1 / 70 / 30 7.5 / 70 / 30
	Flow rate	Helium at 25 mL/min, 10 mL/min makeup	0.4 mL/min
	Injection volume	2 µL	5 µL
	Instrument	Hewlett-Packard HP5890A	Agilent 6460 LC-MS/MS
Detection	Quantitative detection	Nitrogen-phosphorus	Tandem mass spec, positive ionization m/z: 392.2 → 331.1
	LOQ	0.02 mg/kg (cucurbit veg., fruiting veg., hops, caneberry)	0.05 mg/kg (hops) 0.02 mg/kg (onion)
	Whole method linearity (r^2)	Not reported	R^2 > 0.99

Table 2 Summary of recovery of famoxadone from caneberry, green onion, cucurbit vegetables, fruiting vegetables, barley, and wheat

Crop	Matrix	Fortification level [mg/kg]	Individual recoveries [percent]	Range of recoveries [percent]	Mean recovery [percent]	RSD [percent]	Reference
Caneberry	Berries	0.02	95, 105, 120, 125, 125, 130	95 -130	117	12	08776
		0.5	52, 72, 76, 78, 82, 88, 88, 92, 100, 100, 100	52 -100	84	17	
		5	82, 82, 80, 80, 88	80 -88	82	4	
		10	100, 100, 110	100 -110	103	6	
Onion, green	Whole plant	0.02	78, 78, 82	78 -82	79	3	08303
		0.5	88, 90, 91	88 -91	90	2	
		5	80, 86, 92	80 -92	86	7	
Cucumber	Fruit	0.02	76, 82, 85, 93, 98, 103, 104, 104, 108, 110, 110, 114, 119	76 -119	100	13	4340-97
		0.05	94, 98, 103, 110	94 -110	101	7	
		0.1	97, 101, 111	97 -111	103	7	
		0.2	98, 99, 102, 104, 106, 109	98 -109	103	4	
Cantaloupe	Fruit	0.02	88, 100, 102, 106, 106, 107, 109, 110, 114, 115, 118, 120	88 -120	108	8	
		0.04	96, 101	96 -101	99		
		0.1	110				
		0.2	114				
		0.5	94, 107, 108, 109, 110, 111, 112, 113, 114, 116	94 -116	109	6	
Summer squash	Fruit	0.02	91, 94, 100, 102, 106	91 -106	99	6	
		0.05	108				

Crop	Matrix	Fortification level [mg/kg]	Individual recoveries [percent]	Range of recoveries [percent]	Mean recovery [percent]	RSD [percent]	Reference
		0.1	112, 119	112 -119	116		
		0.5	104, 106, 120	104 -120	110	8	
Tomato	Fruit	0.02	82, 84, 89, 99, 103, 110, 113	82 -113	97	13	3723
		0.25	75				
		0.4	81, 83, 87	81 -87	84	4	
		5	72, 81, 90	72 -90	81	11	
Peppers	Fruit	0.02	91, 93, 106, 114, 129	91 -129	107	15	
		0.2	97, 117	97 -117	107		
		0.4	81, 83, 89	81 -89	84	5	
		5	81				
Peppers/Tomato	Fruit	0.02	77, 77, 78, 78, 80, 81, 84, 85, 86, 96, 101	77 -101	84	9	
		0.05	71, 75, 83, 83, 83, 85, 98, 101, 107, 108, 110	71 -110	91	15	
		0.2	77, 78, 80, 84, 85, 86, 86, 88, 92	77 -92	84	6	
		0.5	65, 74, 75, 92, 92, 102, 106, 116, 117	65 -117	93	20	
Tomato	Fruit	0.02	71, 83, 89, 90	71 -90	83	11	9822
		0.2	67, 83, 92	67 -92	81	16	
		1	74				
Winter wheat	Green forage	0.1	84, 95, 101, 105, 108, 114	84 -114	101	10	AMR 2831-93
		0.5	98				
		1	81				
		5	70, 88, 91, 91	70 -91	85	12	
	Grain	0.1	83, 95, 112	83 -112	97	15	
		0.5	80, 101, 102	80 -102	94	13	
	Straw	0.1	96, 101	96 -101	99		
		5	90, 96	90 -96	93		
		15	76, 76, 80, 88	76 -88	80	7	
Winter barley	Green forage	0.1	89, 93, 101, 105, 120	89 -120	102	12	AMR 2971-94
		0.5	87				
		5	80, 84, 85, 93	80 -93	86	6	
	Grain	0.1	106, 110	106 -110	108		
		0.5	101, 112	101 -112	107		
	Straw	0.1	91				
		5	104				
		15	94, 101, 103, 111	94 -111	102	7	
Hops	Dry cones	0.05	69, 69, 71, 76, 78, 80	69 -80	74	6	A7796
		0.5	74, 84, 84, 88	74 -88	83	7	
		5	102, 104, 109	102 -109	105	3	
		50	104, 105, 106	104 -106	105	1	
		0.4	76, 82	76 -82	79	5	07796
		0.5	70, 75, 76, 77, 82, 83	70 -83	77	6	
		5	92, 92, 94, 95	92 -95	93	2	
		50	88, 90, 92, 98, 100, 100, 101	88 -101	96	6	

STABILITY OF RESIDUES IN STORED ANALYTICAL SAMPLES

Data on the stability of famoxadone was provided for caneberry, cucurbit vegetables, onion, and hops. Data were generated concurrently with the field trials cited below.

The results (Table 3) indicate that residues are stable in high water, high acid, and high oil content commodities during frozen storage for at least the storage durations examined (0.8 to 2 years, depending on the commodity).

Table 3 Summary of stability data for residues of famoxadone residues under frozen conditions

Matrix	Storage time, days [A)]	Procedural recovery, percent	Fortification level, mg/kg	Residue remaining, mg/kg	Mean percent remaining
Caneberry	215-216	92, 100	0.5	96, 100, 102	99
Cucumber	0	91, 107	0.3	102, 106	104
	28	103, 104	0.3	93	93
	96	87, 88	0.3	100, 103	102
	180	99, 102	0.3	95, 98	96
	313	94, 99	0.3	90, 99	94
Onion, dry bulb	873 [B)]	88	0.5	79, 90, 91	87
Onion, green	796	90	0.5	71, 77, 82	77
Hops	252	76, 82	0.4	70, 85	78

Notes:

[A)] 0-day data were provided only for cucumber.

[B)] Two days prior to the termination of the storage stability study, a complete thaw of samples, with temperatures above 0°C, occurred for about 36 hours.

USE PATTERN

Registered labels describing the use of famoxadone were submitted to the present Meeting for caneberries, onions, cucurbit vegetables, fruiting vegetables, wheat, barley, and hops (Table 4).

Table 4 Registered uses of famoxadone submitted to the 2022 JMPR

Use site	Country	Formulation		Application					PHI, days
		Conc.	Type	Rate, kg ai/ha/applic	Rate, kg ai/ha/year	Water, L/ha*	Max No.	Interval, days	
Caneberries [A)]	United States	25 percent	DF	0.175	1.26	187 / 47	n.s.	5	0
Bulb vegetables [B)]				0.175	1.47	187 / 47	n.s.	5	3
Cucurbit vegetables [C)]				0.175	0.56	187 / 47	4	5	3
Tomatoes				0.14	1.26	187 / 47	n.s.	5	3
Peppers (incl. bell and chili)				0.175	1.26	187 / 47	n.s.	5	3
Hops				0.14	0.84	187 / 47	6	6	7

Notes:

* Ground / aerial application.

[A)] US Subgroup 13-07A: Blackberries, black and red raspberries, loganberries, wild raspberries.

[B)] US Group 3-07: Chive, Chinese, fresh leaves; chive, fresh leaves; daylily, bulb; elegans hosta; fritillaria, bulb; fritillaria, leaves; garlic, bulb; garlic, great-headed, bulb; garlic, serpent, bulb; kurrat; lady's leek; leek; leek, wild; lily, bulb; onion, Beltsville bunching; onion, bulb; onion, Chinese, bulb; onion, fresh; onion, green; onion, macrostem; onion, pearl; onion, potato, bulb; onion, tree, tops; onion, Welsh, tops; shallot, bulb; shallot, fresh leaves.

[C)] US Crop Group 9: Chayote, Chinese waxgourd, citron melon, cucumber, gherkin, edible gourd, *Momordica* spp, muskmelon, pumpkin, Summer squashes, Winter squashes, watermelon.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on caneberries, onion (bulb and green), cucurbit vegetables (cucumber, cantaloupe. Summer squash), peppers, tomatoes, and hops. In addition, the Meeting received residue decline data for wheat and barley forage as support for green onion (high-water crop category).

The field trial reports included method validation data, as recoveries from spiked samples at levels reflecting those observed in the field trial samples; dates from critical events during the study, including application, harvest, storage, and analysis; as well as detailed information on the field site and treatment parameters. Analytical reports were sufficiently detailed and included example chromatograms and example calculations. Samples were analysed by the method described above for plant commodities.

The field trial study designs included control plots. Measured residues from control plots were <LOQ and are not included in the summary tables in this evaluation. In all cases, the applied material was co-formulated with cymoxanil.

When calculating average residues, values below the LOQ were assumed to be at the LOQ. In the summary tables, residue values leading to maximum residue estimations and used for long-term dietary risk assessment are underlined. The highest individual values selected for estimating acute dietary risks are in bold font. Values reported are below the method limit of detection are reported as ND.

Supervised trials for famoxadone:

Category	Crop	Table
Berries and other small fruits	Cane berries	Table 5
Bulb vegetables	Bulb onions	Table 6
	Green onions	
Cucurbit vegetables	Cucumber	Table 7
	Summer squash	
	Cantaloupe	
Fruiting vegetables	Tomatoes	Table 8
	Peppers	
Herbs	Hops	Table 9

Cane berries

Seven field trials were conducted in Canada (three trials) and the United States (four trials) during the 2003 season (Carpenter, D., 2006, Report 08766) using a WG formulation. Treatment consisted of six foliar applications of ca. 0.84 kg ai/ha, on generally a 7-day interval. Harvest occurred 0 days after the last application. Deviations to the application parameters were noted for the three trials conducted in Canada.

Following harvest, samples (≥ 454 g) samples were frozen within 1.1 to 4.5 hours of collection and shipped, frozen, to the analytical laboratory. Upon arrive at the facility, samples were put into frozen storage. Prior to analysis, samples were homogenized in the presence of dry ice and then returned to frozen storage. Samples were stored for a maximum of 181 days prior to analysis.

Samples were analysed for residues of famoxadone using Method AMR 3705-95 RV2. Concurrent recovery data indicate that the method is suitable, with an LOQ of 0.02 mg/kg.

Table 5 Results of famoxadone residue trials in cane berries (2003)

Location (Trial ID)	Crop (Variety)	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha			Famoxadone	
Critical GAP (US)	--	n.s. [5]	ns	0.175 (1.26/yr)	187	--	0	--	
Abbotsford, British Columbia, Canada (BC08)	Raspberry (Esquimalt)	1 [-] 2 [7] 3 [6] 4 [12] 5 [7] 6 [6]	0.032 0.031 0.030 0.029 0.030 0.031	0.212 0.210 0.210 0.212 0.211 0.217	653 679 687 736 703 692	Berry	0	1.05, 1.04 [1.0]	08766
Madera, California, United States (CA101)	Boysenberry (Boysenberg)	1 [-] 2 [8] 3 [6] 4 [7] 5 [7] 6 [7]	0.045 0.045 0.045 0.045 0.045 0.045	0.210 0.210 0.211 0.209 0.210 0.210	469 469 469 466 469 468	Berry	0	6.6, 5.5 [6.0]	
Oxnard, California, United States (CA102)	Raspberry (Isabel)	1 [-] 2 [7] 3 [7] 4 [7] 5 [6] 6 [8]	0.041 0.041 0.041 0.041 0.041 0.041	0.212 0.210 0.215 0.214 0.217 0.210	520 515 528 525 532 513	Berry	0	0.48, 0.41 [0.44]	
Boston, Ontario, Canada (ON17)	Raspberry (Nova)	1 [-] 2 [7] 3 [6] 4 [7] 5 [7] 6 [8]	0.042 0.042 0.042 0.042 0.042 0.042	0.208 0.210 0.203 0.210 0.209 0.212	497 502 484 502 498 507	Berry	0	0.68, 0.98 [0.83]	
Aurora, Oregon, United States (OR14)	Blackberry (Marion)	1 [-] 2 [7] 3 [8] 4 [7] 5 [7] 6 [7]	0.056 0.056 0.056 0.056 0.056 0.056	0.211 0.210 0.208 0.208 0.212 0.210	377 375 372 371 379 374	Berry	0	3.2, 2.0 [2.6]	
St. Paul d'Abbotsford, Quebec, Canada (QC19)	Raspberry (Kilarine)	1 [-] 2 [1] 3 [7] 4 [8] 5 [8] 6 [7] 7 [8]	0.046 0.046 0.045 0.046 0.045 0.046 0.046	0.105 0.108 0.216 0.198 0.206 0.227 0.200	230 237 474 433 453 497 436	Berry	0	1.8, 0.45 [1.1]	
Burlington, Washington, United States (WA12)	Raspberry (Meeker)	1 [-] 2 [7] 3 [7] 4 [7] 5 [7] 6 [7]	0.056 0.056 0.056 0.056 0.056 0.056	0.216 0.207 0.212 0.214 0.213 0.216	386 369 379 380 380 383	Berry	0	2.2, 1.7 [2.0]	

Dry bulb and green onion

Twelve field trials (eight dry bulb onion and 4 green onion) were conducted in the United States during the 2002 growing season (Carpenter, D., 2007, Report 08303). Treatment generally consisted of seven foliar applications of famoxadone (WG formulation) at ca. 0.22 kg ai/ha with a retreatment interval of 5–6 days. Harvest occurred 3 days after the last application.

Following harvest, samples (≥ 12 bulb onions; and approximately 1.8 kg green onion) were frozen within 2.5 hours (bulb onion) or 1 hour (green onion) of collection and transported, frozen, to the laboratory. In preparation for analysis, samples were homogenized in the presence of dry ice and returned to frozen storage. Samples were maintained in frozen storage except for the dry bulb onion samples (including storage stability samples) which thawed for approximately 36 hours toward the end of the study. Dry bulb onions were stored for a maximum of 916 days and green onions for a maximum of 795 days prior to analysis. The limit of demonstrated stability in frozen dry bulb onion is 873 days.

Samples were analysed for residues of famoxadone using the modified version of Method AMR 3705-95 RV2, described above. Concurrent recovery data for green onion indicate that the method is suitable.

Table 6 Results of famoxadone residue trials in dry bulb and green onion (2002)

Location (Trial ID)	Crop (Variety)	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha			Famoxadone	
Critical GAP (United States)	--	n.s. [5]	ns	0.175 (1.47/yr)	187	--	3	--	
Salinas, California (CA*06) [A)]	Dry Bulb onion (Ruby)	1 [-] 2 [6] 3 [6] 4 [6] 5 [5] 6 [6] 7 [4]	0.0195 0.020 0.018 0.0175 0.0175 0.015 0.0145	0.1955 0.193 0.1905 0.1955 0.194 0.192 0.194	561 547 591 620 615 710 758	Bulb	3	ND, ND [ND]	08303
Salinas, California (CA*07) [B)]	Dry Bulb onion (Caballero)	1 [-] 2 [4] 3 [5] 4 [6] 5 [6] 6 [6] 7 [5]	0.0205 0.0205 0.0195 0.0195 0.018 0.0175 0.0175	0.197 0.193 0.191 0.1905 0.1975 0.1955 0.1895	537 531 547 541 613 620 601	Bulb	3	ND, ND [ND]	
Fort Collins, Colorado (CO02)	Dry Bulb onion (Vantage)	1 [-] 2 [4] 3 [4] 4 [5] 5 [5] 6 [4] 7 [4]	0.032 0.032 0.032 0.032 0.032 0.032 0.032	0.1915 0.188 0.1905 0.1875 0.1935 0.188 0.1905	335 329 334 328 339 329 333	Bulb	1	ND, ND [ND]	
							3	0.079, 0.077 [0.078]	
							7	0.056, 0.05 [0.053]	
							14	ND, ND [ND]	
Parma, Idaho (ID01)	Dry Bulb onion (Vaquero)	1 [-] 2 [6] 3 [7] 4 [7]	0.028 0.028 0.0275 0.028	0.1905 0.1875 0.1875 0.1865	380 373 379 371	Bulb	3	ND, ND [ND]	

Location (Trial ID)	Crop (Variety)	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha			Famoxadone	
		5 [6] 6 [7]	0.028 0.028	0.191 0.192	381 383				
Freeville, New York (NY01)	Dry Bulb onion (Millenium)	1 [-] 2 [8] 3 [7] 4 [7] 5 [8] 6 [6]	0.065 0.017 0.017 0.017 0.0205 0.0205	0.745 0.1935 0.182 0.1825 0.1985 0.203	652 630 592 594 544 557	Bulb	1	ND, ND [ND]	
							3	0.06, ND [0.06]	
							8	ND, ND [ND]	
							15	ND, ND [ND]	
Aurora, Oregon (OR02)	Dry Bulb onion (Santos F1)	1 [-] 2 [4] 3 [5] 4 [4] 5 [4] 6 [6] 7 [5]	0.035 0.035 0.0345 0.035 0.0345 0.0345 0.0345	0.189 0.188 0.19 0.187 0.187 0.192 0.1905	304 303 306 301 302 310 307	Bulb	3	ND, ND [ND]	
Weslaco, Texas (TX*04)	Dry Bulb onion (Don Victor)	1 [-] 2 [6] 3 [4] 4 [6] 5 [6] 6 [6] 7 [6]	0.028 0.0295 0.0295 0.0295 0.0295 0.0295 0.0295	0.1865 0.1875 0.1895 0.19 0.187 0.1905 0.1875	374 355 362 359 357 360 358	Bulb	2	0.23, 0.20 [0.215]	
Prosser, Washington (WA*02)	Dry Bulb onion (Pinnacle)	1 [-] 2 [4] 3 [5] 4 [5] 5 [4] 6 [4] 7 [6]	0.023 0.0225 0.023 0.023 0.023 0.023 0.023	0.1875 0.1855 0.19 0.1945 0.184 0.1875 0.184	461 458 460 472 444 460 447	Bulb	2	ND, ND [ND]	
Salinas, California (CA*05)	Green onion (White Knight)	1 [-] 2 [6] 3 [6] 4 [5] 5 [6] 6 [4] 7 [4]	0.0195 0.018 0.0175 0.0175 0.015 0.0145 0.0145	0.1905 0.1885 0.1955 0.1875 0.192 0.1915 0.1925	541 584 620 593 710 749 752	Whole plant	4	1.4, 1.3 [1.35]	
Bridgeton, New Jersey (NJ37)	Green onion (Long white bunching)	1 [-] 2 [6] 3 [4] 4 [6] 5 [6] 6 [6] 7 [6]	0.0245 0.0245 0.0245 0.0245 0.0245 0.0245 0.0245	0.1905 0.184 0.184 0.194 0.195 0.184 0.189	438 423 423 446 448 423 435	Whole plant	4	4.6, 4.1 [4.35]	
Weslaco, Texas (TX*05)	Green onion (Ringer grand)	1 [-] 2 [5] 3 [5] 4 [6] 5 [6] 6 [6]	0.0285 0.028 0.0295 0.0295 0.0295 0.0295	0.1865 0.1875 0.187 0.1885 0.1895 0.1885	369 375 357 360 358 361	Whole plant	3	16, 14 [15]	

Location (Trial ID)	Crop (Variety)	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha			Famoxadone	
		7 [6]	0.0295	0.188	356				
Arlington, Wisconsin (WI39)	Green onion (South port white globe)	1 [-]	0.025	0.187	416	Whole plant	3	4.1, 3.4 [3.75]	
		2 [5]	0.0275	0.1895	387				
		3 [5]	0.026	0.186	402				
		4 [5]	0.0265	0.1905	404				
		5 [5]	0.0265	0.193	409				
		6 [6]	0.025	0.1905	428				
		7 [6]	0.025	0.191	425				

Notes:

A) Applications began on 4 Sept 2002

B) Applications began on 26 Aug 2002

Fruiting vegetables, Cucurbits

Seventeen field trials were conducted in the United States during the 1997 and 1998 growing seasons (Nathan, E., 1999, Report AMR 4340-97). Treatment consisted of up to seven foliar applications at either 0.14 or 0.21 kg ai/ha, on a 5-day interval. Samples were harvested 3 days after the last application.

Following harvest, samples (generally 12 fruits for cucumbers and cantaloupe, 24 fruits for summer squash) were put into put into frozen storage within 2.5 hours of collection. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 263 days prior to analysis.

Samples were analysed for residues of famoxadone using the method Method AMR 3705-95. Concurrent recovery data indicate that the method is suitable.

Table 7 Residues of famoxadone in cucurbit vegetables from residue trials in the United States (1997-1998)

Location (Trial ID)	Crop (Variety)	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha			Famoxadone	
Critical GAP (United States)	--	4 [5]	ns	0.175 (0.56/yr)	187	--	3	--	
Winterville, GA (1)	Cucumber (Straight Eight)	1 [-]	0.0713	0.140	196	Fruit	3	0.058, 0.031 [0.044]	AMR 4340-97
		2 [5]	0.0681	0.140	206				
		3 [5]	0.0713	0.140	196				
		4 [5]	0.0713	0.140	196				
		5 [5]	0.0749	0.140	187				
		6 [6]	0.0749	0.140	187				
		7 [6]	0.0713	0.140	196				
		1 [-]	0.107	0.210	196	Fruit	3	0.054, 0.075 [0.064]	
		2 [5]	0.102	0.210	206				
		3 [5]	0.107	0.210	196				
		4 [5]	0.107	0.210	196				
		5 [5]	0.112	0.210	187				
		6 [6]	0.112	0.210	187				
		7 [6]	0.107	0.210	196				
Shawsville, VA	Cucumber	1 [-]	0.0651	0.140	215	Fruit	3	0.033, 0.02 [0.027]	

Location (Trial ID)	Crop (Variety)	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha			Famoxadone	
(2)	(Straight Eight)	2 [5]	0.0651	0.140	215				
		3 [5]	0.0651	0.140	215				
		4 [5]	0.0651	0.140	215				
		5 [5]	0.0651	0.140	215				
		6 [5]	0.0651	0.140	215				
		7 [5]	0.0651	0.140	215				
		1 [-]	0.0977	0.210	215	Fruit	3	0.039, 0.056 [0.048]	
		2 [5]	0.0977	0.210	215				
		3 [5]	0.0977	0.210	215				
		4 [5]	0.0977	0.210	215				
		5 [5]	0.0977	0.210	215				
		6 [5]	0.0977	0.210	215				
		7 [5]	0.0977	0.210	215				
Bradenton, FL (3)	Cucumber (Summertime)	1 [-]	0.0499	0.140	281	Fruit	-1	ND, ND [ND]	
		2 [5]	0.0499	0.140	281				
		3 [4]	0.0499	0.140	281				
		4 [8]	0.0499	0.140	281				
		5 [5]	0.0499	0.140	281				
		6 [5]	0.0499	0.140	281				
		7 [5]	0.0499	0.140	281				
							0	< 0.02, < 0.02 [< 0.02]	
							1	0.022, < 0.02 [0.021]	
							3	< 0.02, < 0.02 [< 0.02]	
							7	ND, ND [ND]	
							15	ND, ND [ND]	
							21	ND, ND [ND]	
							28	ND, ND [ND]	
		1 [-]	0.0749	0.210	281	Fruit	-1	< 0.02, < 0.02 [< 0.02]	
		2 [5]	0.0749	0.210	281				
		3 [4]	0.0749	0.210	281				
		4 [8]	0.0749	0.210	281				
		5 [5]	0.0749	0.210	281				
		6 [5]	0.0749	0.210	281				
		7 [5]	0.0749	0.210	281				
							0	0.046, 0.022 [0.034]	
							1	0.027, 0.05 [0.038]	
							3	< 0.02, < 0.02 [< 0.02]	
							7	ND, ND [ND]	
							15	ND, ND [ND]	
							21	ND, ND [ND]	
							28	ND, ND [ND]	
Paynesville, MN (4)	Cucumber (Bush Baby)	1 [-]	0.0749	0.140	187	Fruit	3	0.039, 0.042 [0.040]	
		2 [5]	0.0749	0.140	187				
		3 [4]	0.0749	0.140	187				
		4 [5]	0.0749	0.140	187				
		5 [5]	0.0749	0.140	187				
		6 [5]	0.0749	0.140	187				
		7 [5]	0.0749	0.140	187				
		1 [-]	0.112	0.210	187	Fruit	3	0.04, 0.068 [0.054]	
		2 [5]	0.112	0.210	187				
		3 [4]	0.112	0.210	187				
		4 [5]	0.112	0.210	187				
		5 [5]	0.112	0.210	187				
		6 [5]	0.112	0.210	187				
		7 [5]	0.112	0.210	187				
Marysville, OH (5)	Cucumber (Burpee Hybrid II)	1 [-]	0.0651	0.140	215	Fruit	3	0.098, 0.032 [0.065]	
		2 [6]	0.0651	0.140	215				
		3 [6]	0.0651	0.140	215				
		4 [5]	0.0651	0.140	215				

Location (Trial ID)	Crop (Variety)	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha			Famoxadone	
		5 [5]	0.0624	0.140	224				
		6 [5]	0.0651	0.140	215				
		7 [5]	0.0599	0.140	234				
		1 [-]	0.0977	0.210	215	Fruit	3	0.091, 0.14 [0.12]	
		2 [6]	0.0977	0.210	215				
		3 [6]	0.0977	0.210	215				
		4 [5]	0.0977	0.210	215				
		5 [5]	0.0936	0.210	224				
		6 [5]	0.0977	0.210	215				
		7 [5]	0.0899	0.210	234				
Donna, TX (6)	Cucumber (Poinsette)	1 [-]	0.0374	0.140	374	Fruit	3	0.1, 0.14 [0.12]	
		2 [5]	0.0374	0.140	374				
		3 [6]	0.0374	0.140	374				
		4 [4]	0.0374	0.140	374				
		5 [6]	0.0374	0.140	374				
		6 [5]	0.0374	0.140	374				
		7 [4]	0.0374	0.140	374				
		1 [-]	0.0562	0.210	374	Fruit	3	0.19, 0.13 [0.16]	
		2 [5]	0.0562	0.210	374				
		3 [6]	0.0562	0.210	374				
		4 [4]	0.0562	0.210	374				
		5 [6]	0.0562	0.210	374				
		6 [5]	0.0562	0.210	374				
		7 [4]	0.0562	0.210	374				
East Brunswick, NJ (7)	Cantaloupe (Pulsar)	1 [-]	0.0713	0.140	196	Fruit	3	0.17, 0.17 [0.17]	
		2 [4]	0.0483	0.140	290				
		3 [6]	0.0483	0.140	290				
		4 [6]	0.0394	0.140	355				
		5 [4]	0.0365	0.140	384				
		6 [5]	0.0288	0.140	486				
		7 [5]	0.0288	0.140	486				
		1 [-]	0.107	0.210	196	Fruit	3	0.21, 0.21 [0.21]	
		2 [4]	0.0725	0.210	290				
		3 [6]	0.0725	0.210	290				
		4 [6]	0.0591	0.210	355				
		5 [4]	0.0548	0.210	384				
		6 [5]	0.0432	0.210	486				
		7 [5]	0.0432	0.210	486				
Marysville, OH (8)	Cantaloupe (Burpee Hybrid PM7)	1 [-]	0.0651	0.140	215	Fruit	3	0.12, 0.2 [0.16]	
		2 [5]	0.0651	0.140	215				
		3 [5]	0.0624	0.140	224				
		4 [5]	0.0624	0.140	224				
		5 [5]	0.0599	0.140	234				
		6 [5]	0.0599	0.140	234				
		7 [5]	0.0576	0.140	243				
		1 [-]	0.0977	0.210	215	Fruit	3	0.46, 0.11 [0.29]	
		2 [5]	0.0977	0.210	215				
		3 [5]	0.0936	0.210	224				
		4 [5]	0.0936	0.210	224				
		5 [5]	0.0899	0.210	234				
		6 [5]	0.0899	0.210	234				
		7 [5]	0.0864	0.210	243				
Donna, TX (9)	Cantaloupe (PS 39094 Hybrid)	1 [-]	0.0624	0.140	224	Fruit	-1	0.066, 0.065 [0.066]	
		2 [5]	0.0651	0.140	215				
		3 [5]	0.0624	0.140	224				
		4 [6]	0.0651	0.140	215				
		5 [4]	0.0651	0.140	215				
		6 [5]	0.0651	0.140	215				
		7 [5]	0.0651	0.140	215				
							0	0.14, 0.16 [0.15]	

Location (Trial ID)	Crop (Variety)	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha			Famoxadone	
							1	0.12, 0.12 [0.12]	
							3	0.11, 0.12 [0.12]	
							7	0.1, 0.11 [0.11]	
							14	0.056, -- [0.056]	
		1 [-]	0.0936	0.210	224	Fruit	-1	0.18, 0.16 [0.17]	
		2 [5]	0.0977	0.210	215				
		3 [5]	0.0936	0.210	224				
		4 [6]	0.0977	0.210	215				
		5 [4]	0.0977	0.210	215				
		6 [5]	0.0977	0.210	215				
		7 [5]	0.0977	0.210	215				
							0	0.24, 0.23 [0.24]	
							1	0.18, 0.15 [0.16]	
							3	0.2, 0.2 [0.20]	
							7	0.17, 0.2 [0.18]	
							14	0.048, -- [0.048]	
Glenn, CA (10)	Cantaloupe (Charentais Melons)	1 [-]	0.0681	0.140	206	Fruit	3	0.09, 0.096 [0.093]	
		2 [6]	0.0713	0.140	196				
		3 [6]	0.0749	0.140	187				
		4 [5]	0.0749	0.140	187				
		5 [5]	0.0749	0.140	187				
		6 [6]	0.0713	0.140	196				
		7 [6]	0.0749	0.140	187				
		1 [-]	0.0977	0.210	215	Fruit	3	0.16, 0.17 [0.16]	
		2 [6]	0.107	0.210	196				
		3 [6]	0.112	0.210	187				
		4 [5]	0.112	0.210	187				
		5 [5]	0.112	0.210	187				
		6 [6]	0.107	0.210	196				
		7 [6]	0.112	0.210	187				
Fresno, CA (11)	Cantaloupe (Hale's Best Jumbo)	1 [-]	0.0300	0.140	468	Fruit	3	0.15, 0.14 [0.15]	
		2 [4]	0.0300	0.140	468				
		3 [5]	0.0200	0.140	702				
		4 [5]	0.0200	0.140	702				
		5 [4]	0.0200	0.140	702				
		6 [6]	0.0200	0.140	702				
		7 [5]	0.0200	0.140	702				
		1 [-]	0.0449	0.210	468	Fruit	3	0.31, 0.26 [0.29]	
		2 [4]	0.0449	0.210	468				
		3 [5]	0.0300	0.210	702				
		4 [5]	0.0300	0.210	702				
		5 [4]	0.0300	0.210	702				
		6 [6]	0.0300	0.210	702				
		7 [5]	0.0300	0.210	702				
Porterville, CA (12)	Cantaloupe (Magnum 45 (PMR-45))	1 [-]	0.0499	0.140	281	Fruit	3	0.15, 0.12 [0.14]	
		2 [6]	0.0499	0.140	281				
		3 [6]	0.0499	0.140	281				
		4 [6]	0.0499	0.140	281				
		5 [6]	0.0499	0.140	281				
		6 [6]	0.0499	0.140	281				
		7 [6]	0.0483	0.140	290				
		1 [-]	0.0749	0.210	281	Fruit	3	0.2, 0.13 [0.16]	
		2 [6]	0.0749	0.210	281				
		3 [6]	0.0749	0.210	281				
		4 [6]	0.0749	0.210	281				
		5 [6]	0.0749	0.210	281				
		6 [6]	0.0749	0.210	281				
		7 [6]	0.0725	0.210	290				
Hamburg, PA	Summer Squash	1 [-]	0.0713	0.140	196	Fruit	3	< 0.02, < 0.02 [< 0.02]	

Location (Trial ID)	Crop (Variety)	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha			Famoxadone	
(13)	(Seneca Supreme)	2 [5]	0.0516	0.140	271				
		3 [5]	0.0441	0.140	318				
		4 [4]	0.0405	0.140	346				
		5 [5]	0.0357	0.140	393				
		6 [5]	0.0319	0.140	440				
		7 [5]	0.0306	0.140	458				
		1 [-]	0.107	0.210	196	Fruit	3	0.035, < 0.02 [0.028]	
		2 [5]	0.0775	0.210	271				
		3 [5]	0.0661	0.210	318				
		4 [4]	0.0607	0.210	346				
		5 [5]	0.0535	0.210	393				
		6 [5]	0.0478	0.210	440				
		7 [5]	0.0459	0.210	458				
Rose Hill, NC (14)	Summer Squash (Embassy)	1 [-]	0.0749	0.140	187	Fruit	3	0.064, 0.057 [0.060]	
		2 [6]	0.0749	0.140	187				
		3 [4]	0.0499	0.140	281				
		4 [5]	0.0499	0.140	281				
		5 [5]	0.0499	0.140	281				
		6 [6]	0.0499	0.140	281				
		7 [5]	0.0374	0.140	374				
		1 [-]	0.112	0.210	187	Fruit	3	0.073, 0.054 [0.064]	
		2 [6]	0.112	0.210	187				
		3 [4]	0.0749	0.210	281				
		4 [5]	0.0749	0.210	281				
		5 [5]	0.0749	0.210	281				
		6 [6]	0.0749	0.210	281				
		7 [5]	0.0562	0.210	374				
Bradenton, FL (15)	Summer Squash (Pic N Pic)	1 [-]	0.0300	0.140	468	Fruit	3	0.19, 0.15 [0.17]	
		2 [5]	0.0300	0.140	468				
		3 [5]	0.0300	0.140	468				
		4 [5]	0.0300	0.140	468				
		5 [5]	0.0300	0.140	468				
		6 [5]	0.0300	0.140	468				
		7 [5]	0.0300	0.140	468				
		1 [-]	0.0449	0.210	468	Fruit	3	0.081, 0.076 [0.078]	
		2 [5]	0.0449	0.210	468				
		3 [5]	0.0449	0.210	468				
		4 [5]	0.0449	0.210	468				
		5 [5]	0.0449	0.210	468				
		6 [5]	0.0449	0.210	468				
		7 [5]	0.0449	0.210	468				
Paynesville, MN (16)	Summer Squash (Dividend)	1 [-]	0.0749	0.140	187	Fruit	3	0.1, 0.083 [0.092]	
		2 [5]	0.0749	0.140	187				
		3 [5]	0.0749	0.140	187				
		4 [4]	0.0749	0.140	187				
		5 [5]	0.0749	0.140	187				
		6 [5]	0.0749	0.140	187				
		7 [5]	0.0749	0.140	187				
		1 [-]	0.112	0.210	187	Fruit	3	0.37, 0.15 [0.26]	
		2 [5]	0.112	0.210	187				
		3 [5]	0.112	0.210	187				
		4 [4]	0.112	0.210	187				
		5 [5]	0.112	0.210	187				
		6 [5]	0.112	0.210	187				
		7 [5]	0.112	0.210	187				
Madera, CA (17)	Summer Squash (Hybrid Squash 010)	1 [-]	0.0499	0.140	281	Fruit	3	0.24, 0.20 [0.22]	
		2 [4]	0.0499	0.140	281				
		3 [6]	0.0499	0.140	281				
		4 [6]	0.0499	0.140	281				
		5 [5]	0.0300	0.140	468				

Location (Trial ID)	Crop (Variety)	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha			Famoxadone	
		6 [4]	0.0300	0.140	468				
		7 [4]	0.0300	0.140	468				
		1 [-]	0.0749	0.210	281	Fruit	3	0.19, 0.2 [0.20]	
		2 [4]	0.0749	0.210	281				
		3 [6]	0.0749	0.210	281				
		4 [6]	0.0749	0.210	281				
		5 [5]	0.0449	0.210	468				
		6 [4]	0.0449	0.210	468				
		7 [4]	0.0449	0.210	468				

Fruiting vegetables other than cucurbits

Two studies were submitted to the Meeting. In the first study (McClory, J., 2001, DuPont-3723), field trials were conducted in the United States during the 2000/2001 growing season with tomato (n=13) and bell and non-bell peppers (n=11). Treatment consisted of six applications of a WG formulation, each at 0.21 kg ai/ha on approximately a 5-day interval. Samples were harvested approximately 3 DALA. For tomatoes and bell peppers, samples were 24 fruits (or 12 fruits from large-fruiting varieties); for non-bell peppers, samples were 2 kg of fruits.

Following harvest, samples were bagged and put into put into frozen storage within 2 hours of collection. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 181 days prior to extraction.

In the second study (Hornshuh, M., 2003, Dupont-9822), four trials were conducted in the United States during the 2002 growing season with tomato. Application, timing, and harvest parameters were the same as described in the previous study, and samples were placed into storage within 3 hours of collection. Samples were stored for a maximum of 148 days prior to extraction.

For both studies, samples were analysed for residues of famoxadone using the Method AMR 3705-95. Concurrent recovery data indicate that the method is suitable.

Table 8 Residues of famoxadone in tomato and pepper from residue trials in the United States

Location (Trial ID) Year	Crop (Variety)	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha		Famoxadone	
Tomato Critical GAP (United States)	--	n.s. [5]	ns	0.140 (1.26/yr)	187	3		
North Rose, NY (1) 2000	Tomato (Mountain Pride)	1 [-]	0.0894	0.210	235	2	0.14, 0.34 [0.24]	DuPont-3723
		2 [5]	0.0901	0.210	233			
		3 [5]	0.0921	0.210	228			
		4 [5]	0.0897	0.210	234			
		5 [5]	0.0901	0.210	233			
		6 [5]	0.0901	0.210	233			
Athens, GA (2) 2000	Tomato (Better Boy)	1 [-]	0.0847	0.210	248	3	0.34, 0.32 [0.33]	
		2 [5]	0.0742	0.210	283			
		3 [5]	0.0742	0.210	283			

Location (Trial ID) Year	Crop (Variety)	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha		Famoxadone	
		4 [5]	0.0737	0.210	285			
		5 [5]	0.0727	0.210	289			
		6 [5]	0.0698	0.210	301			
Hobe Sound, FL (3) 2000	Tomato (Florida 47)	1 [-]	0.0579	0.210	363	3	0.14, 0.13 [0.14]	
		2 [5]	0.0605	0.210	347			
		3 [5]	0.0541	0.210	388			
		4 [5]	0.0547	0.210	384			
		5 [5]	0.0590	0.210	356			
		6 [5]	0.0574	0.210	366			
Bradenton, FL (4) 2000	Tomato (Sunpride)	1 [-]	0.0225	0.210	935	3	0.18, 0.16 [0.17]	
		2 [4]	0.0225	0.210	935			
		3 [5]	0.0225	0.210	935			
		4 [5]	0.0225	0.210	935			
		5 [5]	0.0225	0.210	935			
		6 [5]	0.0225	0.210	935			
Rochelle, IL (5) 2000	Tomato (Jet Star)	1 [-]	0.0886	0.210	237	3	0.17, 0.13 [0.15]	
		2 [5]	0.0886	0.210	237			
		3 [6]	0.0695	0.210	302			
		4 [4]	0.0693	0.210	303			
		5 [5]	0.0695	0.210	302			
		6 [5]	0.0682	0.210	308			
Madera, CA 2000 (6)	Tomato (HyPeel 108)	1 [-]	0.0450	0.210	467	3	0.32, 0.28 [0.30]	
		2 [5]	0.0455	0.210	462			
		3 [5]	0.0462	0.210	455			
		4 [5]	0.0452	0.210	465			
		5 [5]	0.0452	0.210	465			
		6 [5]	0.0454	0.210	463			
Fresno, CA (7) 2000	Tomato (Roma)	1 [-]	0.0750	0.210	280	3	0.24, 0.22 [0.23]	
		2 [5]	0.0758	0.210	277			
		3 [5]	0.0745	0.210	282			
		4 [5]	0.0764	0.210	275			
		5 [5]	0.0747	0.210	281			
		6 [5]	0.0747	0.210	281			
Glenn, CA (8) 2000	Tomato (HyPeel 108)	1 [-]	0.0917	0.210	229	3	0.63, 0.79 [0.71]	
		2 [6]	0.0938	0.210	224			
		3 [5]	0.0950	0.210	221			
		4 [4]	0.0942	0.210	223			
		5 [5]	0.0925	0.210	227			
		6 [6]	0.0933	0.210	225			
Porterville, CA (9) 2000	Tomato (UF-6203)	1 [-]	0.0739	0.210	284	3	0.47, 0.34 [0.40]	
		2 [5]	0.0737	0.210	285			
		3 [5]	0.0742	0.210	283			
		4 [5]	0.0742	0.210	283			
		5 [5]	0.0742	0.210	283			
		6 [5]	0.0742	0.210	283			
Easton, CA (10) 2000	Tomato (Shady Lady)	1 [-]	0.0300	0.210	701	3	0.52, 0.48 [0.50]	
		2 [4]	0.0300	0.210	701			
		3 [5]	0.0300	0.210	701			
		4 [5]	0.0300	0.210	701			
		5 [4]	0.0300	0.210	701			
		6 [4]	0.0300	0.210	701			
Le Grand, CA	Tomato	1 [-]	0.0561	0.210	374	3	0.14, 0.15 [0.15]	

Location (Trial ID) Year	Crop (Variety)	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha		Famoxadone	
(11) 2000	(U370)	2 [5] 3 [5] 4 [5] 5 [5] 6 [5]	0.0561 0.0561 0.0561 0.0561 0.0561	0.210 0.210 0.210 0.210 0.210	374 374 374 374 374			
Maxwell, CA (12) 2000	Tomato (9280)	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [5]	0.113 0.113 0.113 0.113 0.112 0.112	0.210 0.210 0.210 0.210 0.210 0.210	186 186 186 186 187 187	3	0.21, 0.13 [0.17]	
Maricopa, AZ (13) 2000	Tomato (Early Girl)	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [4]	0.0714 0.0737 0.0755 0.0742 0.0714 0.0781	0.210 0.210 0.210 0.210 0.210 0.210	294 285 278 283 294 269	3	0.14, 0.14 [0.14]	
Glenn, CA (1) 2002	Tomato (H8892)	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [5]	0.0968 0.0950 0.0950 0.0977 0.0959 0.0950	0.210 0.210 0.210 0.210 0.210 0.210	217 221 221 215 219 221	3	0.52 (0.43, 0.61)[A], 0.71 (0.59, 0.83)[A] [0.615]	DuPont-9822
Porterville, CA (2) 2002	Tomato (Early Girl)	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [5]	0.0372 0.0372 0.0368 0.0376 0.0372 0.0374	0.210 0.210 0.210 0.210 0.210 0.210	565 564 570 558 565 562	3	0.39, 0.33 [0.36]	
Fresno, CA (3) 2002	Tomato (Roma)	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [5]	0.0769 0.0761 0.0758 0.0772 0.0769 0.0755	0.210 0.210 0.210 0.210 0.210 0.210	273 276 277 272 273 278	3	0.43, 0.42 [0.42]	
Madera, CA (4) 2002	Tomato (HP303)	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [5]	0.0448 0.0446 0.0439 0.0441 0.0446 0.0446	0.210 0.210 0.210 0.210 0.210 0.210	469 471 478 476 471 471	3	0.63, 0.74 [0.68]	
Peppers Critical GAP (United States)	--	n.s. [5]	ns	0.175 (1.26/yr)	187	3	--	
Rose Hill, NC (14) 2000	Pepper (Jupiter Sterling) [Bell]	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [5]	0.0886 0.0901 0.0905 0.0909 0.0890 0.0886	0.210 0.210 0.210 0.210 0.210 0.210	237 233 232 231 236 237	3	0.22, 0.18 [0.20]	DuPont-3723
Bradenton, FL	Pepper	1 [-]	0.0449	0.210	468	3	0.57, 0.79 [0.68]	

Location (Trial ID) Year	Crop (Variety)	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha		Famoxadone	
(15) 2000	(Y3R Camelot) [Bell]	2 [4] 3 [5] 4 [5] 5 [5] 6 [5]	0.0449 0.0449 0.0449 0.0449 0.0449	0.210 0.210 0.210 0.210 0.210	468 468 468 468 468			
Rochelle, IL (16) 2000	Pepper (Sweet California Wonder) [Bell]	1 [-] 2 [5] 3 [5] 4 [5] 5 [6] 6 [4]	0.0890 0.0890 0.0886 0.0886 0.0695 0.0693	0.210 0.210 0.210 0.210 0.210 0.210	236 236 237 237 302 303	3	0.085, 0.070 [0.078]	
Donna, TX (17) 2001	Pepper (Capistrano) [Bell]	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [4]	0.0938 0.0938 0.0938 0.0938 0.0938 0.0938	0.210 0.210 0.210 0.210 0.210 0.210	224 224 224 224 224 224	4	0.36, 0.33 [0.34]	
Madera, CA (18) 2000	Pepper (Jupiter) [Bell]	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [5]	0.0451 0.0453 0.0453 0.0450 0.0455 0.0462	0.210 0.210 0.210 0.210 0.210 0.210	466 464 464 467 462 455	3	0.37, 0.35 [0.36]	
Yuma, AZ (19) 2000	Pepper (Indra) [Bell]	1 [-] 2 [5] 3 [5] 4 [6] 5 [5] 6 [7]	0.0747 0.0747 0.0747 0.0747 0.0747 0.0747	0.210 0.210 0.210 0.210 0.210 0.210	281 281 281 281 281 281	3	0.67, 0.59 [0.63]	
Rincon, NM (20) 2000	Pepper (Keystone Resistant Giant #3) [Bell]	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [5]	0.0633 0.0665 0.0616 0.0629 0.0654 0.0642	0.210 0.210 0.210 0.210 0.210 0.210	332 316 341 334 321 327	3	0.15, 0.18 [0.16]	
Dill City, OK (21) 2000	Pepper (Serrano) [Non-bell]	1 [-] 2 [6] 3 [5] 4 [4] 5 [5] 6 [5]	0.0955 0.0917 0.0929 0.0925 0.0921 0.0942	0.210 0.210 0.210 0.210 0.210 0.210	220 229 226 227 228 223	3	3.7, 3.6 [3.6]	
Claude, T x(22) 2000	Pepper (Big Jim Numex) [Non-bell]	1 [-] 2 [5] 3 [5] 4 [5] 5 [5] 6 [5]	0.0605 0.0605 0.0593 0.0616 0.0602 0.0587	0.210 0.210 0.210 0.210 0.210 0.210	347 347 354 341 349 358	3	0.54, 0.40 [0.47]	
Berthoud, CO (23) 2000	Pepper (New Mexico 6) [Non-bell]	1 [-] 2 [5] 3 [5] 4 [5] 5 [5]	0.101 0.101 0.101 0.101 0.101	0.210 0.210 0.210 0.210 0.210	207 207 207 208 207	3	0.46, 0.56 [0.51]	

Location (Trial ID) Year	Crop (Variety)	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., kg ai/hL	Rate, kg ai/ha	L/ha		Famoxadone	
		6 [5]	0.100	0.210	209			
Madera, CA (24) 2000	Pepper (Santa Fe Grande) [Non-bell]	1 [-]	0.0451	0.210	466	3	0.73, 0.46 [0.60]	
		2 [5]	0.0453	0.210	464			
		3 [5]	0.0453	0.210	464			
		4 [5]	0.0450	0.210	467			
		5 [5]	0.0455	0.210	462			
		6 [5]	0.0462	0.210	455			

Notes:

[A)] Replicate analyses.

Hops

Four field trials were conducted in the United States during the 2000 (Thompson, D., 2005, Report No. 07796; three trials) or 2014 (Homa, K., 2016, Report A7796; one trial) seasons using a DF formulation of famoxadone. Treatment consisted of six foliar applications, each at ca. 0.28 kg ai/ha, on a 7-day interval. Samples of mature hop cones were harvested at approximately 7 DALA and dried using a crop dryer or a kiln. After drying, samples (approximately 0.5 kg) were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 205 days prior to analysis. Concurrent storage stability (Report 07796) samples demonstrated that residues were stable for at least 253 days of frozen storage.

Samples were analysed for residues of famoxadone using Method AMR 3705-95. Concurrent recovery data indicate that the method is suitable.

Table 9 Residues of famoxadone in dried hops cones from residue trials in the United States

Location (Trial ID) Year	Crop (Variety)	Application			DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha		Famoxadone	
Critical GAP (United States)	--	6 [6]	0.14 (0.84/yr)	187	7	--	
Prosser, WA (00-WA21) 2000	Hops (Nugget)	1 [-]	0.29	1338	8	15, 16 [15]	07796
		2 [7]	0.28	1319			
		3 [7]	0.28	1319			
		4 [6]	0.28	1871			
		5 [8]	0.29	1338			
		6 [6]	0.29	1300			
Hubbard, OR (00-OR29) 2000	Hops (Nugget)	1 [-]	0.27	664	6	39, 44 [42]	
		2 [7]	0.28	655			
		3 [7]	0.28	664			
		4 [7]	0.28	655			
		5 [7]	0.29	664			
		6 [7]	0.29	683			
Parma, ID (00-ID17)	Hops (Nugget)	1 [-]	0.27	907	8	44, 47 [46]	
		2 [7]	0.28	954			

Location (Trial ID) Year	Crop (Variety)	Application			DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha		Famoxadone	
2000		3 [7] 4 [7] 5 [7] 6 [7]	0.28 0.29 0.28 0.28	954 973 954 954			
Prosser, WA (14-WA477) 2014	Hops (Tomahawk)	1 [-] 2 [7] 3 [7] 4 [7] 5 [7] 6 [6]	0.56 0.56 0.56 0.56 0.55 0.56	1459 1450 1450 1459 1450 1450	8	26, 26 [26]	A7796
		1 [-] 2 [7] 3 [7] 4 [7] 5 [7] 6 [6]	0.28 0.28 0.28 0.28 0.28 0.28	1450 1450 1441 1450 1459 1450	1	14, 14 [14]	
					3	18, 18 [18]	
					7	14, 15 [14]	
					14	8.9, 10 [9.4]	
					21	8.1, 10 [9.0]	

Forage of cereals

Field trial residue decline studies in winter barley (Dubey, L., *et al*., 1996, Report AMR 2971-94) and winter wheat (Dubey, L., *et al*., 1996, Report AMR 2831-93) were submitted to the Meeting as support for the high-water content commodities above. Field trials for cereal foods and feeds were evaluated by the 2003 Meeting and Codex MRLs have been established for cereal commodities.

Two field trials were conducted in *winter barley* during the 1994 growing season in France and Germany (one trial each). Application consisted of two treatments, each at 0.2 kg ai/ha, on a 30-day interval. Samples of green forage (ca. 1 kg each) were collected immediately prior to and at multiple times after the last application. Samples were placed into frozen storage after collection from the field and during transportation to the analytical facility. Samples were homogenized using a cutter and stored for up to 386 days prior to analysis. Residues of famoxadone were analysed using Method AMR 2801-93, which was evaluated by the 2003 Meeting and found to be suitable.

Two field trials were conducted in *winter wheat* during the 1994 growing season in Germany and France (one trial each). Application consisted of three treatments, each at 0.2 kg ai/ha, on a 30-day interval. Samples of green forage (ca. 1 kg each) were collected immediately prior to and at multiple times after the last application. Samples were placed into frozen storage after collection from the field and during transportation to the analytical facility. Samples were homogenized using a cutter and stored for up to 318 days prior to analysis. Residues of famoxadone were analysed using Method AMR 2801-93, which was evaluated by the 2003 Meeting and found to be suitable.

Table 10 Residues of famoxadone in winter barley and winter wheat forage from residue trials in France and Germany

Location (Trial ID) Year	Crop (Variety)	Application			DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha		Famoxadone	
Sancergues, Cher FR (1) 1994	Winter barley (Plaisant)	1 [-] 2 [12]	0.2 0.2	208-210	-1 h	2.80, 3.07, 3.09 [2.99]	AMR 2971-94
					+ 2h	8.26, 9.31, 7.44 [8.34]	
					3	6.48, 6.08, 5.22 [5.93]	
					7	3.85, 4.34, 5.21 [4.47]	
					14	2.41, 2.86, 3.20 [2.82]	
					21	2.92, 2.84, 2.60 [2.79]	
Seehausen, Saxonia DE (2) 1994	Winter barley (Masto)	1 [-] 2 [8]	0.2 0.2	300	-0 h	1.87	
					+0 h	7.05	
					3	4.47	
					7	3.23	
					14	2.73	
					22	2.25	
Villeneuve, St. Nicolas FR	Winter wheat (Soisson)	1 [-] 2 [5] 3 [24]	0.2 0.2 0.2	188-214	-1 h	2.00, 1.96, 1.95 [1.97]	AMR 2831-93
					+1 h	5.92, 6.36, 5.83 [6.04]	
					3	4.72, 4.43, 3.01 [4.05]	
					7	4.78, 4.13, 3.93 [4.28]	
					14	3.11, 2.96, 3.35 [3.14]	
					21	2.10, 2.11, 3.53 [2.58]	
Kirchheim, Rheinland Pfalz, DE	Winter wheat (Greif)	1 [-] 2 [7] 3 [20]	0.2 0.2 0.2	409-414	7	5.60, 6.41, 6.80 [6.27]	
					14	4.35, 5.04, 3.33 [4.24]	
					21	3.60, 4.06, 6.22 [4.63]	

APPRAISAL

Famoxadone (ISO common name) is an oxazolidinedione fungicide belonging to the quinol inhibitor family, which inhibits mitochondrial respiration of fungi. It was evaluated for the first time by JMPR 2003, which established an acceptable daily intake (ADI) of 0–0.006 mg/kg bw and an acute reference dose (ARfD) of 0.6 mg/kg bw. Famoxadone was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional MRLs in 2021 and rescheduled to the 2022 JMPR.

The definition of the residue for compliance with MRLs and for dietary assessment is famoxadone. The residue is fat-soluble.

The current Meeting received information on analytical methods and supervised residue trials to support new MRLs in cane berries, bulb vegetables, cucurbit vegetables, fruiting vegetables, and hops.

Methods of residue analysis

The Meeting received method validation and concurrent recovery data for use of Method AMR 2801-93 (reviewed by 2003 JMPR) and Method AMR 3705-95 RV2. All methods were demonstrated to have adequate performance for recovery of famoxadone, with an LOQ of 0.02 mg/kg in most commodities tested; exceptions are Winter barley and wheat forage (0.1 mg/kg) and dried hops cones (0.05 mg/kg).

Stability of residues in stored analytical samples

Concurrent storage stability data were provided for cane berry, cucumber, bulb onion, green onion, and hops (dried cones). Residues of famoxadone were stable in the tested commodities for at least the tested storage durations:

Cane berry: at least 216 days (7 months),

Cucumber: at least 313 days (10.3 months),

Bulb onion: at least 873 days (28.7 months),

Green onion: at least 796 days (26.2 months), and

Hops (dried cones): at least 252 days (8.3 months).

Results of supervised residue trials on crops

The Meeting received data from supervised residue trials and GAP information on cane berries; bulb and green onion; cucumber, cantaloupe, and summer squash; tomatoes and peppers, and hops. In addition, the meeting received residue decline data for forage of Winter barley and Winter wheat to supplement data submitted to the 2003 Meeting and to provide support for other high-water commodities for which residue decline data were not available.

Cane berries

The GAP for cane berries is from the United States. The label allows multiple applications at a maximum rate of 0.175 kg ai/ha, on a 5-day interval, with a 0-day PHI. The maximum number of applications is not specified. Based on the listed annual limit of 1.26 kg ai/ha, the application pattern for the cGAP is one application at 0.035 kg ai/ha followed by 7 applications at 0.175 kg ai/ha.

Field trials in the United States were conducted with 6 applications on generally a 7-day interval (generally ranging from 6–8 days with one trial having one application done at a 12-day interval) at a rate of approximately 0.21 kg ai/ha, with harvest 0 DALA. The Meeting agreed that an application 35 days before harvest would not contribute significantly to residues and that the difference in retreatment

interval between the cGAP and the trials for one application would not affect residue levels by more than 25 percent. Therefore, the Meeting agreed that the field trials for cane berries are suitable for estimating residues.

Residues of famoxadone in cane berries from independent trials approximating the critical GAP were (n=7): 0.44, 0.83, 1.0, 1.1, 2.0, 2.6, and 6.0 mg/kg.

Noting that the registered use corresponds to the Codex Subgroup of cane berries (FB 2005), the Meeting estimated a maximum residue level of 10 mg/kg for famoxadone in the Subgroup of cane berries, an STMR of 1.1 mg/kg, and an HR of 6.6 mg/kg (from a single sample).

Bulb vegetables

The GAP for bulb vegetables is from the United States and consists of multiple applications each at 0.175 kg ai/ha, on a 5-day interval, with a 3-day PHI. A maximum number of applications is not specified. Based on the maximum rate per crop cycle of 1.47 kg ai/ha, the application pattern for the cGAP is one application at 0.07 kg ai/ha followed by 8 applications at 0.175 kg ai/ha.

Onion, bulb

Field trials in the United States were conducted with 6 or 7 applications on generally a 6-day interval (ranging from 4-8 days) at a rate of approximately 0.19 kg ai/ha, with harvest 2-3 DALA. The Meeting agreed that the initial applications from the cGAP would not likely contribute significantly to residues at harvest and that the trials were suitable for estimating residues.

From independent trials approximating the critical GAP, residues of famoxadone in bulb onion were (n=7): < 0.02 (4), 0.06, 0.078, and 0.22 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg, an STMR of 0.02 mg/kg, and an HR of 0.23 mg/kg (from a single sample) for famoxadone in the subgroup of bulb onion.

Onion, green

Field trials in the United States were conducted with 7 applications on generally a 6-day interval (ranging from 4-6 days) at a rate of approximately 0.19 kg ai/ha, with harvest 3 or 4 DALA. The Meeting agreed that the initial applications from the cGAP would not likely contribute significantly to residues at harvest and that the trials were suitable for estimating residues.

From independent trials approximating the critical GAP, residues of famoxadone in green onion were (n=4): 1.4, 3.8, 4.4, and 15 mg/kg.

The Meeting agreed that four trials was insufficient for estimating residues in green onions.

Cucurbit vegetables

The 2003 JMPR recommended maximum residue level for famoxadone in cucumber and summer squash, each at 0.2 mg/kg based on a GAP from Italy. The Meeting received a more critical GAP from the United States consisting of 4 applications each at 0.175 kg ai/ha, on a 5-day interval, with a 3-day PHI. The new GAP applies to the group of cucurbit vegetables.

Field trials in the United States were conducted in cucumber, summer squash, and melon with 7 applications on generally a 5-day interval (ranging from 4–8 days) at approximate rates of either 0.14 or 0.21 kg ai/ha in side-by-side trials, with harvest 3 DALA. Residue-decline data for cucurbit vegetables were insufficient to provide a robust estimation of half-life. The Meeting agreed that for cucumber and summer squash, the first three applications from the field trials would not lead to significant residues at

harvest due to significant growth dilution and that the trials for those crops are suitable for making recommendations. As both 0.14 and 0.21 kg ai/ha fall within 25 percent of the cGAP application rate of 0.175 kg ai/ha, the Meeting chose whichever result from the side-by-side trials was greater when making its residue estimates. For melons, fruit development is slower than for cucumber and squashes, and the Meeting agreed that contributions to residues at harvest from the first few applications could not be excluded; therefore, the trials in melons did not reflect the cGAP.

Fruiting vegetables, Cucurbits – Cucumbers and Summer Squashes

Cucumber

Residues of famoxadone in cucumber from independent trials were (n=6): < 0.02, 0.048, 0.054, 0.064, 0.12, and 0.16 mg/kg.

Summer squash

Residues of famoxadone in summer squash from independent trials were (n=5): 0.028, 0.064, 0.17, 0.22, and 0.26 mg/kg.

The Meeting noted that the recommended representative commodities for the subgroup of cucumber and summer squashes are cucumber and summer squash and that the median residues from cucumber and summer squash trials are within five-fold. The residue populations are different (Mann-Whitney H test). Based on the dataset from summer squash, the Meeting estimated a maximum residue level of 0.6 mg/kg, an STMR of 0.17 mg/kg, and an HR of 0.37 mg/kg (from a single sample) to make a recommendation for the subgroup of fruiting vegetables, cucurbits – cucumbers and summer squashes and withdrew its previous separate recommendations for cucumber and summer squash.

Tomatoes

The GAP for tomato is from the United States and consists of multiple applications each at 0.14 kg ai/ha, on a 5-day interval, with a 3-day PHI. A maximum number of applications is not specified. Based on the listed limit of 1.26 kg ai/ha per crop cycle, the application pattern for the cGAP is 9 applications at 0.14 kg ai/ha. The 2003 Meeting recommended a maximum residue level of 2 mg/kg in tomato based on registered uses in Greece (up to 8 applications at 0.11 kg/ha, 3-day PHI).

Field trials in the United States were conducted with 6 applications, each at 0.21 kg ai/ha, generally on a 5-day retreatment interval (ranging from 4-6 days), with harvest 3 DALA. The Meeting agreed that the first three applications from the field trials would not lead to significant residues at harvest due to significant growth dilution and that the trials are suitable for estimating residues.

Residues of famoxadone in tomatoes from independent trials conducted at an exaggerated rate were (n=17): 0.14 (2), 0.15 (2), 0.17 (2), 0.23, 0.24, 0.30, 0.33, 0.40, 0.42, 0.50, 0.62, 0.68, and 0.71 mg/kg.

After scaling (factor = 0.67), residues were (n=17): 0.093 (2), 0.10 (2), 0.11 (2), 0.15, 0.16, 0.20, 0.22, 0.24, 0.27, 0.28, 0.33, 0.41, 0.45, and 0.47 mg/kg.

The Meeting noted that these residues are accommodated by the 2003 recommendation and confirmed its previous recommendation of a maximum residue level of 2 mg/kg, and STMR of 0.1 mg/kg and an HR of 1.1 mg/kg for famoxadone in tomato.

Peppers

The GAP for peppers is from the United States and consists of multiple applications each at 0.175 kg ai/ha, on a 5-day interval, with a 3-day PHI. A maximum number of applications is not specified. Based on

the listed limit of 1.26 kg ai/ha per crop cycle, the application pattern for the cGAP is one application at 0.035 kg ai/ha followed by 7 applications at 0.175 kg ai/ha.

Field trials in the United States were conducted with 6 applications on generally a 5-day interval (ranging from 4–7 days) at a rate of 0.21 kg ai/ha, with harvest 3 DALA. The Meeting agreed that an application 35 days before harvest would not contribute significantly to residues at harvest and that the trials are suitable for estimating residues of famoxadone in peppers.

Residues of famoxadone in bell and *chili* peppers from independent trials approximating the critical GAP were (n=11): 0.078, 0.16, 0.20, 0.34, 0.36, *0.47*, *0.51*, *0.60*, 0.63, 0.68, and *3.6* mg/kg.

The Meeting noted that the registration allows for use on bell and chili peppers, but not on other members of the Subgroup of peppers. The Meeting estimated a maximum residue level of 5 mg/kg, an STMR of 0.47 mg/kg, and an HR of 3.7 mg/kg (from a single sample) for famoxadone in each of peppers, sweet (including pimento or pimiento) and peppers, chili.

For dried chili peppers, the Meeting used the default factor of 10 to estimate a maximum residue level of 50 mg/kg for famoxadone in peppers chili, dried, an STMR of 4.7 mg/kg, and an HR of 37 mg/kg.

Hops

The critical GAP for hops is from the United States and consists of six applications, each at 0.14 kg ai/ha, on a 6-day interval, with a 7-day PHI.

Residues of famoxadone in hops (dried cones) from independent trials approximating the critical GAP but at a 2× rate were (n=5): 14, 15, 26, 42, and 46 mg/kg. After scaling to an application rate of 0.14 kg ai/ha, residues were (n=5): 7.0, 7.5, 13, 21, and 23 mg/kg.

The Meeting estimated a maximum residue level of 50 mg/kg and an STMR of 13 mg/kg for hops (dry).

Fate of residues during processing

No new data on fate of residues during processing were submitted to the Meeting. The 2003 Meeting evaluated the effects of processing on residues in tomato commodities. The current Meeting confirmed its previous recommendations with respect to tomato RAC, and by extension, processed tomato commodities.

Residues in animal commodities

The recommendations made by the current Meeting did not include animal feed items; therefore, the Meeting confirmed its previous recommendations for residues in animal commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *famoxadone.*

The residue is fat-soluble.

Table 11 Recommendations for residues of famoxadone from the 2022 JMPR

CCN	Crop/Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
VC 0424	Cucumber	W	0.2	--	--
MU 1100	Hops, dried	50	--	13	--
HS 0444	Peppers chili, dried	50		4.7	37
VO 0444	Peppers, chili	5	--	0.47	3.7
VO 0445	Peppers, sweet (including pimento or pimiento)	5	--	0.47	3.7
VC 0431	Squash, Summer	W	0.2	--	--
VA 2031	Subgroup of bulb onions	0.4	--	0.02	0.23
FB 2005	Subgroup of cane berries	10	--	1.1	6.6
VC 2039	Subgroup of fruiting vegetables, cucurbits – cucumbers and summer squashes	0.6	--	0.17	0.37
VO 0448	Tomato	2	2	0.1	1.1

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for famoxadone is 0–0.006 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for famoxadone were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 1–20 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of famoxadone from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for famoxadone is 0.6 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for famoxadone were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs varied from 0–20 percent of the ARfD for children and 0–9 percent of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of famoxadone from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Report Code	Author(s)	Year	Study Title
07796	Thompson, D.C.	2005	Famoxadone + cymoxanil: magnitude of the residue on hops. Report No. IR-4 PR No. 07796. GLP, Unpublished
A7796	Kathryn, H.	2016	Famoxadone + cymoxanil: magnitude of the residue on hops. Report No. IR-4 PR No. A7796. GLP, Unpublished
08303	Carpenter, D.H.	2007	Famoxadone + cymoxanil: Magnitude of the residue on onion (dry bulb and green). Report No. IR-4 PR No. 08303. GLP, Unpublished
08766	Carpenter, D.H.	2006	Famoxadone + cymoxanil: Magnitude of the residue on caneberry. Report No. IR-4 PR No. 08766. GLP, Unpublished
AMR 2831-93	Dubey, L., Jernberg, K., Lee, P.	1996	Residue Decline Pattern of DPX-JE874 in/on Winter Wheat in France and Germany – Season 1994. GLP, Unpublished
AMR 2971-94	Dubey, L., Jernberg, K., Lee, P.	1996	Residue Decline Pattern of DPX-JE874 in/on Winter Barley in France and Germany – Season 1994. GLP, Unpublished

Report Code	Author(s)	Year	Study Title
AMR 3705-95, revision 2	DeMario, D., Westberg, G.L., Hill, S.J., Nathan, E.C.	1998	Analytical Method for the determination of DPX-JE874 and cymoxanil residues in various matrices. Report No. AMR 3705-95, revision 2. GLP, Unpublished
AMR 4340-97	Nathan, E.C. III	1999	Magnitude of residues of famoxadone and cymoxanil in cucurbits following application of DPX-KP481 fungicide at maximum label rates. Report No. AMR 4340-97. GLP, Unpublished
DuPont-3723	McClory, J.P.	2001	Magnitude of residues of cymoxanil and famoxadone in fruiting vegetables except for cucurbits following application of DPX-KP481 50WG fungicide at maximum label rates – United States, 2000. Report No. DuPont-3723. GLP, Unpublished
DuPont-9822	Hornshuh, J.	2003	Magnitude of residues of cymoxanil and famoxadone in tomatoes following application of DPX-KP481 50WG fungicide at maximum label rates – United States, 2002. Report No. DuPont-9822. GLP, Unpublished

FENAZAQUIN (297)

First draft prepared by Dr M Lee, Andong National University, Republic of Korea

EXPLANATION

Fenazaquin is a quinazoline insecticide/acaricide. Its mode of action involves contact and ovicidal activity against a broad spectrum of mites in various crops. It was first evaluated by JMPR in 2017 for toxicology and residues and then a follow-up evaluation of additional uses was made by the 2019 Extra Meeting.

The 2017 JMPR established an ADI of 0–0.05 mg/kg bw and an ARfD of 0.1 mg/kg bw, applying to fenazaquin, tertiarybutylphenylethanol (TBPE) and 4-hydroxyquinazoline. Further, the Meeting concluded that 2-hydroxy-fenazaquin acid is present as a major metabolite in rat feces, therefore, it is covered by studies of the parent compound.

In 2017, JMPR defined the residue definitions for plant and animal commodities. For plant commodities, the residue definition is fenazaquin for compliance with the MRL and for dietary risk assessment. For animal commodities, correction of the definition for compliance with MRL was made by the 2019 Extra JMPR. The current residue definition for animal commodities is the sum of fenazaquin and 2-hydroxy-fenazaquin acid for compliance with the MRL and the sum of fenazaquin, 2-hydroxy-fenazaquin acid and tautomeric forms of 4-hydroxyquinazoline for dietary risk assessment. The residue is fat-soluble.

Fenazaquin was scheduled at the Fifty-second Session of the CCPR for evaluation of additional uses by the 2022 JMPR. The Meeting received information on residue trials on avocado, berries (blueberry, raspberry, grape, strawberry), citrus fruits (lemon, orange, grapefruit), pome fruits (apple, pear), stone fruits (cherry, peach, plum), fruiting vegetables (cucumber, cantaloupe, Zucchini squash, tomato, peppers), beans and peas (with pods, succulent without pods and dried) and mint. Processing and storage stability studies were also submitted. In addition, a confined rotational crop study and a new analytical method were provided.

Confined rotational crop study

A confined rotational crop study with fenazaquin was conducted using lettuce, radish and wheat at 30 day, 120, and 365 day plant back intervals [Dohn, D.R., 2010, Report 637-001]. Two radiolabelled forms of [$^{1\,4}$C-phenyl] fenazaquin and [^{14}C-quinazoline] fenazaquin were applied to the bare soil (sandy loam) of test plots located outdoors with a target application rate of 505 g/ha (actually, 550–556 g/ha). The applications for the 30 day PBI (February 3, 2009) occurred 90 days after the applications for the 120 day PBI. The applications for the 365 day PBI were made on the same day with the applications for the 30 day PBI.

Crop samples were homogenized in the presence of dry ice. The samples (total radioactive residues, >0.01 mg/kg of fenazaquin equivalents) were extracted twice with acetonitrile:water (1:1, v/v) and once with acetonitrile. The combined supernatants were partitioned twice with dichloromethane and the post extracted solids were subjected to combustion analysis. The aqueous and dichloromethane phases from the partitioning (or only aqueous phase) were analysed by HPLC using authentic reference standards: fenazaquin, 2-oxy-fenazaquin, fenazaquin acid, 4-hydroxyquinazoline (metabolite unique to the quinazoline label) and TBPE (metabolite unique to the phenyl label). The presence fenazaquin, 2-oxy-fenazaquin, and fenazaquin acid were confirmed by TLC in extracts from the 30 day PBI radish roots. Total radioactive residues (TRRs) were measured by combustion analysis/LSC.

Table 1 Total Radioactive Residues in rotational crops measured by combustion analysis

RAC	30-day PBI		120-day PBI		365-day PBI	
	Quinazoline label	Phenyl label	Quinazoline label	Phenyl label	Quinazoline label	Phenyl label
Immature lettuce	0.050±0.002	0.055±0.001	0.043±0.001	0.035±0.001	0.004±0.000	0.007±0.000
Mature lettuce	0.056±0.002	0.067±0.004	0.044±0.001	0.034±0.001	0.012±0.001	0.008±0.000
Radish roots	0.104±0.003	0.095±0.005	0.047±0.002	0.055±0.002	0.008±0.001	0.011±0.001
Radish tops	0.030±0.001	0.028±0.001	0.020±0.002	0.021±0.002	0.007±0.000	0.016±0.001
Wheat forage	0.037±0.001	0.044±0.003	0.067±0.003	0.029±0.001	0.009±0.000	0.129±0.004
Wheat hay	0.125±0.003	0.185±0.009	0.100±0.003	0.079±0.003	0.013±0.001	0.189±0.002
Wheat straw	0.116±0.008	0.243±0.006	0.128±0.003	0.104±0.003	0.025±0.002	0.187±0.006
Wheat grain	0.047±0.001	0.069±0.004	0.045±0.001	0.039±0.000	0.010±0.001	0.017±0.001

Notes:

Values are from combustion/LSC of five replicate sub-samples.

The TRRs from the two labels were in similar levels, and gradually declined with increasing PBIs, except for phenyl label wheat forage and hay (highest at 365 day PBI), phenyl label wheat straw (highest 30 day PBI and lowest 120 day PBI) and quinazoline label wheat forage and straw (highest at 120 day PBI).

In the study report, the percent TRR values on each component, subtotal extracted and PES were derived from the sum of subtotal extracted and PES. The ratio (all cases with label and PBI) of the reported percent TRR over the percent TRR calculated with the combustion analysis TRR value was 91.0–98.2 percent in lettuce (immature, mature), 83.3–125 percent in radish (roots, tops), 86.7–97.1 percent in wheat grain and 88.9–105 percent in wheat animal commodity (forage, hay, straw). As these differences did not give significant effects on the interpretation on results of this study, herein the percent TRR value from the study report was used as it is.

Extractability of residues (all cases with label and PBI) using organic solvents was 58.8–61.1 percent TRR in immature lettuce, 51.2–63.9 percent TRR in mature lettuce, 72.7–80.6 percent TRR in radish roots, 61.9–85.7 percent TRR in radish tops, 65.7–91.4 percent TRR in wheat forage, 49.6–69.6 percent TRR in wheat hay, 47.2–72.4 percent TRR in wheat straw and 28.6–49.3 percent TRR in wheat grain.

Table 2 Residues of fenazaquin in rotational crops

RAC	30-day PBI				120-day PBI			
	Quinazoline label		Phenyl label		Quinazoline label		Phenyl label	
	mg/kg	Percent TRR	mg/kg	Percent TRR	mg/kg	Percent TRR	mg/kg	Percent TRR
Immature lettuce	0.004	8.7	0.004	7.4	0.003	7.5	n.a.	n.a.
Mature lettuce	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Radish roots	0.031	29	0.026	28	0.007	15.9	0.009	15.5
Radish tops	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	0.001	5.0
Wheat forage	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Wheat hay	<0.000	0.0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Wheat straw	0.002	1.8	0.002	0.8	<0.000	0.0	<0.000	0.0
Wheat grain	n.a.	n.a.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.

Notes:

n.a. Not analysed.

n.d. Not detected.

Parent fenazaquin was found at a greater level than 10 percent TRR or 0.01 mg/kg only in 30 day PBI radish roots, representing phenyl label 28.6 percent TRR (0.026 mg/kg) and quinazoline label 29.0 percent TRR (0.031 mg/kg). In the radish roots of 120 day PBI, fenazaquin residues were found at < 0.01 mg/kg (16 percent TRR) in both labels (for 365 day PBI, phenyl label sample, extracted but not analysed by HPLC and quinazoline label sample, not extracted).

Table 3 Residues of metabolite 4-hydroxyquinazoline in rotational crops

RAC	4-hydroxyquinazoline (quinazoline label)					
	30-day PBI		120-day PBI		365-day PBI	
	mg/kg	Percent TRR	mg/kg	Percent TRR	mg/kg	Percent TRR
Immature lettuce	0.002	4.3	0.002	5.0	n.a.	n.a.
Mature lettuce	0.002	3.8	0.002	4.7	n.a.	n.a.
Radish roots	0.009	8.4	0.006	13.6	n.a.	n.a.
Radish tops	0.001	4.0	0.001	4.8	n.a.	n.a.
Wheat forage	0.003	8.6	0.009	13.8	n.a.	n.a.
Wheat hay	0.01	8.0	0.011	11.6	0.001	7.7
Wheat straw	0.005	4.4	0.012	9.4	0.001	3.4
Wheat grain	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

Notes:

n.a. Not analysed.

For metabolites, 4-hydroxyquinazoline was detected in all tested crops except for wheat grain, representing low levels of ≤ 13.8 percent TRR or ≤0.012 mg eq/kg. The other identified metabolites (2-oxy-fenazaquin, fenazaquin acid and 4-*tert*-butylphenyl alcohol), where measured, were found at very low levels of ≤4.5 percent TRR or ≤ 0.004 mg eq/kg. In addition to these metabolites, a polar peak (< 0.013 mg eq/kg) and multiple minor components (each <0.016 mg eq/kg, except for two components in wheat hay of 0.023 mg eq/kg and 0.035 mg eq/kg and one component in wheat straw of 0.064 mg eq/kg) were present in the rotational crops.

RESIDUE ANALYSIS

Analytical methods

A new method for analysis of fenazaquin was submitted to the JMPR, which was used in storage stability tests of apple, peach and tomato. The method involved a sample separation method by QuEChERS and determination by LC-MS/MS (m/z 307→57 for quantification, m/z 307→131 for qualification). Method validation results, included in the storage stability study, showed that mean recovery of fenazaquin fortified at levels of 0.01 and 0.1 mg/kg in apple, peach and tomato ranged from 86-118 percent (RSDs, ≤12 percent). The LOQ for fenazaquin was 0.01 mg/kg in the matrices. A separate method validation study [Lakaschus, S., Amann, S., 2012; Report No. S11-03100] on the new analytical method was submitted, which revealed the method is sufficiently acceptable for analysis of fenazaquin residues in the matrices of plant commodity.

In all the crop field trials and processing studies (mint, orange, plum, and tomato processing except for grape), the analytical method used consisted of processes of extraction with acetonitrile, a

partition between water and methylene chloride, clean-up step using SPE cartridge, and determination by LC-MS/MS (m/z 307.0→161.2 for quantification, m/z 307.0→147.2 for confirmation). The method was based on the method included in the Report 024119-1, considered validated by the 2017 JMPR. Method validation results from the submitted study reports to this JMPR showed that the mean recovery of fenazaquin from various plant matrices fortified at levels of 0.01-0.1/0.5/1.0/2.0 mg/kg ranged from 72–118 percent (RSDs, ≤17 percent)). In concurrent recovery, most of the individual values ranged from 70–120 percent. The LOQ value was 0.01 mg/kg in the matrices.

For some storage stability tests (grape, cucurbit, orange) and a grape processing study, the analytical method used for the analysis of fenazaquin consisted of processes extraction by macerating and heating (for cucurbit, without heating) with acetonitrile:water (90:10, v/v; for orange peel, 75:25, v/v), partitioning into hexane, clean-up step by SPE cartridge (florisil and sequentially aminopropyl) and determination by GC-MS (selected ion, 145.1, 160.0 amu; for grape processing study, additionally 117.0 amu). The method was based on the method R A4167, considered as validated by the 2017 JMPR. Method validation results on grape processing matrices showed that mean recovery of fenazaquin at fortification levels of 0.01 and 1.0 mg/kg ranged from 74–93 percent (RSDs, ≤13 percent). The concurrent recovery values were in the range of 75–101 percent. The LOQ value was 0.01 mg/kg in the matrices.

All analytical methods mentioned above are considered acceptable for the analysis of residue. The recovery results from method validation and concurrent recovery test are shown in Table 4 and Table 5, respectively.

For plant commodities, information on other residues than parent such as fenazaquin dimer (included in the study reports submitted) were not reviewed in this JMPR evaluation

Table 4 Recovery of fenazaquin in method validation

Matrix	Fortification level, mg/kg	Individual values, percent	Mean value, percent	RSD, percent	Method used
Apple (storage stability)	0.01 0.1	87, 92, 98 87, 87, 91	92 88	5 2	QuEChERS, LC-MS/MS
Apple	0.01 0.1	100, 101, 102, 103, 104 106, 106, 106, 107, 108	102 107	2 1	QuEChERS, LC-MS/MS
Avocado, whole	0.01 0.5	97, 111, 120 96, 99, 105	109 100	9 4	LC-MS/MS, Report 024119-1
Avocado, flesh	0.01 0.5	73, 85, 92 70, 73, 74	83 72	9 2	LC-MS/MS, Report 024119-1
Beans (snap)	0.01 0.5	83, 85, 86 79, 80, 80	85 80	1 1	LC-MS/MS, Report 024119-1
Beans (lima), succulent shelled	0.01 0.5	87, 93, 96 76, 77, 77	92 77	4 1	LC-MS/MS, Report 024119-1
Beans (pinto), dry	0.01 0.5	70, 74, 76, 84, 95, 117 68, 68, 68, 71, 71, 73, 83, 88, 89	86 75	15 12	LC-MS/MS, Report 024119-1
Cantaloupe	0.01 0.1	85, 86, 87 73, 76, 81	86 77	1 4	LC-MS/MS, Report 024119-1
Cucumber	0.01 0.1	95, 99, 102 90, 93, 95	99 93	3 2	LC-MS/MS, Report 024119-1
Grapes	0.01 0.1	98, 102, 119 91, 95, 95	106 94	9 2	LC-MS/MS, Report 024119-1
Grapes	0.01 0.1	81, 83, 84 77, 78, 79	83 78	2 1	QuEChERS, LC-MS/MS

Matrix	Fortification level, mg/kg	Individual values, percent	Mean value, percent	RSD, percent	Method used
Grapes, wine	0.01 1.0	81, 82, 83, 83, 90 86, 92, 94, 95, 96	83 93	4 5	R A4167 (GC-MS)
Grapes, juice	0.01 1.0	71, 75, 91 72, 75, 76	79 74	13 2	R A4167 (GC-MS)
Grapes, raisins	0.01 1.0	73, 77, 80, 83, 101 71, 74, 74, 74, 79	83 75	13 4	R A4167 (GC-MS)
Grapes, pomace	0.01 1.0	75, 86, 93 70, 78, 80	85 76	11 7	R A4167 (GC-MS)
Lemon, whole	0.01 0.10 2.0	85, 87, 87 79, 82, 84 80, 81, 83	87 82 82	1 3 2	LC-MS/MS, Report 024119-1
Mint (peppermint), tops, fresh	0.01 0.1	68, 71, 80 77, 78, 78	73 78	7 1	LC-MS/MS, Report 024119-1
Mint (peppermint), oil	0.01 0.1	108, 110, 112 96, 96, 100	110 97	1 2	LC-MS/MS, Report 024119-1
Orange, whole	0.01 0.1	89, 89, 89, 90, 91 83, 84, 85, 87, 87	90 85	1 2	QuEChERS, LC-MS/MS
Orange, pulp	0.01 0.1	93, 96, 96 103, 103, 105	95 104	2 1	QuEChERS, LC-MS/MS
Orange, peel	0.01 0.1	90, 95, 106 106, 109, 110	97 108	8 2	QuEChERS, LC-MS/MS
Peach	0.01 0.1	103, 107, 109 101, 109, 109	106 106	3 4	QuEChERS, LC-MS/MS
Peach (storage stability test)	0.01 0.1	104, 112, 137 95, 97, 98	118 97	12 1	QuEChERS, LC-MS/MS
Peas (snap)	0.01 0.5	93, 96, 98 82, 83, 83	95 83	2 1	LC-MS/MS, Report 024119-1
Peas (garden), succulent shelled[b]	0.01 0.5	92, 96, 99 81, 84, 85	96 83	3 2	LC-MS/MS, Report 024119-1
Peas, dry	0.01 0.5	64, 80, 83 72, 72, 75	76 73	11 2	LC-MS/MS, Report 024119-1
Peas, vines	0.01 0.5	65, 71, 73, 73, 86, 90 77, 77, 79, 101, 106, 109	76 91	13 17	LC-MS/MS, Report 024119-1
Peas, hay	0.01 0.5	86, 86, 89 77, 89, 92	87 86	2 8	LC-MS/MS, Report 024119-1
Pepper, sweet	0.01 0.1	97, 101, 101 80, 82, 89	100 84	2 5	LC-MS/MS, Report 024119-1
Raspberries	0.01 0.1	90, 93, 95 74, 82, 89	93 81	2 8	LC-MS/MS, Report 024119-1
Summer squash (zucchini)	0.01 0.1	88, 101, 108 92, 93, 94	99 93	8 1	LC-MS/MS, Report 024119-1
Strawberry	0.01 0.1	80, 105, 112 90, 93, 99	99 94	14 4	LC-MS/MS, Report 024119-1
Tomato (storage stability)	0.01 0.1	94, 98, 99 85, 85, 87	97 86	2 1	QuEChERS, LC-MS/MS
Tomato	0.01 0.1	96, 108, 110 89, 101, 107	105 99	7 9	QuEChERS, LC-MS/MS

Table 5 Concurrent recovery of fenazaquin

Matrix	Fortification level, mg/kg	Individual values, percent	Mean value, percent	RSD, percent
Apple	0.01 0.1 2	81, 106, 110, 129 79, 81, 85, 87 74, 77, 79, 84	107 83 79	19 4 5
Avocado, whole	0.01 0.5 1	64, 95 67 108	65	
Avocado, flesh	0.01 0.5 1	72, 93 72 99	83	
Beans (green snap)	0.01 0.5	69, 88, 91 73, 78, 84	83 78	14 7
Beas (lima), succulent shelled	0.01 0.5	86, 94, 113 70, 84, 86	98 80	14 11
Beans (pinto), dry	0.01 0.1 0.5	78, 94, 99 82, 84 75	90 83	12
Blueberries	0.01 0.1 2	61, 66, 80, 93 71, 88, 99 93	75 86	19 16
Cantaloupe	0.1 0.5	91, 92 90, 95	92 93	
Cucumber	0.1 0.5	87, 88 79, 85	88 82	
Cherry	0.01 0.1 1	80, 93, 96, 103, 106 84 79, 84, 90, 93, 119	96 93	11 17
Grapefruit, whole	0.01 0.5	72 70		
Grapefruit, flesh	0.01 0.5	78 83		
Grapes	0.01 0.5	98, 103, 104 96, 98, 100	102 98	3 2
Grapes (processing study)*	0.01 1.0	75 97		
Grapes, wine*	0.01 1.0	81 84		
Grapes, juice*	0.01 0.2	86 93		
Grapes, raisins*	0.01 2.0	101 89		
Lemon, whole	0.01 0.5	96 88		
Lemon, flesh	0.01 0.5	100, 108 90, 96	104 93	
Mint (peppermint), tops, fresh	0.01 0.1 0.5 10 25	132 72, 80, 101 75, 89 76 69	 84 82	 18

Matrix	Fortification level, mg/kg	Individual values, percent	Mean value, percent	RSD, percent
Mint (peppermint), oil	0.01	86		
	10	89		
Orange, whole	0.01	90, 98, 102, 109	100	8
	0.02	79		
	0.5	91, 93, 94, 97, 103	96	5
	2	112		
Orange, flesh	0.01	94, 97, 98, 112	100	8
	0.5	90, 91, 92, 94	92	2
Orange, juice	0.01	126		
	2	83		
Orange, dried pulp	0.01	102		
	2	91		
Orange oil	0.005	108		
	150	113		
Peach	0.01	64, 68, 89	74	18
	0.08	95		
	0.1	76, 85, 111	91	20
	0.8	83		
	1.0	71		
	2.0	86		
Pear	0.01	95, 96, 101	97	3
	0.1	87, 99	93	
	0.5	70, 89	80	
Peas (green snap)	0.01	93, 102	98	
	0.1	80		
	0.5	72		
Peas (garden), succulent shelled	0.01	70, 73, 84	76	10
	0.1	72		
	0.5	64, 76	70	
Peas, dry	0.005	97, 99, 113	103	8
	0.01	87		
	0.25	84, 91, 92, 97	91	6
	0.5	90		
Peas, vine	0.005	101, 104, 108	104	3
	0.01	108		
	0.25	115, 116, 119	117	2
	0.5	68, 82	75	
	5.0	76		
Peas, hay	0.01	72, 79, 93, 95	85	13
	0.5	75, 78, 80, 80	78	3
	30	77		
Pepper, sweet	0.01	96, 110	103	
	0.5	84, 95	90	
Pepper (chili)	0.01	99		
	0.5	90		
Plum	0.01	82, 85, 115	94	19
	0.08	100		
	0.1	83, 95, 97	92	8
	0.8	69		
	2.0	83		

Matrix	Fortification level, mg/kg	Individual values, percent	Mean value, percent	RSD, percent
Prune	0.01 0.1 4.0	83, 124 98 80	104	
Raspberries	0.01 0.08 0.1 1.6	77, 82 84 63, 93 88	80 78	
Summer squash (zucchini)	0.1 0.5	86, 106 82, 100	96 91	
Strawberry	0.01 0.1 0.2 1 2	76, 84, 97, 107 82, 87, 99 84 83 98	91 89	15 10
Tomato fruit	0.01 0.1 0.5	94, 95, 103, 111, 119 88, 90, 91, 100 80	104 92	10 6
Tomato paste	0.01 0.1 0.5	92, 112 82 84	102	
Tomato puree	0.01 0.1	104 95		

Notes:

* Using the analytical method R A4167 (GC-MS).

For the other matrices, using the analytical method included in the Report 024119-1 (LC-MS/MS).

STABILITY OF RESIDUES IN STORED ANALYTICAL SAMPLES

The stability of fenazaquin residues on frozen storage was studied in apple, tomato, peach, cucumber, melon, grape and orange with results shown in Table 6. In all studies, fenazaquin was fortified at 0.1 mg/kg. Residues of fenazaquin were not detected in the control samples, except for orange (control values in orange samples, not reported).

Residues of fenazaquin were stable for at least 12 months in apple, tomato and peach when frozen stored at below -18 °C [Lakaschus & Gizler, 2013; Report S11-03099]; for at least 379 days in cucumber, at least 372 days in melon pulp and at least 376 days in melon peel at -10 to -27 °C [Butcher, 1994; Report GHE-P-3798]; at least 389 days in grape at -16 to -27 °C [Gambie, et al., 1994; Report GHE-P-3404]; at least 399 days in orange flesh and at least 371 days in orange peel at -27 to -15 °C [Gambie & Draper, 1993; Report GHE-P-3154].

Table 6 Storage stability results of fenazaquin at a fortification of 0.01 mg/kg

Commodity	Storage interval (days or months)	Procedural recovery, percent	Residues remaining, percent[a]	Report No.
Apple	0 days 3 months 6 months 12 months	 99, 104 96, 91 99, 94	99, 101, 96 92, 90 74, 70 84, 81	S11-03099

Commodity	Storage interval (days or months)	Procedural recovery, percent	Residues remaining, percent[a]	Report No.
Tomato	0 day		98, 99, 95	S11-03099
	3 months	94, 93	87, 80	
	6 months	86. 84	82, 82	
	12 months	93, 89	84, 84	
Peach	0 day		99, 87, 93	S11-03099
	3 months	99, 101	94, 90	
	6 months	84, 88	87, 81	
	12 months	104, 106	100, 100	
Cucumber	0 days	112, 116	114	GHE-P-3798
	83 days	118, 112	118	
	379 days	92, 101	90	
Melon, flesh	0 days	109, 100	101	GHE-P-3798
	80 days	107, 97	104	
	372 days	105, 100	98	
Melon, peel	0 days	108, 109	109	GHE-P-3798
	84 days	104, 103	99	
	376 days	98, 98	96	
Grape	0 days	96, 90	95	GHE-P-3404
	125 days	89, 90	84	
	244 days	105, 92	91	
	389 days	112, 106	102	
Orange flesh[b]	0 days	98, 104	106, 112	GHE-P-3154
	77 days	82, 88	81, 85	
	399 days	80, 81	86, 86	
Orange peel[b]	0 days	94, 101	102, 107	GHE-P-3154
	89 days	74, 75	72, 73	
	371 days	98, 95	87, 89	

Notes:

[a] Recovery was not corrected by procedural recovery.

[b] In orange (flesh, peel), all results were corrected for control values (not reported).

USE PATTERN

The Meeting received information on the GAP of fenazaquin registered in the United States. The information with regard to this evaluation by the Meeting are summarised in Table 7.

Table 7 Registered use of fenazaquin (SC 200 g/L) in United States

Crop	Application				PHI, days	Remarks
	Method	No.	Volume, L/ha	Rate. kg ai/ha		
Citrus fruit Group (10-10) (Grapefruit, Lemon, Orange)	Foliar spray	1	≥935 G*	0.359-0.538 (0.538 kg ai/ha/year)	7	Not before petal fall
Pome fruit Group (11-10) (Apple, Pear)	Foliar spray	1	≥468 G*	0.359-0.538 (0.538 kg ai/ha/year)	7	Not before petal fall
Stone fruit Group (12-12) (Cherry, Peach, Plum)	Foliar spray	1	≥468 G*	0.359-0.538	3	Not before petal fall
	Post-harvest foliar spray	1		0.359-0.538		Max 1.08 kg ai/ha/season
Caneberry Subgroup (13-07A)	Foliar spray	1	≥468 G*	0.359-0.538	7	

Crop	Application				PHI, days	Remarks
	Method	No.	Volume, L/ha	Rate. kg ai/ha		
(Raspberry)				(0.538 kg ai/ha/year)		
Bushberry Subgroup (13-07B) (Blueberry)	Foliar spray	1	≥468 G*	0.359-0.538 (0.538 kg ai/ha/year)	7	
Small fruit vine climbing Subgroup, except Fuzzy kiwifruit (13-07F) (Grape)	Foliar spray	1	≥468 G*	0.359-0.538 (0.538 kg ai/ha/year)	7	Not before petal fall
Low growing berry Subgroup (13-07G) (Strawberry)	Foliar spray	1	≥468 G*	0.359-0.538 (0.538 kg ai/ha/year)	1	Not before petal fall
Avocado	Foliar spray	1	≥935 G*	0.359-0.538 (0.538 kg ai/ha/year)	7	
Cucurbit vegetables Group (9) (Cucumber, Cantaloupe, Summer squash)	Foliar spray	1	≥234 G*	0.359-0.538 (0.538 kg ai/ha/year)	3	
Fruiting vegetables Group (8-10) (Tomato, Pepper)	Foliar spray	1	≥234 G*	0.359-0.538 (0.538 kg ai/ha/year)	3	
Edible-podded legume vegetable Subgroup (6A) (Snap bean, snap pea)	Foliar spray	1	≥187 G*	0.359-0.538 (0.538 kg ai/ha/year)	7	
Succulent pea and bean Subgroup (6B) (Lima bean, Garden pea)	Foliar spray	1	≥187 G*	0.359-0.538 (0.538 kg ai/ha/year)	7	
Dried shelled pea and bean (except soybean) Subgroup (6C) (Pinto bean, Australian winter pea)	Foliar spray	1	≥187 G*	0.359-0.538 (0.538 kg ai/ha/year)	7	
Mint (Peppermint, spearmint)	Foliar spray	1	≥187 G*	0.359-0.538 (0.538 kg ai/ha/year)	7	Apply before bloom

Notes:

The table shows the GAP information on the crops (avocado and the crops in the parenthesis) for which residue trials were submitted.

* Ground application (air application is also allowed with different spray volume).

Restrictions on all crops:

Only ground sprayer, airblast and aerial applications are permitted.

Do not plant rotational crops, other than those on this label, within 30 days of this application. Do not plant root, tuber and bulb vegetables within 120 days of this application.

Crop groups:

Citrus fruit group (10-10): Australian desert lime; Australian finger-lime; Australian round lime; Brown River finger lime; calamondin; citron; citrus hybrids; grapefruit; Japanese summer grapefruit; kumquat; lemon; lime; Mediterranean mandarin; mount white lime; New Guinea wild lime; orange, sour; orange, sweet; pummelo; Russell River lime; satsuma mandarin; sweet lime; tachibana orange; Tahiti lime; tangelo; tangerine (mandarin); tangor; trifoliate orange; uniq fruit; cultivars, varieties, and/or hybrids of these.

Pome fruit group 11-10: Apple; azarole; crabapple; loquat; mayhaw; medlar; pear; pear, Asian; quince; quince, Chinese; quince, Japanese; tejocote; cultivars, varieties, and/or hybrids of these.

Stone fruit group 12-12: Apricot; apricot, Japanese; capulin; cherry, black; cherry, Nanking; cherry, sweet; cherry, tart; Jujube, Chinese; nectarine; peach; plum; plum, American; plum, beach; plum, Canada; plum, cherry; plum, Chickasaw; plum, Damson; plum, Japanese; plum, Klamath; plum, prune; plumcot; sloe; cultivars, varieties, and/or hybrids of these.

Caneberry subgroup 13-07A: Blackberry; loganberry; raspberry, black and red; wild raspberry; cultivars, varieties, and/or hybrids of these.

Bushberry subgroup 13-07B: Aronia berry; blueberry, highbush; blueberry, lowbush; buffalo currant; Chilean guava; cranberry, highbush; currant, black; currant, red; elderberry; European barberry; gooseberry; honeysuckle, edible; huckleberry; jostaberry; Juneberry (Saskatoon berry); lingonberry; native currant; salal; sea buckthorn; cultivars, varieties, and/or hybrids of these

Small fruit vine climbing subgroup, except fuzzy kiwifruit 13-07F: Amur river grape; gooseberry; grape; kiwifruit, hardy; maypop; schisandra berry; cultivars, varieties, and/or hybrids of these.

Low growing berry subgroup 13-07G: Bearberry; bilberry; blueberry, lowbush; cloudberry; cranberry; lingonberry; muntries; partridgeberry; strawberry; cultivars, varieties, and/or hybrids of these.

Cucurbit vegetables group 9: Chayote (fruit); Chinese waxgourd (Chinese preserving melon); citron melon; cucumber; gherkin; gourd, edible (includes hyotan, cucuzza, hechima, Chinese okra); *Momordica* spp (includes balsam apple, balsam pear, bittermelon, Chinese cucumber); muskmelon (includes cantaloupe); pumpkin; squash, summer; squash, winter (includes butternut squash, calabaza, hubbard squash, acorn squash, spaghetti squash); watermelon.

Fruiting vegetables group 8-10: African eggplant; bush tomato; bell pepper; cocona; currant tomato; eggplant; garden huckleberry; goji berry; groundcherry; martynia; naranjilla; okra; pea eggplant; pepino; non-bell pepper; roselle; scarlet eggplant; sunberry; tomatillo; tomato; tree tomato; cultivars, varieties, and/or hybrids of these

Edible-podded legume vegetable subgroup 6A: Bean (*Phaseolus* spp.) (includes runner bean, snap bean, wax bean); bean (*Vigna* spp.) (includes asparagus bean, Chinese longbean, moth bean, yardlong bean); jackbean; pea (*Pisum* spp.) (includes dwarf pea, edible-podded pea, snow pea, sugar snap pea); pigeon pea; soybean (immature seed); sword bean.

Succulent pea and bean subgroup 6B: Bean (*Phaseolus* spp.) (includes lima bean (green)); broad bean (succulent); bean (*Vigna* spp.) (includes blackeyed pea, cowpea, southern pea); pea (*Pisum* spp.) (includes English pea, garden pea, green pea); pigeon pea.

Dried shelled pea and bean (except soybean) subgroup 6C: Dried cultivars of bean (*Lupinus* spp.) (includes grain lupin, sweet lupin, white lupin, and white sweet lupin); (*Phaseolus* spp.) (includes field bean, kidney bean, lima bean (dry), navy bean, pinto bean, tepary bean); bean (*Vigna* spp.) (includes adzuki bean, blackeyed pea, catjang, cowpea, Crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean); broad bean (dry); chickpea; guar; lablab bean (hyacinth bean); lentil; pea (*Pisum* spp.) (includes field pea); pigeon pea.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received residue trials on avocado, berries (blueberry, raspberry, grape, strawberry), citrus fruits (lemon, orange, grapefruit), pome fruits (apple, pear), stone fruits (cherry, peach, plum), fruiting vegetables (cucumber, cantaloupe, Zucchini squash, tomato, peppers), beans and peas (with pods, succulent without pods and dried) and mint. The detailed information is summarised in Tables 8–23.

Group	Subgroup	Commodity	Table No.
FC 0001 Citrus Fruits	FC 0002 Lemons and Limes	FC 0204 Lemon	Table 8
	FC 0004 Oranges, Sweet, Sour	FC 0208 Orange, Sweet	Table 8
	FC 0005 Pummelo and Grapefruits	FC 0203 Grapefruit	Table 8
FP 0009 Pome Fruits		FP 0226 Apple FP 0230 Pear	Table 9
FS 0012 Stone Fruits	FS 0013 Cherries		Table 10
	FS 0014 Plums		Table 10
	FS 2001 Peaches		Table 10
FB 0018 Berries and other small fruits	FB 2005 Cane berries	FB 0272 Raspberries, Red, Black	Table 11

Group	Subgroup	Commodity	Table No.
	FB 2006 Bush berries	FB 0020 Blueberries	Table 11
	FB 2008 Small fruit vine climbing	FB 0269 Grapes	Table 12
	FB 2009 Low growing berries	FB 0275 Strawberry	Table 13
FI 0030 Assorted tropical and sub-tropical fruits – inedible peel	FI 2022 Assorted tropical and subtropical fruits - inedible smooth peel - large	FI 0326 Avocado	Table 14
VC 0045 Fruiting vegetables, Cucurbits	VC 2039 Fruiting vegetables, Cucurbits – Cucumbers and Summer squashes	VC 0424 Cucumber	Table 15
		VC 0431 Squash, Summer	
	VC 2040 Fruiting vegetables, Cucurbits – Melons, Pumpkins and Winter Squashes	VC 0046 Melons, except Watermelon	Table 16
VO 0050 Fruiting vegetables, other than Cucurbits	VO 2045 Tomatoes	VO 0448 Tomato	Table 17
	VO 0051 Peppers	VO 0444 Peppers, Chili	Table 18
		VO 0445 Peppers, Sweet	
VP 0060 Legume vegetables	VP 2060 Beans with pods		Table 19
	VP 2061 Peas with pods		Table 19
	VP 2062 Succulent beans without pods		Table 20
	VP 2063 Succulent peas without pods		Table 20
VD 0070 Pulses	VD 2065 Dry beans		Table 21
	VD 2066 Dry peas		Table 21
HH 0092 Herbs	HH 2095 Herbs	HH 0738 Mint	Table 22
		AL 0072 Pea hays	Table 23
		Pea vines	Table 23

Citrus fruits

Supervised residue trials on lemon (five trials), orange (12 trials) and grapefruit (six trials) were conducted in the United States during 2008-2009 [Belcher, T.I., 2010; Report No. GR08-576].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.50–0.51 kg ai/ha in lemon, 0.50–0.53 kg ai/ha in orange and 0.50–0.51 kg ai/ha in grapefruit. The foliar spray including a non-ionic surfactant was made seven or eight days prior to normal fruit harvest, using tractor mounted airblast orchard sprayers or miniblast sprayers. The spray volume was 635–1909 L/ha in lemon, 321–2194 L/ha in orange and 478–1518 L/ha in grapefruit. Duplicate samples from treated plot were

collected, each sample comprising of at least 24 fruit, weighing a minimum of 2 kg. Collected samples were stored frozen until analysis with a maximum storage period of 326 days. A subsample of fruit collected from treated each plot was peeled and flesh sample was also analysed. No fenazaquin residue was detected in the flesh samples of lemon, orange and grapefruit. Orange trials included two decline studies (1, 3, 7, 10, and 14 days after treatment) and one processing study (Trial 08-576.11).

Table 8 Residue concentration of fenazaquin from residue trials on lemon, orange and grapefruit in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥ 935	1			PHI=7	
Lemon							
Ft. Pierce, FL, 2008 08-576.4 (Eureka)	0.50	786	1	Mature Fruit	Whole fruit	7	0.11, 0.12 (0.12)
Sanger, CA, 2009 08-576.15 (Lizbon 8A)	0.51	1909	1	Mature Fruit	Whole fruit	7	0.13, 0.09 (0.11)
Arroyo Grande, CA, 2008 08-576.16 (Lizbon)	0.51	651	1	Mature Fruit	Whole fruit	7	0.02, 0.02 (0.02)
Yuma, AZ, 2008 08-576.19 (Lizbon)	0.51	2068	1	Mature Fruit	Whole fruit	7	0.05, 0.03 (0.04)
Richgrove, CA, 2008 08-576.22 (Lizbon)	0.50	635	1	Advanced Ripening, BBCH 85	Whole fruit	7	0.08, 0.08 (0.08)
Orange							
Hobe Sound, FL, 2008[a] 08-576.1 (Hamlin)	0.51	321	1	Mature fruit	Whole fruit	1 3 7 10 14	0.17, 0.18 (0.18) 0.15, 0.14 (0.15) 0.17, 0.18 (0.18) 0.09, 0.08 (0.09) 0.11, 0.10 (0.11)
Hobe Sound, FL, 2008[a] 08-576.2 (Hamlin)	0.51	2194	1	Mature fruit	Whole fruit	7	0.19, 0.26 (0.23)
Hobe Sound, FL, 2009 08-576.3 (Pineapple)	0.52	1534	1	Mature fruit	Whole fruit	7	0.12, 0.10 (0.11)
Oviedo, FL, 2008 08-576.7 (Navel)	0.50	838	1	BBCH 85	Whole fruit	7	0.10, 0.14 (0.12)
Clermont, FL, 2009[b] 08-576.8 (Mid Sweet)	0.50	2136	1	Mature fruit	Whole fruit	8	0.08, 0.10 (0.09)
Clermont, FL, 2009[b] 08-576.9 (Valencia)	0.51	478	1	Mature fruit	Whole fruit	7	0.07, 0.07 (0.07)
Groveland, FL, 2008 08-576.11 (Navel)	0.51	552	1	Mature fruit	Whole fruit	7	0.22, 0.15 (0.19)
Haines City, FL, 2008 08-576.12 (Hamlins)	0.52	1060	1	Mature fruit	Whole fruit	7	0.11, 0.10 (0.11)
Alamo, TX, 2008 08-576.13 (N-33)	0.51	750	1	Mature fruit	Whole fruit	7	0.07, 0.08 (0.08)

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
Madera, CA, 2008 08-576.17 (Navel)	0.51	479	1	Mature fruit	Whole fruit	1 3 7 10 14	0.24, 0.17 (0.21) 0.19, 0.12 (0.16) 0.13, 0.17 (0.15) 0.06, 0.04 (0.05) 0.06, 0.08 (0.07)
Redlands, CA, 2009 08-576.18 (Navel)	0.53	2990	1	Mature fruit	Whole fruit	7	0.14, 0.11 (0.13)
Porterville, CA, 2008 08-576.21 (Valencia)	0.51	1749	1	Mature fruit	Whole fruit	7	0.14, 0.15 (0.15)
Grapefruit							
Hobe Sound, FL, 2009 08-576.5 (Red)[c]	0.51	658	1	Mature fruit	Whole fruit	7	0.11, 0.10 (0.11)
Hobe Sound, FL, 2009[c] 08-576.6 (White)	0.50	1474	1	Mature fruit	Whole fruit	7	0.03, 0.05 (0.04)
Clermont, FL, 2009 08-576.10 (Flame)	0.50	478	1	Mature fruit	Whole fruit	8	0.05, 0.01 (0.03)
Alamo, TX, 2008 08-576.14 (Rio Red)	0.51	1518	1	Mature fruit	Whole fruit	7	0.06, 0.07 (0.07)
Redlands, CA, 2009 08-576.20 (Mash Ruby)	0.51	750	1	Mature fruit	Whole fruit	7	0.13, 0.14 (0.14)
Porterville, CA, 2008 08-576.23 (Mellogold)	0.51	1515	1	Mature fruit	Whole fruit	7	0.05, 0.02 (0.04)

Notes:

[a] Not independent trials, conducted at the same location with the same treatment date.

[b] Not independent trials, conducted at the same location with the same harvest date.

[c] Not independent trials, conducted at the same location with a close treatment date, 3 days apart.

Fenazaquin residues were not detected (<0.01 mg/kg) in the flesh samples of lemon, orange and grapefruit,

Pome fruits

Supervised field trials on apple (12 trials) and pear (six trials) trees were conducted in the United States in 2008 [Riley, M., 2010; Report No. GR08-575].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.50–0.53 kg ai/ha in apple and 0.50–0.51 kg ai/ha in pear. The foliar spray including a non-ionic surfactant was made seven days prior to normal harvest, using tractor-mounted airblast orchard sprayers or miniblast sprayers. The spray volume was 542–2068 L/ha in apple and 471–1876 L/ha in pear. Duplicate samples from treated plot were collected, each sample comprising of at least 24 fruit, weighing a minimum of 2 kg. Collected samples were stored frozen until analysis with a maximum storage period of 181 days. Apple trials included two decline studies (1, 3, 7, 9 or 10, and 14 days after treatment).

Table 9 Residue concentration of fenazaquin from residue trials on apple and pear in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥468	1			PHI=7	
Apple							
North Rose, NY,2008[a] 08.575.1 (Cortland – Block 167C)	0.51	610	1	BBCH 85	Whole fruit	7	0.05, 0.07 (0.06)
North Rose, NY, 2008[a] 08.575.3 (Rome – Black 153W)	0.50	2068	1	BBCH 85	Whole fruit	7	0.07, 0.10 (0.09)
Hereford, PA, 2008 08.575.2 (Star Krimson)	0.50	744	1	BBCH 87	Whole fruit	1 3 7 10 14	0.31, 0.24 0.16, 0.23 0.09, 0.13 (0.11) 0.10, 0.08 0.07, 0.08
Chula, GA, 2008 08.575.4 (Anna Apple)	0.50	542	1	Mature fruit	Whole fruit	7	0.12, 0.14 (0.13)
Zion, MNI, 2008 08.575.5 (Honey Crisp)	0.51	971	1	Mature fruit	Whole fruit	7	<0.01, <0.01 (<0.01)
Fitchburg, WI, 2008 08.575.6 (Red Chief Red Delicious)	0.51	687	1	Just before ripe	Whole fruit	7	0.03, 0.03 (0.03)
Paradise, UT, 2008 08.575.7 (Golden Delicious)	0.53	942	1	Mature fruit	Whole fruit	7	0.08, 0.07 (0.08)
Porterville, CA, 2008 08.575.8 (Granny Smith)	0.51	848	1	BBCH 89	Whole fruit	7	0.06, 0.04 (0.05)
Ephrata, WA, 2008 08.575.9 (Red Delicious0	0.51	804	1	BBCH 89	Whole fruit	7	0.18, 0.12 (0.15)
Payette, ID, 2008 08.575.10 (Red Delicious)	0.51	563	1	Advanced ripening	Whole fruit	1 3 7 9 14	0.34, 0.30 0.19, 0.18 0.12, 0.07 (0.10) 0.14, 0.10 (0.12) 0.12, 0.12
Rockland, ID, 2008 08.575.11 (Macintosh)	0.51	1056	1	Mature fruit	Whole fruit	7	0.05, 0.03 (0.04)
Blackfoot, ID, 2008 08.575.12 (Honey Crisp)	0.50	1629	1	Mature fruit	Whole fruit	7	0.02, <0.01 (0.02)
Pear							
North Rose, NY, 2008 08.575.13 (Bosc)	0.51	608	1	BBCH 85	Whole fruit	7	0.13, 0.16 (0.15)
Madera, CA, 2008 08.575.14 (Asian)	0.51	1608	1	Near mature fruit	Whole fruit	7	0.12, 0.12 (0.12)
Lindsay, CA/2008 08.575.15 (Olymic)	0.51	900	1	Mature fruit	Whole fruit	7	0.14, 0.13 (0.14)
Ephrata, WA, 2008 08.575.17 (Bartlett)	0.50	471	1	BBCH 85	Whole fruit	7	0.29, 0.26 (0.28)

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
Ephrata, WA, 2008[a] 08.575.16 (Concord)	0.51	796	1	BBCH 87	Whole fruit	7	0.24, 0.21 (0.23)
Ephrata, WA, 2008[a] 08.575.18 (Bartlett)	0.51	1876	1	BBCH 87	Whole fruit	7	0.24, 0.21 (0.23)

Notes:

[a] Not independent trials, conducted at the same location with treatment dates <1 month apart.

Stone fruits

Supervised residue trials on cherry (six trials), plum (six trials) and peach (nine trials) were conducted in the United States during 2008–2009 [Carringer, S.J., 2010; Report No. TCI-08-215].

Treated plots received one pre-harvest foliar application of the test substance (SC 200 g/L) at a rate of 0.50 kg ai/ha in cherry, 0.50–0.53 kg ai/ha in plum and 0.48–0.52 kg ai/ha in peach. The foliar spray including a non-ionic surfactant was made three days prior to normal fruit harvest, using airblast application equipment. The spray volume was 608–1833 L/ha in cherry, 496–1908 L/ha in plum and 514–1946 L/ha in peach. Duplicate samples from treated plot were collected, comprising of at least 24 fruit, weighing a minimum of 2 kg (cherry samples, ≥ 1 kg). Collected samples were stored frozen until analysis with a maximum storage period of 127 days. Trials of cherry, plum and peach included each one decline study (0, 3, 7, 12 or 14 days after treatment). In one trial (Trial TCI-08-215-17), an additional plot was treated for a processing study.

Table 10 Residue concentration of fenazaquin from residue trials on cherry, plum and peach in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥468	1			PHI=3	
Cherry							
Conklin, MI, 2008[a] TCI-08-215-01 (Sweet/ Sam)	0.50	1740	1	BBCH 87	Fruit	3	0.49, 0.49 (0.49)
Conklin, M, 2008[a] TCI-08-215-02 (Tart / Montmorency)	0.50	608	1	BBCH 89	Fruit	3	0.97, 0.86 (<u>0.91</u>)
Marengo, IL, 2009 TCI-08-215-03 (Tart Cherry/North Star	0.50	627	1	BBCH 87	Fruit	3	0.28, 0.23 (<u>0.26</u>)
Plainview, Cam, 2009 TCI-08-215-04 (Sweet/ Tulare)	0.50	1833	1	BBCH 89	Fruit	0 3 7 14	0.46, 0.68 0.37, 0.58 (<u>0.47</u>) 0.30, 0.30 0.091, 0.15
Royal City, WA, 2008 TCI-08-215-05 (Sweet/ Bing)	0.50	655	1	BBCH 89	Fruit	3	0.66, 0.45 (<u>0.56</u>)

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
Weiser, ID, 2009 TCI-08-215-06 (Tart/ Montmorency)	0.50	1132	1	BBCH 89	Fruit	3	0.71, 0.96 (0.84)
Plums							
Conklin, MI, 2008 TCI-08-215-16 (Stanley)	0.51	580	1	BBCH 87	Fruit	3	0.18, 0.17 (0.18)
Poplar, CA, 2008 TCI-08-215-17 (French Plum (Prunes))	0.50	1908	1	BBCH 89	Fruit	3	0.25, 0.22 (0.24)
Ducor, CA, 2008 TCI-08-215-18 (Black Cat)	0.53	636	1	BBCH 87	Fruit	0 3 7 14	0.015, 0.012 <0.01, <0.01 (<0.01) <0.01, <0.01 <0.01, <0.01
Exeter, CA, 2008 TCI-08-215-19 (Flavour Fall)	0.50	1366	1	BBCH 87	Fruit	3	0.11, 0.090 (0.11)
Dinuba, CA, 2008 TCI-08-215-20 (Fryer's)	0.52	496	1	BBCH 89	Fruit	3	0.021, 0.01 (0.016)
Monmouth, OR, 2008 TCI-08-215-21 (Moyer)	0.52	1525	1	BBCH 87	Fruit	3	0.19, 0.17 (0.18)
Peach							
Alton, NY, 2008 TCI-08-215-07 (Glohaven)	0.50	561	1	BBCH 85	Fruit	3	0.27, 0.24 (0.26)
Monetta, SC, 2008 TCI-08-215-08 Contender)	0.50	1300	1	BBCH 81	Fruit	3	0.35, 0.52 (0.44)
Chula, GA, 2008 TCI-08-215-09 (Hawthorne)	0.50	514	1	BBCH 87	Fruit	3	0.51, 0.32 (0.41)
Montezuma, GA, 2008 TCI-08-215-10 (Flame Prince)	0.50	1946	1	BBCH 89	Fruit	3	0.23, 0.25 (0.24)
Conklin, MI, 2008 TCI-08-215-11 (Bellaire)	0.50	608	1	BBCH 87	Fruit	3	0.38, 0.38 (0.38)
D'Hanis, TX, 2008 TCI-08-215-12 (La Feliciana)	0.52	1497	1	BBCH 85	Fruit	3	1.2, 0.57 (0.89)
Porterville, CA, 2008 TCI-08-215-13 (Fay Alberta)	0.49	599	1	BBCH 87	Fruit	0 3 7 12	0.29, 0.33 0.29, 0.12 (0.20) 0.091, 0.11 0.075, 0.085
Exeter, CA, 2008 TCI-08-215-14 (Klamt)	0.50	1730	1	BBCH 89	Fruit	3	0.86, 0.45 (0.65)
Strathmore, CA, 2008 TCI-08-215-15 (Ceres Carson)	0.48	589	1	BBCH 87	Fruit	3	0.22, 0.20 (0.21)

Notes:

[a] Not independent trials, conducted at the same location with a close treatment date, 13 days apart.

Berries and other small fruits

Raspberries and blueberries

Supervised residue trials on raspberries (five trials) and blueberries (high bush, six trials) were conducted in the United States in 2008 [Wyatt D.R., 2010; Report No. TCI-08-214].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.50–0.53 kg ai/ha in raspberries and 0.50–0.52 kg ai/ha in blueberries. The foliar spray including a non-ionic surfactant was made six or seven days prior to normal fruit harvest, using airblast or handheld vertical boom sprayers. The spray volume was 571–1871 L/ha in raspberries and 524–1235 L/ha in blueberries. Duplicate samples from treated plot were collected, each sample comprising of a minimum of 0.5 kg. Collected samples were stored frozen until analysis with a maximum storage period of 82 days. The trials of raspberry and blueberry included each one decline study (0, 7, 10, and 14 days after treatment).

Table 11 Residue concentration of fenazaquin from residue trials on raspberry and blueberry in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥468	1			PHI=7	
Raspberry							
Penn Yan, NY, 2008 TCI-08-214-01 (Heritage)	0.50	1871	1	BBCH 89	Fruit	7	0.32, 0.42 (0.36)
Conklin, MI, 2008 TCI-08-214-02 (Nova)	0.50	571	1	Maturity	Fruit	7	0.128, 0.18 (0.18)
Jefferson, OR, 2008 TCI-08-214-03 (Coho)	0.50	617	1	BBCH 89	Fruit	0 7 10 14	0.23, 0.40 (0.32) 0.18, 0.19 (0.18) 0.18, 0.15 (0.17) 0.18, 0.1183 (0.12)
Albany, OR, 2008 TCI-08-214-04 (Meeker)	0.53	1796	1	BBCH 89	Fruit	7	0.20, 0.29 (0.24)
Gervais, OR, 2008 TCI-08-214-05 (Meekers)	0.52	627	1	BBCH 89	Fruit	7	0.23, 0.14 (0.18)
Blueberry, high bush							
Penn Yan, NY, 2008 TCI-08-214-06 (Blueray)	0.50	935	1	BBCH 85	Fruit	0 7 10 14	1.5, 1.5 (1.5) 0.42, 0.40 (0.41) 0.31, 0.26 (0.28) 0.18, 0.16 (0.17)
Lahaska, PA, 2008 TCI-08-214-07 (Ozark Blue)	0.52	1235	1	Mature	Fruit	6	0.10, 0.24 (0.17)
Kinston, NC, 2008 TCI-08-214-08 (Blue Haven)	0.52	561	1	BBCH 87	Fruit	6	0.21, 0.26 (0.23)
Conklin, MI, 2008 TCI-08-214-09 (Blue Ray)	0.50	561	1	Maturity	Fruit	7	0.21, 0.25 (0.23)
Fremont, MI, 2008 TCI-08-214-10 (Blue Crop)	0.50	1122	1	Maturity	Fruit	7	0.24, 0.23 (0.24)
Corvallis, OR, 2008 TCI-08-214-11 (Duke)	0.52	524	1	BBCH 89	Fruit	7	0.31, 0.31 (0.31)

Grapes

Supervised residue trials on grapes (12 trials) were conducted in the United States in 2008 [Belcher, T.I., 2010; Report No. GR08-577].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.50–0.51 kg ai/ha. The foliar spray including a non-ionic surfactant was made seven days prior to normal fruit harvest, using tractor mounted or backpack airblast sprayers. The spray volume was 422–697 L/ha. Duplicate samples from treated plot were collected, each sample comprising of at least 12 grape bunches, weighing a minimum of 1 kg. Collected samples were stored frozen until analysis with a maximum storage period of 281 days.

Table 12 Residue concentration of fenazaquin from residue trials on grapes in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥468	1			PHI=7	
Dundee, NY, 2008 Trial 08-577.1 (Vidal Blanc)	0.51	477	1	Advanced Ripening	Fruit	7	0.35, 0.28 (0.32)
Orefield, PA, 2008 Trial 08-577.2 (Fredonia)	0.50	563	1	BBCH 85	Fruit	7	0.20, 0.23 (0.22)
Ukiah, CA, 2008 Trial 08-577.3 (Merlot)	0.50	657	1	Mature Fruit	Fruit	7	0.22, 0.18 (0.20)
Plainview, CA, 2008 Trial 08-577.4 (Crimson)	0.50	646	1	BBCH 85	Fruit	7	0.18, 0.18 (0.18)
Rich rave, CA, 2008 Trial 08-577.5 (Crimson)	0.51	697	1	BBCH 88	Fruit	7	0.08, 0.12 (0.10)
Madera, CA, 2008 Trial 08-577.6 (Thompson Seedless)	0.50	472	1	BBCH 85	Fruit	7	0.06, 0.08 (0.07)
Madera, CA, 2008 Trial 08-577.7 (Thompson Seedless)	0.50	558	1	Mature grapes	Fruit	7	0.19, 0.17 (0.18)
Paso Robles, CA, 2008 Trial 08-577.8 (Petite Syrah)	0.50	578	1	BBCH 89	Fruit	7	0.05, 0.04 (0.05)
Soledad, CA, 2008 Trial 08-577.9 (Chenin Blanc)	0.51	422	1	BBCH 89	Fruit	7	0.05, 0.05 (0.05)
Mecca, CA, 2008 Trial 08-577.10 (Thompson Seedless)	0.51	667	1	Brix -16	Fruit	7	0.40, 0.26 (0.33)
Ephrata, WA, 2008 Trial 08-577.11 (White Riesling)	0.50	560	1	BBCH 85	Fruit	7	0.32, 0.32 (0.32)
Ephrata, WA, 2008 Trial 08-577.12 (Petite Syrah)	0.50	684	1	BBCH 85	Fruit	7	0.16, 0.40 (0.28)

Strawberry

Supervised residue trials on strawberry (eight trials) were conducted in the United States during 2008-2009 [Wyatt, D.R., 2010; Report No. TCI-08-213].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.49–0.52 kg ai/ha. The foliar spray including a non-ionic surfactant was made one days prior to normal fruit harvest, using ground broadcast equipment. The spray volume was 767–917 L/ha. Duplicate samples from treated plot were collected, each sample weighing a minimum of 1.1 kg. Collected samples were stored frozen until analysis with a maximum storage period of 110 days. The trials included one decline study (0, 1, 7 and 10 days after treatment).

Table 13 Residue concentration of fenazaquin from residue trials on strawberry in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥468	1			PHI=1	
New Tripoli, PA, 2008 TCI-08-213-01 (All Star)	0.49	917	1	BBCH 87	Fruit	1	0.42, 0.28 (0.35)
Seven Springs, NC, 2008 TCI-08-213-02 (Camarosa)	0.49	823	1	BBCH 89	Fruit	1	0.61, 0.70 (0.65)
Oviedo, FL, 2009 TCI-08-213-03 (Camarosa)	0.52	767	1	BBCH 87	Fruit	1	1.2, 1.2 (1.2)
Sparta, MI, 2008 TCI-08-213-04 (All star)	0.50	842	1	BBCH 87	Fruit	1	0.36, 0.46 (0.41)
Porterville, CA, 2008 TCI-08-213-05 (Diamante)	0.50	879	1	BBCH 85	Fruit Fruit Fruit Fruit	0 1 7 10	0.39, 0.61 (0.50) 0.52, 0.52 (0.52) 0.26, 0.28 (0.27) 0.19, 0.19 (0.19)
Salinas, CA, 2008 TCI-08-213-06 (Albion)	0.52	870	1			1	0.63, 0.49 (0.56)
Watsonville, CA, 2008 TCI-08-213-07 (Camarosa)	0.52	870	1			1	0.59, 0.32 (0.46)
Junction City, OR, 2008 TCI-08-213-08 (Shukson)	0.50	795	1			1	0.059, 0.097(0.078)

Assorted tropical and sub-tropical fruits – inedible peel

Avocado

Supervised residue trials on avocado (five trials) were conducted in the United States in 2009 [Belcher, T., 2010; Report No. 09-02399]. Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.47–0.51 kg ai/ha. The foliar spray including a non-ionic surfactant was made seven days prior to normal fruit harvest, using ground application airblast equipment. The spray volume was 482–2716 L/ha. Duplicate samples from treated plot were collected and each sample was comprised of at least 24 fruit. Collected samples were stored frozen until analysis with a maximum storage period of 211 days for whole and peeled avocado.

Table 14 Residue concentration of fenazaquin from residue trials on avocado in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥935	1			PHI=7	
Carpenteria, CA, 2009 09-02399.1 (Haas)	0.50	1405	1	Mature	Peel+flesh Flesh	7	0.049, 0.049 (0.049) 0.013, <0.01 (0.01)
Portewille, CA, 2009 09-02399.2 (Zutano)	0.48	742	1	Mature BBCH 88	Peel+flesh Flesh	7	0.028, 0.046 (0.037) <0.01, <0.01 (<0.01)
Hemet, CA, 2009 09-02399.3 (Lane Haas)	0.51	858	1	Mature	Peel+flesh Flesh	7	0.050, 0.040 (0.045) <0.01, <0.01 (<0.01)
Fallbrook, CA, 2009 09-02399.4 (Bacon)	0.47	2716	1	Mature	Peel+flesh Flesh	7	0.035, 0.129 (0.082) <0.01, <0.01 (<0.01)
Homestead, FL, 2009 09-02399.5 (Monroe)	0.51 (Concentrate)	482	1	Mature	Peel+flesh Flesh	7	0.021, 0.043 (0.032) <0.01, <0.01 (<0.01)
	0.51 (Dilute)	1368	1	Mature	Peel+flesh Flesh	7	<0.01, 0.037 (0.023) <0.01, <0.01 (<0.01)

Fruiting vegetables, Cucurbits

Cucumber, Summer squash and melons

Supervised residue trials on cucumber (six trials), summer squash (zucchini five trials) and cantaloupe (six trials) were conducted in the United States in 2008 [Korpalski, S.J., 2010; Report No. GR08-582].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.50–0.52 kg ai/ha in cucumber, 0.50–0.51 kg ai/ha in summer squash and 0.49–0.52 kg ai/ha in melons. The foliar spray including a non-ionic surfactant was made three days prior to normal fruit harvest, using backpack or tractor-mounted sprayers. The spray volume was 183–236 L/ha in cucumber, 186–191 L/ha in summer squash and 185–194 L/ha in melons. Duplicate samples from treated plot were collected and each sample consisted of at least 12 fruits. Collected samples were stored frozen until analysis with a maximum storage period of 274 days.

Table 15 Residue concentration of fenazaquin from residue trials on cucumber and summer squash in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥234	1			PHI=3	
Cucumber							
Elko, SC, 2008 Trial 08-582.2 (Talladega)	0.50	183	1	Mature fruit	Whole fruit	3	0.03, 0.06 (0.05)
Chula. GA, 2008 Trial 08-582.3 (Thunder)	0.51	188	1	Mature fruit	Whole fruit	3	0.19, 0.14 (0.17)

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
Hobe Sound, FL, 2008 Trial 08-582.7 (Dasher II)	0.51	236	1	Mature fruit	Whole fruit	3	0.04, 0.03 (0.04)
Lime Springs, IA, 2008 Trial 08-582.9 (Bush Champion)	0.52	190	1	Mature fruit	Whole fruit	3	0.03, 0.03 (0.03)
Campbell, MN, 2008 Trial 08-582.10(Speedway)	0.51	187	1	Mature fruit	Whole fruit	3	0.05, 0.07 (0.06)
Trial 08-582.12, 2008 Hinton, OK (Poinsett 76)	0.50	186	1	Mature fruit	Whole fruit	3	0.08, 0.05 (0.07)
Summer squash							
Brodheadsville, PA, 2008 Trial 08-582.1 (Judgement 3)	0.51	189	1	Mature fruit	Whole fruit	3	0.04, 0.04 (0.04)
Chula. GA, 2008 Trial 08-582.4 (Justice III)	0.50	187	1	Mature fruit	Whole fruit	3	0.16, 0.10 (0.13)
Quincy, FL, 2008 Trial 08-582.6 (Justice III)	0.51	186	1	Mature fruit	Whole fruit	3	0.06, 0.05 (0.06)
Campbell, MN, 2008 Tria108-582.11 (Spineless Beauty)	0.51	188	1	Mature fruit	Whole fruit	3	0.06, 0.10 (0.08)
Madera, CA, 2008 Trial 08-582.15 (Black Beauty)	0.50	191	1	Mature fruit	Whole fruit	3	0.08, 0.07 (0.08)

Table 16 Residue concentration of fenazaquin from residue trials on melons (cantaloupe) in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥234	1			PHI=3	
Chula. GA, 2008 Trial 08-582.5 (Athena)	0.50	188	1	Mature fruit	Whole fruit	3	0.08, 0.06 (0.07)
Richland. IA, 2008 Trial 08-582.8 (Delicious 51)	0.50	185	1	Mature fruit	Whole fruit	3	0.17, 0.12 (0.15)
Hinton, OK, 2008 Trial 08-582.13 (PMR 45)	0.52	188	1	Mature fruit	Whole fruit	3	0.12, 0.06 (0.09)
Paso Robles, CA, 2008 Trial 08-582.14 (Hale's Best)	0.50	186	1	Mature fruit	Whole fruit	3	0.07, 0.03 (0.05)
Madera, CA, 2008 Trial 08-582.16 (Hearts of Gold)	0.50	188	1	Mature fruit	Whole fruit	3	0.05, 0.05 (0.05)
Porterville, CA, 2008 Trial 08-582.17 (Hale's Best Jumbo)	0.49	194	1	Mature fruit	Whole fruit	3	0.02, 0.02 (0.02)

Fruiting vegetables, other than Cucurbits

Tomato and peppers (Chili and sweet)

Supervised residue trials on tomatoes (12 trials), sweet pepper (six trials) and chili pepper (three trials) were conducted in the United States in 2008 [Carringer, S.J., 2010; Report No. TCI-08-216].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.50–0.52 kg ai/ha. The foliar spray including a non-ionic surfactant was made three days prior to normal fruit harvest in tomato, 0.49–0.52 kg ai/ha in sweet pepper and 0.50 kg ai/ha in chili pepper, using ground broadcast equipment. The spray volume was 131-309 L/ha in tomato, 112–281 L/ha in sweet pepper and 140–281 L/ha in chili pepper. Duplicate samples from treated plot were collected and each sample was comprised of a minimum of 12 large or 24 small fruit weighing at least 2 kg. Collected samples were stored frozen until analysis with a maximum storage period of 278 days. The trials on tomato and sweet pepper included each one decline study (0, 2 or 3, 7 and 14 days after treatment). Samples from the one trial (TCI-08-216-08) were used for a processing study.

Table 17 Residue concentration of fenazaquin from residue trials on tomato in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥234	1			PHI=3	
Brodheadsville, PA, 2008 TCI-08-216-01 (Red Pride)	0.50	187	1	Maturity	Whole fruit	3	0.046, 0.047 (0.046)
Seven Springs, NC, 2008 TCI-08-216-02 (Inbar)	0.50	271	1	BBCH 89	Whole fruit	3	0.034, 0.043 (0.038)
Oviedo FL, 2008 TCI-08-216-03 (Better Boy)	0.50	281	1	BBCH 79	Whole fruit	3	0.060, 0.081 (0.071)
Quincy, FL, 2008 TCI-08-216-04 (Crista)	0.50	234		BBCH 82	Whole fruit	3	0.039, 0.020 (0.029)
Conklin, MI, 2008 TCI-08-216-05 (Sunoma) cherry tomato	0.50	281	1	BBCH 84	Whole fruit	3	0.039, 0.034 (0.037)
Paso Robles, CA, 2008 TCI-08-216-06 (Sun Gold) cherry tomato	0.50	309	1	BBCH 85	Whole fruit	3	0.19, 0.18 (0.19)
Hughson, CA, 2008 TCI-08-216-07 (Classy Lady)	0.50	281	1	BBCH 89	Whole fruit	3	0.031, 0.027 (0.029)
Porterville, CA, 2008[a] TCI-08-216-08 (Sun 6117)_	0.50	131	1	BBCH 89	Whole fruit	3	0.042, 0.074 (0.058)
Lemoore, CA, 2008 TCI-08-216-09 (AB2)	0.50	131	1	BBCH 88	Whole fruit	0 3 7 14	0.12, 0.084 0.058, 0.071 (0.065) 0.039, 0.044 0.035, 0.025
Huron, CA, 2008 TCI-08-216-10 (Sun Brite)	0.53	131	1	BBCH 89	Whole fruit	3	0.057, 0.047 (0.052)
Five Points, CA, 2008 TCI-08-216-11 (UC 825)	0.52	299	1	BBCH 89	Whole fruit	3	0.064, 0.059 (0.061)
Porterville, CA, 2008[a] TCI-08-216-12 (9665)	0.50	178	1	BBCH 89	Whole fruit	3	0.028, 0.027 (0.027)

Notes:

[a] Not independent trials, conducted at a close location (5.4 miles apart) with a close treatment date (3 days apart)

Table 18 Residue concentration of fenazaquin from residue trials on peppers (Chili and sweet) in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥234	1			PHI=3	
Chili pepper							
Levelland, TX/2008 TCI-08-216-17 (Jalapeno M)	0.50	281	1	Maturity	Whole fruit	3	0.089, 0.074 (0.082)
Porterville, CA/2008 TCI-08-216-20 (Fresno)	0.50	140	1	BBCH 89	Whole fruit	3	0.22, 0.15 (0.19)
King City, CA/2008 TCI-08-216-21 (Chochi)	0.50	281	1	BBCH 79	Whole fruit	3	0.13, 0.12 (0.12)
Sweet pepper							
Seven Springs, NC/2008 TCI-08-216-13 (Heritage)	0.50	271	1	BBCH 89	Whole fruit	3	0.092, 0.066 (0.079)
Quincy, FL/2008 TCI-08-216-14 (Aristotle)	0.52	187	1	Maturity	Whole fruit	3	0.046, 0.061 (0.054)
Carlyle, IL/2008 TCI-08-216-15 (Big Bertha)	0.50	159	1	BBCH 89	Whole fruit	3	0.078, 0.034 (0.056)
Uvalde, TX/2008 TCI-08-216-16 (Camelot)	0.50	206	1	BBCH 72	Whole fruit	3	0.047, 0.065 (0.056)
Porterville, CA/2008 TCI-08-216-18 (Excalibur)	0.49	112	1	BBCH 87	Whole fruit	0 2 7 14	0.017, 0.017 <0.010, 0230 (0.017) 0.010, 0.026 (0.018) 0.014, 0.010
San Ardo, CA/2008 TCI-08-216-19 (Moody)	0.50	281	1	BBCH 79	Whole fruit	3	0.13, 0.11 (0.12)

Legume vegetables

Beans and peas, with pods

Supervised residue trials on beans with pods (snap bean nine trials) and peas with pods (snap peas three trials) were conducted in the United States in 2009 [Korpalski, S.J., 2010; Report No. 09-00522].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.50–0.53 kg ai/ha in beans with pods and 0.48–0.53 kg ai/ha in peas with pods. The foliar spray including a non-ionic surfactant was made seven days prior to normal harvest, using ground boom sprayers with multiple nozzles. The spray volume was 187–194 L/ha in beans with pods and 180–197 L/ha in peas with pods. Duplicate samples from treated plot were collected and each sample ranged from approximately 1 to 2 kg. Collected samples were stored frozen until analysis with a maximum storage period of 238 days.

Table 19 Residue concentration of fenazaquin from residue trials on beans and peas, with pods in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥187	1			PHI=7	
Beans with pods (snap bean)							
Germansville, PA, 2009 09-522.01 (Yellow snap bean / Eureka PVP)	0.52	190	1	Pod set	Beans with pods	7	0.16,0.16, 0.18, 0.17 (0.17)
Chula, GA, 2009 09-522.02 (Green snap bean/ Terminator)	0.50	189	1	Maturity	Beans with pods	7	0.086, 0.077, 0.13, 0.10 (0.099)
Newberry, FL, 2009 09-522.03 (Green snap bean/ Bronco)	0.50	188	1	Maturity	Beans with pods	7	0.14, 0.097, 0.090, 0.084 (0.10)
Campbell, MN, 2009 09-522.04 (Green snap bean / Greencrop)	0.50	187	1	BBCH79	Beans with pods	7	0.083, 0.10 (0.094)
Fitchburg, WI, 2009 09-522.05 (Green snap bean/Long Tendergreen)	0.53	194	1	Flowering to 6-in pods	Beans with pods	7	0.067, 0.088, 0.10, 0.11 (0.090)
American Falls, ID, 2009 09-522.06 (Green snap bean/Bean Strike)	0.53	193	1	BBCH 78	Beans with pods	7	0.18, 0.18 (0.18)
Peas with pods (snap pea)							
Paso Robles, CA, 2009 09-522.07 (Oregon Sugar Pod 11)	0.53	197	1	BBCH 79	Peas with pods	7	0.047, 0.034 (0.041)
Garey, CA, 2009 09-522.08 (Mammoth Melting)	0.50	187	1	Mature Pods	Peas with pods	7	0.17, 0.099 (0.13)
Payette, ID, 2009 09-522.09 (Oregon Sugar Pod II)	0.48	180	1	BBCH 77	Peas with pods	7	0.088, 0.11 (0.10)

Succulent beans and peas, without pods

Supervised residue trials on succulent beans without pods (lima bean six trials) and succulent peas without pods (garden pea 5 trials) were conducted in the United States in 2009 [Korpalski, S.J., 2010; Report No.09-00521].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.50–0.52 kg ai/ha in succulent beans without pods and 0.50 kg ai/ha in succulent peas without pods. The foliar spray including a non-ionic surfactant was made seven days prior to normal harvest, using ground boom sprayers with multiple nozzles. The spray volume was 184–192 L/ha in succulent beans with pods and 188–192 L/ha in succulent peas without pods. Duplicate samples from treated plot were collected and each sample ranged from approximately 0.32–2.75 kg (0.32-0.45 kg shelled bean in the Trial 09-

521.04; ≥1 kg in the other trials). After shelling, samples were stored frozen until analysis with a maximum storage period of 233 days.

Table 20 Residue concentration of fenazaquin from residue trials on succulent beans and peas, without pods in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥187	1			PHI=7	
Succulent beans without pods (lima bean)							
Chula, GA, 2009 09-521.01 (Cangreen)	0.52	192	1	Mature	Shelled Beans	7	<0.01, <0.01 (<u>≤0.01</u>)
Alcolu, SC, 2009 09-521.02 (Jackson Wonder)	0.50	188	1	Mature Pods	Shelled Beans	7	0.017, 0.016 (<u>0.017</u>)
Montezuma, GA, 2009 09-521.03 (Cangreen)	0.50	184	1	Mature	Shelled Beans	7	<0.01, <0.01 (<0.01)*
Campbell, MN, 2009 09-521.04 (Jackson Wonder Bush))	0.50	187	1	BBCH75 (50 percent of pods at typical length)	Shelled Beans	7	<0.01, <0.01 (<u>≤0.01</u>)
Fresno, CA, 2009 09-521.05 (Ford Hook 244)	0.50	185	1	BBCH 89	Shelled Beans	7	<0.01, <0.01 (<u>≤0.01</u>)
Payette, ID, 2009 09-521.06 (Henderson Baby Lima)	0.50	189	1	Individual beans fully formed	Shelled Beans	7	<0.01, <0.01 (<u>≤0.01</u>)
Succulent peas without pods (garden pea)							
Elko, SC, 2009 09-521.07 (Wando)	0.50	191	1	Mature Pods/Peas	Shelled peas	7	<0.01, <0.01 (<u>≤0.01</u>)
Conklin, MI, 2009 09-521.08 (Mr. Big)	0.50	192	1	BBCH 79	Shelled peas	7	<0.01, <0.01 (<u>≤0.01</u>)
Richmond, WI, 2009 09-521.09 (Jun)	0.50	188	1	Mature Peas	Shelled peas	7	<0.01, <0.01 (<u>≤0.01</u>)
Campbell, MN, 2009 09-521.10 (Wando)	0.50	192	1	BBCH 79	Shelled peas	7	<0.01, <0.01 (<u>≤0.01</u>)
Payette, ID, 2009 09-521.11 (Green Arrow)	0.50	188	1	Green Ripe	Shelled Peas	7	<0.01, <0.01 (<u>≤0.01</u>)

Notes:

* Shelled bean sample collected, <1 kg.

Pulses

Beans and peas, dry

Supervised residue trials on dry beans (pinto bean nine trials) and dry peas (Australian winter pea five trials) were conducted in the United States in 2009 [Korpalski, S.J., 2010; Report No.09-00523].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.50–0.52 kg ai/ha in dry beans and 0.50 kg ai/ha in dry peas. The foliar spray including a non-ionic surfactant

was made seven or eight days prior to normal harvest, using ground boom sprayers with multiple nozzles. The spray volume was 183–194 L/ha in dry beans and 169–188 L/ha in dry peas. Duplicate samples from treated plot were collected and each shelled sample weighted at least 1 kg. Before shelling, beans samples from four trials (09-523.02, 09-523.06, 09-523.07 and 09-523.09) were field dried for 1, 5, 8 and 13 days, respectively, under ambient temperature or 0–1 °C, and pea samples from one trial (Trial 09-523-14) were air dried for 3 days under ambient conditions. The other bean and pea samples were shelled on the same day with harvest. Bean and pea samples were stored frozen until analysis with a maximum storage period of 144 days. For collection of pea vine and hay samples, treatment was made to another plot.

Table 21 Residue concentration of fenazaquin from residue trials on beans and peas, dry in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥187	1			PHI=7	
Dry beans (pinto bean)							
Centerville, SD, 2009 09-523.01 (Montrose)	0.50	183	1	BBCH 96 (80 percent of leaves fallen)	Shelled dry beans	7	0.012, 0.022 (0.016)
York, NE, 2009 09-523.02 (Chase)	0.50	186	1	BBCH 88 (80-90 percent pods ripe)	Shelled dry beans	7	0.013, 0.022 (0.018)
Campbell, MN, 2009 09-523.03 (Lariat)	0.50	187	1	BBCH 87 (70 percent of beans hard)	Shelled dry beans	7	<0.01, <0.01 (<0.01)
Larned, KS, 2009 09-523.04 (Chase)	0.52	194	1	BBCH 88 (80 percent pods ripe)	Shelled dry beans	7	0.055, 0.12 (0.088)
York, NE, 2009 09-523.05 (Chase)	0.5	188	1	BBCH 88 (80-90 percent pods ripe)	Shelled dry beans	7	0.024, 0.042 (0.033)
Larned, KS, 2009 09-523.06 (Chase)	0.51	190	1	BBCH 87 (70 percent pods ripe)	Shelled dry beans	7	0.020, 0.036 (0.028)
Jerome, ID, 2009 09-523.07 (Othello)	0.51	191	1	BBCH 86 (60 percent pods ripe)	Shelled dry beans	7	<0.01, <0.01 (<0.01)
Fresno, CA, 2009 09-523.08 (variety unknown)	0.50	186	1	BBCH 87 (70 percent pods ripe)	Shelled dry beans	7	0.015, 0.013 (0.014)
American Falls, ID, 2009 09-523.09 (Chase)	0.50	190	1	BBCH 79 Individual beans easily visible	Shelled dry beans	8	0.17, 0.17 (0.17)
Dry peas (Austrian winter pea)							
Grand Island, NE, 2009 09-523.10 (Austrian Field Pea)	0.50	181	1	BBCH 87 (70 percent pods ripe)	Shelled dry peas	7	<0.01, <0.01 (<0.01)
Ephrata, WA, 2009 09-523.11 (Austrian Winter Pea)	0.50	187	1	BBCH 88 (80 percent pods ripe)	Shelled dry peas	7	0.050, 0.053 (0.052)

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
Hood River, OR, 2009 09-523.12 (Austrian Winter Pea, Fenn variety)	0.50	169	1	Pods fully mature	Shelled dry peas	7	0.010, 0.011 (0.011)
Payette, ID, 2009 09-523.13 (Austrian Winter Pea)	0.50	188	1	BBCH 88 (80 percent pods ripe)	Shelled dry peas	7	0.012, 0.014 (0.013)
Jerome, ID, 2009 09-523.14 (Austrian Winter Pea)	0.50	186	1	BBCH 88 (80 percent pods ripe)	Shelled dry beans	7	0.013, 0.015 (0.014)

Herbs

Mint

Supervised residue trials on mint (five trials; four peppermint and one spearmint) were conducted in the United States in 2008 [Wyatt, D.R., 2010; Report No. TCI-08-217].

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.49–0.52 kg ai/ha. The foliar spray including a non-ionic surfactant was made seven days prior to normal harvest, using ground broadcast equipment. The spray volume was 140–215 L/ha. Duplicate samples from treated plot were collected and each sample weighted a minimum of 1 kg. Mint samples (fresh tops, leaves and stems) were stored frozen until analysis with a maximum storage period of 155 days. The trials included one decline study (0, 7, 10 and 14 days after treatment). In one trial (TCI-08-217-05), an additional plot was treated for a processing study (peppermint oil).

Table 22 Residue concentration of fenazaquin from residue trials on mint in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥187	1	Before bloom		PHI=7	
Paynesville, MN, 2008 TCI-08-217-01 (Spearmint /Mentha Spicata)	0.50	168	1	BBCH 48 (80 percent of leaf mass reached)	Tops, fresh	7	5.2, 5.5 (5.3)
St. Johns, MI, 2008 TCI-08-217-02 (Peppermint /Black Mitchum)	0.49	215	1	Vegetative, 20-30 leaves	Tops, fresh	7	0.87, 1.0 (0.93)
George, WA, 2008 TCI-08-217-03 (Peppermint /Todds Mitchem)	0.52	140	1	BBCH 55, pre-bloom	Tops, fresh	0 7 10 14	6.7, 8.5 0.63, 0.52 (0.57) 0.67, 0.31 0.23, 0.17
Prinville, OR, 2008 TCI-08-217-04 (Peppermint /M-83-7)	0.49	187	1	BBCH 51 (flower buds)	Tops, fresh	7	0.73, 0.55 (0.64)
Culver, OR, 2008 TCI-08-217-05 (Peppermint /N-83-7)	0.50	187	1	BBCH 51 (flower buds)	Tops, fresh	7	1.7, 1.4 (1.6)

Animal feeds

Pea vines and hay

Supervised residue trials on dry pea [Korpalski, S.J., 2010; Report No.09-00523] included an additional plot for collecting vine and hay samples.

Treated plots received one foliar application of the test substance (SC 200 g/L) at a rate of 0.50–0.52 kg ai/ha. The foliar spray including a non-ionic surfactant was made seven days prior to normal harvest, using ground boom sprayers with multiple nozzles. The spray volume was 186–198 L/ha. Duplicate samples from treated plot were collected and each sample weighted 1.1–1.8 kg in vines and 0.57–0.82 kg in hay. Hay samples were field dried for 5–13 days under ambient conditions (moisture content after drying, 10–20 percent). Collected samples were stored frozen until analysis with a maximum storage period of 140 days in vines and 219 days in hay.

Table 23 Residue concentration of fenazaquin from residue trials on pea vines and hay in the United States

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
GAP: United States	0.359-0.538 (0.538 kg ai/ha/year)	≥187	1			PHI=7	
Pea vines (Austrian winter pea)							
Grand Island, NE, 2009 09-523.10 (Austrian Field Pea)	0.50	186	1	BBCH 70 (early pod development)	Pea Vines	7	0.21, 0.31 (0.26)
Ephrata, WA, 2009 09-523.11 (Austrian Winter Pea)	0.50	188	1	BBCH 67 (flowering declining)	Pea Vines	7	1.4, 1.6 (1.5)
Hood River, OR, 2009 09-523.12 (Austrian Winter Pea, Fenn variety)	0.50	189	1	Early pod formation	Pea Vines	7	1.6, 1.8 (1.7)
Payette, ID, 2009 09-523.13 (Austrian Winter Pea)	0.50	187	1	Early pod formation	Pea Vines	7	1.9, 1.7 (1.8)
Jerome, ID, 2009 09-523.14 (Austrian Winter Pea)*	0.52	198	1	BBCH 51 (first flower buds)	Pea Vines	7	4.5, 3.1 (3.8)
Pea hays (Austrian winter pea)							
Grand Island, NE, 2009 09-523.10 (Austrian Field Pea)	0.50	186	1	BBCH 70 (early pod development)	Pea Hay	7	0.78, 0.78 (0.78)
Ephrata, WA, 2009 09-523.11 (Austrian Winter Pea)	0.50	188	1	BBCH 67 (flowering declining)	Pea Hay	7	4.2, 6.4 (5.3)
Hood River, OR, 2009 09-523.12 (Austrian Winter Pea, Fenn variety)	0.50	189	1	Early pod formation	Pea Hay	7	4.1, 9.4 (6.8)

Location, Year Trial ID (Variety)	Application				Portion analysed	DAT	Fenazaquin, mg/kg (mean)
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage			
Payette, ID, 2009 09-523.13 (Austrian Winter Pea)	0.50	187	1	Early pod formation	Pea Hay	7	8.6, 10 (9.5)
Jerome, ID, 2009 09-523.14 (Austrian Winter Pea)*	0.52	198	1	BBCH 51 (first flower buds)	Pea Hay	7	23, 21 (22)

Notes:

* Growth in the trial (09-523.14), where planted in late summer and treated in the fall, was slower than the other trials where early treatment occurred at the typical early-summer timing.

FATE OF RESIDUES IN STORAGE AND PROCESSING

The Meeting received processing studies simulating commercial practices on orange, plum, grapes, tomato and mint. Details on field trial were described in the section of residues resulting from supervised trials on crops, except for grape.

Mint (oil)

In one supervised field trial on mint [TCI-08-217], an additional plot was treated at 2.67 kg ai/ha (5× the maximum label rate) and a sample of fresh mint (tops and leaves) was taken 7 days after treatment. After drying in the field for 3 days, this mint hay sample (20 kg) was further air-dried for 3 hours to achieve a moisture content of < 50 percent and duplicate sub-samples were taken for analysis. The dried mint hay (15 kg) was subjected to steam distillation (steam injected for 1–2 hours in steam retort) to produce peppermint oil (35 mL).

Orange (juice, oil and dried pulp)

Sample of orange was obtained from a plot treated at 2.53 kg ai/ha (5× the maximum label rate) [GR08-576]. The harvest orange was processed into juice, dried pulp and oil fractions. The residue concentration in the peeled orange (flesh) was also analysed.

Plum (prune)

Sample of plum was obtained from a plot treated at 2.50 kg ai/ha (5× the max. label rate) [TCI-08-215]. The fresh prunes were washed and then air dried at 68–79 ˚C until when the appropriate moisture content (approximately 27 percent) was achieved.

Grape (wine, juice and raisin)

Three residue trials were conducted during the 2007 growing season in Southern and Northern France [Simek, I., 2009, Report No. R A7167]. In each trial, one foliar application with a SC formulation (200 g/L) was treated on the plot at a rate of 1.0 kg ai/ha. Grapes were harvested 21 days after application. The grape samples were processed into red wine, juice (clarified and pasteurized) and raisin (moisture content, 16 percent).

Tomato (past and puree)

Sample of tomato was obtained from a plot treated at 2.54 kg ai/ha (5× the max. label rate) [TCI-08-216]. The harvest tomato was processed into paste and puree.

Table 24 Residue concentration of fenazaquin in processed products

Location, Year Trial ID (Variety)	Application				Harvest time, DAT	Matrix analysed	Fenazaquin, mg/kg	Pf
	Rate, kg ai/ha	Water, L/ha	No.	Growth stage				
Groveland, FL, 2008 08-576.11 (Navel)	2.53 SC200	547	1		7	Whole orange (RAC) Orange, flesh Orange juice Orange oil Dried pulp	1.05 <0.01 0.01 82.64 0.19	 0.01 78.7 0.18
Poplar, CC, 2008 TCI-08-215-17 (French plum (Prunes))	2.50 SC200		1		3	Plum fruit (RAC) Prunes	0.698 3.37	 4.8
Bellocq, France, 2007 A7167SA1 (Tannat)	1.0 SC200	829	1	BBCH 85-89	21	Grapes (RAC) Red wine Grapes (RAC) Juice Raisin	0.41 <0.01 0.33 0.12 0.96	 <0.02 0.36 2.9
Duras, France, 2007 A7167DR1 (Cabernet Franc)	1.0 SC200	796	1	BBCH 85-89	21	Grapes (RAC) Red wine Grapes (RAC) Juice Raisin	0.42 <0.01 0.43 0.06 0.95	 <0.02 0.14 2.2
St Aubin de Luigné, France, 2007 A7167BM1 (Cabernet Sauvignon)	1.0	820	1	BBCH 85	21	Grapes (RAC) Red wine Grapes (RAC) Juice Raisin	0.39 <0.01 0.38 0.05 0.84	 <0.03 0.13 2.2
Porterville, CA, 2008 TCI-08-216-08 (Sun 6117)	2.54		1		3	Tomato (RAC) Tomato paste Tomato puree	0.217 0.195 0.0886	 0.90 0.41
Culver, OR, 2008 TCI-08-217-05 (Peppermint/N-83-7)	2.67		1		7	Fresh mint (RAC) Mint hay Mint oil	Not analysed 20.5 9.51	 - -

Notes:

Residue concentration of fenazaquin is a mean value of duplicate analyses, except for grape studies.

APPRAISAL

Fenazaquin is a quinazoline insecticide/acaricide. It was first evaluated by JMPR in 2017 for toxicology and residues. Subsequently, additional uses were evaluated by the 2019 Extra JMPR Meeting.

The 2017 JMPR established an ADI of 0–0.05 mg/kg bw and an ARfD of 0.1 mg/kg bw, applying to fenazaquin, tertiarybutylphenylethanol (TBPE), 4-hydroxyquinazoline and 2-hydroxy-fenazaquin acid. Residue definition for plant commodities is fenazaquin for compliance with the MRL and for dietary risk assessment. For animal commodities, the residue definition is the sum of fenazaquin and 2-hydroxy-fenazaquin acid for compliance with the MRL and the sum of fenazaquin, 2-hydroxy-fenazaquin acid, and tautomeric forms of 4-hydroxyquinazoline for dietary risk assessment. The residue is fat-soluble.

Fenazaquin was scheduled at the Fifty Second Session of the CCPR for evaluation of additional uses by the 2022 JMPR. The Meeting received information on residue trials (avocado, berries, citrus fruits, pome fruits, stone fruits, fruiting vegetables, beans and peas, and mint), processing and storage stability. In addition, a confined rotational crop study and a new analytical method were provided.

Confined rotational crop study

A confined rotational crop study with [^{14}C-phenyl] and [^{14}C-quinazoline] fenazaquin was conducted using lettuce, radish and wheat at 30, 120, and 365 day plant back intervals. Radiolabelled fenazaquin was applied to the bare soil at a rate of 550–556 g/ha. Total radioactive residues from the two labels were in similar levels, and gradually declined with increasing PBIs in the food commodities. The total radioactive residues were 0.004–0.055 mg eq/kg in immature lettuce, 0.008–0.067 mg eq/kg in mature lettuce, 0.008–0.104 mg eq/kg in radish roots, 0.007–0.030 mg eq/kg in radish tops, 0.009–0.129 mg eq/kg in wheat forage, 0.013–0.189 mg eq/kg in wheat hay, 0.025–0.243 mg eq/kg in wheat straw and 0.010–0.069 eq/kg in wheat grain.

Extractability of residues using organic solvents was 58.8–61.1 percent TRR in immature lettuce, 51.2–63.9 percent TRR in mature lettuce, 72.7–80.6 percent TRR in radish roots, 61.9–85.7 percent TRR in radish tops, 65.7–91.4 percent TRR in wheat forage, 49.6–69.6 percent TRR in wheat hay, 47.2–72.4 percent TRR in wheat straw and 28.6–49.3 percent TRR in wheat grain.

Only in 30-day PBI radish roots, parent fenazaquin was found at a greater level than 10 percent TRR or 0.01 mg/kg, representing 28.6–29.0 percent TRR (0.026–0.031 mg/kg). In all tested rotational crops except for wheat grain, metabolite 4-hydroxyquinazoline was detected, but did not exceed the level of 13.8 percent TRR or 0.012 mg eq/kg. The other identified metabolites were also found in the crops, but at low levels of below 4.5 percent TRR or 0.004 mg eq/kg, in addition, many minor components present at very low levels below 0.016 mg eq/kg were found, except for two components in wheat hay (0.023 mg eq/kg and 0.035 mg eq/kg) and one component in wheat straw (0.064 mg eq/kg).

The Meeting concluded that type and amount of residues in rotational crops would not impact on the current residue definition for plant commodities, and that significant residues are not expected in leafy vegetable, root and tuber vegetable or cereals grown as rotational crops.

Environmental fate

The 2017 JMPR concluded that fenazaquin is moderately persistent in soil under field conditions (DT_{50} values ranging from 26-114 days) and that the photolysis of fenazaquin in soil, under sunlight conditions, was an important degradation pathway (soil surface DT_{50} of 15 days).

Methods of analysis

Analysis of fenazaquin residue was conducted using analytical methods evaluated and considered suitable by previous JMPRs. The recovery test results from the studies submitted to the current Meeting were acceptable and the LOQs of fenazaquin were 0.01 mg/kg. In some storage stability tests, a new method employing QuEChERs and LC-MS/MS was used. The Meeting considered this method was sufficiently validated with LOQ of 0.01 mg/kg in apple, tomato, grape, peach and orange matrices.

Stability of residues in stored analytical samples

The Meeting received storage stability studies for fenazaquin in high water commodities (apple, cucumber, melon, peach, tomato) and high acid commodities (grape, orange). The results demonstrated that residues of fenazaquin were stable for at least 12 or 13 months in the stored frozen matrices, respectively.

In the previous JMPR, the Meeting considered fenazaquin residues are stable when stored frozen for up to 3.5 months for high starch commodity (maize grain) and at least 17 months for high oil commodity (almond nutmeat). For high protein commodities, the information was not available to the Meeting.

The frozen sample storage intervals in the field trials were all within the acceptable storage stability periods.

Results of supervised residue trials on crops

The US GAP provided to the Meeting permits a single foliar application of 0.538 kg ai/ha (0.538 kg ai/ha/year) fenazaquin on all registered crops. All residue trials submitted to this Meeting were conducted with a single foliar application in the United States.

Citrus fruits

The US GAP (1×0.538 kg ai/ha, 7-day PHI) covers the commodities in the Codex citrus fruits group and residue trials on lemon, orange and grapefruit matched this GAP.

Orange

In whole oranges, residues were (n= 10 from independent trials): 0.08, 0.09, 0.11, 0.11, 0.12, 0.13, 0.15, 0.15, 0.19 and 0.23 mg/kg. In flesh, residues of fenazaquin were not detected, all <0.01 mg/kg (n=10).

The Meeting estimated a maximum residue level of 0.4 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.01 mg/kg for the Subgroup of oranges, sweet, sour. The median (whole fruit) residue was 0.125 mg/kg.

Lemon

In whole lemons, residues were (n=5): 0.02, 0.04, 0.08, 0.11 and 0.12 mg/kg. In flesh, residues of fenazaquin were not detected, all <0.01 mg/kg (n=5).

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.01 mg/kg for the Subgroup of lemons and limes. For kumquat, the Meeting estimated an STMR of 0.08 mg/kg and an HR of 0.12 mg/kg for whole fruit. The median (whole fruit) residue is 0.08 mg/kg.

Grapefruit

In whole grapefruits, residues were (n=5 from independent trials): 0.03, 0.04, 0.07, 0.11 and 0.14 mg/kg. In flesh, residues of fenazaquin were not detected, all <0.01 mg/kg (n=5)

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.01 mg/kg for the Subgroup of pummelo and grapefruits. The median (whole fruit) residue is 0.07 mg/kg.

Mandarin

For the Subgroup of mandarins, the Meeting agreed to extrapolate the residue data set for lemons to estimate a maximum residue level.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.01 mg/kg for the Subgroup of mandarins. The median (whole fruit) residue is 0.08 mg/kg.

Pome fruits

The US GAP (1×0.538 kg ai/ha, 7-day PHI) covers the commodities in the Codex pome fruit group except Japanese persimmon and residue trials on apple and pear matched this GAP.

In apple, residues were (n=11 from independent trials): <0.01, 0.02, 0.03, 0.04, 0.05, 0.08, 0.09, 0.11, 0.12, 0.13, and 0.15 mg/kg with the highest analytical value of 0.18 mg/kg.

In pear, residues were (n=5 from independent trials): 0.12, 0.14, 0.15, 0.23 and 0.28 mg/kg with highest analytical value of 0.29 mg/kg.

The median residues in the datasets for apple and pear are within a 5-fold difference, however, the Mann-Whitney test showed that the residues were not from the same population. For pears, with higher residues, only five trial results were available, and the Meeting considered that 5 trials were not enough to support a pome fruit group maximum residue level.

For apples, the Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.08 mg/kg and an HR of 0.18 mg/kg.

For pears, the Meeting agreed there were insufficient trials to estimate a maximum residue level.

Stone fruits

The US GAP (1×0.538 kg ai/ha, 3-day PHI) covers the commodities in the Codex stone fruits group and residue trials on cherries, plums and peaches matched this GAP, although residues were measured in fruit without stones.

The 2017 JMPR concluded that for stone fruit, residues measured in fruit without stones would overestimate whole-fruit residues by about 10 percent and that correcting for this factor would lead to the same maximum residue level estimation.

The median residues in the datasets for cherries, plums and peaches are within a 5-fold difference, however, the Kruskal-Wallis test showed that these data sets were not from the same population. Therefore, the Meeting decided to estimate separate recommendations for the subgroups.

Cherry

In cherries (without stones), residues were (n=5 from independent trials): 0.26, 0.47, 0.56, 0.84 and 0.91 mg/kg with highest analytical value of 0.97 mg/kg.

The Meeting agreed there were insufficient trials on cherries to estimate a maximum residue level.

Peach

In peaches (without stones), residues were (n=9): 0.20, 0.21, 0.24, 0.26, 0.38, 0.41, 0.44, 0.65 and 0.89 mg/kg with highest analytical value of 1.2 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR of 0.38 mg/kg and an HR of 1.2 mg/kg for the Subgroup of peaches.

Plums

In plums (without stones), residues were (n=6): <0.01, 0.016, 0.11, 0.18, 0.18 and 0.24 mg/kg with highest analytical value of 0.25 mg/kg

The Meeting estimated a maximum residue level of 0.5 mg/kg, an STMR of 0.145 mg/kg and an HR of 0.25 mg/kg for the Subgroup of plums.

Cane berries

The US GAP (1×0.538 kg ai/ha, 7-day PHI) covers the commodities in the Codex cane berries subgroup and in residue trials on raspberries matching this GAP (PHI 7 days), residues were (n=5): 0.18, 0.18, 0.18, 0.24 and 0.36 mg/kg with highest analytical value of 0.41 mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg, an STMR of 0.18 mg/kg and an HR of 0.41 mg/kg for the Subgroup of cane berries.

Bush berries

The US GAP (1×0.538 kg ai/ha, 7-day PHI) covers the commodities in the Codex bush berries subgroup and in residue trials on blueberries matching this GAP (PHI 7 days), residues were (n=6): 0.17, 0.23, 0.23, 0.24, 0.31 and 0.41 mg/kg with highest analytical value of 0.42 mg/kg.

The Meeting estimated a maximum residue level of 0.8 mg/kg, an STMR of 0.235 mg/kg and an HR of 0.42 mg/kg for the Subgroup of bush berries.

Small fruit vine climbing

The US GAP (1×0.538 kg ai/ha, 7-day PHI) covers the commodities in the Codex small fruit vine climbing subgroup and in residue trials on grapes matching this GAP (PHI 7 days), residues were (n=12): 0.05, 0.05, 0.07, 0.10, 0.18, 0.18, 0.20, 0.22, 0.28, 0.32, 0.32 and 0.33 mg/kg with highest analytical value of 0.40 mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg, an STMR of 0.19 mg/kg and an HR of 0.4 mg/kg for the Subgroup of small fruit vine climbing.

Low growing berries

The US GAP (1×0.538 kg ai/ha, 3-day PHI) covers the commodities in the Codex low growing berries subgroup and in residue trials on strawberries matching this GAP, residues were (n=8): 0.078, 0.35, 0.41, 0.46, 0.52, 0.56, 0.65 and 1.2 mg/kg with highest analytical value of 1.2 mg/kg

The Meeting estimated a maximum residue level of 2 mg/kg, an STMR of 0.49 mg/kg and an HR of 1.2 mg/kg for the Subgroup of low growing berries.

Avocado

The US GAP for avocado is 1×0.538 kg ai/ha, 7-day PHI and in residue trials matching this GAP, residues in avocados (without stones) were (n=5): 0.032, 0.037, 0.045, 0.049 and 0.082 mg/kg. In avocado flesh, residues were (n=5): <0.01 (4) and 0.01 mg/kg.

Based on information available from other avocado residue studies, stones do not make up more than 15 percent of the whole fruit weight and the Meeting concluded that correcting the reported residues to express them on a whole fruit basis would lead to the same maximum residue level estimation.

The Meeting estimated a maximum residue level of 0.15 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.01 mg/kg for avocado.

Fruiting vegetables, Cucurbits

The US GAP (1×0.538 kg ai/ha, 3-day PHI) covers the commodities in the Codex fruiting vegetables, Cucurbits and residue trials on cucumber, summer squash and melons matched the GAP.

In cucumber, residues were (n=6): 0.03, 0.04, 0.05, 0.06, 0.07 and 0.17 mg/kg.

In Summer squash, residues were (n=5): 0.04, 0.06, 0.08, 0.08 and 0.13 mg/kg.

In melons, residues were (n=6): 0.02, 0.05, 0.05, 0.07, 0.09 and 0.15 mg/kg.

The median residues in the datasets for cucumber, summer squash and melons are within a 5-fold difference and the Kruskal-Wallis test showed that the residues were not from different populations. Therefore, the Meeting decided to estimate recommendations for the group of fruiting vegetables, Cucurbits.

The combined residues were (n=17): 0.02, 0.03, 0.04, 0.04, 0.05, 0.05, 0.05, 0.06, 0.06, 0.07, 0.07, 0.08, 0.08, 0.09, 0.13, 0.15 and 0.17 mg/kg with highest analytical value of 0.19 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.06 mg/kg and an HR of 0.19 mg/kg for the Group of fruiting vegetables, Cucurbits.

Tomatoes

The US GAP (1×0.538 kg ai/ha, 3-day PHI) covers the commodities in the Codex subgroup of tomatoes (PHI, 3 days) and in residue trials on tomatoes, residues were (n=11 from independent trials): 0.029, 0.029, 0.037, 0.038, 0.046, 0.052, 0.058, 0.061, 0.065, 0.071 and 0.19 mg/kg with highest analytical value of 0.19 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.052 mg/kg and an HR of 0.19 mg/kg for the Subgroup of tomatoes.

Peppers

The US GAP (1×0.538 kg ai/ha, 3-day PHI) covers the commodities in the Codex subgroup of peppers and in residue trials matching this GAP, residues in sweet peppers (6) and *chili peppers* (3) were (n=9): 0.018, 0.054, 0.056, 0.056, 0.079, *0.082*, *0.12*, 0.12 and *0.19* mg/kg with highest analytical value of 0.22 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.079 mg/kg and an HR of 0.22 mg/kg for the Subgroup of peppers (except martynia, okra and roselle).

For dried chili peppers, the Meeting applied the default concentration factor of 10 to the data set for fresh peppers and estimated a maximum residue level of 3 mg/kg, an STMR of 0.79 mg/kg and an HR of 2.2 mg/kg for dried chili pepper.

Eggplant

The US GAP (1×0.538 kg ai/ha, 3-day PHI) covers the commodities in the Codex subgroup of eggplants and in line with the 2018 JMPR recommendation that residue data on tomatoes or peppers (whichever is higher) could be extrapolated to eggplants, the Meeting agreed to extrapolate the data for peppers to estimate a maximum residue level of 0.3 mg/kg, an STMR of 0.079 mg/kg and an HR of 0.22 mg/kg for the Subgroup of eggplants

Legume vegetables

The US GAP (1×0.538 kg ai/ha, 7-day PHI) covers all commodities in the Codex legume vegetables group (except underground beans and peas) and residue trials on snap beans with pods, snap peas with pods, lima beans without pods and garden pea without pods matched this GAP.

Plant metabolism studies previously evaluated by the JMPR covered fruit (apple, orange, grape) and cereals (maize). As plant metabolism studies covering pulses and oilseeds or a third crop from a group different to fruits or cereal grains were not available, the Meeting could not estimate dietary intake of residues in legume vegetables.

Beans and peas with pods

In snap beans with pods, residues were (n=6): 0.09, 0.094, 0.099, 0.1, 0.17 and 0.18 mg/kg and in snap peas with pods, residues were (n=3): 0.041, 0.1 and 0.13 mg/kg.

The Meeting agreed to combine the data for beans and peas with pods for mutual support to estimate subgroup maximum residue levels.

The combined data set for snap beans and snap peas is (n=9): 0.041, 0.09, 0.094, 0.099, 0.1, 0.1, 0.13, 0.17 and 0.18 mg/kg

The Meeting estimated but did not recommend a maximum residue level of 0.4 mg/kg for the Subgroups of beans with pods and peas with pods because a dietary intake assessment could not be completed.

Beans and peas without pods

In lima beans without pods, residues were (n=5): <0.01 (4) and 0.017 mg/kg and in garden peas without pods, residues were (n=5): <0.01 (5).

The Meeting agreed to combine the data for beans and peas without pods for mutual support to estimate subgroup maximum residue levels.

The Meeting estimated but did not recommend a maximum residue level of 0.02 mg/kg for the Subgroups of beans with pods and peas without pods because a dietary intake assessment could not be completed.

Pulses

The US GAP (1×0.538 kg ai/ha, 7-day PHI) covers all commodities in the Codex dry beans and dry peas subgroups and residue trials on pinto bean (dry) and Australian winter pea (dry) matched this GAP.

Plant metabolism studies previously evaluated by the JMPR covered fruit (apple, orange, grape) and cereals (maize). As plant metabolism studies covering pulses and oilseeds or a third crop from a group different to fruits or cereal grains were not available, the Meeting could not estimate dietary intake of residues in pulses.

Dry beans

In pinto bean (dry), residues were (n=9): <0.01, <0.01, 0.014, 0.016, 0.018, 0.028, 0.033, 0.088 and 0.17 mg/kg in Australian winter pea (dry), residues were (n=5): <0.01, 0.011, 0.013, 0.014 and 0.052 mg/kg.

The median residues in the datasets for are within a 5-fold difference and the Kruskal-Wallis test showed that the residues were from the same population. Therefore, the Meeting decided to estimate subgroup maximum residue levels.

The combined data set is (n=14): <0.01, <0.01, <0.01, 0.011, 0.013, 0.014, 0.014, 0.016, 0.018, 0.028, 0.033, 0.052, 0.088 and 0.17 mg/kg.

The Meeting estimated but did not recommend a maximum residue levels of 0.3 mg/kg for the Subgroup of dry beans (except soya bean) and the Subgroup of dry peas because a dietary intake assessment could not be completed.

Mints

In residue trials on mint matching the US GAP for peppermint and spearmint (1×0.538 kg ai/ha, 7-day PHI), residues in mint (fresh) were (n=5): 0.57, 0.64, 0.93, 1.6 and 5.3 mg/kg with highest analytical value of 5.5 mg/kg.

Plant metabolism studies previously evaluated by the JMPR covered fruit (apple, orange, grape) and cereals (maize). As plant metabolism studies covering leafy commodities or a third crop from a group different to fruits or cereal grains were not available, the Meeting could not evaluate the residue data on mints.

Residues in animal feeds

Pea vines and hays

In the residue trials on peas matching the US GAP (PHI, 7 days), residues in pea vines were (n=5): 0.26, 1.5, 1.7, 1.8 and 3.8 mg/kg with highest analytical value of 4.5 mg/kg and in pea hay, residues were (n=5): 0.78, 5.3, 6.8, 9.5 and 22 mg/kg with highest analytical value of 23 mg/kg

As no recommendations for legume vegetable food commodities were made (because no metabolism study was available for leafy commodities), the Meeting did not estimate animal feed intake from residues in pea vines or hay.

Fate of residues during processing

The Meeting received information on the fate of fenazaquin during processing of on orange, plum, grapes, tomato and mint. In the mint oil processing study, residue data for fresh mint was not reported. Therefore, the Meeting could not estimate a processing factor for mint oil.

Processing factors were calculated for fenazaquin (for maximum residue level estimation and for calculating livestock dietary burdens) and for total residues (for risk assessment).

Where residues concentrated in the processed food commodities, maximum residue levels were estimated using the estimated maximum residue levels for the raw commodities and applying the calculated best estimate processing factors.

Table 25 Estimated maximum residue levels for processed commodities

Commodity	Processing factors		Fenazaquin
	Calculated Processing Factors #	Best Estimate	Maximum Residue Level (mg/kg)
Orange			MRL=0.4
Citrus oil	78.7	78.7	32
Grapes			MRL=0.7
Dried grapes	2.2, 2.2, 2.9	2.2	1.5
Plums			MRL=0.5
Dried prunes	4.8	4.8	2.4

Notes:

The ratio of the residues in the processed item divided by the residue in the Raw Agricultural Commodity.

The Meeting estimated a maximum residue level of 40 mg/kg for citrus oil.

The Meeting estimated a maximum residue level of 1.5 mg/kg for dried grapes.

The Meeting estimated a maximum residue level of 3 mg/kg for dried prunes.

For processed food and feed commodities, STMR-Ps, median-Ps and HR-Ps (where relevant) were calculated using the STMRs or median residues for the raw commodities and applying the calculated best estimate processing factors.

Table 26 Calculated STMR-Ps and HR-Ps for processed food and feed commodities

RAC	Processing factors		Fenazaquin residue (mg/kg)	
	Calculated Processing factors #	Best Estimate	STMR-P	HR-P
Lemon			Median=0.08	
Lemons and lime (subgroup), juice	0.01 (orange)	0.01	0.0008	-
Orange			Median=0.125	
Oranges (subgroup), juice	0.01 (orange)	0.01	0.00125	-
Citrus oil	78.7 (orange)	78.7	9.84	
Citrus pulp, dry	0.18 (orange)		Median=0.15 0.027	
Grapefruit			Median=0.0.07	-
Pummelo and grapefruit (subgroup), juice	0.01 (orange)	0.01	0.0007	-
Mandarins			Median=0.0.08	-
Mandarin (subgroup), juice	0.01 (orange)	0.01	0.0008	-
Grapes			STMR=0.19	HR=0.4
Red wine	<0.02, <0.02, <0.03	<0.02	0.0038	
Juice	0.13, 0.14, 0.36	0.14	0.027	
Dried grapes	2.2, 2.2, 2.9	2.2	0.42	0.88
Plums			STMR=0.145	HR=0.25
Dried prunes	4.8	4.8	0.7	1.2
Tomato			STMR=0.052	
Paste	0.9	0.9	0.047	-
Puree	0.41	0.41	0.021	

Notes:

The ratios of the residue in the processed item divided by the residue in the Raw Agricultural Commodity.

Residues in animal commodities

Farm animal feeding studies

The 2019 JMPR evaluated a 28-day dairy cow feeding study where animals were dosed with 12.5 ppm, 37.5 ppm or 125 ppm fenazaquin by capsule. Milk samples were taken daily, milk and cream samples were taken from the Day-25 collection and analysed for fenazaquin. Liver, muscle, kidney and fat samples were taken about 8 hours after the last dose and analysed for fenazaquin and 2-OH fenazaquin acid.

Residues of fenazaquin in milk plateaued after 3 days and in tissues were highest in fat and lower in liver and kidney and in muscle were <LOQ at the highest dose. Similarly, residues of 2-OH fenazaquin acid were <LOQ in muscle. The highest levels of 2-OH fenazaquin acid were found in liver, with lesser amounts in kidney.

Table 27 Residues in milk and tissues from cattle dosed for 28 days with fenazaquin in the diet

Tissue	Dose level, ppm	Residues, mg eq/kg [a]									
		Fenazaquin		2-OH fenazaquin acid		4-OH quinazoline [b]		Fenazaquin + 2-OH fenazaquinacid		Fenazaquin + 2-OH fenazaquinacid + 4-OH quinazoline	
		max	mean	max	mean	max	mean	max	mean	max	mean
Liver	12.5	Not analysed		0.017	0.014	0.018	0.011	0.027	0.024	0.045	0.039
	37.5	<0.01	<0.01	0.052	0.049	0.039	0.037	0.062	0.059	0.1	0.096
	125	0.059	0.033	0.15	0.13	0.12	0.094	0.21	0.16	0.33	0.25
Kidney	12.5	Not analysed		0.009	0.009	<0.002	<0.002	<0.019	<0.019	<0.021	<0.021
	37.5	<0.01	<0.01	0.027	0.024	0.018	0.018	0.037	0.034	0.055	0.052
	125	0.022	0.013	0.061	0.056	0.018	0.014	0.074	0.071	0.092	0.089
Muscle	12.5	Not analysed		<0.009	<0.009	Assumed 0		<0.019	<0.019	<0.019	<0.019
	37.5	Not analysed		<0.009	<0.009	Assumed 0		<0.019	<0.019	<0.019	<0.019
	125	<0.01	<0.01	<0.009	<0.009	Assumed 0		<0.019	<0.019	<0.019	<0.019
Fat [c]	12.5	0.056	0.045	Not analysed		Assumed 0		0.056	0.045	0.056	0.045
	37.5	0.12	0.11	Not analysed		Assumed 0		0.12	0.11	0.12	0.11
	125	0.42	0.31	Not analysed		Assumed 0		0.42	0.31	0.42	0.31
Milk	37.5	<0.01	<0.01	<0.0025 [d]	<0.0025 [d]	<0.015	<0.015	<0.00125	<0.0125	<0.0275	<0.0275
	125	0.046	0.031	0.0115 [d]	0.00775 [d]	0.069	0.0465	0.0575	<0.0125	0.13	<0.0175

Notes:

[a] For fenazaquin and 2-OH fenazaquin acid reported as not analysed or <LOQ mg/kg, residues were assumed to be 0.01 mg/kg and combined residues are listed as <combined fenazaquin-equivalent LOQs only when all residues were reported as <0.01 mg/kg.

[b] Calculated from 2-OH fenazaquin acid by a factor of 0.75 (liver) or 0.25 (milk) from the goat metabolism study evaluated by the 2017 JMPR. Residues in fat were assumed to be zero.

[c] Perirenal fat (highest concentrations).

[d] For milk, residues calculated from fenazaquin by a factor of 0.25 (for 2-OH fenazaquin acid) or 1.5 (for 4-quinazoline).

Farm animal dietary burden

The 2019 JMPR estimated maximum and mean dietary burdens of 0.133 ppm in cattle (arising from consumption of almond hulls). For the additional uses considered by the current Meeting, citrus pulp, dry, tomato pomace and apple pomace are relevant for farm animal dietary burden calculation (cattle only).

The Meeting considered that residues of 2-hydroxy-fenazaquin acid and 4-hydroxyquinoline detectable in those feeds would have little effect on levels of dietary burdens and estimated dietary burdens based on only fenazaquin residues.

Table 28 Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden: fenazaquin ppm, of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0.002	0.0025	0.04	0.04	0.18❶	0.18❷	-	-
Dairy cattle	0.15	0.15	0.022	0.022	0.17❸	0.17❹	-	-

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues.

❷ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❸ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for mammalian milk.

For dietary risk assessment, the Meeting estimated residues using the approach adopted by the 2019 Extra JMPR, using conversion factors calculated from the metabolism study to estimate unmeasured residues of 2-OH fenazaquin acid (0.25×fenazaquin) and 4-OH quinazoline (1.5×fenazaquin) in milk and unmeasured residues of 4-OH quinazoline in liver (0.75×2-OH-fenazaquin acid) and kidney (0.25×2-OH-fenazaquin acid).

Table 29 Maximum residue level, STMR and HR in mammalian animal commodities

	Feed level (ppm) for milk	Residues (mg eq/kg) in milk	Feed level (ppm) for tissues	Residues (mg eq/kg) in			
				Muscle	Liver	Kidney	Fat
MRL (beef or dairy cattle) based on fenazaquin+2-OH fenazaquin acid							
Feeding study	37.5	<0.0125	12.5	<0.019	0.027	<0.019	0.056
Dietary burden and highest residue	0.17	<0.000057	0.18	<0.00027	0.00039	<0.00027	0.0008
STMR (beef or dairy cattle) based on fenazaquin+2-OH fenazaquin acid+4-OH quinazoline							
Feeding study	37.5	<0.0275	12.5	<0.019	0.039	<0.021	0.045
Dietary burden and highest residue	0.17	<0.00013	0.18	<0.00027	0.00056	<0.0003	0.00065
HR (beef or dairy cattle) based on fenazaquin+2-OH fenazaquin acid+4-OH quinazoline							
Feeding study	37.5	<0.0275	12.5	<0.019	0.045	<0.019	0.056
Dietary burden and highest residue	0.18	<0.00013	0.18	<0.00027	0.00065	<0.0003	0.00081

Based on the anticipated residues, the Meeting estimated maximum residue levels of 0.02 (*) mg/kg for milks; milk fats; meat (from mammals other than marine mammals; as fat); edible offal (mammalian) and mammalian fats (except milk fats).

The Meeting estimated STMRs of 0 mg/kg for milks and meat (from mammals other than marine mammals and 0.00065 mg/kg for mammalian fats (except milk fats).

The Meeting estimated HRs of 0 mg/kg for meat (from mammals other than marine mammals and 0.00081 mg/kg for mammalian fats (except milk fats).

For mammalian edible offal, the Meeting estimated an STMR of 0.00056 mg/kg and a HR of 0.0065 mg/kg (based on residues in liver).

For poultry, since none of the feed items were applicable, the Meeting did not estimate maximum residue levels for eggs or poultry commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL and for dietary risk assessment for plant commodities: *Fenazaquin.*

Definition of the residue for compliance with the MRL for animal commodities: *Sum of fenazaquin and 2-hydroxy-fenazaquin acid, expressed as fenazaquin equivalents.*

Definition of the residue for dietary risk assessment for animal commodities: *Sum of fenazaquin, and 2-hydroxy-fenazaquin acid and tautomeric forms of 4-hydroxyquinazoline, expressed as fenazaquin equivalents.*

The residue is fat-soluble.

Table 30 Residue levels suitable for establishing maximum residue limits and for IEDI and IESTI assessments

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FC 0002	Lemons and Limes (incl. Citron), Subgroup of	0.3		0.01 (Kumquat, 0.08)	0.01 (Kumquat, 0.12)
FCT7001	Lemons and Limes (incl. Citron), Subgroup of, juice			0.0008	0.01
FC 0004	Oranges, Sweet, Sour (incl. Orange-like hybrids), Subgroup of	0.4		0.01	0.01
JF 0004	Oranges, Sweet, Sour (incl. Orange-like hybrids), Subgroup of, juice			0.00125	
FC 0005	Pummelo and Grapefruits (incl. Shaddock-like hybrids, among others Grapefruit), Subgroup of	0.3		0.01	0.01
JF 0203	Pummelo and Grapefruits (incl. Shaddock-like hybrids, among others Grapefruit), Subgroup of, juice			0.0007	
FC 0003	Mandarins (incl. Mandarin-like hybrids), Subgroup of	0.3		0.01	0.01

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FCT7003	Mandarins (incl. Mandarin-like hybrids), Subgroup of, juice			0.0008	
OR 0001	Citrus oil, edible	32		9.84	
AB 0001	Citrus pulp, dry			0.027	
FP 0226	Apples	0.3		0.08	0.18
FS 0014	Plums, Subgroup of	0.5		0.145	0.25
DF 0014	Prune, dried	3		0.7	1.2
FS 2001	Peaches (incl. Nectarine and Apricots), Subgroup of	1.5		0.38	1.2
FB 2005	Cane berries, Subgroup of	0.7		0.18	0.41
FB 2006	Bush berries, Subgroup of	0.8		0.235	0.42
FB 2008	Small fruit vine climbing, Subgroup of	0.7		0.19	0.40
DF 0269	Dried grapes (=Currants, Raisins and Sultanas)	1.5		0.42	0.88
	Grape wine (red)			0.0038	
JF 0269	Grape juice			0.027	
FB 2009	Low growing berries, Subgroup of	2		0.49	1.2
FI 0326	Avocado	0.15		0.01	0.01
VC 0045	Fruiting vegetables, Cucurbits, Group of	0.3		0.060	0.19
VO 2045	Tomatoes, Subgroup of	0.3		0.052	0.19
DM 0448	Tomato paste			0.047	
DM 0448	Tomato puree			0.021	
VO 0051	Peppers, Subgroup of (excl. martynia, okra and roselle)	0.3		0.079	0.22
HS 0444	Peppers Chili, dried	3		0.79	2.2
VO 2046	Eggplants, Subgroup of	0.3		0.079	0.22
MO 0105	Edible offal (Mammalian)	0.02 (*)		0.00056 (liver)	0.0065 (liver)
MF 0100	Mammalian fats (except milk fats)	0.02 (*)		0.00065	0.00081
MM 0095	Meat (from mammals other than marine mammals)	0.02 (*) (fat)		0	0
ML 0106	Milks	0.02 (*) (fat)		0	
FM 0183	Milk fats	0.02 (*) (fat)		0	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for fenazaquin is 0–0.05 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for fenazaquin were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 0–2 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of fenazaquin from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for fenazaquin is 0.1 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for fenazaquin were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs varied from 0–60 percent of the ARfD for children and 0–20 percent of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of fenazaquin from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Author(s)	Year	Report No.	Study Title
Belcher, T.	2010	09-02399	Magnitude of the Residue of Fenazaquin and Fenazaquin Dimer on Avocados
Belcher, T.	2010	GR08-577	Magnitude of the residue of GWN-1708 on grapes
Belcher, T.	2010	GR08-576	Magnitude of the residue of fenazaquin and fenazaquin dimer on citrus: Raw and Processed commodities
Butcher, S.	1994	GHE-P-3798 / DOC. No. 645-008	The Stability of Fenazaquin in fortified Cucurbits stored under frozen conditions
Carringer, S. J.	2010	TCI-08-215	Magnitude and Decline of the Residue of Fenazaquin and Fenazaquin Dimer in or on Stone Fruit Raw Agricultural and Processed Commodities Following One Application of GWN-1708-2008.
Carringer, S. J.	2010	TCI-08-216	Magnitude and decline of the residue of fenazaquin and fenazaquin dimer in or on fruiting vegetable raw agricultural and processed commodities following one application of GWN-1708--2008
Carringer, S. J.	2015	TCI-12-349	Residues of fenazaquin in or on Almonds following one application of GWN-1708 1.67 SC (2012)
Cassidy, P.	2009	024119-1	Analytical Method Report for the Analysis of Fenazaquin and Fenazaquin Dimer in RAC Crops.
Cassidy, P.	2010	023643-1	Stability of fenazaquin and fenazaquin dimer in crops after freezer storage.
Cassidy, P.	2011	023643-2	Stability of fenazaquin and fenazaquin dimer in crops after freezer storage.
Dohn, D.R.	2010	637-001	A Confined Rotational Crop Study With [^{14}C]- Fenazaquin (2 Radiolabels) Using Lettuce, Radish, and Wheat at 30, 120 and 365 Day Plant Back Intervals
Gambie, A.R., Draper, R.	1993	GHE-P-3154 / Doc. No. 645-002	The Stability of Fenazaquin in Fortified Orange Peel and Flesh Under Frozen Conditions.
Gambie, A.R., Butcher, S., Laurie, S.	1994	GHE-P-3404 / Doc. No.: 645-005	The Stability of Fenazaquin in Fortified Grapes Stored Under Frozen Conditions.
Korpalski, S.	2010	GR08-582	Magnitude of Residue of GWN-1708 on Cucurbit Vegetables
Korpalski, S.	2010	09-00522	Magnitude of the residue of GWN-1708 on edible-podded beans and peas
Korpalski, S.	2010	09-00521	Magnitude of the residue of GWN-1708 on succulent shelled beans and peas
Korpalski, S.	2010	09-00523	Magnitude of the residue of GWN-1708 on dry beans and peas
Lakaschus, S., Amann, S.	2012	S11-03100	Fenazaquin, 4-OHQ, TBPE – Validation of an analytical method for the determination of fenazaquin, 4-hydroxyquinazoline (4-OHQ) and p-tert butylphenylethanol (TBPE) in acid and water-rich-matrices
Lakaschus, S., Gizler, A.	2013	S l 1-03099 / Doc. No. 645-020	Freezer Storage Stability of Fenazaquin, 4-Hydroxyquinazoline (4-OHQ) and p-tert-Butylphenylethanol (TBPE)
Oden, K	2012	028973-1	Validation of the Residue Analytical Method for Detection of Fenazaquin in Raw Agricultural Commodities Using a Primary and Secondary Transition Ion. Ricerca Document 028973-1, Ricerca, Inc.

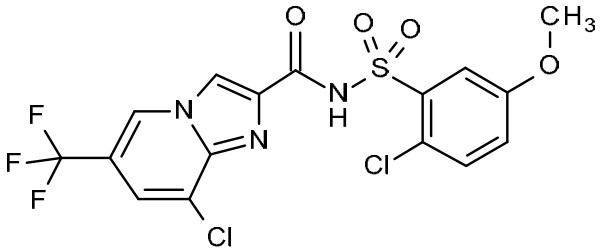
Author(s)	Year	Report No.	Study Title
Perny, A.	2007	Doc. No. 432-024	Validation of the Analytical Method for the Determination of Fenazaquin Residue in Grape Processed Fractions – Wine, Raisins, Grape Juice and Pomace
Riley, M.	2010	GR08-575	Magnitude and decline of the residue of fenazaquin and fenazaquin dimer on pome fruit
Simek, I.	2009	R A7167	Determination of fenazaquin residues in grapes (RAC and processed products) following treatments with a SC formulation containing 200 g/L fenazaquin under field conditions in Europe in 2007
Wyatt, D.	2010	TCI-08-214	Magnitude and Decline of the Residue of Fenazaquin and Fenazaquin Dimer in or on Berry Raw Agricultural Commodities Following One Application of GWN-1708--2008
Wyatt, D.	2010	TCI-08-213	Magnitude and decline of the residue of Fenazaquin and Fenazaquin dimer in or on Strawberry Raw Agricultural Commodities following one Application of GWN-1708--2008
Wyatt, D.	2010	TCI-08-217	Magnitude and Decline of the Residue of Fenazaquin and Fenazaquin Dimer in or on Mint Raw Agricultural Commodities and Processed Commodities Following One Application of GWN-1708--2008

FLUAZAINDOLIZINE (327)

First draft prepared by Dr D.J. MacLachlan, Department of Agriculture, Fisheries and Forestry, Canberra Australia

Fluazaindolizine is a nematicide for the control of plant parasitic nematodes. At the Fifty-first Session of CCPR it was scheduled for the evaluation as a new compound in 2021 and rescheduled to the 2022 JMPR. Fluazaindolizine is to be used for annual crops (e.g., fruiting vegetables, cucurbits, root vegetables, row crops) and certain perennial crops (e.g., citrus, tree nuts, stone fruits). Application methods include drip, drench, in furrow spray with or without soil incorporation either before or at planting, with the option for follow-up in crop treatment.

IDENTITY

Common name:	Fluazaindolizine
IUPAC name:	8-Chloro-N-[(2-chloro-5-methoxyphenyl)sulfonyl]-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide
CAS name:	8-Chloro-N-[(2-chloro-5-methoxyphenyl)sulfonyl]-6-(trifluoromethyl)-imidazo[1,2-a]pyridine-2-carboxamide
CAS number:	1254304-22-7
Synonyms:	DPX-Q8U80
Structural formula:	
Molecular formula:	$C_{16}H_{10}Cl_2F_3N_3O_4S$
Molecular weight:	468.24 g/mol

Physical and chemical properties

Property	Results	Reference
Physical state	Solid	Reddy 2013 DuPont-36641, Pushpalatha 2017 DuPont-49059
Colour	PAI: off-white. TGAI: manufacturing batches from different sources often varied in colour (light yellowish grey, reddish brown, beige, brown) even though purity was still quite high.	Reddy 2013 DuPont-36641, Pushpalatha 2017 DuPont-49059
Odour	Odourless at room temperature (PAI); odourless at 20 ± 0.5 °C (TGAI)	Reddy 2013 DuPont-36641 Pushpalatha 2017 DuPont-49059
Melting point:	218.5±0.0 °C (PAI)	Kumar 2013a DuPont-36643, Revision No. 2
Boiling point:	The test material is a solid which decomposes before reaching boiling. Test compound decomposition started at 260 °C and it was completely decomposed by the time temperature reached at 310 °C.	Kumar 2013b DuPont-36642, Revision No. 1
Relative density	1.6818±0.1080 at an average recorded temperature of 20±4 °C(PAI).	Reddy 2013 DuPont-36638
Bulk density	0.619±0.001 g/mL (TGAI). The tap density was found to be 0.795±0.001 g/mL.	Anand 2016 DuPont-46269

Property	Results	Reference
pH of a 1 % aqueous suspension	4.44 ± 0.01 at 20 ± 0.5 °C (PAI). 4.17-4.71 (batches of TGAI)	Reddy 2013 DuPont-36637 Pushpalatha 2017 DuPont-49059
vapour pressure	PAI (Gas Saturation method): 2.04×10^{-7} Pa at 20 °C 4.12×10^{-7} Pa at 35 °C 6.02×10^{-7} Pa at 50 °C (extrapolation): 2.12×10^{-7} Pa at 20 °C 2.57×10^{-7} Pa at 25 °C	Anand 2014 DuPont-36640
Vapour pressure of the metabolites:	IN-F4106: 4.45×10^{-5} Pa at 20 °C and 4.95×10^{-5} Pa at 25 °C. IN-QEK31: 2.88×10^{-8} Pa at 20 °C and 3.69×10^{-8} Pa at 25 °C IN-VM862: 1.319 Pa	Pushpalatha 2016a DuPont-42578 Pushpalatha 2016b DuPont-40413 Pushpalatha 2017 DuPont-48540
Henry's Law constant at 20 °C (calculated):	pH 4: 4.5 Pa/m^3/mol pH 7: 4.6 Pa/m^3/mol pH 9: 0.35 Pa/m^3/mol	Anand 2014 DuPont-36640
Octanol-water partition coefficients	pH 4, log K_{ow} = 2.24±0.07; pH 7, log K_{ow} = -0.16±0.01; pH 9, log K_{ow} = -0.71±0.03; in distilled water log K_{ow} = 0.81±0.06	Pushpalatha 2013 DuPont-35462
Octanol-water partition coefficients metabolites	IN-A5760 mean log K_{ow} 0.22 IN-F4106 mean log K_{ow} 0.73 IN-VM862 mean log K_{ow} 1.77 IN-QEK31 pH 4, log K_{ow} 0.58±0.03 pH 7, log K_{ow} -1.29±0.01 pH 9, log K_{ow} 1.52±0.00 IN-REG72 pH 4, log K_{ow} 1.84±0.09 pH 7, log K_{ow} 0.03±0.01 pH 9, log K_{ow} -0.49±0.02	Anand 2016 DuPont-39230
Solubility in water	PAI (measured at 20 °C): 0.0561±0.0036 g/L in distilled water; 0.0221±0.0013 g/L at pH 4; 2.1479±0.0310 g/L at pH 7, 2.8455±0.0888 g/L at pH 9	Pushpalatha 2013 DuPont-35461
Solubility in water, metabolites	(measured at 20°C) IN-QEK31 0.16±0.01 g/L distilled water 0.25±0.02 g/L pH 4 1.45±0.02 g/L pH 9 IN-F4106 0.83±0.04 g/L distilled water 0.81±0.01 g/L pH 4 1.06±0.05 g/L pH 9 IN-REG72 0.057±0.001 g/L distilled water 0.19±0.001 g/L pH 4 2.9±0.14 g/L pH 9 IN-A5760 9.72±0.17 g/L distilled water 8.84±0.34 g/L pH 4 10.27±0.36 g/L pH 9 IN-VM862 0.33±0.00 g/L distilled water 0.38±0.01 g/L pH 4 0.45±0.01 g/L pH 9	Pushpalatha 2015a DuPont-38078 Pushpalatha 2015b DuPont-38079 Pushpalatha 2015c DuPont-38581 Pushpalatha 2015d DuPont-40417 Pushpalatha 2016 DuPont-39514
Solubility in organic solvents	PAI (measured at 20 °C) Acetonitrile 35.05±3.31 g/L Methanol 3.47±0.196 g/L Acetone 99.76±3.73 g/L Ethyl acetate 27.62±1.08 g/L 1,2-dichloroethane 19.29±3.53 g/L o-xileno 1.247±0.041 g/L	Moorthy 2012 DuPont-35460

Property	Results	Reference
	n-octanol 2.00±0.19 g/L n-hexane 0.002±0.001 g/L	
Hydrolysis	Both imidazo[1,2-a]pyridine-5,8a-^{14}C]fluazaindolizine and [Phenyl-^{14}C(U)]fluazaindolizine are: Stable at pH 4 (<10 percent degradation at 50°C for 30 days) Stable at pH 7 (No degradation at 50°C for 30 days) Stable at pH 9 (No degradation at 50°C for 30 days)	Anand 2013 DuPont-35131
Hydrolysis of metabolites	The hydrolysis of IN-QEK31 at 50±0.5°C after 5 days of incubation was <3 percent in pH 4, 7 and 9 buffer solutions. IN-QEK31 is considered to be stable ($t_{1/2}$ at 25°C >1 year) at pH 4, 7 and 9. The hydrolysis of IN-F4106 at 50±0.5°C after 5 days of incubation was <6 percent in pH 4, 7 and 9 buffer solutions. IN-F4106 is considered to be stable ($t_{1/2}$ at 25°C>1 year) at pH 4, 7 and 9.	Yogeesha 2015 DuPont-40399 Manikandan 2015 DuPont-42585
Photolysis	Photolytic half-life of fluazaindolizine (radiolabelled at two different sites) in sterile aqueous buffer solution (pH 4 and 9) at 20 °C were 1.1 days and 1.2 days, respectively in a continuous irradiated system. Conversion to 12-hour sunlight days results in half-life of 2.3 days. A large number of degradation products were formed, and more than a dozen were identified. No single product exceeded >10 percent of the applied amounts.	Bell & Jewkes 2015 DuPont-37450
Dissociation constant	pK_a = 5.6 ± 0.07 at 20 °C	Anand 2013 DuPont-35459
Dissociation constant (metabolites)	IN-F4106 pK_a = 9.46± 0.03 at 20 °C IN-QEK31 pK_a = 4.55 ± 0.01 at 20 °C	Pushpalatha 2016a DuPont-40416 Pushpalatha 2016b DuPont-42582
photochemical oxidative degradation	The estimated rate constant for addition of hydroxyl radicals (OH) to the pyrimidine and phenyl aromatic rings of fluazaindolizine in the gas phase is 3.9×10^{-12} cm^3 molecule^{-1} sec^{-1}. The estimated half-life in 12-hour daylight is ~32.6 hours.	Sharma 2018 DuPont-36631
Surface tension	67 ± 0.1 dynes/cm at an average recorded temperature of 20.5 °C	Manikandan 2013 DuPont-36639
Molar extinction coefficient	Neutral: ε =6839 L mol^{-1} cm^{-1} (log ε = 3.83) at 298nm Acidic: ε = 50155 L mol^{-1} cm^{-1} (log ε = 4.70) at 235nm Basic: ε = 50207 L mol^{-1} cm^{-1} (log ε = 4.70) at 235 nm	Shubha 2015 DuPont-37833

Notes:

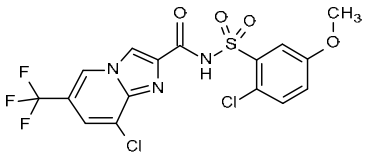
PAI: pure active ingredient

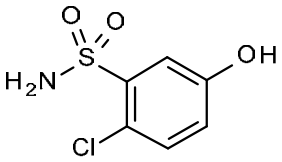
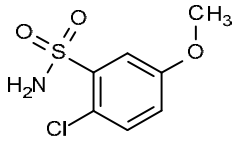
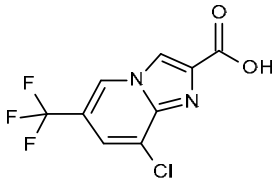
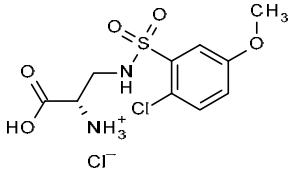
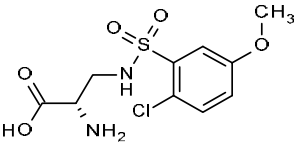
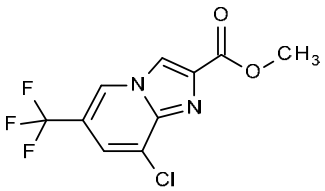
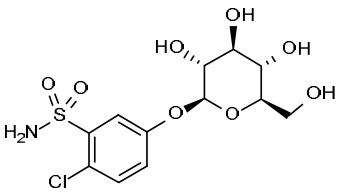
TGAI: technical grade active ingredient

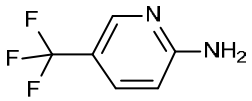
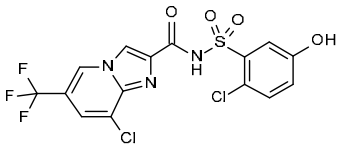
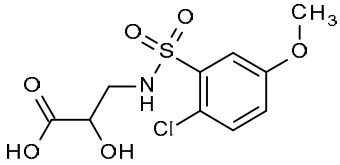
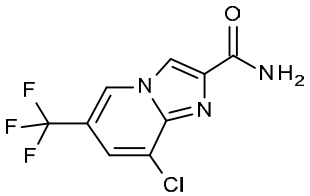
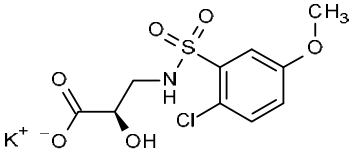
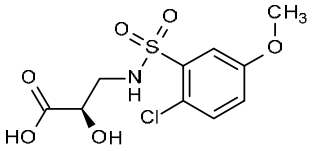
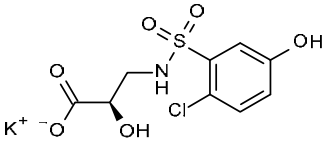
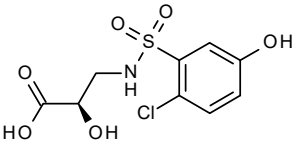
METABOLISM AND ENVIRONMENTAL FATE

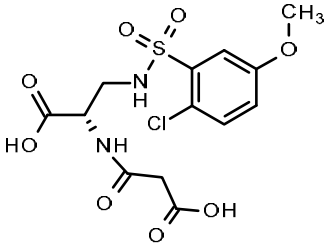
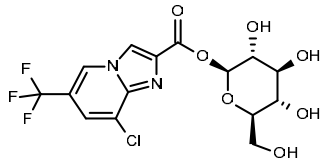
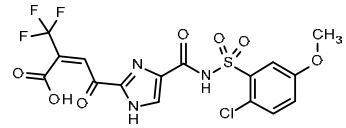
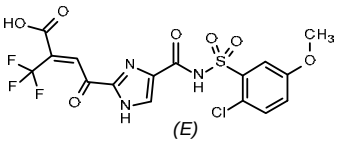
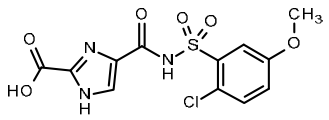
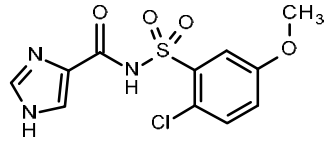
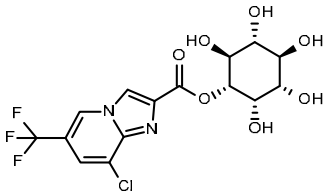
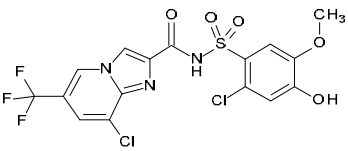
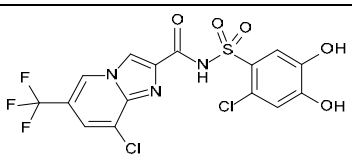
The metabolite summary below provides a reference for the numbering scheme used in the current evaluation.

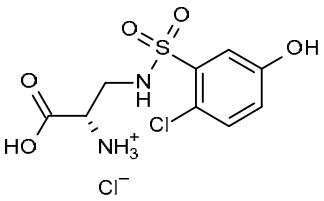
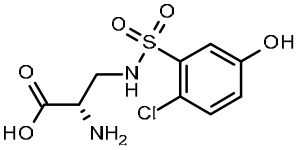
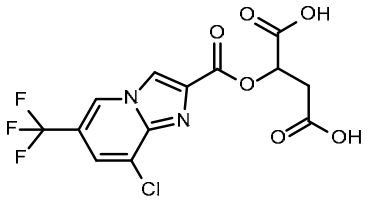
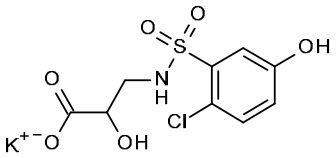
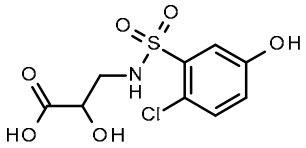
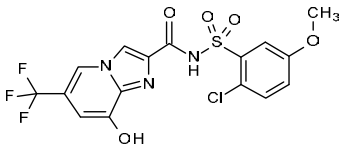
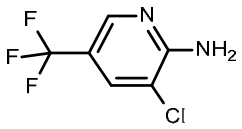
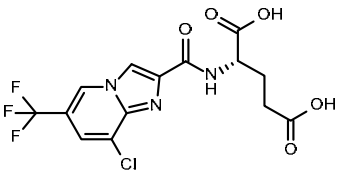
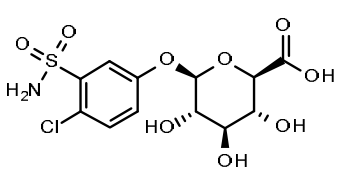
Degradation compounds from metabolism of fluazaindolizine in plants, animals, and the environment

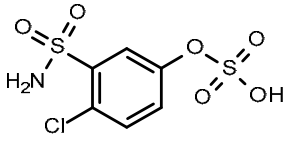
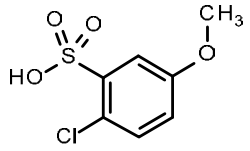
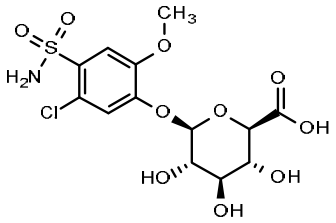
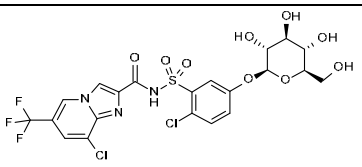
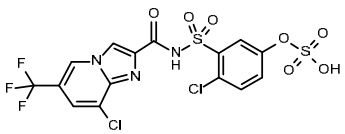
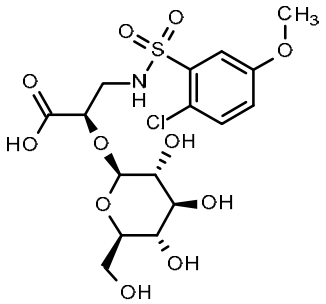
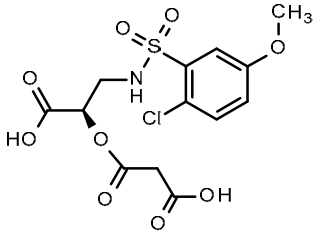
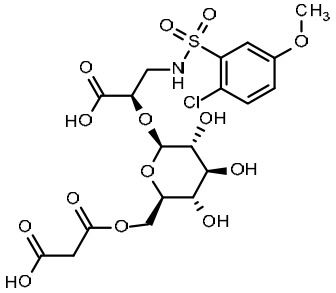
Code Names, MW	Chemical name, CAS number	Chemical structure	Where found
Fluazaindolizine $C_{16}H_{10}Cl_2F_3N_3O_4S$ MW: 468.23 DPX-Q8U80	8-Chloro-N-[(2-chloro-5-methoxyphenyl)sulfonyl]-6-(trifluoromethyl)-imidazo[1,2-a]pyridine-2-carboxamide CAS number: 1254304-22-7		Parent, active substance

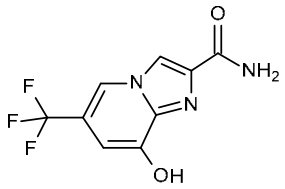
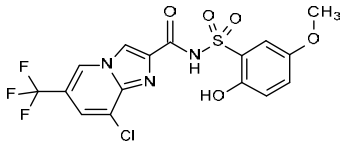
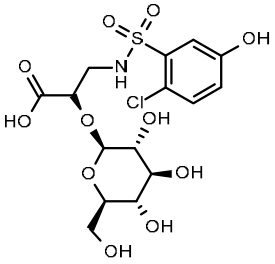
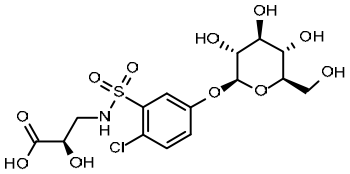
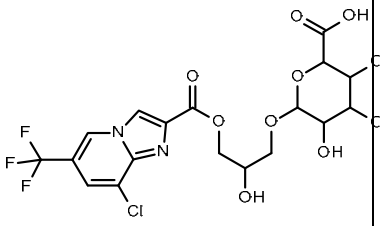
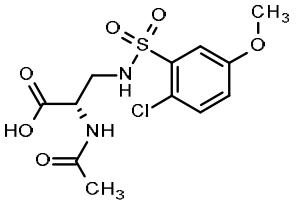
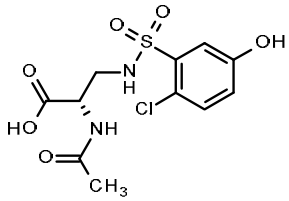
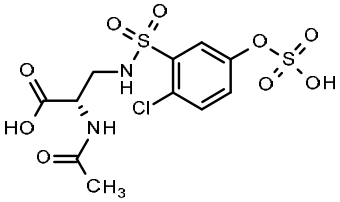
Code Names, MW	Chemical name, CAS number	Chemical structure	Where found
IN-A5760 $C_6H_6ClNO_3S$ MW: 207.64	2-chloro-5-hydroxybenzenesulfonamide CAS number: 86093-06-3		Soil (aerobic, anaerobic) Water/sediment systems Crops (tomato, soya bean, potato, sugarcane, carrot [as conjugate IN-R3Z85]) Livestock (goat) Rat, mouse Livestock (goat from IN-QZY47)
IN-F4106 $C_7H_8ClNO_3S$ MW: 221.66	2-chloro-5-methoxybenzenesulfonamide CAS number: 502187-53-3		Soil (aerobic, anaerobic, photolysis) Water/sediment systems Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach) Livestock (goat, hen) Rat, mouse Livestock (goat from IN-QZY47, IN-TMQ01)
IN-QEK31 $C_9H_4ClF_3N_2O_2$ MW: 264.59	8-Chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylic acid CAS number: 353258-35-2		Soil (aerobic, anaerobic, aqueous photolysis) Water/sediment systems Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach) Livestock (goat, hen) Rat, mouse
IN-QZY47 (*S*-enantiomer) IN-F4106 serine conjugate (HCl salt) $C_{10}H_{14}Cl_2N_2O_5S$ MW: 345.2 (free base) $C_{10}H_{13}ClN_2O_5S$ MW: 308.74	(HCl salt) 3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-L-alanine hydrochloride CAS number: 1928754-04-4 (free base) 3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-L-alanine CAS number: 1928754-03-3	 Hydrochloride salt  Free base	Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach)
IN-R2W56 IN-QEK31 methyl ester $C_{10}H_6ClF_3N_2O_2$ MW: 278.62	8-Chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylic acid methyl ester CAS number: 1430228-25-3		Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach) Livestock (goat, hen)
IN-R3Z85 IN-A5760 glucose conjugate $C_{12}H_{16}ClNO_8S$ MW: 369.78	2-chloro-5-(β-D-glucopyranosyloxy)benzenesu-fonamide CAS number: 1928754-13-5		Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach)

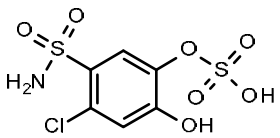
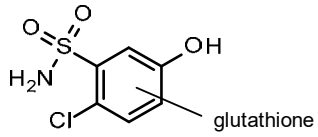
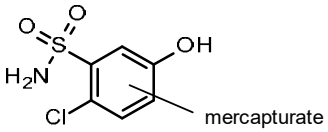
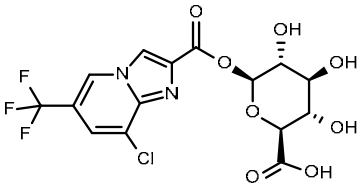
Code Names, MW	Chemical name, CAS number	Chemical structure	Where found
IN-R6P21 $C_6H_5F_3N_2$ MW: 162.11	5-(trifluoromethyl)pyridin-2-amine CAS number: 74784-70-6		Aqueous photolysis
IN-REG72 $C_{15}H_8Cl_2F_3N_3O_4S$ MW: 454.21	8-Chloro-N-[(2-chloro-5-hydroxyphenyl)sulfonyl]-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide CAS number: 1254306-61-0		Soil (aerobic, anaerobic, photolysis) Water/sediment systems Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach) Livestock (goat, hen) Rat, mouse
IN-RSU03 (racemate) $C_{10}H_{12}ClNO_6S$ MW: 309.72	3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-2-hydroxypropanoic acid CAS number: 1928754-00-0		Racemic analytical standard (demonstrated to be *R* enantiomer IN TMQ01 in crops) Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach)
IN-RYC33 $C_9H_5ClF_3N_3O$ MW: 263.60	8-Chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide CAS number: 1228376-01-9		Soil (rotational crop soil), aqueous photolysis Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach) Livestock (hen) Rat
IN-TMQ01 (*R*-enantiomer of IN-RSU03) *R*-enantiomer of IN-RSU03 (potassium salt) $C_{10}H_{11}ClNO_6SK$ MW: 347.81 (free acid) $C_{10}H_{13}ClN_2O_5S$ MW: 309.72	(potassium salt): 3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-(2*R*)-hydroxypropanoic acid, potassium salt CAS number: 1928754-02-2 (free acid): 3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-(2*R*)-hydroxypropanoic acid CAS number: 1928754-02-1	 Potassium salt  Free acid	Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach)
IN-TQD54 (*R*-enantiomer of IN-UNS90) (potassium salt) $C_9H_9ClNO_6SK$ MW: 333.79 (free acid) $C_9H_{10}ClNO_6S$ MW: 295.70	(potassium salt): 3-[[(2-chloro-5-hydroxyphenyl)sulfonyl]amino]-(2*R*)-hydroxypropanoic acid potassium salt CAS number: 1928754-12-4 (free acid): 3-[[(2-chloro-5-hydroxyphenyl)sulfonyl]amino]-(2*R*)-hydroxypropanoic acid CAS number: 1928754-11-3	 Potassium salt  Free acid	Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach) Livestock (goat from IN-QZY47)

Code Names, MW	Chemical name, CAS number	Chemical structure	Where found
IN-TUT81 IN-QZY47 malonyl conjugate $C_{13}H_{15}ClN_2O_8S$ MW: 394.79	*N*-(carboxyacetyl)-3-[[(2-chloro-5-methoxyphenyl)sulfonyl]-amino]-L-alanine CAS number: 1928754-18-0		Crops (soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach)
IN-UGA20 IN-QEK31 glucose conjugate $C_{15}H_{14}ClF_3N_2O_7$ MW: 426.7	β-D-glucopyranose 1-[8-chloro 6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylate] CAS number: 1928754-17-9		Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach) More complex carbohydrate conjugates were also formed in most crops.
IN-UGA22 $C_{16}H_{11}ClF_3N_3O_7S$ MW: 481.8	4-[4-[[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]carbonyl]-1H-imidazol-2-yl]-4-oxo-2-(trifluoromethyl)-(2E/Z)-butenoic acid	 *(Z)*  *(E)*	Aqueous photolysis products, postulated E/Z geometric isomers
IN-UGA26 $C_{12}H_{10}ClN_3O_6S$ MW: 359.7	4-[[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]carbonyl]-1H-imidazole-2-carboxylic acid		Aqueous photolysis
IN-UHC58 $C_{11}H_{10}ClN_3O_4S$ MW: 315.7	N-[(2-chloro-5-methoxyphenyl)sulfonyl]-1H-imidazole-4-carboxamide		Aqueous photolysis
IN-UHD13 IN-QEK31 inositol conjugate $C_{15}H_{14}ClF_3N_2O_7$ MW: 426.7	[(2*S*,3*R*,5*S*,6*S*)-2,3,4,5,6-pentahydroxycyclohexyl]1-[8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylate] CAS number: 1928754-19-1		Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach)
IN-UHD20 Hydroxy-DPX-Q8U80; Q8U80-OH $C_{16}H_{10}Cl_2F_3N_3O_5S$ MW: 484.2404	8-Chloro-N-[(2-chloro-4-hydroxy-5-methoxyphenyl)sulfonyl]-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide CAS number: 1928754-15-7		Rat, mouse
IN-UHD21 Hydroxy-IN-REG72 REG72-OH $C_{15}H_8Cl_2F_3N_3O_5S$ MW: 470.2133	8-Chloro-N-[(2-chloro-4,5-dihydroxyphenyl)sulfonyl]-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide CAS number: 1928754-16-8		Rat, mouse

Code Names, MW	Chemical name, CAS number	Chemical structure	Where found
IN-UJV12 (*S*-enantiomer) (HCl salt) $C_9H_{12}Cl_2N_2O_5S$ MW: 331.17 (free base) $C_{13}H_{15}ClN_2O_8S$ MW: 294.71	(hydrochloride salt): 3-[[(2-Chloro-5-hydroxyphenyl)sulfonyl]amino]-L-alanine hydrochloride CAS number: 1928754-08-8 (free base): 3-[[(2-Chloro-5-hydroxyphenyl)sulfonyl]amino]-L-alanine	 Hydrochloride salt  Free base	Crops (soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach)
IN-UJU44 QEK31 malic acid conjugate $C_{13}H_8ClF_3N_2O_6$ MW: 380.66	2-[[[8-Chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridin-2-yl]carbonyl]oxy]butanedioic acid CAS number: 1928754-14-6		Rotational crops (wheat, radish)
IN-UNS90 (racemate) (potassium salt) $C_9H_9ClNO_6S.K$ MW: 333.79 (free acid) $C_9H_{10}ClNO_6S$ MW: 295.70	(potassium salt): 3-[[(2-Chloro-5-hydroxyphenyl)sulfonyl]amino]-2-hydroxypropanoic acid potassium salt CAS number: 1928754-10-2 (free acid): 3-[[(2-Chloro-5-hydroxyphenyl)sulfonyl]amino]-2-hydroxypropanoic acid CAS number: 1928754-09-9	 Potassium salt  Free acid	Racemic analytical standard (found to be *R*-enantiomer IN-TQD54) Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach)
IN-URA40 $C_{16}H_{11}ClF_3N_3O_5S$ MW: 449.8	N-[(2-Chloro-5-methoxyphenyl)sulfonyl]-8-hydroxy-6-(trifluoromethyl)-imidazo[1,2-a]pyridine-2-carboxamide		Aqueous photolysis
IN-VM862 $C_6H_4ClF_3N_2$ MW: 196.56	3-Chloro-5-(trifluoromethyl)pyridin-2-amine CAS number: 79456-26-1		Soil (aqueous photolysis) Crops (carrot) Rotational crops (wheat)
IN-WUK12 Glutamic acid conjugate of IN-QEK31 $C_{14}H_{11}ClF_3N_3O_5$ MW: 393.7	N-[[8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-yl]carbonyl]-L-glutamic acid		Rotational crops (radish)
No code IN-A5760 glucuronide conjugate $C_{12}H_{14}ClNO_9S$ MW: 383.8	2-Chloro-5-(β-L-glucopyranuronosyloxy)benzenesulfonamide		Rat Livestock (goat from IN-QZY47), (Rat from IN-F4106)

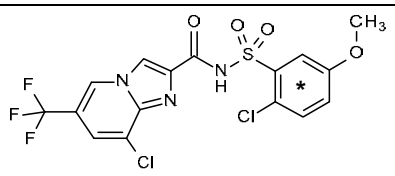
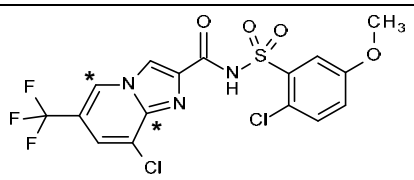
Code Names, MW	Chemical name, CAS number	Chemical structure	Where found
No code IN-A5760 sulfate conjugate $C_6H_6ClNO_6S_2$ MW: 287.7	2-Chloro-5-(sulfooxy)benzenesulfonamide		Livestock (hen) Rat, mouse Livestock (goat from IN-QZY47) (Rat from IN-F4106)
No code 2-Chloro-5-methoxy-benzenesulfonic acid $C_7H_8ClO_4S$ MW: 222.65	2-Chloro-5-methoxybenzenesulfonic acid		Aqueous photolysis (tentative ID)
No code Hydroxy IN-F4106 glucuronide $C_{13}H_{16}ClNO_{10}S$ MW: 413.8	2-Chloro-4-(β-L-glucopyranuronosyloxy)-5-methoxy-benzenesulfonamide		Rat
No code IN-REG72 glucose conjugate $C_{21}H_{18}Cl_2F_3N_3O_9S$ MW: 616.4	8-Chloro-N-[(2-chloro-5-(β-D-glucopyranosyl) phenyl) sulfonyl]-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide		Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (radish, wheat, spinach)
No code IN-REG72 sulfate conjugate $C_{15}H_8Cl_2F_3N_3O_7S_2$ MW: 534.3	8-Chloro-N-[[2-chloro-5-(sulfooxy)phenyl]sulfonyl]-6-(trifluoromethyl) imidazo[1,2-a]pyridine-2-carboxamide		Rat
No code IN-RSU03 glucose conjugate IN-TMQ01 glucose conjugate $C_{16}H_{22}ClNO_{11}S$ MW: 471.9	3-[[(2-Chloro-5-methoxyphenyl)sulfonyl]amino]-(2*R*)-(β-D-glucopyranosyloxy)propanoic acid		Crops (tomato, carrot, potato, sugarcane) Rotational crops (radish, wheat, spinach) (expected to be as *R*-enantiomer IN-TMQ01)
No code IN-RSU03 malonyl conjugate (IN-TMQ01 malonyl conjugate) $C_{13}H_{14}ClNO_9S$ MW: 395.8	mono[1-carboxy-(2*R*)-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]ethyl]-propanedioic acid ester		Crops (carrot, potato [foliage]) (expected to be as *R*-enantiomer IN-TMQ01)
No code IN-RSU03 glucose malonyl conjugate (IN-TMQ01 glucose malonyl conjugate) $C_{19}H_{24}ClNO_{14}S$ MW: 557.9	(2*R*)-[[6-O-(2-carboxyacetyl)-β-D-glucopyranosyl]oxy]-3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]- propanoic acid		Crop rotation (wheat) (expected to be as *R*-enantiomer IN-TMQ01)

Code Names, MW	Chemical name, CAS number	Chemical structure	Where found
No code: Hydroxylated-IN-RYC33 Hydroxy-QEK31 $C_9H_6F_3N_3O_2$ MW: 245.16	8-Hydroxy-6-(trifluoromethyl)-imidazo[1,2-a]pyridine-2-carboxamide		Aqueous photolysis (tentative ID)
No code PH-hydroxy-DPX-Q8U80 $C_{16}H_{11}ClF_3N_3O_5S$ MW: 449.8	8-Chloro-N-[(2-hydroxy-5-methoxyphenyl)sulfonyl]-6-(trifluoromethyl)-imidazo[1,2-a]pyridine-2-carboxamide		Aqueous photolysis (tentative ID)
No code IN-UNS90 glucose conjugate (IN-TDQ54 glucose conjugate) $C_{15}H_{20}ClNO_{11}S$ MW: 457.8	3-[[(2-Chloro-5-hydroxyphenyl)sulfonyl]amino]-(*2R*)-(β-D-glucopyranosyloxy)-propanoic acid		Crops (tomato, carrot, potato, sugarcane) Rotational crops (radish, wheat, spinach) (expected to be as *R*-enantiomer IN-TQD54)
No code IN-UNS90 phenolic glucose conjugate (IN-TDQ54 phenolic glucose conjugate) $C_{15}H_{20}ClNO_{11}S$ MW: 457.8	3-[[[2-Chloro-5-(β-D-glucopyranosyloxy)phenyl]sulfonyl]amino]-(*2R*)-hydroxypropanoic acid		Crops (tomato, carrot, soya bean, potato, sugarcane) Rotational crops (wheat) (expected to be as *R*-enantiomer IN-TQD54)
No code IN-QEK31 glycerol glucuronide $C_{18}H_{18}ClF_3N_2O_{10}$ MW: 514.8	Imidazo[1,2-a]pyridine-2-carboxylic acid, 8-chloro-6-(trifluoromethyl)-, 3-(hexopyranuronosyloxy)-2-hydroxypropyl ester		Crops (soya bean, carrot) Rotational crops (spinach)
No code Acetylated IN-QZY47 QZY47 acetyl conjugate $C_{12}H_{15}ClN_2O_6S$ MW: 350.80	*N*-Acetyl-3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-L-alanine		Rotational crops (spinach, radish) (Rat from IN-QZY47)
No code Acetylated IN-UJV12 $C_{11}H_{13}ClN_2O_6S$ MW: 336.80	*N*-Acetyl-3-[[(2-chloro-5-hydroxyphenyl)sulfonyl]amino]-L-alanine		(Rat from IN-QZY47)
No code IN-UJV12 acyl sulfate $C_{11}H_{13}ClN_2O_9S_2$ MW: 416.8	N-acetyl-3-[[[2-chloro-5-(sulfooxy)phenyl]sulfonyl]-amino]- L-alanine		(Rat from IN-QZY47)

Code Names, MW	Chemical name, CAS number	Chemical structure	Where found
No code HO-A5760-sulfate $C_6H_6ClNO_7S_2$ MW: 303.7	2-Chloro-4-hydroxy-5-(sulfooxy)-benzenesulfonamide		(Rat from IN-QZY47)
No code IN-A5760 glutathione conjugate $C_{16}H_{21}ClN_4O_9S_2$ MW: 512.9			Livestock (goat from IN-QZY47)
No code IN-A5760 mercapturate conjugate $C_{11}H_{13}ClN_2O_6S_2$ MW: 368.8			(Rat from IN-QZY47)
No code: IN-QEK31 glucuronic acid conjugate $C_{15}H_{12}ClF_3N_2O_8$ MW: 440.7	1-O-[[8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridin-2-yl]carbonyl]-β-D-glucopyranuronic acid		(Rat from IN-QEK31)

Plant metabolism

Fluazaindolizine is a nematicide that is typically incorporated into the soil at planting, with additional applications to the soil shortly thereafter. Primary plant metabolism studies were conducted with [^{14}C]-fluazaindolizine in tomato, carrot, potato, soya bean, and sugar cane and provided examples of metabolism in several crop groups including fruit/fruiting vegetables, root/tuber, oil seed and grass crops.

	
[phenyl-^{14}C(U)]fluazaindolizine or [Ph-^{14}C]-fluazaindolizine)	[imidazo[1,2-a]pyridine-5,8a-^{14}C]fluazaindolizine or [IP-5,8a-^{14}C]-fluazaindolizine)

The protocol for analysis of samples was similar across the plant metabolism studies using phenyl and/or imidazo ^{14}C-labelled fluazaindolizine.Samples were homogenised (milled) with dry ice, and total radioactive residues (TRR) determined. Portions of milled samples were extracted with methanol:water (7:3) and the extracts concentrated under a stream of nitrogen or using a rotary evaporator. For carrot and potato extracts, volatile metabolites evaporated during the concentration step were trapped by passing sequentially through an acetone/dry ice cold trap and a methanol:water (7:3) trap. The unextracted radioactivity in the post-extracted solids (PES) was determined by combustion analysis. Extracts containing ≥0.010 mg eq/kg) were analysed by liquid chromatography.

Endogenous materials in the concentrated methanol extracts were removed from the samples by solid phase extraction (SPE). The concentrated extracts were loaded onto a Varian Bond Elut C18 extraction cartridge preconditioned with acetonitrile followed by 10 mM ammonium acetate. Sample was then eluted with acetonitrile:10 mM ammonium acetate (7:3). The elutes were then concentrated to 100 percent aqueous prior to loading onto Waters Oasis HLB cartridges, washed with 10 mM ammonium acetate, before being eluted with acetonitrile:10 mM ammonium acetate (9:1) followed by 100 percent acetonitrile.

Conjugates in selected extracts were hydrolysed using 1M HCl at 80°C overnight to determine the core fluazaindolizne-related structure of various conjugates (exocon). Selected extracts from tomato fruit from plants grown in soil treated with [IP-5,8a-^{14}C]fluazaindolizine were also incubated in a β-glucosidase solution in pH 5 acetate buffer for *ca* 48 hrs at *ca.* 37 ºC.

PES from selected soya bean and carrot samples were dried under ambient conditions, rehydrated and with water prior to extraction with acetonitrile:water (1:1) at 40 °C/ultrasonication, followed by sequential treatment with Driselase (cell degrading enzyme mix containing cellulase, laminarinase, xylanase) in acetate buffer (37 °C, pH 4.6, 24 hours), 0.1 M HCl (50–60 °C, 6 hours), 1.3 M HCl (80 °C, 20 hours) and 0.1 M NaOH (80 °C, 4 hours). PES from selected sugarcane samples were further extracted with acetonitrile:water (1:1), water, Driselase and 0.1 M HCl.

Identification of the principal ^{14}C-residues was accomplished by HPLC and LC-MS with reference to authenticated putative metabolite standards where available. Fluazaindolizine and IN-F4106, IN-UGA20, IN-TUT81, IN-UNS90 (IN-TQD54), IN-R3Z85, IN-RUS03, IN-REG72, IN-QEK31, IN-RYC33, IN-R2W56, IN-UDH13, IN-A5760 and IN-QZY47 were confirmed by comparison of their retention times and where concentrations permitted, accurate mass determination, chlorine isotopic pattern and mass spectral fragments (MS/MS) to authentic synthesised standards. Chiral analysis was carried out on a number of metabolites (IN-RSU03, IN-QZY47, IN-UNS90).

Tomato

Hobbs et al. (2017; DuPont-34946) investigated the metabolic fate of [Ph-^{14}C(U)]-fluazaindolizine and [IP-5,8a-^{14}C]-fluazaindolizine in glasshouse grown tomatoes (cv Red Alert). Tomato plants were transplanted into sandy loam soil (pH 6.0, percent OM 5.1, 67 percent sand, 17 percent silt, 16 percent clay, CEC 15 meq/100g) approximately 2 hours after a soil drench application with an SC formulation of [Ph-^{14}C]- or [IP-5,8a-14C]fluazaindolizine, at a nominal application rate of 1.5 kg ai/ha. A further application at a nominal application rate of 0.5 kg ai/ha was made 30 days after the initial application. The total achieved treatment rates were 1.93 kg ai/ha and 1.92 kg ai/ha for the [Ph-^{14}C] and [IP-5,8a-^{14}C] labels, respectively.

Red Alert is an indeterminate tomato variety, therefore, red ripe fruit were available for sampling at various times during the growing period between 71 days (41 days after application 2 41DAA2, BBCH 82) and 92 days (62DAA2, BBCH 89) after transplanting of the seedlings. Foliage was taken for analysis 60 days after transplant (30DAA2). Fruit and foliage were taken for analysis 71, 80 and 92 days after transplanting and corresponded to an early ripe sample (41DAA2), a medium ripe sample (50DAA2) and a full ripe sample (62DAA2). Samples were kept under frozen storage (*ca.* -20 °C) until analysis. Plant samples were generally extracted within 42 days (fruit) or 195 days (foliage) of harvest and the extracts were stored for no more than 38 days before initial chromatography. A selected sample from the [Ph-^{14}C]-fluazaindolizine experiment (62DAA2 fruit and foliage), as well as a selected sample from the [IP-5,8a-^{14}C]-fluazaindolizine experiment (62DAA2 fruit and foliage) were re-extracted and profiled to demonstrate stability of the residue in the stored samples. The profile remained the same for at least 53 months for fruit and 39 months for foliage for both radiolabels.

TRR in tomato fruit from the [Ph-^{14}C]-fluazaindolizine experiment fluctuated from 0.070 mg eq/kg at 41DAA2 to 0.079 and 0.066 mg eq/kg at the 50DAA2 and 62DAA2 sampling intervals, respectively while TRR in foliage increased from 2.344 mg eq/kg at the 30DAA2 to 5.743 mg eq/kg at 50DAA2 sampling before declining to 1.857 mg eq/kg at 62DAA2 (Table 1).

TRR in tomato fruit from the [IP-5,8a-^{14}C]-fluazaindolizine experiment increased from 0.029 mg eq/kg at 41DAA2 and 50DAA2 sampling intervals to 0.037 at 62DAA2, while the foliage fluctuated from 0.596, 0.577, 0.919 and 0.438 mg eq/kg respectively at the same intervals (Table 1).

The extractability of ^{14}C with methanol:water was good (80–98 percent TRR).

Table 1 TRR (mg eq/kg) in tomato plant matrices following two applications of [^{14}C]-fluazaindolizine

Days after planting	Sample	[Ph-^{14}C]-fluazaindolizine		[IP-5,8a-^{14}C]-fluazaindolizine	
		Fruit	Foliage	Fruit	Foliage
60	30DAA2	NA	2.344	NA	0.596
71	41DAA2	0.070	4.232	0.029	0.577
80	50DAA2	0.079	5.743	0.029	0.919
92	62DAA2	0.066	1.857	0.037	0.438

Identification and characterisation of radioactive residues [Ph-^{14}C]fluazaindolizine

Tomato fruit

Parent fluazaindolizine was detected only in the 62DAA2 tomato fruit sample, at 0.4 percent TRR (<0.001 mg/kg) (Table 2). The principal residue extracted from the fruit samples was the glucose conjugate of IN-A5760 (IN-R3Z85), which constituted 45.0 percent TRR (0.032 mg eq/kg) in the 41DAA2 fruit sample, rising to 48.6 percent TRR (0.038 mg eq/kg) in the 50DAA2 fruit, and decreasing to 24.4 percent TRR (0.016 mg eq/kg) in the 62DAA2 fruit sample with a corresponding increase in unconjugated IN-A5760, 13.0 percent TRR (0.009 mg eq/kg).

The glucose conjugate of IN-RSU03 was the second most abundant metabolite present within [Ph-^{14}C]-treated tomato fruit, constituting 20.3 and 19.7 percent TRR (0.014 and 0.016 mg eq/kg) in the 41DAA2 and 50DAA2 fruit samples respectively, and to 22.6 percent TRR (0.015 mg eq/kg) in the 62DAA2 fruit.

Two glucose conjugates of IN-UNS90, were present mostly in the 41DAA2 to 50DAA2 tomato fruit samples (up to 12.7 percent TRR, 0.010 mg eq/kg), with lesser amounts found in the 62DAA2 sample (<1 percent TRR, <0.001 mg eq/kg). In the fruit, the major glucose conjugate of IN-UNS90 was proposed to be conjugated *via* the 2-hydroxy propanoic acid moiety. IN-F4106 was present between 2.6 and 7.9 percent TRR (0.002–0.005 mg eq/kg) in the various fruit samples.

Other identified metabolites in the fruit included IN-RSU03, IN-REG72 and IN-UNS90 (each ≤ 2.0 percent TRR, ≤ 0.002 mg eq/kg). Up to 16 minor unknown metabolites were found in the mature fruit, representing an aggregate total of 22.5 percent TRR and 0.013 mg eq/kg. No individual unknown metabolite was greater than 2.6 percent TRR or 0.002 mg eq/kg.

Analysis of acid-hydrolysed tomato fruit extracts from the 62DAA2 [Ph-^{14}C]-experiment demonstrated hydrolysis of IN-R3Z85 and the glucose conjugates of IN-UNS90 and IN-RSU03, primarily to IN-A5760, IN-RSU03, and IN-UNS90.

Table 2 Identification of TRR (% TRR) in tomato fruit from plants grown in soil after application of [Ph-^{14}C]-fluazaindolizine

	41DAA2	50DAA2	62DAA2	
TRR (mg eq/kg)	0.070	0.079	0.066	Post hydrolysis
Extracted (methanol:water 7:3)	93.4	92.3	98.0	
Fluazaindolizine	-	-	0.4	-
IN-REG72	-	-	0.8	-
Total whole molecule compounds	-	-	1.2	-
IN-RSU03 (IN-TMQ01)	2.0	-	1.4	19.1
Glucose Conjugate of IN-RSU03 (IN-TMQ01)	20.3	19.7	22.6	-
Total IN-RSU03 (IN-TMQ01) metabolites	22.3	19.7	24.0	19.1
IN-UNS90 (IN-TQD54)	-	-	1.5	11.0
Glucose Conjugate of IN-UNS90 (IN-TQD54)	10.0	12.7	0.3	-
Phenol-Glucose Conjugate of IN-UNS90 (IN-TQD54)	2.0	-	0.7	-
Total IN-UNS90 (IN-TQD54) metabolites	12.0	12.7	2.5	11.0
Glucose conjugate of IN-A5760 (IN-R3Z85)	45.0	48.6	24.4	-
IN-A5760	2.1	-	13.0	50.1
Total IN-A5760 metabolites	47.1	48.6	37.4	50.1
IN-F4106	2.6	2.6	7.9	3.4
Unretained	8.3	7.6	-	
Total unidentified metabolites	1.1[A]	-	18.1[B]	7.5
Unextracted	6.7	7.7	2.0	
Total identified or characterised by HPLC	93.4	91.1	91.2	

Notes:

[A] Consisting of a single component.

[B] Unidentified consisting of 16 components. None >2.6 percent TRR, 0.002 mg eq/kg.

- Not detected in this sample.

Tomato foliage

The majority of residues were extracted from the [Ph-^{14}C] tomato foliage by initial methanol:water extractions, 92.6–94 percent TRR (Table 3). Unextracted residues accounted for 6.0–7.4 percent TRR.

Fluazaindolizine was detected in the 41DAA2 foliage (0.5 percent TRR) but not at later harvest points. More than 10 metabolites were found in the foliage and their proportion of ^{14}C remained relatively constant. The principal metabolite was the glucose conjugate of IN-RSU03 (46.7 percent–53.9 percent TRR). Other metabolites included IN-QZY47 (5.7–7.4 percent TRR), IN-A5760 (6.0–6.4 percent TRR) and its glucose conjugate IN-R3Z85 (4.5–6.0 percent TRR), the malonic acid conjugate of IN-QZY47 (IN-TUT81, 2.7–3.1 percent TRR), IN-RSU03 (3.2–5.0 percent TRR) and IN-F4106 (3.9–4.9 percent TRR) together with IN-UNS90 (1.8–2.1 percent TRR) and two glucose conjugates of IN-UNS90, one proposed to be conjugated *via* the phenol (3.2 to 4.0 percent) and the other conjugated *via* the 2-hydroxy propanoic acid moiety (2.9–3.0 percent TRR).

Acid hydrolysis of tomato foliage extracts from the 50DAA2 [Ph-^{14}C]-experiment resulted in hydrolysis of the glucose conjugates of IN-UNS90, IN-RSU03 and IN-R3Z85 with the resulting chromatogram showing enhancement of unconjugated metabolites, free IN-A5760, IN-RSU03, and IN-UNS90 (Table 3).

The observation of the methyl ester of IN-RSU03 (8.4 percent TRR) observed in the acid hydrolysed foliage extract is proposed to be due to the incomplete removal of methanol prior to the hydrolysis step resulting in the esterification of IN-RSU03 and is therefore an artifact of the analysis protocol.

Table 3 Identification of TRR (%TRR) from tomato foliage from [Ph-^{14}C]-fluazaindolizine experiment

	41DAA2	50DAA2		62DAA2
TRR (mg eq/kg)	4.232	5.743	Post hydrolysis	1.857
Extracted (methanol:water 7:3)	94.0	93.5		92.6
Fluazaindolizine	0.5	-	-	-
IN-REG72		-	0.9	-
Total whole molecule compounds	0.5	-	0.9	-
IN-RSU03 (IN-TMQ01)	5.0	4.1	33.7	3.2
Methyl ester of IN-RSU03 (IN-TMQ01)			8.4[A]	
Glucose conjugate of IN-RSU03 (IN-TMQ01)	46.7	48.2	7.1	53.9
Total IN-RSU03 (IN-TMQ01) metabolites	51.7	52.3	49.3	57.1
Malonic acid conjugate of IN-QZY47 (IN-TUT81)	2.7	2.9	7.0	3.1
IN-QZY47	7.4	6.9	0.7	5.7
Total IN-QZY47 metabolites	10.1	9.8	7.7	8.8
IN-UNS90 (IN-TQD54)	1.8	1.7	6.2	2.1
Glucose conjugate of IN-UNS90 (IN-TQD54)	2.9	2.9	0.5	3.0
Phenol-glucose conjugate of IN-UNS90 (IN-TQD54)	3.7	4.0	0.4	3.2
Total IN-UNS90 (IN-TQD54) metabolites	8.4	8.6	7.1	8.3
IN-A5760	6.4	6.2	14.1	6.0
Glucose conjugate of IN-A5760 (IN-R3Z85)	5.1	6.0	-	4.5
Total IN-A5760 metabolites	11.5	12.2	14.1	10.5
IN-F4106	4.3	3.9	5.8	4.9
Unretained	0.6	0.5		0.3
Total unidentified metabolites	4.8[B]	3.7[C]	6.1	-
Unextracted	6.0	6.5		7.4
Total identified or characterised by HPLC	91.9	91.0		89.9

Notes:

[A] – Artifact due to formation of methyl ester of IN-RSU03 during sample processing.

[B] Unidentified consisting of six components. None >1.3 percent TRR, 0.056 mg eq/kg.

[C] Unidentified consisting of six components. None >1.2 percent TRR, 0.067 mg eq/kg.

- Not detected in this sample.

Identification and characterisation of radioactive residues [IP-5,8a-^{14}C]fluazaindolizine

Tomato fruit

Extractability of ^{14}C in fruit using methanol:water was good (92.8–95.6 percent TRR). Fluazaindolizine was detected in small amounts in both the 41DAA2 and 50DAA2 tomato fruit samples, at 0.8 and 0.9 percent TRR (<0.001 mg/kg), respectively (Table 4). The principal residue was the glucose conjugate of IN-QEK31 (identified as IN-UGA20), which constituted 39.9–51.4 percent TRR. Free IN-QEK31 became increasingly conjugated over time as it decreased as a proportion of the ^{14}C from 14.3 percent TRR in the 41DAA2 fruit sample to 4.5 percent and 7.1 percent TRR in the tomato fruit sampled 50DAA2 and 62DAA2, respectively. Other identified metabolites included inositol conjugate of IN-QEK31 (IN-UHD13), methyl ester of IN-QEK31 (IN–R2W56), IN-RYC33, and IN-REG72 (all ≤ 2.3 percent TRR) (Table 4).

Acid hydrolysis of 50DAA2 tomato fruit extracts resulted in conversion/hydrolysis of fluazaindolizine, IN-REG72, IN-UHD13, and IN-UGA20, IN-RYC33 and methyl ester of IN-QEK31 (IN-R2W56) to IN-QEK31 (Table 4). Treatment of a sample of the 50DAA2 tomato fruit extract with β-glucosidase resulted in hydrolysis of IN-UHD13 and IN-UGA20 with resulting increase in IN-QEK31.

Table 4 Identification of TRR (% TRR) from tomato fruit in various extracts and fractions after application of [IP-5,8a-^{14}C]-fluazaindolizine to soil

	41DAA2	50DAA2	62DAA2	
TRR (mg eq/kg)	0.029	0.029	0.037	Post hydrolysis
Extracted (methanol:water 7:3)	93.2	92.8	95.6	
Fluazaindolizine	0.8	0.9	-	-
IN-REG72	0.7	-	-	-
Total whole molecule compounds				-
Inositol conjugate of IN-QEK31 (IN-UHD13)	2.1	1.3	2.3	-
Glucose conjugate of IN-QEK31 (IN-UGA20)	39.9	45.1	51.4	-
IN-QEK31	14.3	4.5	7.1	72.5
Methyl ester of IN-QEK31 (IN-R2W56)	0.8	1.2	0.9	-
Total IN-QEK31 metabolites	57.1	52.1	61.7	72.5
IN-RYC33	1.4	1.2	1.1	-
Unretained	10.2	12.1	7.2	
Total unidentified metabolites	22.9[A]	26.3[B]	25.5[C]	23.1
Unextracted	6.8	7.2	4.4	
Total identified or characterised by HPLC	93.2	92.8	95.6	

Notes:

[A] Unidentified consisting of five components. None >9.1 percent TRR, 0.003 mg eq/kg.

[B] Unidentified consisting of seven components. None >8.4 percent TRR, 0.002 mg eq/kg.

[C] Unidentified consisting of seven components. None >3.9 percent TRR, 0.004 mg eq/kg.

- Not detected in this sample.

Tomato foliage

The majority of residues were extracted from the [IP-5,8a-^{14}C] tomato foliage by initial extractions, 80.2-83.9 percent TRR (0.366–0.770 mg eq/kg: Table 15). The remaining unextracted residues determined by oxidative combustion accounted for 16.1–19.8 percent TRR (0.071–0.148 mg equiv/kg).

Fluazaindolizine was detected in all sample timepoints, at 2.7–3.7 percent TRR (Table 5). The principal residues were IN-QEK31 (9.9–15.8 percent TRR) and its glucose conjugate IN-UGA20, (13.6–14.0 percent TRR). Other identified metabolites in foliage included inositol conjugate of IN-QEK31 (IN-UHD13), methyl ester of IN-QEK31 (IN-R2W56), IN-RYC33, and IN-REG72 (all ≤ 5.7 percent TRR, ≤ 0.049 mg eq/kg).

Analysis of acid hydrolysed 50DAA2 tomato foliage extracts from the [IP-5,8a-^{14}C]-labelled experiment demonstrated hydrolysis of fluazaindolizine, IN-UGA20 (and the various rearranged glucose conjugates of IN-QEK31) and nearly all of the various unidentified metabolites to IN-QEK31. Nearly all unidentified metabolites were conjugated through the ester linkage (Table 5). Chiral HPLC determined IN-RSU03 was present as the *R*-enantiomer (IN-TMQ01). A proposed metabolic pathway of fluazaindolizine in tomato is shown in Figure 1.

Table 5 Identification of TRR (% TRR) in tomato foliage after application of [IP-5,8a-^{14}C]fluazaindolizine

	41DAA2	50DAA2		62DAA2
TRR (mg eq/kg)	0.577	0.919	Post hydrolysis	0.438
Extracted (methanol:water 7:3)	80.2	83.9		83.7
Fluazaindolizine	3.0	3.7	-	2.7
IN-REG72	0.9	3.2	1.2	-
Total whole molecule metabolites		6.9	1.2	2.7
Inositol conjugate of IN-QEK31 (IN-UHD13)	4.6	5.4	4.3	5.7
Glucose conjugate of IN-QEK31 (IN-UGA20)	14.2	13.6	1.5	14.0

	41DAA2	50DAA2		62DAA2
TRR (mg eq/kg)	0.577	0.919	Post hydrolysis	0.438
IN-QEK31	15.8	9.9	36.4	15.2
Methyl ester of IN-QEK31 (IN-R2W56)	1.2	2.4	8.1	3.1
Total IN-QEK31 metabolites	35.8	31.3	50.3	38.0
IN-RYC33	3.1	2.7	-	2.1
Unretained	2.9	2.9		3.7
Total unidentified metabolites	32.1[A]	40.1[B]	32.3	37.2[C]
Unextracted	19.8	16.1		16.3
Total identified or characterised by HPLC	77.7	83.9		83.7

Notes:

[A] Unidentified consisting of 21 components. None >5.5 percent TRR, 0.032 mg eq/kg.

[B] Unidentified consisting of 28 components. None >4.4 percent TRR, 0.040 mg eq/kg.

[C] Unidentified consisting of 15 components. None >7.8 percent TRR, 0.034 mg eq/kg.

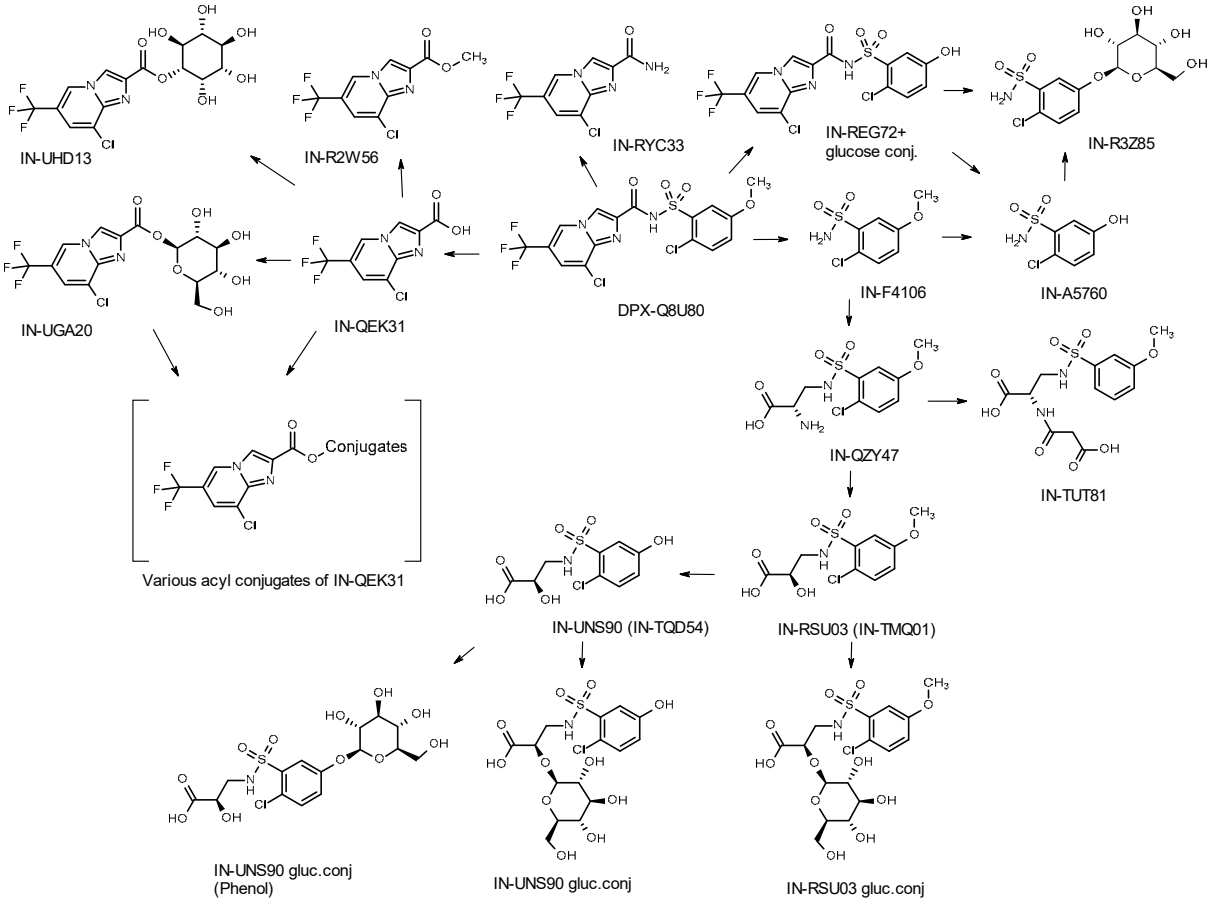


Figure 1 Proposed metabolic pathway of fluazaindolizine in tomato plant

In summary, fluazaindolizine is taken up by tomato plants and found in fruit and foliage. Fluazaindolizine is *O*-demethylated to form IN-REG72. Fluazaindolizine and IN-REG72 are hydrolysed at the amide bond resulting in IN-F4106 (and/or IN-A5760 and its glucose conjugate), and IN-QEK31. IN-F4106 is conjugated with endogenous serine to form IN-QZY47. The free amino moiety of IN-QZY47 is conjugated with malonic acid to form IN-TUT81which is found only in foliage. IN-QZY47 is mostly deaminated to the lactic acid derivative IN-RSU03, which was found only as the *R*-enantiomer, IN-TMQ01.

IN-RSU03 can undergo *O*-demethylation to form IN-UNS90 (*R*-enantiomer designated as IN-TQD54) which appears to be mostly found in the conjugated form.

IN-QEK31 is conjugated to glucose *via* an ester bond to form IN-UGA20. Some degree of acyl-migration of the glucose ester is observed. A glycerol glucuronide conjugate of IN-QEK31 was also identified as well. More complex carbohydrate esters of IN-QEK31 are proposed to form as numerous radioactive peaks are found with retention characteristics consistent with glucose or inositol (IN-UHD13) esters. The methyl ester of IN-QEK31 (IN-R2W56) was formed to a small extent (≤ 3.1 percent TRR) and could be an artefact of the extraction methodology (using methanol in the procedure).

IN-RYC33, is found in fruit and foliage, is formed either by hydrolysis of fluazaindolizine at the sulfonamide bond in the plant or taken up directly as a metabolite from the soil. All of the metabolites containing the imidazo-pyridine rings are readily hydrolysed to IN-QEK31 under acidic conditions.

Soya beans

The metabolic fate of [Ph-^{14}C(U)]-fluazaindolizine and [IP -5,8a-^{14}C]-fluazaindolizine in soya beans was studied by Begley and Hobbs (2016 DuPont-34948).

Soya bean seeds (cv Elena) were sown into treated sandy loam soil (pH 7.3, percentOM 2.4, 65 percent sand, 20 percent silt, 15 percent clay, CEC 10 meq/100g) *ca.* 1 hour after a soil drench application of an SC formulation of [Ph-^{14}C]-fluazaindolizine or [IP-5,8a-^{14}C]-fluazaindolizine, at a nominal application rate of 1000 g ai/ha. The achieved treatment rates were 1.00 kg ai/ha for both the [Ph-^{14}C] and [IP-5,8a-^{14}C]-experiments.

Plants were taken for analysis 48, 75 and 112 days after planting and corresponded to a forage (48DAA, BBCH63), hay samples (75DAA, BBCH69) and seed (112DAA, BBCH99) at crop maturity. Samples were stored frozen until analysis and extracted, and analysed within 1 month of harvest.

Extractability of ^{14}C with methanol:water was good (78–91 percent TRR). Further sequential extraction using water, acetonitrile:water, and treatment with driselase (a cell wall degrading enzyme), 0.1M HCl, 1.3M HCl and 0.1M NaOH released a further 7.1–22.3 percent TRR.

TRR in soya bean plants from the [Ph-^{14}C]-fluazaindolizine experiment were 0.435–0.660 mg eq/kg while TRR in soya bean plants from the [IP-5,8a-^{14}C]-fluazaindolizine experiment ranged from 0.763 to 2.018 mg eq/kg (Table 6).

Table 6 TRR (mg eq/kg) in soya bean plant matrices following one application at plant of [^{14}C]fluazaindolizine

Days after planting	Sample type	[Ph-^{14}C]fluazaindolizine	[IP-5,8a-^{14}C]fluazaindolizine
48	Forage (BBCH 61)	0.435	0.763
75	Hay (BBCH 75)	0.660	1.042
112	Seed (maturity, BBCH 99)	0.271	2.018

Soya bean forage

Extractability of ^{14}C with methanol:water in the [Ph-^{14}C] experiment soya bean forage was good (90.1 percent TRR; Table 7). Further ^{14}C was released following additional treatments (7.2 percent TRR).

Fluazaindolizine accounted for 7.2 percent TRR in extracts (7.0 percent TRR in methanol:water; 0.2 percent TRR further treatments). The principal component of the ^{14}C in the extracts in [Ph-^{14}C] experiment was a malonyl conjugate of IN-QZY47 (IN-TUT81) at 56.5 percent TRR. Other identified metabolites included IN-QZY47 and IN-F4106 (both ≤4.9 percent TRR) with IN-REG72 and IN-RSU03 (IN-

TMQ01) as low level metabolites accounting for ≤1 percent TRR. A polar metabolite accounting for 4.2 percent TRR was tentatively assigned as a conjugate of IN-UJV12. Multiple unidentified metabolites were also detected accounting for an aggregate total of 17.6 percent TRR but each individually ≤ 2.6 percent TRR, ≤ 0.011 mg eq/kg.

Following hydrolysis of the methanol:water forage extract, the malonyl conjugate (IN-TUT81) was hydrolysed to IN-QZY47 (Table 7). An increase in IN-F4106 and IN-A5760 was also observed after hydrolysis. While some formation of IN-F4106 can be attributed to the hydrolysis of fluazaindolizine and IN-REG72, it is apparent that several other minor unidentified metabolites or conjugates are also cleaved under these conditions and released as IN-F4106 and IN-A5760. Small amounts of IN-RSU03 (IN-TMQ01) and IN-UJV12 are also released by acid hydrolysis.

Soya bean hay

Extractability of ^{14}C in hay with methanol:water was good in the [Ph-^{14}C] experiment (90.6 percent TRR Table 7). A further 7.0 percent TRR was released with additional treatments.

Fluazaindolizine accounted for 6.1 percent TRR and was found only in the hay methanol:water extracts. The principal extracted residue in the [Ph-^{14}C] experiment was a malonyl conjugate of IN-QZY47 (IN-TUT81) accounting for 56.4 percent TRR. Other identified metabolites included IN-QZY47 and IN-F4106 (both ≤ 5.3 percent TRR, ≤ 0.034 mg eq/kg) and IN-REG72 and IN-RSU03 (IN-TMQ01) both at less than 1.2 percent TRR. A polar metabolite was observed accounting for 2.4 percent TRR and was tentatively assigned as a conjugate of IN-UJV12. Multiple unidentified metabolites were also detected; all ≤ 3.4 percent TRR, ≤ 0.022 mg eq/kg.

Following hydrolysis of the hay extract the malonyl conjugate (IN-TUT81) was hydrolysed to IN-QZY47. An increase in IN-F4106 and IN-A5760 was also observed after hydrolysis. While some formation of IN-F4106 and IN-A5760 could be attributed to the hydrolysis of fluazaindolizine and IN-REG72, it was apparent that minor other unidentified metabolites or conjugates were also cleaved under these conditions and liberated mostly as IN-F4106 (17.8 percent TRR) with small amounts of IN-A5760 (3.5 percent TRR). A polar metabolite with retention time of approximately 8.5 minutes was mostly hydrolysed to IN-UJV12.

Soya bean seed

Extractability of ^{14}C in seed with methanol:water in the [Ph-^{14}C] experiment was good with 80.6 percent TRR recovered (Table 7). A further 17.4 percent TRR was released using additional treatments.

The principal extracted residue identified in seed in the [Ph-^{14}C]fluazaindolizine experiment was parent fluazaindolizine (46.2 percent TRR of which 45.0 percent TRR in the methanol:water extract). A malonyl conjugate of IN-QZY47 (IN-TUT81) was the only other residue exceeding 10 percent TRR (15.3 percent TRR). Other metabolites were IN-REG72 (9.0 percent TRR) and IN-F4106 (1.9 percent TRR). Multiple unidentified metabolites were detected; none >1.3 percent TRR.

Following hydrolysis of the seed methanol water extract, mostly IN-F4106 was formed which was mostly attributed to the hydrolysis of fluazaindolizine (Table 7). The malonyl conjugate (IN-TUT81) was hydrolysed to IN-QZY47. Other hydrolysis products including IN-A5760 (6.0 percent TRR, originating from IN-REG72) as well as small amounts of IN-UJV12 from hydrolysis of more complex conjugates were observed. As the increase in IN-F4106 was slightly more than what was expected from fluazaindolizine, it is apparent that minor other unidentified metabolites or conjugates were also cleaved under HCl hydrolysis conditions and released as IN-F4106.

Table 7 Identification of TRR (% TRR) in various soya bean extracts and fractions after application of [Ph-^{14}C]-fluazaindolizine

Component	Forage TRR= 0.435 mg eq/kg		Hay TRR = 0.660 mg eq/kg		Seed TRR = 0.271 mg eq/kg	
	Pre hydrolysis	Post hydrolysis	Pre hydrolysis	Post hydrolysis	Pre hydrolysis	Post hydrolysis
Extracted (methanol:water)	90.1		90.6		80.6	
Fluazaindolizine	7.0	-	6.1	-	46.2	-
IN-REG72	0.7	-	1.2	-	9.0	-
Total intact molecule metabolites	7.7	-	7.3	-	55.2	-
Malonyl conjugate of IN-QZY47 (IN-TUT81)	53.9	17.5	56.4	9.1	15.3	0.076
IN-QZY47	4.9	42.3	5.3	52.5	-	0.184
Total IN-QZY47 associated metabolites	61.4	59.8	61.7	61.6	15.3	0.26
IN-UJV12 Conjugates[A]	4.2	0.5	2.4	-	-	-
IN-RSU03	1.0	1.5	0.5	-	-	-
IN-A5760	-	3.2	-	3.5	-	6.0
IN-F4106	1.9	11.7	2.7	17.8	1.9	53.5
IN-UNS90/IN-UJV12		5.8		7.7		2.1
Total unidentified metabolites	16.6[B]	7.6[C]	18.3[B]	-	9.4	1.9[D]
Unretained	1.0		2.1		2.8	
Unextracted with methanol:water	9.9		9.4		19.3	
Aqueous soak 1+ acetonitrile:water	3.9		4.0		4.0	
Fluazaindolizine	0.2		-		1.2	
IN-REG72	-		-		0.4	
Malonyl conjugate of IN-QZY47 (IN-TUT81)	2.6		2.9		-	
IN-QZY47	0.2		0.2		-	
IN-F4106	0.2		-		-	
Unidentified metabolites	0.1		0.8		1.3	
Unretained	0.7		0.3		1.1	
Aqueous soak 2	0.2		0.3		0.6	
Driselase	0.8		0.7		3.1	
0.1M HCl	0.7		0.6		1.5[E]	
1.3M HCl	1.1		1.0		2.1	
0.1M NaOH	0.5		0.4		0.8[E]	
Remaining	2.7		2.3		1.8	

Notes:

[A] Hydrolysis product confirmed by LC-MS as IN-UJV12

[B] Total unidentified metabolites consist of multiple components – none greater than 3.4 percent TRR, 0.022 mg eq/kg: several of which appear to be hydrolysed with 1M HCl to IN-A5760 and IN-F4106

[C] Consisting of 17 components, none greater than 2.3 percent TRR (0.010 mg eq/kg).

[D] Consisting of 2 components, none greater than 1.3 percent TRR (0.004 mg eq/kg).

[E] Data obtained from measurements that were <LOQ.

Identification and characterisation of radioactive residues [IP-5,8a-^{14}C]fluazaindolizine

Soya bean forage

Extractability of ^{14}C in forage from the [IP-5,8a-^{14}C] experiment using methanol:water was good (89.5 percent TRR; Table 8). Further treatments released an additional 8.0 percent TRR.

Fluazaindolizine was exclusively extracted from the forage into the methanol:water extract, accounting for 6.9 percent TRR (0.053 mg/kg). The principal extracted residue identified was IN-QEK31 accounting for 40.5 percent TRR (0.309 mg/kg) in the methanol water extract, with a small amount (1.4 percent TRR) in the acetonitrile extract for a total of 41.9 percent TRR (0.320 mg/kg). A glycerol glucuronide conjugate of IN-QEK31 accounting for 9.5 percent TRR (0.072 mg/kg) was identified as a major metabolite by LC-MS. IN-UGA20 and acyl migrated glucose ester isomers accounted for 11.8 percent TRR (0.092 mg eq/kg), and were found as several closely eluting peaks in the radiochromatogram. IN-UHD13, an inositol conjugate of IN-QEK31, was tentatively identified as a minor metabolite (1.5 percent TRR, 0.012 mg eq/kg) based on similar retention time to the reference standard. Other identified metabolites include, IN-RYC33 (5.7 percent TRR, 0.043 mg eq/kg) and minor metabolites IN-REG72, and methyl ester of IN-QEK31 (IN-R2W56) (both ≤ 1.7 percent TRR, ≤ 0.013 mg eq/kg). Multiple unidentified metabolites were also detected; all ≤ 2.0 percent TRR, ≤ 0.015 mg eq/kg. A minor volatile metabolite was trapped during the concentration of forage extracts and accounted for 0.6 percent TRR, 0.004 mg eq/kg. This component was thought to be IN-VM862, based on its chromatographic behaviour, and analysis of a volatile metabolite formed in the crop rotation study (DuPont-34945, Revision No. 1).

Acid hydrolysis of forage samples showed that the various conjugates of IN-QEK31, as well as IN-REG72, IN-RYC33, methyl ester of IN-QEK31 (IN-R2W56) and fluazaindolizine, readily hydrolysed to IN-QEK31 using 1N HCl (Table 8). The majority of the other minor unidentified metabolites were also hydrolysed to IN-QEK31 using 1N HCl.

Soya bean hay

Extractability of ^{14}C in hay with methanol:water in the [IP-5,8a-^{14}C] experiment was good with 86.6 percent TRR recovered (Table 8). Further treatments released an additional 10.3 percent TRR with 3.1 percent TRR remaining in the solids.

Fluazaindolizine was exclusively extracted in [IP-5,8a-^{14}C] hay methanol:water extracts at low concentrations (4.8 percent TRR). The principle extractable residue identified was IN-QEK31 accounting for 39.0 percent TRR with the majority extracted into methanol:water (36.2 percent TRR). A glycerol glucuronide conjugate of IN-QEK31 was identified accounting for 7.5 percent TRR. IN-UHD13 an inositol conjugate of IN-QEK31 was tentatively assigned to 1.7 percent TRR. The multiplex of peaks associated with IN-UGA20 (the glucose conjugate of IN-QEK31) accounted for 14.8 percent TRR. IN-UHD13, IN-REG72, IN-RYC33 and methyl ester of IN-QEK31 (IN-R2W56) were also identified at low levels (≤ 3.2 percent TRR, ≤ 0.033 mg eq/kg).

Multiple unidentified metabolites were also detected in the hay, (≤ 2.3 percent TRR, ≤ 0.024 mg eq/kg). The majority of the minor unidentified metabolites as well as glycerol glucuronide conjugate of IN-QEK31, IN-UGA20 and IN-UHD13 were hydrolysed to IN-QEK31 using 1M HCl (Table 8).

Soya bean seed

Extractability of ^{14}C in seed with methanol:water in the [IP-5,8a-^{14}C] experiment was good with 77.7 percent TRR recovered (Table 8). A further 22.3 percent TRR was released using additional treatments.

Parent molecule fluazaindolizine was found primarily in the methanol water fraction with a trace amount in the further extracts and accounted for a total of 8.3 percent TRR (0.167 mg/kg). The principle extractable residue identified was IN-QEK31 accounting for 65.8 percent TRR in the methanol water extract and 20.2 percent TRR in the further extracts for a total of 86.0 percent TRR (1.736 mg/kg). IN-UGA20, IN-REG72 and IN-R2W56 (≤ 1.7 percent TRR, ≤ 0.034 mg/kg), were also identified by comparison

of retention time with known reference standards. No radioactive residues were found at the HPLC retention times of IN-RYC33 in seed samples.

Sample residues were further characterised by acid hydrolysis and the major residues confirmed and identified by LC-MS analysis. Acid hydrolysis demonstrated that fluazaindolizine and nearly all of the minor components were hydrolysed to IN-QEK31, an indication that they were conjugates of this metabolite (Table 8).

Table 8 Identification of TRR (% TRR)in various soya bean extracts and fractions after application of [IP-5,8a-^{14}C]fluazaindolizine

	Forage TRR=0.763 mg eq/kg		Hay TRR= 1.042 mg eq/kg		Seed TRR=2.018 mg eq/kg	
Component	Pre hydrolysis	Post hydrolysis	Pre hydrolysis	Post hydrolysis	Pre hydrolysis	Post hydrolysis
Extracted methanol:water	89.5		86.6		77.7	
Fluazaindolizine	6.9	-	4.8	-	8.0	-
IN-REG72	1.4	-	0.9	-	1.2	-
Total intact molecule metabolites	8.3	-	5.7	-	9.2	-
Glycerol glucuronide conjugate of IN-QEK31	9.2	-	7.5	-	-	-
Inositol conjugate of IN-QEK31 (IN-UHD13)	1.5	-	1.7	-	-	-
Glucose conjugate of IN-QEK31 (IN-UGA20)	11.6	-	14.8	-	1.7	-
IN-QEK31	40.5	72.5	36.2	67.1	65.8	75.2
methyl ester of IN-QEK31 (IN-R2W56)	1.7	0.2	3.2	-	1.0	-
IN-RYC33	5.7	0.6	2.8	-	-	-
Total IN-QEK31 associated metabolites	70.2	73.3	66.2	67.1	68.5	75.2
Total IN-QEK31 associated metabolites in acetonitrile:water further ext.	-	1.9	-	2.8	-	20.2
RT *ca.* 11 min		2.6		3.0		1.6
IN-VM862	0.6					
Unretained	0.9		2.1		-	
Total unidentified metabolites	10.1[A]	13.0[B]	12.6[A]	16.8[C]	-	0.8[D]
Unextracted methanol:water[C]	10.6		13.4		22.3	
Aqueous soak1 +acetonitrile:water	0.4		0.5		0.8	
Fluazaindolizine	-		-		0.3	
Glycerol glucuronide conjugate of IN-QEK31	0.3		-		-	
Glucose conjugate of IN-QEK31 (IN-UGA20)	0.2		-		-	
Unretained	0.4		1.5		-	
Unidenified metabolites	1.9		1.1		-	
Aqueous soak 2	0.4		0.5		0.8	
Driselase	0.6		1.0		0.6	
0.1M HCl	1.1		1.2		0.1[E]	
1.3M HCl	1.2		1.6		0.2[E]	
0.1M NaOH	0.5		0.6		0.1[E]	
Remaining	2.7		3.1		(<0.1[F])	

Notes:

[A] Total unidentified metabolites consist of multiple components – none greater than 2.3 percent TRR, 0.024 mg eq/kg: most minor unknown metabolites are hydrolysed with 1M HCl to IN-QEK31

[B] Consisting of 27 components, none greater than 1.3 percent TRR (0.010 mg eq/kg).

[C] Consisting of 16 components, none greater than 4.3 percent TRR (0.045 mg eq/kg).

[D] Consisting of a single component.

[E] Data obtained from measurements that were <LOQ.

[F] Insufficient volume of tissue remained following the further extracts to analyse remaining unextractable residues. All initially identified residues from the PES were observed in the further extracts.

Chiral analysis of metabolites

Chiral HPLC determined the S-enantiomeric configuration of IN-QZY47, consistent with naturally occurring L-serine. The metabolic pathway for fluazaindolizine in soya bean plants following soil application is presented in Figure 2 and is proposed based on the metabolites identified in forage, hay, and soya bean seed. Metabolites in the figure are depicted in their free acid or free base forms.

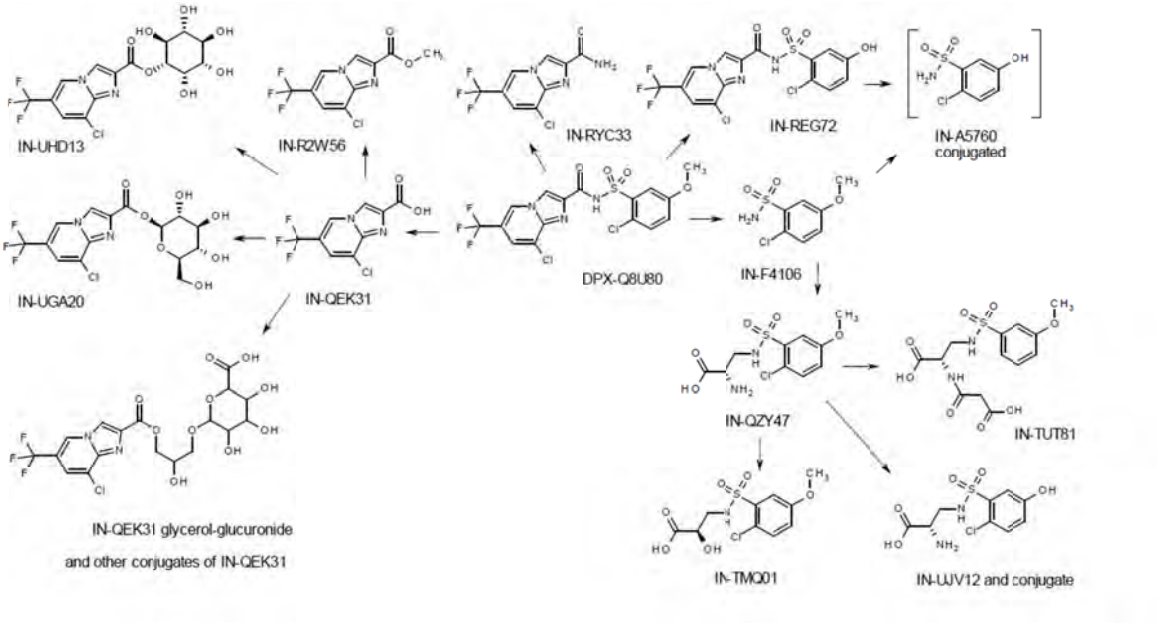


Figure 2 Proposed metabolic pathway of fluazaindolizine in soya bean plant

Carrots

The metabolic fate of [phenyl-^{14}C(U)]fluazaindolizine and [imidazo[1,2-a]pyridine-5,8a-^{14}C]fluazaindolizine in glasshouse grown carrot plants (cv. F1 Bangor) was studied by Hobbs et al. (2016 DuPont-34947).

Carrot seeds were sown into treated sandy loam soil (pH 6, percentOM 5.1, 67 percent sand, 17 percent silt, 16 percent clay, CEC 15 meq/100 g) *ca.* one hour after a soil drench application with an SC formulation of [Ph-^{14}C]fluazaindolizine or [IP-5,8a-^{14}C]fluazaindolizine, at a nominal application rate of 1500 g ai/ha. Seeds were sown in four rows approximately 10 cm apart at a depth of *ca.* 13 mm. A further application was made to the same plots 30 days after the initial application at a nominal application rate of 0.5 kg ai/ha. The total achieved treatment rates were 2.00 kg ai/ha and 1.96 kg ai/ha for the [Ph-^{14}C] and [IP-5,8a-^{14}C] labels, respectively.

Plants were taken for analysis 30, 73 and 93 days after planting and corresponded to a foliage only sample (30 days after application 1, 30DAA1; BBCH 42), an immature root and foliage sample

(43DAA2, BBCH 45) and root and foliage sample at crop maturity (63DAA2, BBCH 49). Samples were kept under frozen storage (*ca.* -20°C) until analysis.

Plant samples were generally extracted within 50 days of harvest and the extracts were stored for no more than 52 days before initial chromatography. A mature foliage sample from the [Ph-^{14}C]fluazaindolizine experiment and an immature foliage sample from the [IP-5,8a-^{14}C]fluazaindolizine experiment were re-extracted and profiled. The profiles remained qualitatively the same after 3 years storage. The results are shown in Table 9.

Table 9 TRR (mg eq/kg) in carrot plant matrices following one or two applications of [^{14}C]fluazaindolizine

Days after planting	Sample type	[Ph-^{14}C]-fluazaindolizine		[IP-5,8a-^{14}C]-fluazaindolizine	
		Foliage	Root	Foliage	Root
30	30DAA1	4.434	NS	3.169	NS
73	Immature 43DAA2	0.659	0.135	0.278	0.073
93	Mature 63DAA2	1.174	0.104	0.382	0.068

Notes:

NS = No sample available

Carrot foliage

Extractability of ^{14}C in foliage with methanol:water in the [Ph-^{14}C] experiment was good with 82.6–87.2 percent TRR recovered (Table 10). Unextracted residues in the 63DAA2 sample was subjected to further treatments released an additional 9.4 percent TRR with 4.7 percent TRR remaining in the solids.

Fluazaindolizine, found primarily in the methanol:water extracts, was identified in the early foliage (20.9 percent TRR, 0.926 mg/kg) decreasing in later samples. The principal extracted residue was IN-RSU03 increasing from 23.4 percent TRR at 30DAA1 to 51.1 percent TRR at crop maturity. Additional residues of IN-RSU03 and fluazaindolizine were released in the rehydrated: acetonitrile extracts of the mature foliage; 2.4 percent TRR and 0.2 percent TRR for IN-RSU03 and fluazaindolizine, respectively (Table 10).

Other identified metabolites were the malonyl conjugate of IN-QZY47 (IN-TUT81), glucose conjugate of IN-A5760 (IN-R3Z85), IN-QZY47, IN-F4106, IN-REG72 and IN-UNS90 (all ≤15.6 percent TRR, ≤ 0.691 mg eq/kg) as well as a malonyl conjugate of IN-RSU03 (15.5 percent TRR, 0.182 mg eq/kg), and glucose conjugates of IN-UNS90, IN-RSU03 and IN-REG72 (≤ 2.6 percent TRR, ≤ 0.042 mg eq/kg).

Multiple unidentified metabolites were also detected and accounted for an aggregate total of 2.7-5.2 percent TRR in each sample but each individually was ≤ 1.7 percent TRR (≤ 0.076 mg eq/kg).

Carrot roots

Extractability of ^{14}C in roots from the [Ph-^{14}C] experiment using methanol:water was good (92.8–92.9 percent TRR Table 10). Unextracted residues accounted for 7.1–7.2 percent TRR.

The fluazaindolizine accounted for 8.4 percent TRR in immature carrot roots decreasing to 1.7 percent TRR in mature carrot roots. The principal extracted residues were the malonyl conjugate of IN-RSU03 (35.5–40.0 percent TRR) in addition to unconjugated IN-RSU03 metabolite (15.3–21.8 percent TRR). Other identified metabolites were the malonyl conjugate of IN-QZY47 (IN-TUT81), IN-QZY47, IN-UNS90, the glucose conjugate of IN-RSU03 and a glucose conjugate of IN-UNS90 (all ≤ 25.7 percent TRR, ≤ 0.035 mg eq/kg). Multiple unidentified metabolites were also detected accounting for an aggregate total of 2.4–4.9 percent TRR in each sample but each individually was ≤ 2.6 percent TRR (≤ 0.003 mg eq/kg).

Following hydrolysis of the mature carrot root aqueous methanol extract, the malonyl conjugate and glucose conjugate of IN-RSU03 were hydrolysed to IN-RSU03 (Table 10). The malonyl conjugate of IN-QZY47 (IN-TUT81) was mostly hydrolysed to IN-QZY47, and the glucose conjugate of IN-UNS90 was hydrolysed to IN-UNS90. An increase in IN-F4106 was also observed after hydrolysis, while some formation of IN-F4106 can be attributed to the hydrolysis of fluazaindolizine, it is apparent that other minor unidentified metabolites or conjugates found in the HPLC chromatogram were also cleaved under these conditions and released as IN-F4106 (Table 10).

Table 10 Identification of TRR (%TRR) in carrot root and foliage extracts and fractions after application of [Ph-^{14}C]fluazaindolizine

	43DAA2 Immature Roots	63DAA2 Mature Roots		30DAA1 Foliage	43DAA2 Foliage	63DAA2 Foliage
TRR (mg eq/kg)	0.135	0.104	Post hydrolysis	4.434	0.659	1.174
Extracted (methanol water)	92.9	92.8		82.6	87.2	85.9
Fluazaindolizine	8.4	1.7	-	20.9	7.1	3.2
IN-REG72	-	-	2.3	1.3	0.5	0.4
Glucose Conjugate of IN-REG72	-	-	-	0.6	0.7	0.6
Total whole compounds	8.4	1.7	2.3	22.8	8.3	4.2
IN-RSU03 (IN-TMQ01)[A]	15.3	21.8	59.9	23.4	49.3	51.1
Malonyl Conjugate of IN-RSU03	35.5	40.0	-	8.3	13.4	14.9
Glucose Conjugate of IN-RSU03	0.5	1.3	-	0.4	1.5	1.7
Total IN-RSU03 metabolites	51.3	63.1	59.9	32.1	64.2	67.7
IN-QZY47	1.4	1.0	9.0	0.5	-	-
Malonic acid conjugate of IN-QZY47 (IN-TUT81)	25.7	18.5	5.7	15.6	6.5	6.5
Total IN-QZY47 metabolites	27.1	19.5	14.7	16.1	6.5	6.5
IN-UNS90 (IN-TQD54)	1.3	1.1	4.8	0.4	0.6	0.7
Glucose Conjugate of IN-UNS90	2.4	2.5	-	1.0	2.1	2.4
Total IN-UNS90 metabolites	3.7	3.6	4.8	1.4	2.7	3.1
glucose conjugate of IN-A5760 (IN-R3Z85)	-	-	-	0.1	-	-
IN-F4106	-	-	6.8	4.2	0.3	1.2
Unretained	-	-		0.6	1.0	0.7
Total unidentified metabolites[B]	2.4	4.9	-	5.2	4.1	2.5
Unextracted methanol:water	7.1	7.2		17.4	12.8	14.1
acetonitrile:water	-	-		-	-	4.2
Fluazaindolizine						0.2
IN-RSU03						2.4
Malonyl Conjugate of IN-RSU03						0.6
Malonic acid conjugate of IN-QZY47 (IN-TUT81)						0.4
Glucose conjugate of IN-UNS90						0.2
IN-UNS90						0.1
IN-F4106						0.2
Unretained						0.2
Driselase						1.7
0.1M HCl						0.6
1M HCl						1.6
0.1M NaOH						1.3
Remaining						4.7

Notes:

DAA = days after one or two soil applications.

[A] Only found as the *R*-enantiomer IN-TMQ01.

[B] Total unidentified metabolites consist of multiple components – none greater than 2.6 percent TRR: in the roots and 1.7 percent TRR, in the foliage, several of which appear to be hydrolysed with 1M HCl to IN-F4106.

Identification and characterisation of radioactive residues [IP-5,8a-^{14}C]fluazaindolizine

Carrot foliage

Extractability of ^{14}C in foliage from the [IP-5,8a-^{14}C] experiment using methanol:water was good (67–81 percent TRR Table 11). Unextracted residues accounted for 19.9-32.9 percent TRR. Further treatment of unextracted residues from mature foliage (63DAA2) released an additional 17.7 percent TRR with 15.1 percent TRR remaining in the solids.

Fluazaindolizine in methanol:water extracts from the [IP-5,8a-^{14}C] experiment carrot foliage decreased from 40.6 percent TRR at 30DAA1 foliage to 13.4 percent TRR in the mature foliage. The major metabolite was IN-QEK31 (8.9-21.1 percent TRR), although in later samples IN-QEK31 was increasingly present in the conjugated forms (*i.e.,* the glycerol glucuronide, IN-UGA20, inositol conjugate of IN-QEK31 (IN-UHD13) plus the methylated form IN-R2W56). Other identified metabolites were the glucose conjugate of IN-REG72, IN-RYC33, and IN-VM862 (all ≤ 10.3 percent TRR, ≤ 0.328 mg eq/kg).

Additional residues of IN-QEK31 (0.9 percent TRR) and fluazaindolizine (0.5 percent TRR) were released in the acetonitrile extracts of the mature foliage.

A volatile metabolite was also detected in the immature and mature carrot foliage extracts during the concentration process, accounting for 12.7 percent and 2.4 percent TRR, respectively. This component is thought to be IN-VM862, based on its chromatographic behaviour, and analysis of a volatile metabolite formed during the growth of crops under rotational crops study (DuPont-34954).

Multiple unidentified metabolites were also detected accounting for an aggregate total of 3.5–17.5 percent TRR in each sample but each individually was ≤ 2.7 percent TRR (≤ 0.043 mg eq/kg).

Following hydrolysis of the mature carrot foliage aqueous methanol extract, fluazaindolizine, the glycerol glucuronide of IN-QEK31, the glucose conjugate of IN-REG72, glucose conjugate of IN-QEK31 (IN-UGA20), methyl ester of IN-QEK31 (IN-R2W56) and IN-RYC33 were hydrolysed to IN-QEK31. It was apparent that minor other unidentified metabolites or conjugates were also cleaved under these conditions and released as IN-QEK31 or minor unknown metabolites (Table 11).

Table 11 Identification of TRR (%TRR) in carrot root and foliage extracts and fractions after application of [IP-5,8a-^{14}C]fluazaindolizine

	43DAA2 Roots	63DAA2 Mature Roots	30DAA1 Foliage	43DAA2 Foliage	63DAA2 Foliage	
TRR (mg eq/kg)	0.073	0.068	3.169	0.278	0.382	Post hydrolysis
Extracted (methanol water)	89.1	86.3	69.4	80.1	64.6	
Fluazaindolizine	12.6	11.9	40.6	22.7	12.9	-
IN-REG72	-	-	2.6	-	1.0	-
Glucose conjugate of IN-REG72	-	-	0.7	2.9	4.4	-
Total whole molecule metabolites	12.6	11.9	43.9	25.6	18.3	-
Glycerol glucuronide of IN-QEK31	-	1.3	0.3	2.8	3.7	-
Inositol conjugate of IN-QEK31 (IN-UHD13)	-	-	-	1.1	3.5	-
Glucose conjugate of IN-QEK31 (IN-UGA20)	-	3.3	0.8	2.8	5.7	-
IN-QEK31	66.0	53.7	8.9	21.1	13.5	45.4
methyl ester of IN-QEK31 (IN-	-	1.8	0.8	4.1	2.6	-

	43DAA2 Roots	63DAA2 Mature Roots	30DAA1 Foliage	43DAA2 Foliage	63DAA2 Foliage	
TRR (mg eq/kg)	0.073	0.068	3.169	0.278	0.382	Post hydrolysis
R2W56)						
IN-RYC33	1.8	1.5	10.3	4.6	2.3	-
Total IN-QEK31 metabolites	67.8	61.6	21.1	36.5	31.3	45.4
IN-VM862	-	-	0.3	-	-	-
Volatile	-	3.8	-	12.7	2.4	
Unretained	2.6	5.0	0.3	1.8	1.8	
Total unidentified metabolites	6.1[A]	1.4[B]	3.8[C]	3.5[D]	13.3[E]	19.3
Unextracted methanol:water	10.9	13.7	30.6	19.9	33.0	
acetonitrile:water	-	-	-	-	7.3	
Fluazaindolizine					0.5	
IN-QEK31					0.9	
Glycerol glucuronide of IN-QEK31					0.5	
Inositol conjugate of IN-QEK31 (IN-UHD13)					0.2	
Glucose conjugate of IN-QEK31 (IN-UGA20)					0.4	
IN-RYC33					0.2	
Unretained					0.3	
Total unidentified metabolites					4.2[F]	
Driselase					2.7	
0.1M HCl					1.4	
1M HCl					3.3	
0.1M NaOH					3.0	
Remaining					15.1	

Notes:
DAA = days after one or two soil applications.
[A] Two components, none >3.4 percent TRR, 0.002 mg eq/kg.
[B] Unidentified consisting of a single component.
[C] Unidentified consisting of 6 components. None >1.4 percent TRR, 0.043 mg eq/kg.
[D] Unidentified consisting of 3 components. None >1.5 percent TRR, 0.004 mg eq/kg.
[E] Unidentified consisting of 8 components. None >2.7 percent TRR, 0.010 mg eq/kg.
[F] Unidentified consisting of 22 components. None >0.4 percent TRR, 0.001 mg eq/kg.

Carrot roots

Extractability of ^{14}C in roots from the [IP-5,8a-^{14}C] experiment using methanol:water was good (86.3–89.1 percent TRR Table 11). Unextracted residues accounted for 10.9–13.7 percent TRR.

The principal extracted residue in the [IP-5,8a-^{14}C] experiment carrot roots was IN-QEK31 (53.7–66.0 percent TRR). Fluazaindolizine accounted for 11.9–12.6 percent TRR. Other identified metabolites were the glycerol glucuronide of IN-QEK31, methyl ester of IN-QEK31 (IN-R2W56), glucose conjugate of IN-QEK31 (IN-UGA20) and IN-RYC33 (≤ 3.3 percent TRR, 0.002 mg/kg). A volatile metabolite was also detected in the mature carrot root extracts during the concentration process and accounted for 3.8 percent TRR. Multiple unidentified metabolites were also detected accounting for an aggregate total of 1.4–6.1 percent TRR in each sample but each individually was ≤ 3.4 percent TRR (≤ 0.002 mg eq/kg).

Chiral analysis of metabolites

Chiral HPLC was conducted on IN-RSU03 and it was determined that only the R-enantiomer designated as IN-TMQ01 was present. As O-demethylation would not alter the chiral carbon, by analogy, only the R-enantiomer IN-TQD54 is expected in those metabolites as determined by the racemic standard IN-UNS90.

Proposed metabolic pathway of fluazaindolizine in carrots

The metabolic pathway for fluazaindolizine in carrot following soil application is presented in Figure 3 and is proposed based on the metabolites identified in foliage and roots. Metabolites in the figure are depicted as their free acid or free base forms and not as the various salt forms.

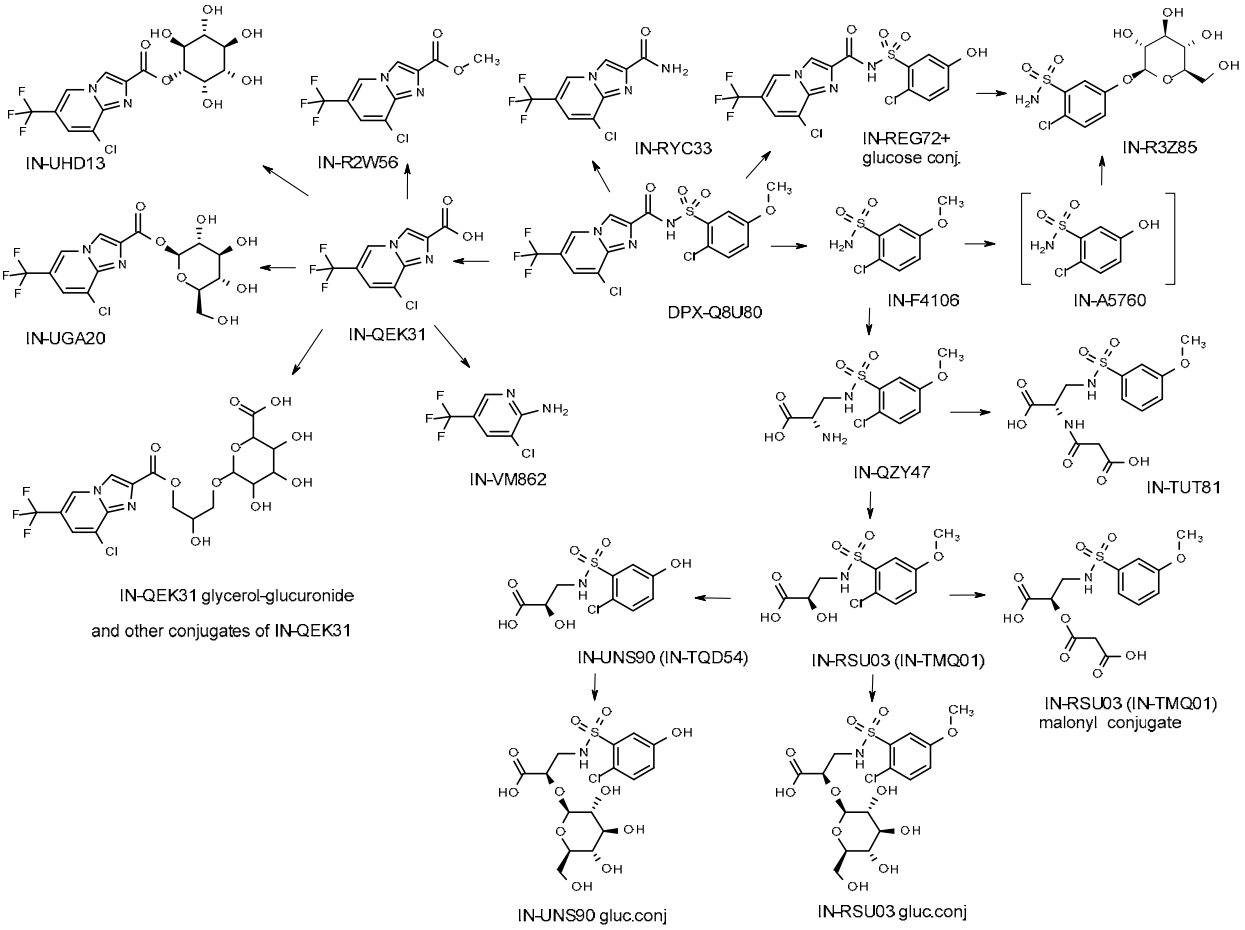


Figure 3 Proposed metabolic pathway of fluazaindolizine in carrots

Potato

The metabolism of [Ph-^{14}C(U)]-fluazaindolizine and [IP -5,8a-^{14}C]-fluazaindolizine in potatoes (cv. Maris Bard) was studied by Hobbs (2017 DuPont-43220). Seed potatoes were sown into treated sandy loam soil (pH 7.3, percent OM 2.4, 65 percent sand, 20 percent silt, 15 percent clay, CEC 10 meq/100 g) ca. 2 hours prior to a soil drench application with an SC formulation of [Ph-^{14}C]- or [IP-5,8a-^{14}C]-fluazaindolizine, at a nominal application rate of 1000 g ai/ha and maintained in a glasshouse. A further application at a nominal rate of 1.00 kg ai/ha was made to the same plots 30 days after the initial application. The total achieved treatment rates were 2.01 kg ai/ha and 2.00 kg ai/ha for the [Ph-^{14}C]- and [IP-5,8a-^{14}C]-fluazaindolizine experiments, respectively.

Foliage was taken for analysis 45 days after planting (15DAA2) for total radioactivity analysis only, tuber and foliage were taken for analysis at 65 and 100 days after planting and corresponded to an immature sample (35DAA2) and a mature sample (70DAA2). Samples were kept under frozen storage (*ca.* -20 °C) until analysis. Tuber samples were extracted within 14 days of harvest and the extracts were stored for no more than 39 days before initial chromatography. Overall, the time between the harvest and initial HPLC analysis was ≤ 53 days. Foliage samples were stored in a freezer set to maintain *ca.* -20 °C

from harvest until extraction. Overall, the time between the harvest and initial HPLC analysis of the foliage samples was ≤ 146 days. The results are shown in Table 12)

Table 12 TRR (mg eq/kg) in potato plant matrices following two applications of [^{14}C]-fluazaindolizine

Days after planting	Sample type	[Ph-^{14}C]-fluazaindolizine		[IP-5,8a-^{14}C]-fluazaindolizine	
		Tuber	Foliage	Tuber	Foliage
45	15DAA2	NA	0.277	NA	0.072
65	Immature 35DAA2	0.085	0.796	0.043	0.159
100	Mature 70DAA2	0.126	5.057	0.069	0.774

Identification and characterisation of radioactive residues [Ph-^{14}C]-fluazaindolizine

Potato tuber

Extractability of ^{14}C in tubers from the [Ph-^{14}C] experiment using methanol:water was good (76.6–80.4 percent TRR Table 13). Unextracted residues accounted for 19.6–23.4 percent TRR. The unextracted residues from mature tubers (70DAA2) were subject to further treatments which released an additional 15.1 percent TRR with 4.5 percent TRR remaining with the solids.

Fluazaindolizine was identified in the early tubers (6.8 percent TRR, 0.006 mg/kg) decreasing in later samples to where it was not detected. The principal extracted residue identified in [Ph-^{14}C]fluazaindolizine potato tubers was IN-QZY47 (15.3–15.4 percent TRR). IN-TUT81 (the malonyl conjugate of IN-QZY47) was present at 6.1–6.7 percent TRR) in immature and mature samples.

Other identified metabolites were IN-F4106, IN-A5760, IN-UNS90, IN-RSU03, glucose conjugate of IN-A5760 (IN-R3Z85), IN-REG72 and IN-UJV12 (all ≤6.6 percent TRR, ≤ 0.005 mg eq/kg). A number of metabolites were subsequently identified by LC-MS analysis including a glucose conjugate of IN-UNS90 (≤11.3 percent TRR, ≤ 0.014 mg eq/kg), and glucose conjugates of IN-RSU03 (≤10.1 percent TRR, ≤ 0.013 mg eq/kg).

Multiple unidentified metabolites were also detected in tuber samples accounting for aggregate totals of 16.4-24.2 percent TRR (0.013-0.031 mg eq/kg); each component ≤5.6 percent TRR (≤ 0.007 mg eq/kg). The majority of these multiple unidentified metabolites appeared to be more complex conjugates of IN-F4106 and/or IN-A5760, as determined after acid hydrolysis.

Following hydrolysis of the mature potato tuber aqueous methanol extract, the glucose conjugate of IN-RSU03 was hydrolysed to IN-RSU03. The malonyl conjugate of IN-QZY47 (IN-TUT81) was hydrolysed to IN-QZY47, and the glucose conjugate of IN-UNS90 was hydrolysed to IN-UNS90 (Table xx). An increase in IN-F4106, IN-UJV12 and IN-A5760 were also observed after hydrolysis. Other minor unidentified metabolites or conjugates found were also cleaved under these conditions releasing IN-F4106, IN-UJV12 and IN-A5760.

Potato foliage

Extractability of ^{14}C in foliage from the [Ph-^{14}C] experiment using methanol:water was good (76.0 percent TRR Table 13). Unextracted residues accounted for 23.9 percent TRR and were not examined further.

Fluazaindolizine was identified in low quantities in the potato foliage sample (0.2 percent TRR; 0.010 mg/kg). The principle extracted residues identified were metabolites associated with IN-RSU03 (including the malonyl and glucose conjugates) accounting in total for 12.0 percent TRR (0.606 mg eq/kg) and metabolites associated with IN-QZY47 (including the malonyl conjugate, IN-TUT81 and the acetyl conjugate) accounting in total for 14.9 percent TRR (0.750 mg eq/kg).

Other identified metabolites were IN-F4106, IN-A5760, IN-UNS90, glucose conjugate of IN-A5760 (IN-R3Z85) and IN-UJV12 (all ≤8.3 percent TRR, ≤ 0.422 mg eq/kg). A number of metabolites were subsequently identified by LC-MS analysis including a glucose conjugate of IN-UNS90 (5.6 percent TRR, 0.283 mg eq/kg) and malonyl and glucose conjugates of IN-RSU03 (≤8.1 percent TRR, ≤ 0.410 mg eq/kg).

Multiple unidentified metabolites were also detected accounting for an aggregate total of 21.2 percent TRR (1.076 mg eq/kg) in each foliage sample but each individually was ≤ 1.8 percent TRR (≤ 0.092 mg eq/kg). The majority of these multiple unidentified metabolites appeared to be more complex conjugates of IN-QZY47, IN-F4106 and/or IN-A5760, as determined after acid hydrolysis. As expected, following hydrolysis of the mature potato foliage aqueous methanol extract, the glucose and malonyl conjugates of IN-RSU03 were hydrolysed to IN-RSU03. The malonyl conjugate of IN-QZY47 (IN-TUT81) and acetyl conjugate of IN-QZY47 were hydrolysed to IN-QZY47, and the glucose conjugate of IN-UNS90 was hydrolysed to IN-UNS90 (Table 13). An increase in IN-F4106, IN-UJV12 and IN-A5760 were also observed after hydrolysis; it was apparent that other minor unidentified metabolites or conjugates were also cleaved under these conditions and released as IN-F4106, IN-UJV12 and IN-A5760 (Table 13).

Table 13 Identification of TRR (% TRR) from various potato tuber and foliage extracts and fractions after application of [Ph-^{14}C]-fluazaindolizine

	Immature Tuber 35DAA2	Mature Tuber 70DAA2		Mature Foliage 70DAA2	
TRR (mg eq/kg)	0.085	0.126	Post hydrolysis	5.057	Post hydrolysis
Extracted (methanol water)	76.6	80.4		76.0	
SPE fraction, retained+unretained	47.1+29.5				
Fluazaindolizine	6.8	-	-	0.2	-
IN-REG72	0.4	-	-	-	-
Total whole molecule metabolites	7.2	-	-	0.2	-
IN-RSU03 (IN-TMQ01)	1.0	-	11.6	2.4	12.4
Malonyl Conjugate of IN-RSU03 (IN-TMQ01)	-	-	-	1.5	-
Glucose Conjugate of IN-RSU03	6.5+0.6	10.1	-	8.1	-
Total IN-RSU03 metabolites	8.1	10.1	11.6	12.0	12.4
IN-QZY47	14.8+0.5	15.4	22.9	5.3	15.1
Malonic acid conjugate of IN-QZY47 (IN-TUT81)	2.4+3.7	6.7	0.8	5.2	0.7
Acetyl Conjugate of IN-QZY47	-	-	-	4.4	-
Total IN-QZY47 metabolites	21.4	22.1	23.2	14.9	15.8
IN-UNS90 (IN-TQD54)	1.1+0.2	0.8	15.1	3.3	9.9
Glucose Conjugate of IN-UNS90 (IN-TQD54)	0.9+7.8	11.3	5.0	5.6	4.6
Total IN-UNS90 metabolites	10.0	12.1	20.1	8.9	14.5
Glucose conjugate of IN-A5760 (IN-R3Z85)	0.7+0.6	1.2	-	8.3	-
IN-A5760	1.9+0.2	0.6	3.9	4.2	12.1
Total IN-A5760 metabolites	3.4	1.8	3.9	12.5	12.1
IN-F4106	6.4+0.2	1.1	6.0	3.2	4.2
IN-UJV12	0.8+0.6	2.8	6.3	1.6	5.2
Unretained		6.1		1.6	
Total unidentified metabolite	3.5+12.9[A]	24.2[B]	8.7[D]	21.2[C]	9.7[E]
Unextracted methanol:water	23.4	19.6		23.9	
Acetonitrile:water	-	4.6		-	
Water		1.4			
Driselase		1.7			
Amylase		3.9			
1M HCl		3.5			

	Immature Tuber 35DAA2	Mature Tuber 70DAA2		Mature Foliage 70DAA2	
TRR (mg eq/kg)	0.085	0.126	Post hydrolysis	5.057	Post hydrolysis
Remaining		4.5			

Notes:

[A] Unidentified consisting of 18 components. None >3.9 percent TRR (0.003 mg eq/kg).

[B] Unidentified consisting of 16 components. None >5.6 percent TRR (0.007 mg eq/kg).

[C] Unidentified consisting of 31 components. None >1.8 percent TRR (0.092 mg eq/kg).

[D] Unidentified consisting of 12 components. None >1.6 percent TRR (0.002 mg eq/kg).

[E] Unidentified consisting of 14 components. None >1.5 percent TRR (0.075 mg eq/kg).

NOTE the x+y results are for SPE fraction, retained + unretained where relevant!

Identification and characterisation of radioactive residues [IP-5,8a-^{14}C]fluazaindolizine

Potato tuber

Extractability of ^{14}C in tubers from the [IP-5,8a-^{14}C] experiment using methanol:water was good (80.8-84.1 percent TRR Table 14). Unextracted residues accounted for 15.9-19.2 percent TRR. The unextracted residues from mature tubers (70DAA2) were subject to further treatments which released an additional 10.8 percent TRR with 8.4 percent TRR remaining with the solids.

Fluazaindolizine was identified in the early tubers (9.3 percent TRR, 0.004 mg/kg) with non-detectable residues found in the mature tubers. The principal extracted residue was IN-QEK31 (59.3-65.0 percent TRR). Other identified metabolites were glucose conjugate of IN-QEK31 (IN-UGA20), inositol conjugate of IN-QEK31 (IN-UHD13) and methyl ester of IN-QEK31 (IN-R2W56), (all ≤ 3.5 percent TRR, ≤ 0.002 mg eq/kg). Multiple unidentified metabolites were also detected accounting for an aggregate total of 3.6-8.2 percent TRR in each sample but each individually was ≤ 1.7 percent TRR (≤ 0.001 mg eq/kg). A volatile component was trapped only in the mature potato extract (2.7 percent TRR; 0.002 mg eq/kg) but was not characterised further due to its low level.

Following hydrolysis of the mature potato tubers aqueous methanol extract the fluazaindolizine and IN-R2W56 were hydrolysed to IN-QEK31 (Table 14).

Potato foliage

Extractability of ^{14}C in foliage from the [IP-5,8a-^{14}C] experiment using methanol:water was good (73.8 percent TRR Table 14). Unextracted residues accounted for 26.2 percent TRR.

Fluazaindolizine was identified in small quantities in the foliage (1.9 percent TRR, 0.015 mg/kg). The principle extractable residue identified was IN-QEK31 accounting for 18.5 percent TRR (0.143 mg eq/kg). Other identified metabolites were IN-REG72, glucose conjugate of IN-QEK31 (IN-UGA20), IN-RYC33 and methyl ester of IN-QEK31 (IN-R2W56), (all ≤6.3 percent TRR, ≤ 0.049 mg eq/kg). Multiple unidentified metabolites were also detected accounting for an aggregate total of 37.7 percent TRR but each individually was ≤ 3.5 percent TRR (≤ 0.027 mg eq/kg).

Following hydrolysis of the mature potato foliage aqueous methanol extract the following metabolites were hydrolysed to IN-QEK31: fluazaindolizine, IN-REG72, IN-UGA20, IN-R2W56 and IN-RYC33 (Table 14). It was apparent that other minor unidentified metabolites or conjugates also cleaved under these conditions releasing IN-QEK31 or minor unknown metabolites.

Table 14 Identification of TRR (% TRR) in various potato tuber and foliage extracts and fractions after application of [IP-5,8a-^{14}C]fluazaindolizine

	Immature Tuber 35DAA2	Mature Tuber 70DAA2	Mature Foliage 70DAA2	Mature Foliage (70DAA2)
TRR (mg eq/kg)	0.043	0.069	0.774	Post hydrolysis
Extracted (methanol water)	84.1	80.8	73.8	
Fluazaindolizine	9.3	-	1.9	-
IN-REG72	-	-	0.7	-
Total intact molecule metabolites	9.3	-	2.6	-
Inositol conjugate of IN-QEK31 (IN-UHD13)	-	0.8	-	2.5
Glucose conjugate of IN-QEK31 (IN-UGA20)	0.9	0.9	6.3	0.4
IN-QEK31	65.0	59.3	18.5	47.0
Methyl ester of IN-QEK31 (IN-R2W56)	3.5	2.9	1.0	-
IN-RYC33	-	-	0.9	-
Total IN-QEK31 metabolites	69.4	63.9	26.7	49.5
Total unidentified metabolites	3.6[A]	8.2[A]	37.7[A]	23.3[B]
Unextracted (methanol:water)	15.9	19.2	26.2	
Acetonitrile:water	-	2.4	-	
Water		0.7		
Driselase		2.1		
Amylase		5.6		
remaining		8.4		

Notes:

[A] Total unidentified metabolites consist of multiple components – none greater than 1.7 percent TRR, 0.001 mg eq/kg in the tubers and 3.5 percent TRR, 0.027 mg eq/kg in the foliage, several of which appear to be hydrolysed with 1M HCl to IN-QEK31.

[B] Post-hydrolysis: Unidentified consisting of 11 components. None >5.2 percent TRR (0.041 mg eq/kg).

Chiral analysis of metabolites

Chiral HPLC was conducted on isolates from [Ph-^{14}C]-fluazaindolizine experiment foliage containing components corresponding to the racemic compounds IN-RSU03, IN-UTG08 and IN-UNS90. Only the R-enantiomer of IN-RSU03, designated as IN-TMQ01, was present. IN-UNS90 was also identified as only the R-enantiomer, IN-TQD54, consistent with IN-TMQ01 as O-demethylation would not alter the chiral carbon. Residues measured as IN-QZY47 were confirmed to be in the S configuration, consistent with naturally occurring L-serine.

The metabolic pathway for fluazaindolizine in potato plants following soil application is presented in Figure 4 and was proposed based on the metabolites identified in tubers and foliage. Metabolites in the figure are depicted in their free acid or free base forms.

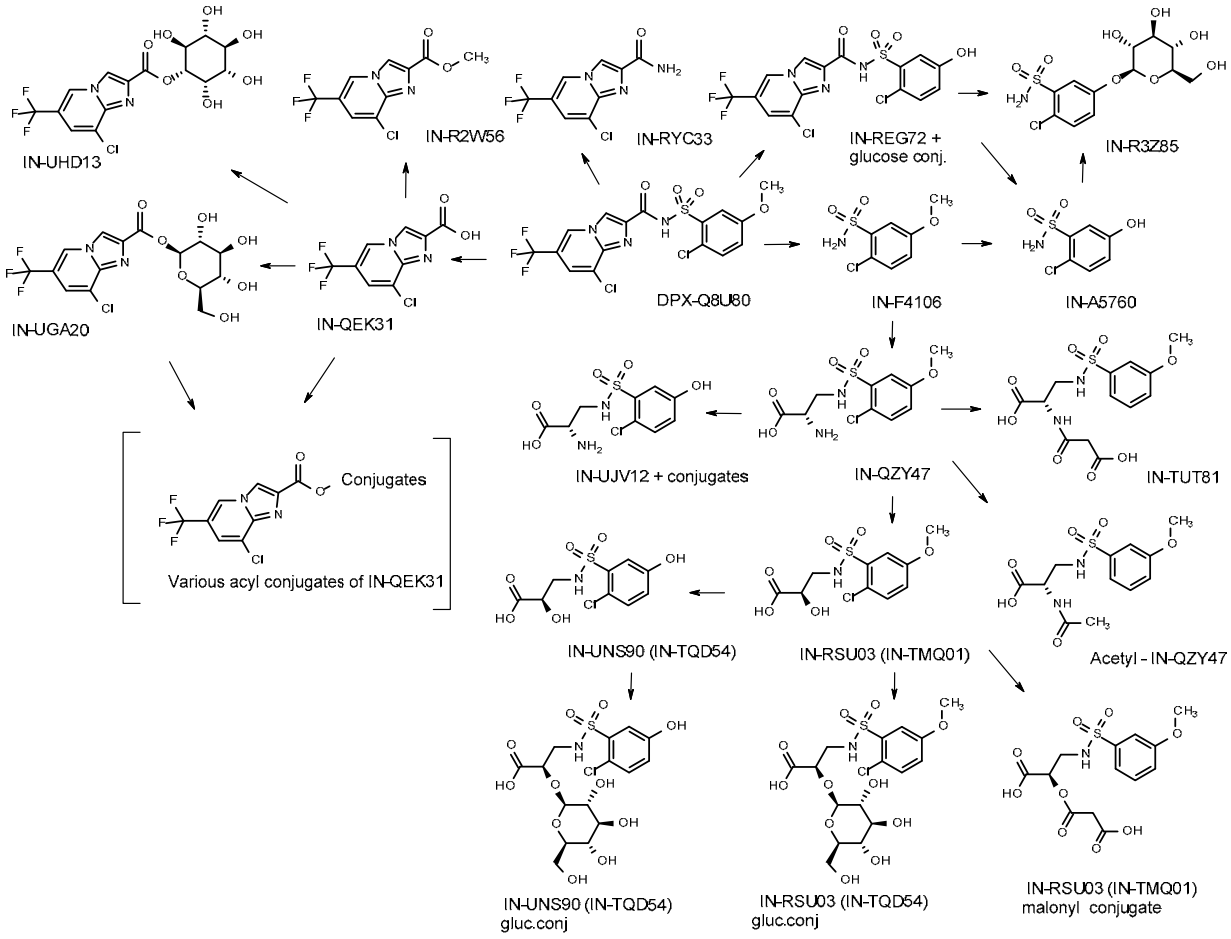


Figure 4 Proposed metabolic pathway of fluazaindolizine in potato plant

Sugar cane

Begley and Lloyd (2017 DuPont-41849) studied the metabolic fate of [phenyl-^{14}C(U)]fluazaindolizine and [imidazo[1,2-a]pyridine-5,8a-^{14}C]fluazaindolizine in sugar cane plants.

Mature sugar cane sets (cv. NC0310) were propagated in a sand/loam mixture until reaching 2–3 leaf stage (BBCH 12) at which time they were transplanted into crates filled with a sandy loam soil (pH 7.3, 2.4 percentOM, 64 percent sand, 21 percent silt, 15 percent clay, CEC 10 meq/100g). [Ph-^{14}C]fluazaindolizine or [IP-5,8a-^{14}C]fluazaindolizine was applied as an SC formulation at a nominal rate of 1.0 kg ai/ha, as a soil drench application within 2 hours of transplant. The achieved application rates were 1.00 kg ai/ha and 1.01 kg ai/ha for [Ph-^{14}C]-fluazaindolizine and [IP-5,8a-^{14}C]-fluazaindolizine, respectively.

Whole plant samples, consisting of mostly foliage, were taken at 51 days after application at BBCH 32 and whole plants above soil level at maturity (BBCH 39) which was 231 days after application. At maturity, the sample was separated into foliage and mature cane.

Samples were stored at *ca.* -20 °C prior to analysis. Plant samples were extracted within 17 days of harvest and the extracts were stored for no more than 10 days before initial chromatography. Overall, the time between the harvest and initial HPLC analysis was 23 days. Sample extracts were also reconstituted in more aqueous solvent and the HPLC analysis repeated, and in these cases, the time between harvest and this HPLC analysis was no greater than 71 days.

Table 15 TRR (mg eq/kg) in sugar cane plant matrices following one application of [^{14}C]-fluazaindolizine

Days after application	Sample type	[Ph-^{14}C]-fluazaindolizine	[IP-5,8a-^{14}C]-fluazaindolizine
51	Foliage	0.162	0.087
231	Foliage	0.069	0.121
231	Cane	0.020	0.052

Identification and characterisation of radioactive residues in sugar cane plants grown in [Ph-^{14}C]fluazaindolizine treated soil

Extractability of ^{14}C in foliage using methanol:water was good (82.9-91.5 percent TRR Table 16) and in mature cane 80.8 percent TRR. Foliage from 51DAA were subject to further solvent (acetonitrile/water), enzyme and acid treatment which increased the ^{14}C recovered to >89 percent TRR with 4.0-10.8 percent TRR remaining in the solids.

Sugar Cane Foliage

The principal extracted residue identified in [Ph-^{14}C] experiment sugar cane foliage was IN-RSU03 (IN-TMQ01) and its glucose and malonyl conjugates. The IN-RSU03 glucose conjugate was the principal conjugate detected accounting for 37.6–40.6 percent TRR (Table 16). The malonyl conjugate was detected at lower levels accounting for 1.6–6.7 percent TRR and IN-RSU03 was also identified at 2.0–9.6 percent TRR. IN-UNS90 was detected at 7.1–12.4 percent TRR and its glucose conjugate at 14.3–15.4 percent TRR (Table 16).

Multiple unidentified metabolites were also detected accounting for an aggregate total of 6.6–7.1 percent TRR in each sample but individually none >2.3 percent TRR (0.004 mg eq/kg).

Acid hydrolysis experiments demonstrated the residues were converted to the expected hydrolysis products. The conjugates of IN-RSU03 being converted to IN-RSU03, the glucose conjugates of IN-UNS90 being converted to IN-UNS90 and glucose conjugate of IN-A5760 (IN-R3Z85) being converted to IN-A5760 (Table 16).

Mature Sugar Cane

The principal extracted residue identified in [Ph-^{14}C] experiment mature sugar cane was IN-RSU03 glucose conjugate (19.4 percent TRR, 0.004 mg/kg, Table 16). Lower levels of the malonyl conjugate of IN-RSU03 were also detected accounting for 2.2 percent TRR. IN-R3Z85 (the glucose conjugate of IN-A5760) was the second highest residue detected (15.8 percent TRR). IN-UNS90 was detected at 4.0 percent TRR and its glucose conjugate at 4.9 percent TRR. The IN-RSU03 conjugates and IN-UNS90 and its conjugates were identified by comparison of retention time with the residues identified by LC-MS in the 51 DAA foliage. The identification of IN-R3Z85 by co-chromatography, in one system, with the provided reference standards, was supported by the acid hydrolysis experiments and the analysis of the 51 DAA foliage by LC-MS through the detection of the in-source fragment, IN-A5760.

IN-QZY47, IN-A5760, IN-TUT81 and IN-UJV12 were also identified by co-chromatography, in one system, with the provided reference standards (all ≤4.9 percent TRR, ≤ 0.001 mg eq/kg).

Multiple unidentified metabolites were also detected accounting for an aggregate total of 19.0 percent TRR (<0.005 mg eq/kg) but individually none >7.0 percent TRR (0.001 mg eq/kg).

Acid hydrolysis experiments showed the residues were converted to the expected hydrolysis products. The glucose conjugate of IN-RSU03 was converted to IN-RSU03, the glucose conjugate of IN-

UNS90 converted to IN-UNS90 and IN-R3Z85 converted to IN-A5760. Acid hydrolysis converted a significant portion of the unidentified metabolites to IN-UJV12 (Table 16).

Table 16 Identification of TRR (% TRR) in various sugar cane extracts and fractions after application of [Ph-^{14}C]fluazaindolizine

	Foliage (51 DAA)	Foliage (231 DAA)	Mature cane (231 DAA)	
TRR (mg eq/kg)	0.162	0.069	0.020	Post hydrolysis
Extracted methanol:water	91.5	82.9	80.8	
Malonic acid conjugate of IN-QZY47 (IN-TUT81)	1.3	1.0	2.3	-
IN-QZY47	-	-	3.8	4.9
Total IN-QZY47 metabolites	1.3	1.0	6.1	4.9
IN-RSU03	9.6	2.0	-	22.1
Glucose Conjugate of IN-RSU03	37.6	40.6	19.4	3.6
Malonyl Conjugate of IN-RSU03	6.7	1.6	2.2	-
Total IN-RSU03 metabolites	53.9	44.2	21.6	25.7
IN-UNS90	7.1	12.4	4.0	12.3
Glucose Conjugate of IN-UNS90	15.4	14.3	4.9	-
Total IN-UNS90 metabolites	22.5	26.7	8.9	12.3
IN-UJV12	-	-	4.9	8.6
IN-A5760	1.5	1.9	4.6	23.3
Glucose conjugate of IN-A5760 (IN-R3Z85)	5.9	1.9	15.8	-
Total IN-A5760 metabolites	7.4	3.8	20.4	23.3
Unretained	-	1.9	2.2	
Total unidentified metabolites	6.6[A]	5.2[A]	16.8[A]	6.1[A]
Unextracted methanol:water	8.5	17.1	19.2	
Acetonitrile:water	1.6	-	3.3	
Water	0.3		1.0	
1M HCl	2.5		4.2	
Remaining	4.0		10.8	

Notes:

[A] Total unidentified metabolites consist of multiple components – none greater than 7.0 percent TRR, 0.001 mg eq/kg in the cane and 2.3 percent TRR, 0.004 mg eq/kg in the foliage.

Identification and characterisation of radioactive residues in sugar cane plants grown in [IP-5,8a-^{14}C]fluazaindolizine treated soil

Extractability of ^{14}C in foliage using methanol:water was good (69.0–76.1 percent TRR Table 17) and in mature cane 82.0 percent TRR. Foliage and mature cane from 51DAA were subject to further solvent (acetonitrile/water), enzyme and acid treatment which increased the ^{14}C recovered to >92 percent TRR with 7.3–9.5 percent TRR remaining in the solids.

Sugar Cane Foliage

The principal extracted residue IN-QEK31 accounted for 27.9 percent TRR in the 51 DAA foliage and decreased to 5.1 percent TRR in the 231 DAA foliage (Table 17). IN-QEK31 was also detected as the glucose conjugate, IN-UGA20, accounting for 15.3 percent TRR in 51 DAA foliage and 8.5 percent TRR in 231 DAA foliage and the methyl ester, IN-R2W56 accounting for 1.3 percent TRR in 51 DAA foliage and 12.4 percent TRR in 231 DAA foliage. The identity of IN-QEK31 and its conjugates IN-UGA20 and IN-R2W56 were confirmed by LC-MS.

IN-RYC33, IN-REG72 and its glucose conjugate were also detected at low levels (≤4.6 percent TRR, ≤ 0.004 mg eq/kg). The glucose conjugate of IN-REG72 was tentatively identified by LC-MS. Multiple unidentified metabolites were also detected accounting for an aggregate total of 25.5-36.7 percent TRR in each sample but individually none >6.2 percent TRR (0.007 mg eq/kg). Acid hydrolysis experiments demonstrated that most of these low-level metabolites were hydrolysed to IN-QEK31 with the post-hydrolysis extract. The IN-QEK31 associated metabolites accounted for 64.5 percent TRR (0.056 mg eq/kg) in 51 DAA foliage and 35.1 percent TRR (0.042 mg eq/kg) in 231 DAA foliage (Table 17).

Mature Sugar Cane

The principal extracted residue identified in sugar cane was IN-QEK31 and associated metabolites. IN-QEK31 accounted for 13.5 percent TRR, the glucose conjugate of IN-QEK31 (IN-UGA20) accounted for 31.7 percent TRR and the methyl ester of IN-QEK31 (IN-R2W56), accounted for 3.9 percent TRR. The identity of IN-QEK31 and its conjugates, IN-UGA20 and IN-R2W56, were confirmed by LC-MS in the [IP-5,8a-^{14}C]-fluazaindolizine 51 DAA foliage extract.

The acid hydrolysis experiments supported the identifications showing that IN-QEK31 and associated metabolites accounted for 65.4 percent TRR (Table 17).

The glucose conjugate of IN-REG72 was also detected at low levels (3.9 percent TRR) and was tentatively identified by retention time match with the metabolite identification in [IP-5,8a-^{14}C]fluazaindolizine 51 DAA foliage extract by LC-MS.

Multiple unidentified metabolites were also detected accounting for an aggregate total of 19.5 percent TRR but individually none >3.1 percent TRR (0.002 mg eq/kg).

Table 17 Identification of TRR (% TRR) in various sugar cane extracts and fractions after application of [IP-5,8a-^{14}C]-fluazaindolizine

	Foliage 51 DAA	Foliage 231 DAA	Mature cane 231 DAA	
TRR (mg eq/kg)	0.087	0.121	0.052	Post hydrolysis
Extracted methanol:water	76.1	69.0	82.0	
IN-QEK31	*27.9*	*5.1*	*13.5*	*61.9*
Glucose conjugate of IN-QEK31 (IN-UGA20)	*15.3*	*8.5*	*31.7*	*3.5*
Methyl ester of IN-QEK31 (IN-R2W56)	*1.3*	*12.4*	*3.9*	-
IN-RYC33	*1.6*	*1.7*		-
Total IN-QEK31 metabolites	*46.1*	*27.7*	*49.1*	*65.4*
Glucose conjugate of IN-REG72	*4.6*	*2.9*	*3.9*	-
IN-REG72		*1.8*		-
RT 19.12 mins				*1.5*
Unretained	*3.9*	*5.8*	*2.9*	-
Total unidentified metabolites	*21.6*[A]	*30.9*[A]	*16.6*[A]	*5.7*[A]
Unextracted methanol:water	23.9	31.0	18.0	
Acetonitrile:water	3.7	-	5.2	
Water	0.8		1.4	
Drielase	3.0			
1M HCl	6.9		4.1	
Remaining	9.5		7.3	

Notes:

[A] Total unidentified metabolites consist of multiple components – none greater than 3.1 percent TRR, 0.002 mg eq/kg in the cane and 6.2 percent TRR, 0.007 mg eq/kg in the foliage.

Proposed metabolic pathway of fluazaindolizine in sugar cane

The metabolic pathway for fluazaindolizine in sugar cane is presented in Figure 5 and is proposed based on the metabolites identified in sugar cane and sugar cane foliage. Metabolites in the figure are depicted in their free acid or base forms.

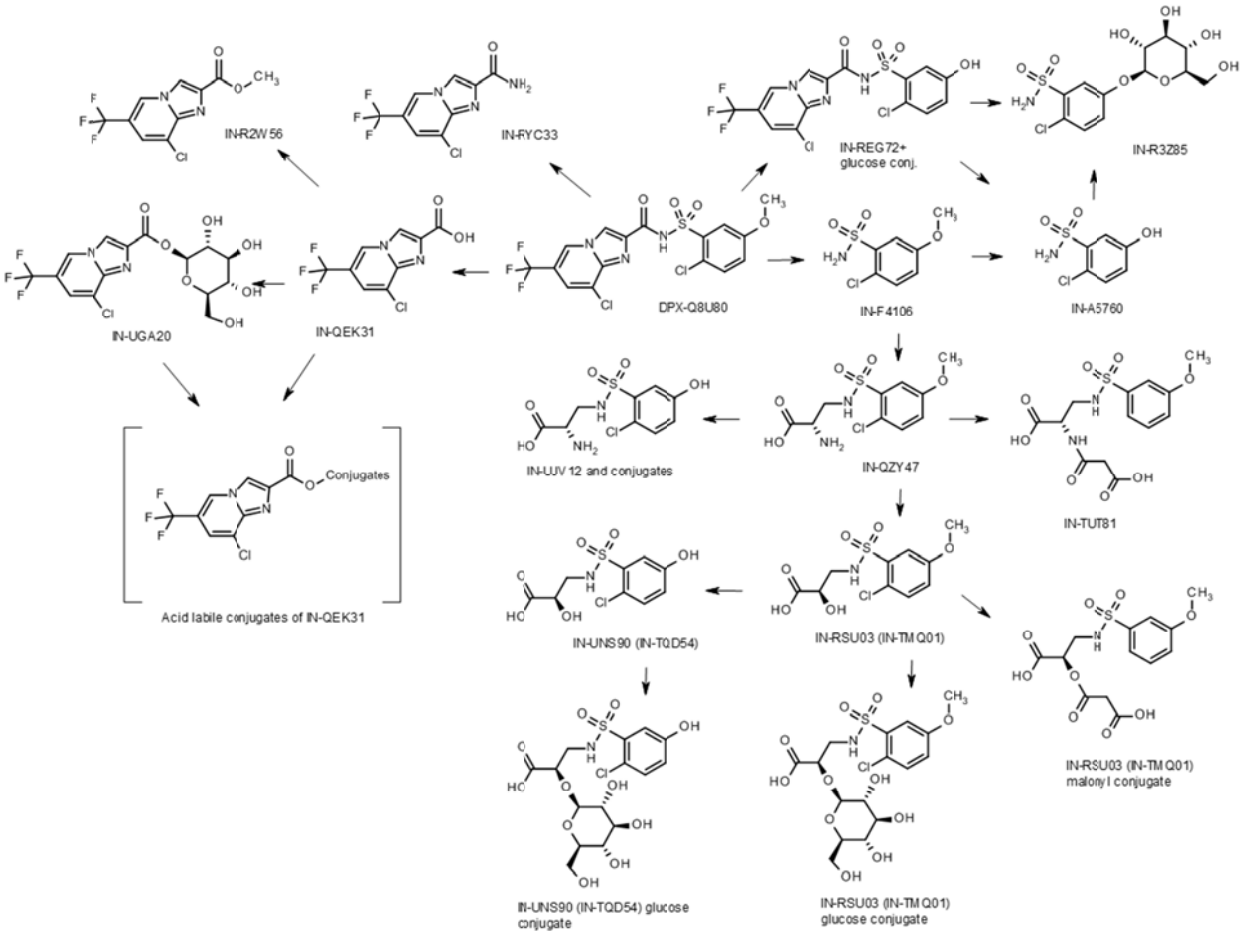


Figure 5 Proposed metabolic pathway of fluazaindolizine in sugar cane

Metabolism in rotational crops

Hobbs (2019 DuPont-34945, Revision No. 1) studied the metabolic fate of [Ph-^{14}C]fluazaindolizine and [IP-5,8a-^{14}C]fluazaindolizine in succeeding and rotated crops of spring wheat, spinach, and radish.

Single spray applications of SC formulations of [Ph-^{14}C]fluazaindolizine or [IP-5,8a-^{14}C]fluazaindolizine, at a nominal application rate of 2.0 kg ai/ha were made to bare sandy loam soil (pH 6.0, 5.1 percentOM, 64 percent sand, 19 percent silt, 17 percent clay, CEC 15 meq/100 g). The total achieved treatment rates were 1.95 and 1.94 kg ai/ha for the [Ph-^{14}C] and [IP-5,8a-^{14}C] labels, respectively.

Soil aging and crop maintenance were performed in a glasshouse. Seeds of wheat (cv Paragon), spinach (cv Renegade F1) and radish (cv Helox F1) were subsequently sown into the soil at 30, 120, and 300 days after application (DAA) and grown to maturity. Wheat forage, hay, straw and grain, spinach foliage, radish foliage (immature and mature foliage for both spinach and radish), and radish roots were sampled. Soil samples were collected following application, at each sowing point and at crop maturity.

Following harvest, samples were stored in a freezer set to maintain -20 °C within 2 hours, maintained under these conditions until processed, and returned to storage in the freezer once processing was complete. Plant samples were generally extracted within 3 months of harvest and the extracts were generally stored for no more than 3 months before initial chromatography; however, some samples were extracted beyond 6 months of harvest and extracts stored for longer than 6 months prior to chromatographic analysis due to additional HPLC method development needed to resolve various metabolites. Some extracts were also reanalysed at later time points using these HPLC methods and enabled efficient isolation of peaks of interest for LC-MS identification.

Soil extraction and analysis

Subsamples (ca. 50 g) of selected soil cores, 0, 71 (30DAA mature radish harvest), 120, and 300DAA, were extracted with acetonitrile:0.2 percent formic acid (aq) 9:1 followed by acetonitrile:0.2 percent formic acid (aq) 4:1 and two further extractions with acetonitrile:0.2 percent formic acid (aq) 1:1 . The unextracted radioactivity (bound radioactivity in the soil) was determined by combustion analysis. Extracted residues were analysed by HPLC. Concentration of the extracts from the [IP-5,8a-^{14}C]fluazaindolizine treated soil were concentrated in conjunction with trapping devises to capture any volatile metabolites as observed in previous soil metabolism studies. Identification of ^{14}C -residues was accomplished by either HPLC, or LC-MS with reference to authenticated analytical standards.

Portions of each homogenised crop sample were then extracted three times using methanol:water (7:3). Where necessary samples were further extracted with acetonitrile:water (1:1, 50 °C, 30 min), subjected to enzymatic digestion with driselase (an enzymatic mixture containing cellulase, laminarinase, and xylanase at 37 °C, 24 h; twice) and extracted with 0.1 M HCl (50 or 60 °C, 6 hrs), 0.1 M NaOH (80 °C, 4 hrs), and 1 M HCl (80 °C, 20 hrs). The results are shown in Table 18.

Table 18 TRR (mg eq/kg) found in soil

Radiolabel	Day 0 Application	Day 30 Sowing	Day 71 Mature Radish	Day 120 Sowing	Day 300 Sowing
[Ph-^{14}C]-fluazaindolizine	3.074	1.746	1.556	0.795	0.491
[IP-5,8a-^{14}C]-fluazaindolizine	4.655	3.628	0.456	0.662	1.307

Fluazaindolizine, IN-F4106, and IN-QEK31 were the major residues extracted from soil which were available for uptake by plants. IN-F4106, and IN-QEK31 exceeded 50 percent of the residue in the 120-300DAA samples, while IN-RYC33, IN-REG72, and IN-A5760 were found between 2.3 and 11.7 percent TRR at various sampling intervals. IN-VM862 was only presnt at low levels (<4 percent TRR determined *via* capture of volatile residues). The major metabolic route in the soil was the hydrolysis of fluazaindolizine at the amide bond, resulting in IN-F4106 and IN-QEK31. Fluazaindolizine was also *O*-demethylated to form IN-REG72, which also was hydrolysed to IN-A5760 and IN-QEK31. Less prominent pathways were hydrolysis of fluazaindolizine at the sulfonamide bond, resulting in IN-RYC33 and the further degradation of IN-QEK31 to IN-VM862.

TRR values expressed as mg/kg equivalents of the parent fluazaindolizine in the various commodities at the 30, 120, and 300DAA plant-back intervals are shown in Table 19.

Table 19 TRR (mg eq/kg) in the various commodities at the 30, 120, and 300 plant-back intervals

		Wheat				Spinach		Radish		
		Forage	Hay	Straw	Grain	Immature	Mature	Immature Foliage	Mature Foliage	Mature Roots
[Ph]	30DAA	1.165	1.433	6.873	0.086	0.254	0.647	0.342	0.328	0.388

		Wheat				Spinach		Radish		
		Forage	Hay	Straw	Grain	Immature	Mature	Immature Foliage	Mature Foliage	Mature Roots
Label	120DAA	0.422	0.334	2.559	0.055	0.052	0.095	0.062	0.054	0.131
	300DAA	0.396	0.531	2.741	0.026	0.087	0.147	0.056	0.103	0.054
[IP-5,8a Label	30DAA	0.411	1.143	3.547	1.517	0.116	0.520	0.329	0.537	0.277
	120DAA	0.198	0.377	1.357	0.521	0.018	0.043	0.049	0.064	0.037
	300DAA	0.609	0.969	4.073	1.296	0.167	0.233	0.092	0.200	0.051

In general, residues were lower with longer PBIs apart from wheat commodities from the [IP-5,8a-^{14}C]fluazaindolizine soil application, where TRRs in the various commodities at the 30 and 300 day PBIs were comparable.

Most residues were readily extracted across all commodities using a methanol:water mixture (70:30). In cases where residues were more extensively incorporated into the crop matrix such as wheat straw or grain samples, additional enzymatic and acid treatments allowed for recovery of >90 percent TRR. Extracted residues containing significant radioactivity (≥0.01 mg equiv/kg) were analysed by HPLC. Identification of ^{14}C-residues was accomplished by either HPLC, or LC-MS with reference to authenticated analytical standards.

Uptake and metabolism of fluazaindolizine in rotated crops

Metabolic pathways of fluazaindolizine and its soil metabolites were similar among all the rotational crops and rotational intervals. Differences observed in the various crops were mainly in the degree and type of more complex conjugation with endogenous constituents.

Spinach

TRR for spinach commodities for the various plant-back intervals are presented in Table 20. The highest concentrations of total radioactivity were found in the mature samples.

Table 20 TRR (mg eq/kg) in spinach 30, 120, and 300 days after a soil drench application of [^{14}C]fluazaindolizine

Days after application	[Ph-^{14}C]-fluazaindolizine		[IP-5,8a-^{14}C]-fluazaindolizine	
	Immature	Mature	Immature	Mature
30	0.254	0.647	0.166	0.520
120	0.052	0.095	0.18	0.043
300	0.087	0.147	0.167	0.233

Identification and characterization of radioactive residues [Ph-^{14}C]-fluazaindolizine in spinach

Extractability of ^{14}C in [Ph-^{14}C] spinach using methanol:water was good (85.2-95.2 percent TRR Table 21). The unextracted residues in the 30DAA sample was subject to further investigations; these further treatments released almost all the ^{14}C unextracted by methanol:water (Table 21).

Fluazaindolizine was found at all plant-back intervals (0.4-14.1 percent TRR, ≤ 0.091 mg/kg) decreasing in later samples. The principal extracted residue was IN-TUT81 (43.9-68.8 percent TRR) which is derived from *N*-malonyl conjugation of IN-QZY47. An acetylated derivative of IN-QZY47 identified by LC-MS accounted for 1.7-8.0 percent TRR (0.001-0.028 mg eq/kg) and IN-QZY47 for 0.9-2.4 percent TRR (0.001-0.010 mg eq/kg). The total of IN-QZY47 derived metabolites (IN-TUT81, acetylated IN-QZY47, and

IN-QZY47) accounted for most of the spinach residues comprising of 49.7-75.7 percent TRR (0.035-0.322 mg eq/kg: Table 22).

Other identified metabolites were the IN-REG72, IN-RSU03, IN-UNS90, IN-A5760, IN-UJV12, glucose conjugate of IN-A5760 (IN-R3Z85), and IN-F4106, (all ≤5.1 percent TRR, ≤ 0.033 mg eq/kg) as well as glucose conjugates of IN-REG72 and IN-RSU03 (≤ 3.3 percent TRR, ≤ 0.021 mg eq/kg; Table 22).

Multiple unidentified metabolites were also detected accounting for an aggregate total of 6.6-14.2 percent TRR in each sample but each individually was ≤ 2.8 percent TRR (≤ 0.013 mg eq/kg). Most of these metabolites were hydrolysed to IN-F4106, IN-RSU03, IN-QZY47, or IN-UNS90 among other known metabolites (Table 22).

Table 21 Extraction of residues in spinach sown 30, 120, and 300 days after application of [Ph–^{14}C]-fluazaindolizine

	30DAA		120DAA		300DAA	
	Immature	Mature	Immature	Mature	Immature	Mature
TRR (mg eq/kg)	0.254	0.052	0.087	0.647	0.095	0.147
Extracted (MeOH/water), % TRR	85.2	90.1	92.9	94.8	95.2	92.8
Unextracted (MeOH/water), % TRR	14.7[A]	9.9	7.0	5.1	4.8	7.2

Notes:

[A] Exhaustive extraction only conducted on immature 30DAA spinach, additional radioactivity released included: Aqueous soak 1 (overnight) 1.1 percent TRR, 0.003 mg eq/kg, Acetonitrile:water 2.3 percent TRR, 0.006 mg eq/kg, Aqueous soak 2 (*ca.* 5 h, ambient) <LOD, Enzyme (Driselase) 8.1 percent TRR, 0.021 mg eq/kg, 0.1M HCl (*ca.* 50-60°C, 6 h) 1.7 percent TRR, 0.004 mg eq/kg, 1.3M HCl (*ca.* 80°C, 20 h) 0.9 percent TRR, 0.002 mg eq/kg, 0.1M NaOH (*ca.* 80°C, 4 h) 0.5 percent TRR, 0.001 mg eq/kg, entire solids were consumed in exhaustive extraction.

Table 22 Identification of TRR (% TRR) from spinach in various extracts and fractions from crops sown 30, 120, and 300 days after application of [Ph-^{14}C]-fluazaindolizine

	30DAA				120DAA		300DAA	
	Immature	Post hydrolysis	Mature	Post hydrolysis	Immature	Mature	Immature	Mature
TRR (mg eq/kg)	0.254		0.052		0.087	0.647	0.095	0.147
Fluazaindolizine	10.3	-	14.1	-	4.4	3.0	1.5	0.4
IN-REG72	1.2	-	2.0	-	-	-	-	-
Glucose Conjugate of IN-REG72	0.8	-	3.3	-	-	1.0	-	0.3
Total whole molecule metabolites	12.3	-	19.4	-	4.4	4.0	1.5	0.7
IN-RSU03	1.9	2.6	5.1	9.2	2.1	1.8	3.1	5.2
Glucose Conjugate of IN-RSU03	2.8	-	0.5	-	-	-	-	-
Total IN-RSU03 metabolites	4.7	2.6	5.6	9.2	2.1	1.8	3.1	5.2
Acetyl conjugate of IN-QZY47	1.7	-	4.3	-	1.8	3.9	4.8	8.0
IN-QZY47	1.8	42.4	1.5	41.7	2.1	2.1	2.4	0.9
Malonic acid conjugate of IN-QZY47 (IN-TUT81)	49.3	-	43.9	-	65.1	68.8	68.5	64.7
Total IN-QZY47 metabolites	52.8	42.4	49.7	41.7	69.0	74.8	75.7	73.6
IN-UNS90	0.9	8.2	1.2	5.5	0.5	-	-	1.0
Total IN-UNS90 metabolites	0.9	8.2	1.2	5.5	0.5	-	-	1.0
IN-A5760	0.7	5.2	0.4	7.1	1.2	-	-	-
Glucose conjugate of IN-A5760 (IN-R3Z85)	1.2	-	1.2	-	1.4	1.1	1.2	1.2
Total IN-A5760 metabolites	1.9	5.2	1.6	7.1	2.6	1.1	1.2	1.2

	30DAA				120DAA		300DAA	
	Immature	Post hydrolysis	Mature	Post hydrolysis	Immature	Mature	Immature	Mature
IN-F4106	3.9	20.9	3.0	27.1	5.1	1.0	-	0.5
IN-UJV12	0.7	2.5	0.5	3.1				
Unidentified	8.6[A]	3.6[G]	14.2[B]	1.1[H]	6.6[C]	6.8[D]	10.2[E]	10.7[F]

Notes:

[A] Unidentified, including unretained components, consisting of 13 components. None individually >1.7 percent TRR, 0.004 mg eq/kg.

[B] Unidentified, including unretained components, consisting of 24 components. None individually >1.9 percent TRR, 0.013 mg eq/kg.

[C] Unidentified, including unretained components, consisting of 6 components. None individually >2.4 percent TRR, 0.001 mg eq/kg.

[D] Unidentified consisting of 8 components. None individually >1.9 percent TRR, 0.002 mg equiv/kg.

[E] Unidentified, including unretained components, consisting of 6 components. None >2.8 percent TRR, 0.002 mg eq/kg.

[F] Unidentified, including unretained components, consisting of 10 components. None individually >2.4 percent TRR, 0.004 mg eq/kg.

[G] Unidentified, including unretained components, consisting of 5 components. None individually >1.1 percent TRR, 0.003 mg eq/kg.

[H] Unidentified consisting of a single component.

Identification and characterization of radioactive residues [IP-5,8a-^{14}C]fluazaindolizine in spinach

Extractability of ^{14}C in [IP-5,8a-^{14}C] spinach using methanol:water was good (81.2–93.2 percent TRR Table 23). The unextracted residues in the 300DAA immature and mature samples were subject to further investigations; these further treatments released 7.1–7.5 percent TRR with ≤ 3.3 percent TRR remaining in the solids (Table 23).

Fluazaindolizine was identified in all samples (2.9–29.0 percent TRR, ≤ 0.114 mg/kg) decreasing in concentration in later samples. The principal extracted residue was IN-QEK31 accounting for 19.2–52.5 percent TRR (0.010–0.115 mg eq/kg) apart from immature spinach at the 30DAA plant-back interval where fluazaindolizine was the principal residue at 29.0 percent TRR (0.034 mg eq/kg; Table 24).

Other identified metabolites were the IN-REG72, inositol conjugate of IN-QEK31 (IN-UHD13), glucose conjugate of IN-QEK31 (IN-UGA20), methyl ester of IN-QEK31 (IN-R2W56), IN-RYC33, and malic acid conjugate IN-QEK31 (IN-UJU44), (all ≤9.4 percent TRR, ≤ 0.045 mg eq/kg) as well as a glucose conjugate of IN-REG72 (≤9.3 percent TRR, ≤ 0.048 mg eq/kg) and a glycerol glucuronide conjugate of IN-QEK31 (≤11.1 percent TRR, ≤ 0.022 mg eq/kg; Table 24).

Multiple unidentified metabolites were also detected accounting for an aggregate total of 10.7-29.4 percent TRR in each sample but each individually was ≤5.3 percent TRR (≤ 0.011 mg eq/kg). Acid hydrolysis cleaved many of these components to IN-QEK31 (Table 24).

Table 23 Extraction of residues in spinach from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]-fluazaindolizine (% TRR)

	30DAA		120DAA		300DAA	
	Immature	Mature	Immature	Mature	Immature	Mature
TRR (mg/eq/kg)	0.116	0.520	0.018	0.043	0.167	0.233
Extracted (MeOH/water)	92.3	93.2	81.2	89.0	89.3	90.1
Unextracted (MeOH/water)	7.7	6.8	18.8	11.1	10.8	9.8
Aqueous soak 1 (overnight)	NC	NC	NC	NC	0.9	0.4
Acetonitrile:water	NC	NC	NC	NC	2.3	1.2
Aqueous soak 2 (*ca.* 5 h, ambient)	NC	NC	NC	NC	<LOQ	<LOQ
Enzyme (Driselase)	NC	NC	NC	NC	<LOQ	1.7

	30DAA		120DAA		300DAA	
	Immature	Mature	Immature	Mature	Immature	Mature
TRR (mg/eq/kg)	0.116	0.520	0.018	0.043	0.167	0.233
0.1M HCl (*ca.* 50-60°C, 6h)	NC	NC	NC	NC	1.5	1.3
1.3M HCl (*ca.* 80°C, 20 h)	NC	NC	NC	NC	2.2	1.9
0.1M NaOH (*ca.* 80°C, 4 h)	NC	NC	NC	NC	0.6	0.6
Remaining	NC	NC	NC	NC	3.3	2.7

Notes:

NC = Not conducted

Table 24 Identification of TRR (% TRR) from spinach in various extracts and fractions from crops sown 30, 120, and 300 days after application of [IP 5,8a-^{14}C]-fluazaindolizine

	30DAA				120DAA		300DAA	
	Immature	Post hydrolysis	Mature	Post hydrolysis	Immature	Mature	Immature	Mature
TRR (mg eq/kg)	0.116		0.520		0.018	0.043	0.167	0.233
Fluazaindolizine	29.0	-	21.9	-	2.9	17.0	8.3	7.6
IN-REG72	1.8	-	2.3	-	-	1.3	1.2	1.4
Glucose conjugate of IN-REG72	6.8	-	9.3	-	-	3.8	5.3	-
Total whole molecule metabolites	37.6	-	33.5	-	2.9	22.1	14.8	9.0
Glycerol glucuronide conjugate of IN-QEK31	1.4	-	4.3	-	11.1	9.0	7.4	7.1
Inositol conjugate of IN-QEK31 (IN-UHD13)	-	-	2.1	-	-	-	-	8.4
Glucose conjugate of IN-QEK31 (IN-UGA20)	-	-	8.6	-	4.1	4.6	5.1	7.6
IN-QEK31	19.2	71.5	22.0	66.3	52.5	27.1	28.3	24.8
Methyl ester of IN-QEK31 (IN-R2W56)	2.4	-	1.6	-		2.8	2.6	2.1
Malic acid conjugate of IN-QEK31 (IN-UJU44)	-	-	-	3.1	-	1.9	1.6	1.7
IN-RYC33	9.4	-	7.7	1.4	-	2.6	4.0	-
Total IN-QEK31 metabolites	32.4	71.5	46.3	70.8	67.7	48	49	51.7
Unidentified	17.7[A]	20.7[G]	11.0[B]	20.0[H]	10.7[C]	12.4[D]	24.0[E]	29.4[F]

Notes:

[A] Unidentified, including unretained components, consisting of 11 components. None >6.6 percent TRR, 0.008 mg eq/kg.

[B] Unidentified, including unretained components, consisting of 7 components. None >2.1 percent TRR, 0.011 mg eq/kg.

[C] Unidentified, including unretained components, consisting of 3 components. None >4.7 percent TRR, 0.001 mg eq/kg.

[D] Unidentified, including unretained components, consisting of 8 components. None >3.9 percent TRR, 0.001 mg eq/kg.

[E] Unidentified, including unretained components, consisting of 8 components. None >5.6 percent TRR, 0.009 mg eq/kg.

[F] Unidentified, including unretained components, consisting of 20 components. None >4.1 percent TRR, 0.010 mg eq/kg.

[G] Unidentified, including unretained components, consisting of 4 components. None >7.4 percent TRR, 0.009 mg eq/kg.

[H] Unidentified, including unretained components, consisting of 5 components. None >8.8 percent TRR, 0.046 mg eq/kg.

Radish

TRR for radish commodities for the various plant-back intervals are presented in Table 25.

Table 25 Total radioactive residues (mg eq/kg) in radish 30, 120, and 300 days after a soil drench application of [^{14}C]-fluazaindolizine

Days after application	[Ph-^{14}C]-fluazaindolizine			[IP-5,8a-^{14}C]-fluazaindolizine		
	Immature foliage	Mature foliage	Mature Roots	Immature Foliage	Mature foliage	Mature Roots
30	0.342	0.328	0.388	0.329	0.537	0.277
120	0.062	0.054	0.131	0.049	0.064	0.037
300	0.056	0.103	0.054	0.092	0.200	0.051

Identification and characterization of radioactive residues [Ph-^{14}C]-fluazaindolizine in radish

Foliage

Extractability of ^{14}C in [Ph-^{14}C] radish foliage using methanol:water was good (88.8-92.9 percent TRR Table 26). The unextracted residues in the 300DAA sample were subject to further investigations; these further treatments released 7.5 percent TRR with ≤ 3.8 percent TRR remaining in the solids (Table 26).

Fluazaindolizine was identified at all plant-back intervals (0.2-11.8 percent TRR, ≤ 0.039 mg/kg) decreasing in later samples. The principal extracted residue was IN-QZY47 (25.8-32.6 percent TRR) which in conjunction with other IN-QZY47 derived metabolites (IN-TUT81 and acetylated IN-QZY47 identified by LC-MS analysis) accounted for 31.3-37.3 percent TRR (0.018–0.112 mg eq/kg; Table 27).

Other identified metabolites were IN-RSU03, IN-UJV12, IN-UNS90, IN-A5760, glucose conjugate of IN-A5760 (IN-R3Z85), and IN-F4106, (all ≤5.8 percent TRR, ≤ 0.018 mg eq/kg) as well as glucose conjugates of IN-REG72, IN-UNS90, and IN-RSU03 (≤34.7 percent TRR, ≤ 0.080 mg eq/kg) and an acetyl conjugate of IN-QZY47 (≤ 2.0 percent TRR, ≤ 0.007 mg eq/kg; Table 27).

Multiple unidentified metabolites, including unretained components which may contain multiple metabolites, were also detected accounting for an aggregate total of 2.7–5.9 percent TRR in each sample but each individually was ≤ 2.9 percent TRR (≤ 0.010 mg eq/kg). Most of these metabolites were hydrolysed to known metabolites such as IN-F4106, IN-RSU03, IN-QZY47 and IN-UNS90 (Table 27).

Table 26 Extraction of residues in radish foliage from crops sown 30, 120, and 300 days after application of [Ph-^{14}C]-fluazaindolizine (% TRR)

	30DAA		120DAA		300DAA	
	Immature	Mature	Immature	Mature	Immature	Mature
TRR (mg eq/kg)	0.342	0.328	0.062	0.054	0.056	0.103
Extracted (MeOH/water)	92.9	92.2	90.6	88.8	92.1	88.8
Unextracted (MeOH/water)	7.0	7.8	9.4	11.1	8.0	11.2
Aqueous soak 1 (overnight)	NC	NC	NC	NC	NC	0.7
Acetonitrile:water	NC	NC	NC	NC	NC	1.2
Aqueous soak 2 (*ca.* 5 h, ambient)	NC	NC	NC	NC	NC	<LOQ
Enzyme (Driselase)	NC	NC	NC	NC	NC	1.6
0.1M HCl (*ca.* 50-60°C, 6 h)	NC	NC	NC	NC	NC	1.1
1.3M HCl (*ca.* 80°C, 20 h)	NC	NC	NC	NC	NC	2.2
0.1M NaOH (*ca.* 80°C, 4 h)	NC	NC	NC	NC	NC	0.7
Remaining	NC	NC	NC	NC	NC	3.8

NC = not conducted

Table 27 Identification of TRR from radish foliage in various extracts and fractions from crops sown 30, 120, and 300 days after application of [Ph–^{14}C]-fluazaindolizine (% TRR)

	30DAA			120DAA		300DAA	
	Immature	Mature	Post hydrolysis	Immature	Mature	Immature	Mature
TRR (mg eq/kg)	0.342	0.328	-	0.062	0.054	0.056	0.103
Fluazaindolizine	3.3	11.8	-	0.3	0.9	0.2	-
IN-REG72	-	0.7	-	-	-	-	-
Glucose conjugate of IN-REG72	1.0	2.1	-	-	0.4	-	0.2
Total whole molecule metabolites	4.3	14.6	-	0.3	1.3	0.2	0.2
IN-RSU03	1.8	1.7	28.0	1.4	1.3	0.9	1.1
Glucose conjugate of IN-RSU03	23.5	12.2	-	22.4	20.3	34.7	18.3
Total IN-RSU03 metabolites	25.3	13.9	28.0	23.8	21.6	35.6	19.4
Acetyl conjugate of IN-QZY47	2.0	0.9	-	-	-	0.4	0.3
IN-QZY47	25.8	26.3	12.8	32.6	29.3	29.1	31.8
Malonic acid conjugate of IN-QZY47 (IN-TUT81)	4.6	4.1	-	4.4	4.3	5.2	5.2
Total IN-QZY47 metabolites	32.4	31.3	12.8	37.0	33.6	34.7	37.3
IN-UNS90	3.0	4.0	17.6	4.4	5.2	4.1	4.8
Glucose conjugate of IN-UNS90	8.5	14.6	-	13.8	15.3	8.0	17.2
Total IN-UNS90 metabolites	11.5	18.6	17.6	18.2	20.5	12.1	22.0
IN-A5760	1.0	1.2	5.3	1.0	1.6	0.4	1.0
Glucose conjugate of IN-A5760 (IN-R3Z85)	5.1	2.8	-	5.1	3.6	5.8	5.1
Total IN-A5760 metabolites	6.1	4.0	5.3	6.1	5.2	6.2	6.1
IN-F4106	4.2	1.5	19.7	1.2	1.2	0.7	1.0
IN-UJV12	0.8	0.6	1.6	1.1	-	-	-
Unidentified	5.9[A]	5.6[B]	4.9[G]	2.9[C]	2.8[D]	2.7[E]	3.0[F]

Notes:

[A] Unidentified, including unretained components, consisting of 6 components, none individually >2.9 percent TRR, 0.010 mg eq/kg.

[B] Unidentified, including unretained components, consisting of 8 components, none individually >1.6 percent TRR, 0.005 mg eq/kg.

[C] Unidentified, including unretained components, consisting of 2 components, none individually >2.2 percent TRR, 0.001 mg eq/kg.

[D] Unidentified, including unretained components, consisting of 2 components, none individually >2.1 percent TRR, 0.001 mg eq/kg.

[E] Unidentified, including unretained components, consisting of 4 components, none individually >1.2 percent TRR, 0.001 mg eq/kg.

[F] Unidentified, including unretained components, consisting of 7 components, none individually >0.6 percent TRR, 0.001 mg eq/kg

[G] Unidentified, including unretained components, consisting of 2 components, none individually >2.5 percent TRR, 0.008 mg eq/kg

Roots

Extractability of ^{14}C in [Ph-^{14}C] radish roots using methanol:water was good (94.3–95.1 percent TRR Table 28).

Fluazaindolizine was identified at all plant-back intervals (1.7-12.2 percent TRR, ≤ 0.047 mg/kg) decreasing in later samples. The principal extracted residue identified was IN-QZY47 (18.0-26.6 percent TRR) which in conjunction with the malonyl and acetylated derivatives of IN-QZY47 (IN-TUT81 and acetylated IN-QZY47) accounted for 36.7-40.3 percent TRR (0.021-0.143 mg eq/kg; Table 29).

Other identified metabolites were IN-RSU03, IN-REG72, IN-UNS90, IN-A5760, glucose conjugate of IN-A5760 (IN-R3Z85), and IN-F4106, (all ≤ 3.3 percent TRR, ≤ 0.013 mg eq/kg) as well as glucose conjugates of IN-REG72, IN-USN90, and IN-RSU03 (≤36.5 percent TRR, ≤ 0.073 mg eq/kg; Table 29).

Multiple unidentified metabolites, including unretained components which may contain multiple metabolites, were also detected accounting for an aggregate total of 7.7-8.8 percent TRR in each sample but each individually was ≤ 3.4 percent TRR (≤ 0.005 mg eq/kg). Most of these metabolites were hydrolysed to known metabolites such as IN-F4106, IN-RSU03, IN-QZY47 and IN-UNS90 (Table 29).

Table 28 Extraction of residues in radish root from crops sown 30, 120, and 300 days after application of [Ph-^{14}C]-fluazaindolizine (%TRR)

	30DAA TRR=0.388 mg eq/kg	120DAA TRR = 0.131 mg eq/kg	300DAA TRR= 0.054 mg eq/kg
Extracted (MeOH/water)	94.6	94.3	95.1
Unextracted (MeOH/water)	5.3	5.7	5.0

Table 29 Identification of TRR (% TRR) from mature radish roots in various extracts and fractions from crops sown 30, 120, and 300 days after application of [Ph–^{14}C]-fluazaindolizine

	30DAA		120DAA	300DAA
TRR (mg eq/kg)	0.388	Post hydrolysis	0.131	0.054
Fluazaindolizine	12.2	-	1.7	2.0
IN-REG72	0.4	-	-	-
Glucose Conjugate of IN-REG72	5.5	-	0.6	0.8
Total whole molecule metabolites	18.1	-	2.3	2.8
IN-RSU03	1.2	23.5	0.7	0.7
Glucose Conjugate of IN-RSU03	18.9	-	28.0	36.5
Total IN-RSU03 metabolites	20.1	23.5	28.7	37.2
Acetyl conjugate of IN-QZY47	-	-		
IN-QZY47	23.1	27.4	18.0	26.6
Malonic acid conjugate of IN-QZY47 (IN-TUT81)	13.6	4.6	22.3	11.7
Total IN-QZY47 metabolites	36.7	32.4	40.3	38.3
IN-UNS90	3.2	5.5	3.0	3.3
Glucose Conjugate of IN-UNS90	2.9	-	4.6	3.4
Total IN-UNS90 metabolites	6.1	5.5	7.6	6.7
IN-A5760	0.8	6.6	0.7	-
Glucose conjugate of IN-A5760 (IN-R3Z85)	1.2	-	1.8	1.1
Total IN-A5760 metabolites	2.0	6.6	2.5	1.1
IN-F4106	1.5	16.3	1.8	1.6
IN-UJV12	-	2.5		
Unidentified	7.7[A]	5.2[D]	8.5[B]	8.8[C]

Notes:

[A] Unidentified, including unretained components, consisting of 12 components, none individually >1.3 percent TRR, 0.005 mg eq/kg.

[B] Unidentified, including unretained components, consisting of 11 components, none individually >2.7 percent TRR, 0.003 mg eq/kg.

[C] Unidentified, including unretained components, consisting of 6 components, none individually >3.4 percent TRR, 0.002 mg eq/kg.

[D] Unidentified, including unretained components, consisting of 3 components, none individually >2.2 percent TRR, 0.008 mg eq/kg.

Identification and characterization of radioactive residues [IP-5,8a-^{14}C]fluazaindolizine in radish

Foliage

Extractability of ^{14}C in [IP-5,8a-^{14}C] radish foliage using methanol:water was good (84.7–91.3 percent TRR Table 30). The unextracted residues in the 30DAA and 300DAA samples were subject to further investigations; these further treatments released 9.9–12.0 percent TRR (Table 30).

Fluazaindolizine was found in all samples (1.0-6.8 percent TRR, ≤ 0.036 mg/kg) decreasing in concentrations at later plant-back intervals. The principal extractable residue identified was IN-UJU44, a malic acid conjugate of IN-QEK31 accounting for 32.6-43.9 percent TRR (0.016-0.236 mg eq/kg; Table 31). The next most abundant metabolite was IN-QEK31 accounting for 10.2-23.9 percent TRR (0.010-0.059 mg eq/kg).

Other identified metabolites were the inositol conjugate of IN-QEK31 (IN-UHD13), glucose conjugate of IN-QEK31 (IN-UGA20), glutamic acid conjugate of IN-QEK31 (IN-WUK12), methyl ester of IN-QEK31 (IN-R2W56), and IN-RYC33 (all ≤7.9 percent TRR, ≤ 0.026 mg eq/kg) together with a glucose conjugate of IN-REG72 (≤ 2.2 percent TRR, ≤ 0.012 mg eq/kg; Table 31).

Multiple unidentified metabolites, including unretained components which may contain multiple metabolites, were also detected accounting for an aggregate total of 7.2-12.2 percent TRR in each sample but each individually was ≤5.7 percent TRR (≤ 0.023 mg eq/kg).

Table 30 Extraction of residues in radish foliage from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]fluazaindolizine (% TRR)

	30DAA		120DAA		300DAA	
	Immature	Mature	Immature	Mature	Immature	Mature
TRR (mg eq/kg)	0.329	0.537	0.049	0.064	0.092	0.200
Extracted (MeOH/water)	91.3	90.0	84.7	88.1	85.3	85.2
Unextracted (MeOH/water)	8.7	10.0	15.2	11.9	14.8	14.7
Aqueous soak 1 (overnight)	NC	0.7	NC	NC	NC	2.6
Acetonitrile:water	NC	4.1	NC	NC	NC	2.4
Aqueous soak 2 (*ca.* 5 h, ambient)	NC	0.2	NC	NC	NC	<LOQ
Enzyme (Driselase)	NC	2.6	NC	NC	NC	1.1
0.1M HCl (*ca.* 50-60°C, 6 h)	NC	1.2	NC	NC	NC	2.7
1.3M HCl (*ca.* 80°C, 20 h)	NC	0.7	NC	NC	NC	2.6
0.1M NaOH (*ca.* 80°C, 4 h)	NC	0.4	NC	NC	NC	0.6
Remaining	NC	NC	NC	NC	NC	2.7

Notes:

NC = Not Conducted.

Table 31 Identification of TRR from radish foliage in various extracts and fractions from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]fluazaindolizine (% TRR)

	30DAA		120DAA		300DAA	
	Immature	Mature	Immature	Mature	Immature	Mature
TRR (mg eq/kg)	0.329	0.537	0.049	0.064	0.092	0.200
Fluazaindolizine	4.5	6.8	1.2	1.6	1.0	1.2
IN-REG72	-	0.5	-	-	-	-
Glucose conjugate of IN-REG72	1.1	2.2	-	-	-	-
Total whole molecule metabolites	5.6	9.5	1.2	1.6	1.0	1.2
Glucose conjugate of IN-QEK31 (IN-UGA20)	4.3	5.4	4.7	3.0	2.6	-
IN-QEK31	17.9	10.2	20.3	23.9	16.9	21.0

	30DAA		120DAA		300DAA	
	Immature	Mature	Immature	Mature	Immature	Mature
methyl ester of IN-QEK31 (IN-R2W56)	0.7	0.5	1.4	1.1	-	1.9
Inositol conjugate of IN-QEK31 (IN-UHD13)	1.8	1.0	3.3	4.1	-	-
Malic acid conjugate IN-QEK31 (IN-UJU44)	42.2	43.9	32.6	34.2	41.0	43.9
Glutamic acid conjugate of IN-QEK31 (IN-WUK12)	-	1.7	5.7	4.5	3.4	2.2
IN-RYC33	7.9	2.8	3.1	2.3	2.7	1.7
Total IN-QEK31 metabolites	74.8	65.5	71.1	73.1	66.6	70.7
Unidentified	7.2[A]	12.2[B]	12.2[C]	10.0[D]	12.2[E]	11.8[F]

Notes:

[A] Unidentified consisting of 2 components, none individually >5.4 percent TRR, 0.018 mg eq/kg.

[B] Unidentified, including unretained components, consisting of 7 components, none individually >4.3 percent TRR, 0.023 mg eq/kg.

[C] Unidentified, including unretained components, consisting of 6 components, none individually >5.7 percent TRR, 0.003 mg eq/kg.

[D] Unidentified, including unretained components, consisting of 4 components, none individually >4.5 percent TRR, 0.003 mg eq/kg.

[E] Unidentified, including unretained components, consisting of 4 components, none individually >4.2 percent TRR, 0.004 mg eq/kg.

[F] Unidentified, including unretained components, consisting of 4 components, none individually >4.5 percent TRR, 0.009 mg eq/kg.

Roots

Extractability of ^{14}C in [IP-5,8a-^{14}C] radish roots using methanol:water was good (86.4-93.7 percent TRR, Table 32).

Fluazaindolizine was found in all samples (6.3–17.3 percent TRR, ≤ 0.048 mg/kg) decreasing in concentration in later samples. The principal extracted residue identified was the malic acid conjugate IN-QEK31 (IN-UJU44) accounting for 13.2–28.8 percent TRR (0.005–0.073 mg eq/kg) with the next most abundant metabolite IN-QEK31 accounting for 4.8–14.0 percent TRR (0.002–0.026 mg eq/kg), and its glucose conjugate IN-UGA20, accounting for 9.9–13.9 percent TRR (0.004–0.039 mg eq/kg; Table 33).

Other identified metabolites were IN-REG72, inositol conjugate of IN-QEK31 (IN-UHD13), glutamic acid conjugate of IN-QEK31 (IN-WUK12), methyl ester of IN-QEK31 (IN-R2W56), and IN-RYC33 (all ≤4.2 percent TRR, ≤ 0.010 mg eq/kg) and a glucose conjugate of IN-REG72 (≤9.3 percent TRR, ≤ 0.026 mg eq/kg; Table 33).

Multiple unidentified metabolites, including unretained components which may contain multiple metabolites, were also detected accounting for an aggregate total of 10.2-25.7 percent TRR in each sample but each individually was ≤8.2 percent TRR (≤ 0.017 mg eq/kg). These metabolites would appear to be mostly conjugates of IN-QEK31 based upon their hydrolysis behaviour (Table 33).

Table 32 Extraction of residues in mature radish roots from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]-fluazaindolizine (% TRR)

	30DAA TRR=0.277 mg eq/kg	120DAA TRR=0.037 mg eq/kg	300DAA TRR= 0.051 mg eq/kg
Extracted (MeOH/water)	93.7	86.4	93.6
Unextracted (MeOH/water)	6.3	13.6	6.4

Table 33 Identification of TRR (% TRR) from mature radish roots in various extracts and fractions from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]-fluazaindolizine

	30DAA		120DAA	300DAA
TRR (mg eq/kg)	0.277	Post hydrolysis	0.037	0.051
Fluazaindolizine	17.3	-	10.2	6.3
IN-REG72	-	-	2.3	-
Glucose conjugate of IN-REG72	9.3	-	4.7	-
Total whole molecule metabolites	26.6	-	17.2	6.3
Glucose conjugate of IN-QEK31 (IN-UGA20)	13.9	-	9.9	11.3
IN-QEK31	9.3	88.7	4.8	14.0
methyl ester of IN-QEK31 (IN-R2W56)	2.0	-	0.7	1.8
Inositol conjugate IN-QEK31 (IN-UHD13)	-	-	3.2	2.5
Malic acid conjugate IN-QEK31 (IN-UJU44)	26.5	-	13.2	28.8
Glutamic acid conjugate of IN-QEK31 (IN-WUK12)	3.5	-	4.2	3.8
IN-RYC33	-	-	-	0.8
Total IN-QEK31 metabolites	55.2	88.7	36	63
Unidentified	10.2[A]	3.4[D]	25.7[B]	18.1[C]

Notes:

[A] Unidentified, including unretained components, consisting of 4 components, none individually >6.0 percent TRR, 0.017 mg eq/kg.

[B] Unidentified, including unretained components, consisting of 5 components, none individually >8.2 percent TRR, 0.003 mg eq/kg.

[C] Unidentified, including unretained components, consisting of 11 components, none individually >3.8 percent TRR, 0.002 mg eq/kg.

[D] Unidentified consisting of a single unretained component.

Wheat

The TRR for spring wheat commodities for the various plant-back intervals are presented in Table 34. The highest levels of total radioactivity were found in straw from wheat grown in the [Ph-^{14}C]-fluazaindolizine treated soil. Radioactive residues in grain were considerably higher in wheat grown in soil treated with [IP-5,8a-^{14}C]-fluazaindolizine as compared to the [Ph-^{14}C] label, indicating cleavage of fluazaindolizine molecule.

Table 34 TRR (mg eq/kg) in wheat sown 30, 120, and 300 days after a soil drench application of [^{14}C]-fluazaindolizine

Days after application	[Ph-^{14}C]-fluazaindolizine				[IP-5,8a-^{14}C]-fluazaindolizine			
	Forage	Hay	Straw	Grain	Forage	Hay	Straw	Grain
30	1.165	1.433	6.873	0.086	0.411	1.143	3.547	1.517
120	0.422	0.334	2.559	0.055	0.198	0.377	1.357	0.521
300	0.396	0.531	2.741	0.026	0.609	0.969	4.073	1.296

Identification and characterization of radioactive residues [Ph-^{14}C]fluazaindolizine in wheat forage, hay, and grain

Forage

Extractability of ^{14}C in [Ph-^{14}C] forage using methanol:water was good (94.5–96.9 percent TRR, Table 35).

Table 35 Extraction of residues in wheat forage from crops sown 30, 120, and 300 days after application of [Ph–^{14}C]-fluazaindolizine

Fraction	30DAA		120DAA		300DAA	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Extracted (MeOH/water)	96.1	1.120	94.5	0.399	96.9	0.383
Unextracted (MeOH/water)	3.9	0.045	5.6	0.024	3.1	0.012
Total	100	1.165	100	0.422	100	0.396

Fluazaindolizine was only identified in small quantities at the 30DAA plant-back interval (0.7 percent TRR, 0.009 mg/kg); it was not found in later plant-back intervals (Table 36). The principal extracted residue identified in [Ph-^{14}C] forage was the glucose conjugate of IN-UNS90 (38.2–41.4 percent TRR). An additional glucose conjugate of IN-UNS90 proposed to be conjugated to the phenolic position accounted for 2.8–3.0 percent TRR. Free IN-UNS90 and its two glucose conjugates accounted for greater than half the residue (53.3–64.0 percent TRR; Table 36).

Other identified metabolites were IN-REG72, IN-RSU03, IN-QZY47, malonic acid conjugate of IN-QZY47 (IN-TUT81), IN-A5760, glucose conjugate of IN-A5760 (IN-R3Z85), and IN-F4106 (all ≤ 3.4 percent TRR). A number of metabolites in the [Ph-^{14}C] experiment forage were observed including the glucose conjugate of IN-RSU03 (≤12.2 percent TRR), a malonyl glucose conjugate on IN-RSU03 (≤ 2.3 percent TRR) and a glucose conjugate of IN-REG72 (≤ 1.7 percent TRR).

Unidentified metabolites, including unretained components which may contain multiple metabolites, were also detected, accounting for an aggregate total of 11.6–14.8 percent TRR in each sample, were comprised of 10-17 individual metabolites but each individually was ≤4.8 percent TRR (≤ 0.027 mg eq/kg). A significant portion of the unknown metabolites in the initial methanol water extracts were shown to hydrolyse with 1M HCl to IN-F4106, IN-A5760, IN-RSU03, and other known metabolites (Table 36).

Table 36 Identification of TRR (% TRR) from wheat forage in various extracts and fractions from crops sown 30, 120, and 300 days after application of [Ph–^{14}C]-fluazaindolizine[1]

Metabolite	30DAA		120DAA	300DAA
TRR (mg/eq/kg)	1.165	Post hydrolysis	0.422	0.396
Fluazaindolizine	0.7	-	-	-
IN-REG72	0.2	-	-	-
Glucose conjugate of IN-REG72	1.1	-	-	1.7
Total whole molecule metabolites	2.0	-	-	1.7
IN-RSU03	2.1	12.9	1.6	1.9
Malonyl Glucose Conjugate of IN-RSU03	0.7	-	2.3	0.7
Glucose Conjugate of IN-RSU03	9.8	-	12.2	8.2
Total IN-RSU03 metabolites	12.6	12.9	16.1	10.8
IN-QZY47	2.9	4.7	1.5	2.1
Malonic acid conjugate of IN-QZY47 (IN-TUT81)	0.6	-	2.6	2.0
Total IN-QZY47 metabolites	3.5	4.7	4.1	4.1
IN-UNS90	16.0	66.8	12.3	21.2
Glucose conjugate of IN-UNS90 (*ca.* 8 min)	41.4 (44.2)	-	38.2	39.8
Phenol glucose conjugate of IN-UNS90 (*ca.* 15 min)	2.8		2.8	3.0
Total IN-UNS90 metabolites	60.2	66.8	53.3	64.0
IN-A5760	1.5	4.1	0.7	1.4
Glucose conjugate of IN-A5760 (IN-R3Z85)	0.9	-	3.4	1.9
Total IN-A5760 metabolites	2.4	4.1	4.1	3.3
IN-F4106	1.9	2.2	1.9	1.3
Unidentified	13.3[A] (18.2[D])	10.0[E]	14.8[B]	11.6[C]

Notes:

[1] the numbers in parentheses are results, where different, of repeat analysis just prior to acid hydrolysis

[A] Unidentified, including unretained components, consisting of 17 components, none >2.3 percent TRR, 0.027 mg eq/kg.

[B] Unidentified, including unretained components, consisting of 10 components, none >4.8 percent TRR, 0.020 mg eq/kg.

[C] Unidentified, including unretained components, consisting of 11 components, none >2.7 percent TRR, 0.011 mg eq/kg.

[D] Unidentified, including unretained components, consisting of 17 components, none >2.3 percent TRR, 0.027 mg eq/kg.

[E] Unidentified consisting of 3 components, none >2.2 percent TRR, 0.026 mg eq/kg.

Hay

Extractability of ^{14}C in [Ph-^{14}C] hay using methanol:water was good (91.6–92.8 percent TRR Table 37). The unextracted residues from 30DAA and 300DAA samples were subject to further investigations; these further treatments released an additional 5.1–7.1 percent TRR from the bound residues with terminal unextracted residues accounting for 0.8–2.0 percent TRR (Table 37).

Fluazaindolizine was only identified in small quantities at the 30DAA plant-back interval (0.5 percent TRR, 0.008 mg/kg) and not in later samples. The principal extracted residue was the glucose conjugate of IN-UNS90 (32.7–37.0 percent TRR) which in conjunction with IN-UNS90 and the phenol glucose conjugate of IN-UNS90 accounted for 46.1–51.2 percent TRR (Table 38). Chiral HPLC analysis was conducted on the isolated IN-UNS90 and it was demonstrated that only the *R*-enantiomer, IN-TQD54, was present.

Other identified metabolites were IN-RSU03, IN-QZY47, malonic acid conjugate of IN-QZY47 (IN-TUT81), IN-A5760, glucose conjugate of IN-A5760 (IN-R3Z85), and IN-F4106 (all ≤ 3.9 percent TRR). Chiral HPLC analysis was conducted on the isolated IN-RSU03 and it was demonstrated that only the *R*-enantiomer, IN-TMQ01, was present.

Other metabolites identified included a glucose conjugate of IN-RSU03 (13.7 percent TRR) and a malonyl glucose conjugate on IN-RSU03 (≤ 1.7 percent TRR).

Multiple unidentified metabolites, including unretained components which may contain multiple metabolites, were also detected accounting for an aggregate total of 16.5-19.1 percent TRR in each sample but each individually was ≤5.2 percent TRR. A significant portion of the unknown metabolites were shown to hydrolyse with 1M HCl to IN-F4106, IN-A5760, IN-RSU03, and other known metabolites in the forage and straw (Table 38).

Table 37 Extraction of residues in wheat hay from crops sown 30, 120, and 300 days after application of [Ph–^{14}C]fluazaindolizine (% TRR)

Fraction	30DAA TRR=1.433 mg eq/kg	120DAA TRR = 0.334 mg eq/kg	300DAA TRR=0.531 mg eq/kg
Extracted (MeOH/water)	92.2	91.6	92.8
Unextracted (MeOH/water)	7.8	8.4	7.1
Aqueous soak 1 (overnight)	0.7	NC	1.5
Acetonitrile:water	3.1	NC	1.9
Aqueous soak 2 (*ca.* 5 h, ambient)	0.5	NC	<LOQ
Enzyme (Driselase)	1.6	NC	<LOQ
0.1M HCl (*ca.* 50-60°C, 6 h)	0.4	NC	0.8
1.0M HCl (*ca.* 80°C, 20 h)	0.5	NC	0.9
0.1M NaOH (*ca.* 80°C, 4 h)	0.2	NC	<LOQ
Remaining	0.8	8.4	2.0

Table 38 Identification of TRR from wheat hay in various extracts and fractions from crops sown 30, 120, and 300 days after application of [Ph–^{14}C]fluazaindolizine (% TRR)[1]

Metabolite	30DAA		120DAA	300DAA
TRR (mg eq/kg)	1.433	Post hydrolysis	0.334	0.531
Fluazaindolizine	0.5 (-)	-	-	-
IN-REG72	- (-)	-	-	-
Glucose conjugate of IN-REG72	2.7 (2.7)	-	1.4	2.7
Total whole molecule metabolites	3.2 (2.7)	-	1.4	2.7
IN-RSU03	1.3 (1.0)	11.0	1.4	1.0
Malonyl Glucose Conjugate of IN-RSU03	1.1 (0.7)	-	1.7	0.7
Glucose Conjugate of IN-RSU03	12.2 (12.9)	-	13.7	12.9
Total IN-RSU03a metabolites	14.6 (14.6)	11.0	16.8	14.6
IN-QZY47	0.9 (0.6)	1.5	1.1	0.6
Malonic acid conjugate of IN-QZY47 (IN-TUT81)	1.1 (0.9)	-	1.2	0.9
Total IN-QZY47 metabolites	2.0 (1.5)	1.5	2.3	1.5
IN-UNS90	10.6 (10.5)	54.3	9.9	10.5
Glucose Conjugate of IN-UNS90 (*ca.* 8 min)	35.1 (40.7)	-	32.7	37.0
Phenol glucose conjugate of IN-UNS90 (*ca.* 15 min)	4.1		3.5	3.7
Total IN-UNS90 metabolites	49.8 (51.2)	54.3	46.1	51.2
IN-A5760	1.2 (-)	5.4	1.1	-
Glucose conjugate of IN-A5760 (IN-R3Z85)	2.8 (3.9)	-	3.2	3.9
Total IN-A5760 metabolites	4.0 (3.9)	5.4	4.3	3.9
IN-F4106	2.7 (2.2)	1.3	2.7	2.2
Unidentified	19.1[A]/16.5[D]	19.4[E]	17.9[B]	16.5[C]

Notes:

[1] the numbers in parentheses are results, where different, of repeat analysis just prior to acid hydrolysis

[A] Unidentified, including unretained components, consisting of 24 components none >4.5 percent TRR, 0.065 mg eq/kg.

[B] Unidentified, including unretained components, consisting of 12 components none >4.4 percent TRR, 0.015 mg eq/kg.

[C] Unidentified, including unretained components, consisting of 9 components none >5.2 percent TRR, 0.028 mg eq/kg.

[D] Unidentified, including unretained components, consisting of 9 components none >5.2 percent TRR, 0.028 mg eq/kg.

[E] Unidentified, consisting of 9 components none >8.6 percent TRR, 0.046 mg eq/kg.

Straw

Extractability of ^{14}C in [Ph-^{14}C] straw using methanol:water was good (72.3–86.0 percent TRR Table 39). The unextracted residues were subject to further investigations; these further treatments released an additional 7.6–26.1 percent TRR with terminal residues remaining in solids accounting for 1.7–6.4 percent TRR (Table 39).

Fluazaindolizine was only identified in small quantities at the 30DAA plant-back interval (0.7 percent TRR, 0.041 mg/kg) and was not found in later plant-back intervals (Table 40). The principal extracted residue identified in [Ph-^{14}C] straw was IN-UNS90 (17.5–27.6 percent TRR). In conjunction with its glucose conjugates these metabolites accounted for 36.8–42.9 percent TRR (Table 40).

Other identified metabolites were IN-REG72, IN-RSU03, IN-QZY47, malonic acid conjugate of IN-QZY47 (IN-TUT81), IN-A5760, glucose conjugate of IN-A5760 (IN-R3Z85), IN-F4106, and IN-UJV12 (all ≤ 5.6 percent TRR, ≤ 0.228 mg eq/kg), together with a glucose conjugate of IN-RSU03 (≤ 12.2 percent TRR) and a malonyl glucose conjugate of IN-RSU03 (≤ 2.8 percent TRR) and a glucose conjugate of IN-REG72 (≤ 2.4 percent TRR).

Multiple unidentified metabolites, including unretained components which may contain multiple metabolites, were also detected accounting for an aggregate total of 21.4–27.6 percent TRR in each sample but each individually was ≤ 7.2 percent TRR (≤ 0.198 mg eq/kg). A significant portion of the unknown metabolites were shown to hydrolyse with 1M HCl to IN-F4106, IN-A5760, IN-RSU03, and other known metabolites (Table 40).

Table 39 Extraction of residues in wheat straw from crops sown 30, 120, and 300 days after application of [Ph–^{14}C]fluazaindolizine

Fraction	30DAA TRR = 6.873 mg eq/kg	120DAA TRR = 2.559 mg eq/kg	300DAA TRR = 2.741 mg eq/kg
Extracted (MeOH/water)	72.3	86.0	73.1
Unextracted (MeOH/water)	27.7	14.0	26.9
Aqueous soak 1 (overnight)	3.5	1.5	7.2
Acetonitrile:water	16.2	1.7	12.1
Aqueous soak 2 (*ca.* 5 h, ambient)	NC	0.6	1.0
Enzyme (Driselase)	3.5	1.7	1.6
0.1M HCl (*ca.* 50-60°C, 6 h)	0.8	0.5	1.3
1.0M HCl (*ca.* 80°C, 20 h)	1.6	1.1	0.9
0.1M NaOH (*ca.* 80°C, 4 h)	0.5	0.5	0.5
Remaining	1.7	6.4	2.3

Notes:

NC = Not conducted.

Table 40 Identification of TRR from wheat straw in various extracts and fractions from crops sown 30, 120, and 300 days after application of [Ph-^{14}C]fluazaindolizine (% TRR)[1]

Metabolite	30DAA		120DAA	300DAA
TRR (mg eq/kg)	6.873	Post hydrolysis	2.559	2.741
Fluazaindolizine	0.7 (0.3)	-	-	-
IN-REG72	0.8 (0.6)	-	1.5	-
Glucose conjugate of IN-REG72	2.4 (1.7)	-	1.5	0.9
Total whole molecule metabolites	3.9 (2.6)	-	3.0	0.9
IN-RSU03	2.7 (1.5)	12.3	1.8	3.4
Malonyl Glucose Conjugate of IN-RSU03	2.4 (2.2)	-	2.8	1.5
Glucose Conjugate of IN-RSU03	12.2 (10.1)	-	11.9	10.5
Total IN-RSU03 metabolites	17.3 (13.8)	14.4	16.5	15.4
IN-QZY47	0.4 (0.2)	0.8	0.7	3.8
Malonic acid conjugate of IN-QZY47 (IN-TUT81)	0.8 (0.6)	-	-	0.6
Total IN-QZY47 metabolites	1.2 (0.8)	0.8	0.7	4.4
IN-UNS90	18.4 (12.7)	34.7	17.5	27.6
Glucose conjugate of IN-UNS90 (*ca.* 8 min)	17.1 (18.8)	-	20.2	6.4
Phenol glucose conjugate of IN-UNS90 (*ca.* 15 min)	5.2		5.2	2.8
Total IN-UNS90 metabolites	40.7 (31.5)	34.7	42.9	36.8
IN-A5760	2.1 (0.9)	6.2	1.6	5.6
Glucose conjugate of IN-A5760 (IN-R3Z85)	1.7 (1.5)	-	2.5	1.7
Total IN-A5760 metabolites	3.8 (2.4)	6.2	4.1	7.3
IN-F4106	3.3 (2.6)	1.7	2.7	3.1
IN-UJV12	0.5 (0.4)	-	<0.1	0.5
Unidentified	27.1[A]/18.4[D]	15.4[E]	21.4[B]	27.6[C]

Notes:

[1] the numbers in parentheses are results, where different, of repeat analysis just prior to acid hydrolysis

[A] Unidentified, including unretained components, consisting of 64 components none >2.4 percent TRR, 0.163 mg eq/kg.

[B] Unidentified, including unretained components, consisting of 14 components none >6.3 percent TRR, 0.162 mg eq/kg.

[C] Unidentified, including unretained components, consisting of 32 components none >7.2 percent TRR, 0.198 mg eq/kg.
[D] Unidentified, including unretained, consisting of 24 components none >2.4 percent TRR, 0.163 mg eq/kg.
[E] Unidentified consisting of 6 components. None >6.2 percent TRR, 0.426 mg eq/kg.

Grain

Extractability of ^{14}C in [Ph-^{14}C] grain using methanol:water was poor (16.0–34.3 percent TRR, Table 41). The unextracted residues were subject to further investigations; these further treatments released an additional 62.9–84.0 percent TRR with terminal residues remaining in solids accounting for 2.8 percent TRR in case of the 300DAA grain sample (Table 41).

It was not possible to obtain accurate profiles from the 120 and 300DAA samples due to the large quantity of endogenous materials and large volumes of sample extract required to release the residue. The profiles obtained demonstrated that the residue was comprised of multiple metabolites although it was not possible to accurately determine their identity. Most of the residues were released by enzyme and/or acidic extractions which may alter the nature of the residue in the extraction process. The tentatively identified metabolites in these samples are presented in Table 42.

Fluazaindolizine was found in small quantities at the 30DAA plant-back interval (3.3 percent TRR, 0.003 mg/kg). The principal extracted residue identified in [Ph-^{14}C] grain was IN-A5760 (3.5–6.1 percent TRR, 0.002–0.005 mg eq/kg).

Other identified metabolites were IN-UNS90 and IN-F4106 (all ≤ 3.3 percent TRR, ≤ 0.003 mg eq/kg). IN-RSU03, IN-QZY47, and IN-REG72 were tentatively observed in the 300DAA grain although due to the low residues these regions could not be positively assigned. The glucose conjugate of IN-RSU03 (0.9 percent TRR, 0.001 mg eq/kg) and glucose conjugates on IN-UNS90 (≤ 2.4 percent TRR, ≤ 0.002 mg eq/kg) were also identified.

Multiple unidentified metabolites, including unretained components which may contain multiple metabolites, were also detected accounting for an aggregate total of 18.0-96.6 percent TRR in each sample but generally each individually was ≤ 15.7 percent TRR (≤ 0.012 mg eq/kg). An unretained region accounted for 52.1 percent TRR and 0.029 mg eq/kg in the 120DAA grain following enzyme digestion and extraction which may consist of multiple components, as observed in other grain fractions where this radioactive region is partially resolved into numerous polar metabolites. As fluazaindolizine and its major phenyl metabolites (IN-F4106 and IN-A5760) are known to mineralise in soil to $^{14}CO_2$ at significant levels, these polar components may result from the reincorporation of the radiolabel into endogenous plant materials.

Table 41 Extraction of residues in wheat grain from crops sown 30, 120, and 300 days after application of [Ph–^{14}C]fluazaindolizine (% TRR)

Fraction	30DAA TRR=0.089 mg eq/kg	120DAA TRR=0.055 mg eq/kg	300DAA TRR=0.026 mg eq/kg
Extracted (MeOH/water)	34.3	16.0	34.3
Unextracted (MeOH/water)	65.7	84.0	65.7
Aqueous soak 1 (overnight)	7.8	<LOD	<LOD
Acetonitrile:water extract	<LOD	<LOD	<LOD
Aqueous soak 2 (*ca.* 5 h, ambient)	<LOD	<LOD	<LOD
Enzyme extract (Driselase)	27.3	52.1	21.5
0.1M HCl (*ca.* 50-60°C, 6 h)	13.5	<LOD	13.7
1.0M HCl (*ca.* 80°C, 20 h)	19.3	31.9	27.7
0.1M NaOH (*ca.* 80°C, 4 h)	<LOD	<LOD	<LOD
Remaining	NC[d]	NC	2.8

Table 42 Identification of TRR from wheat grain in various extracts and fractions from crops sown 30, 120, and 300 days after application of [Ph–^{14}C]fluazaindolizine

	30DAA, TRR= 0.089 mg eq/kg	120DAA, TRR= 0.055 mg eq/kg	300DAA, TRR= 0.026 mg eq/kg
Fluazaindolizine	3.3	-	-
IN-REG72	-	-	7.9
Total whole molecule metabolites	3.3	-	7.9
IN-RUS03	-	-	8.4
Glucose Conjugate of IN-RSU03	0.9	-	-
Total IN-RSU03 metabolites	0.9	-	8.4
IN-QZY47	-	-	2.3
Total IN-QZY47 metabolites	-	-	2.3
IN-UNS90	0.9	-	-
Glucose Conjugate of IN-UNS90 (*ca.* 8 min)	1.1	-	-
Glucose Conjugate of IN-UNS90 (*ca.* 15 min)	2.4	-	-
Total IN-UNS90 metabolites	4.4	-	-
IN-A5760	6.1	3.5	-
IN-F4106	3.3	-	-
Unidentified	47.0[A]	96.6[B]	15.7[C]

Notes:

[A] Unidentified, including unretained components, consisting of 12 components none >13.4 percent TRR, 0.012 mg eq/kg.

[B] Unidentified consisting of a single component of 7.1 percent TRR, 0.004 mg eq/kg, plus unretained regions following enzyme and acid extraction of ≤52.1 percent TRR, ≤ 0.029 mg eq/kg, which may consist of multiple components.

[C] Unidentified, including unretained components, consisting of 3 components none >9.3 percent TRR, 0.002 mg eq/kg.

Identification and characterization of radioactive residues [IP-5,8a-^{14}C]fluazaindolizine in wheat forage, hay, straw, and grain

Forage

Extractability of ^{14}C in [IP-5,8a-^{14}C] forage using methanol:water was good (83.2–89.9 percent TRR Table 43). The unextracted residues were subject to further investigations; these further treatments released an additional 8.4–11.4 percent TRR with terminal residues remaining in solids accounting for ≤ 5.5 percent TRR (Table 43).

Fluazaindolizine was identified in all plant-back intervals (0.6–3.9 percent TRR, ≤ 0.016 mg/kg) decreasing in later samples (Table 44). The principal extracted residue identified in forage was IN-QEK31 accounting for 25.9–40.8 percent TRR (0.051-0.248 mg eq/kg).

Other identified metabolites were the glucose conjugate of IN-QEK31 (IN-UGA20), IN-RYC33, and malic acid conjugate of IN-QEK31 (IN-UJU44), (all ≤ 7.8 percent TRR, ≤ 0.032 mg eq/kg) together with a glucose conjugate of IN-REG72 (≤ 2.8 percent TRR) and multiple conjugates of IN-QEK31 (≤ 2.6 percent TRR).

Multiple unidentified metabolites, including unretained components which may contain multiple metabolites, were also detected accounting for an aggregate total of 28.2–37.2 percent TRR in each sample but each individually was ≤ 6.7 percent TRR (≤ 0.039 mg eq/kg). A significant portion of the unknown metabolites were shown to hydrolyse to IN-QEK31 (Table 44).

Table 43 Extraction of residues in wheat forage from crops sown 30, 120, and 300 days after application of [IP 5,8a ^{14}C]-fluazaindolizine

Fraction	30DAA TRR=0.411 mg eq/kg	120DAA TRR= 0.198 mg eq/kg	300DAA TRR= 0.609 mg eq/kg
Extracted (MeOH/water)	89.9	83.2	87.6
Unextracted (MeOH/water)	10.1	16.9	12.4
Aqueous soak 1 (overnight)	1.3	0.4	0.6
Acetonitrile:water	2.5	1.0	2.3
Aqueous soak 2 (*ca.* 5 h, ambient)	<LOD	0.4	<LOD
Enzyme (Driselase)	2.9	4.2	1.2
0.1M HCl (*ca.* 50-60°C, 6 h)	1.2	1.9	1.8
1.0M HCl (*ca.* 80°C, 20 h)	1.7	2.7	1.8
0.1M NaOH (*ca.* 80°C, 4 h)	0.5	0.8	0.7
Remaining	NC	5.5	4.0

Table 44 Identification of TRR from wheat forage in various extracts and fractions from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]-fluazaindolizine (%TRR)[1]

	30DAA		120DAA	300DAA
TRR (mg eq/kg)	0.411	Post hydrolysis	0.198	0.609
Fluazaindolizine	3.9	-	1.0	0.6
IN-REG72	-	-		
Glucose conjugate of IN-REG72	2.8	-	-	1.8
Total whole molecule metabolites	6.7	-	1.0	2.4
IN-RYC33	7.8	1.8	2.1	1.5
Malic acid conjugate of IN-QEK31 (IN-UJU44)	-		2.0	-
methyl ester of IN-QEK31 (IN-R2W56)	2.4	2.6	1.2	0.6
Conjugate of IN-QEK31 (*ca.* 20 min)[A]	1.9	-	1.2	1.3
Conjugate of IN-QEK31 (*ca.* 21 min)[A]	3.7	-	2.1	2.5
Conjugate IN-QEK31 (*ca.* 27 min)[A]	2.6	-	2.2	-
Glucose conjugate of IN-QEK31 (IN-UGA20)	5.7	-	5.9	3.0
IN-QEK31	40.5 (36.8)	71.5	25.9	42.3
Total IN-QEK31 metabolites	64.6 (60.9)	75.9	42.6	51.2
Unidentified	24.5[C]/8.5[B] 22.4[F]/6.4[B]	13.9[G]/6.4	37.2[D]/17.3[B]	36.9[E]/14.2[B]

Notes:

[1] the numbers in parentheses are results, where different, of repeat analysis just prior to acid hydrolysis

[A] Glucose-polyol and other conjugates of IN-QEK31 as determined by LC-MS.

[B] total residue of multiple unidentified polar metabolites in the 0-5 min region of the chromatogram, which were further analysed in the IP-5,8a straw extracts and demonstrated to incorporate multiple components.

[C] Unidentified, including unretained components, consisting of 13 components none >5.2 percent TRR, 0.021 mg eq/kg.

[D] Unidentified, including unretained components, consisting of 15 components none >6.7 percent TRR, 0.013 mg eq/kg. The unretained residue was demonstrated to incorporate multiple components in the IP-5,8a straw extracts.

[E] Unidentified, including unretained components, consisting of 21 components none >6.5 percent TRR, 0.039 mg eq/kg. The unretained residue was demonstrated to incorporate multiple components in the IP-5,8a straw extracts.

[F] Unidentified, including unretained components, consisting of 13 components none >5.2 percent TRR, 0.021 mg eq/kg.

[G] Unidentified, including unretained components, consisting of 3 components none >3.9 percent TRR, 0.016 mg eq/kg.

Hay

Extractability of ^{14}C in [IP-5,8a-^{14}C] hay using methanol:water was good (76.4–84.7 percent TRR Table 45). The unextracted residues were subject to further investigations; these further treatments released an

additional 10.5–16.1 percent TRR with terminal residues remaining in solids accounting for ≤ 7.5 percent TRR (Table 45).

Fluazaindolizine was identified only in the 30 and 120DAA samples at low concentrations (0.7–1.4 percent TRR, ≤ 0.016 mg/kg; Table 46). The principal extracted residue identified were IN-QEK31 accounting for 17.4–26.5 percent TRR (0.066-0.303 mg eq/kg) and glucose conjugate of IN-QEK31 (IN-UGA20 5.8–16.9 percent TRR, 0.022-0.192 mg eq/kg).

Other identified metabolites were IN-RYC33 (≤ 5.8 percent TRR, ≤ 0.066 mg eq/kg), the inositol conjugate of IN-QEK31 (IN-UHD13) and methyl ester of IN-QEK31 (IN-R2W56), (both ≤ 4.8 percent TRR, ≤ 0.055 mg eq/kg) as well as a glucose conjugate of IN-REG72 (≤8.0 percent TRR, ≤ 0.091 mg/kg) and multiple conjugates of IN-QEK31 (≤ 4.0 percent TRR, ≤ 0.046 mg eq/kg).

Multiple unidentified metabolites, including unretained components which may contain multiple metabolites, were also detected accounting for an aggregate total of 12.5-48.9 percent TRR in each sample but each individually was ≤9.1 percent TRR (≤ 0.088 mg eq/kg). A significant portion of the unknown metabolites were shown to hydrolyse to IN-QEK31 (Table 46).

Several small acidic metabolites are also postulated in [IP-5,8a-^{14}C]fluazaindolizine wheat samples occurring in the polar region of the chromatogram were further divided into distinct multiple metabolites.

Table 45 Extraction of residues in wheat hay from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]fluazaindolizine (% TRR)

Fraction	30DAA TRR=1.143 mg eq/kg	120DAA TRR=0.377 mg eq/kg	300DAA TRR= 0.969 mg eq/kg
Extracted (MeOH/water)	80.5	84.7	76.4
Unextracted (MeOH/water)	19.4	15.3	23.6
Aqueous soak 1 (overnight)	1.2	0.5	2.0
Acetonitrile:water	5.5	1.3	4.3
Aqueous soak 2 (*ca.* 5 h, ambient)	0.5	0.4	0.3
Enzyme (Driselase)	3.2	3.5	1.6
0.1M HCl (*ca.* 50-60°C, 6 h)	2.0	1.6	2.7
1.0M HCl (*ca.* 80°C, 20 h)	2.9	2.4	3.9
0.1M NaOH (*ca.* 80°C, 4 h)	0.7	0.9	1.3
Remaining	3.4	4.8	7.5

Table 46 Identification of TRR from wheat hay in various extracts and fractions from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]fluazaindolizine (% TRR)[1]

	30DAA		120DAA	300DAA
TRR (mg/eq/kg)	1.143	Post hydrolysis	0.377	0.969
Fluazaindolizine	1.4	-	0.7	-
Glucose conjugate of IN-REG72	8.0 (7.7)	-	-	4.8
Total whole molecule metabolites	9.4 (9.1)	-	0.7	4.8
IN-RYC33	5.8	-	1.6	-
methyl ester of IN-QEK31 (IN-R2W56)	1.8	2.6	1.3	2.1
conjugate of IN-QEK31 (*ca.* 20 min)[A]	4.0	-	1.5	4.2
conjugate of IN-QEK31 (*ca.* 22 min)[A]	2.2	-	1.8	3.4
conjugate of IN-QEK31 (*ca.* 27 min)[A]	-	-	2.8	4.1
Inositol conjugate of IN-QEK31 (IN-UHD13)	4.8		-	-
Glucose conjugate of IN-QEK31 (IN-UGA20)	16.9 (16.5)	-	5.8	6.5
IN-QEK31	26.5 (25.3)	68.8	17.4	24.2
Total IN-QEK31 metabolites	62.0 (55.6)	71.4	32.2	44.5

	30DAA		120DAA	300DAA
TRR (mg/eq/kg)	1.143	Post hydrolysis	0.377	0.969
Unidentified[B]	12.5[C]/6.6 11.1[F]/5.2	9.1[G]/6.7	45.5[D]/21.3	37.2[E]/17.4

Notes:

[1] the numbers in parentheses are results, where different, of repeat analysis just prior to acid hydrolysis

[A] Glucose-polyol and other conjugates of IN-QEK31 as determined by LC-MS.

[B] Values indicate the total residue of multiple unidentified polar metabolites in the 0-5 min region of the chromatogram, which were further analysed in the IP-5,8a straw extracts and demonstrated to incorporate multiple components.

[C] Unidentified, including unretained components, consisting of 7 components none >4.2 percent TRR, 0.048 mg eq/kg. The unretained residue was demonstrated to incorporate multiple components in the IP-5,8a straw extracts.

[D] Unidentified, including unretained components, consisting of 21 components none >8.7 percent TRR, 0.033 mg eq/kg. The unretained residue was demonstrated to incorporate multiple components in the IP-5,8a straw extracts.

[E] Unidentified components, consisting of 29 components none >4.3 percent TRR, 0.042 mg eq/kg plus an unretained component of 9.1 percent TRR, 0.088 mg equiv/kg. The unretained residue was demonstrated to be composed of at least 3 components in the IP-5,8a straw extracts.

[F] Unidentified, including unretained components, consisting of 6 components none >4.2 percent TRR, 0.048 mg eq/kg.

[G] Unidentified, including unretained components consisting of 3 components none >4.0 percent TRR, 0.045 mg eq/kg.

Straw

Extractability of ^{14}C in [IP-5,8a-^{14}C] straw using methanol:water was poor (52.8–64.4 percent TRR Table 47). The unextracted residues were subject to further investigations; these further treatments released an additional 27.7-38.9 percent TRR with terminal residues remaining in solids accounting for ≤ 8.3 percent TRR (Table 47).

Fluazaindolizine was found only in the 30 and 120DAA samples (1.4-4.3 percent TRR, ≤ 0.153 mg/kg). The principal extracted residue was IN-QEK31 accounting for 12.3-24.9 percent TRR (0.217-1.018 mg eq/kg: Table 48).

Other identified metabolites were IN-REG72, the inositol acid conjugate IN-QEK31 (IN-UHD13), glucose conjugate of IN-QEK31 (IN-UGA20), methyl ester of IN-QEK31 (IN-R2W56), IN-RYC33, and malic acid conjugate IN-QEK31 (IN-UJU44), (all ≤ 6.0 percent TRR, ≤ 0.246 mg eq/kg) together with a glucose conjugate of IN-REG72 (≤6.3 percent TRR, ≤ 0.224 mg eq/kg) and several more complex conjugates of IN-QEK31 (≤ 3.5 percent TRR, ≤ 0.123 mg eq/kg).

Multiple unidentified metabolites were also detected accounting for an aggregate total of 49.4–61.0 percent TRR in each sample but each individually was ≤ 7.6 percent TRR (≤ 0.228 mg eq/kg). A significant portion of these unknown metabolites were shown to hydrolyse to IN-QEK31 (Table 48). In addition, an unretained region was observed accounting for 7.1–14.0 percent TRR (≤ 0.568 mg eq/kg). Additional chromatography of the polar region of the chromatogram demonstrated that this region was comprised of several components, none greater than 4.3 percent of the TRR.

Table 47 Extraction of residues in wheat straw from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]-fluazaindolizine (% TRR)

	30DAA TRR=3.547 mg eq/kg	120DAA TRR=1.357 mg eq/kg	300DAA TRR=4.0373 mg eq/kg
Extracted (MeOH/water)	62.2	64.4	52.8
Unextracted (MeOH/water)	37.8	35.6	47.2
Aqueous soak 1 (overnight)	1.4	4.7	8.5
Acetonitrile:water extract	16.7	8.2	19.3
Aqueous soak 2 (*ca.* 5 h, ambient)	NC	0.9	1.6

	30DAA TRR=3.547 mg eq/kg	120DAA TRR=1.357 mg eq/kg	300DAA TRR=4.0373 mg eq/kg
Enzyme (Driselase)	4.6	5.6	2.4
0.1M HCl (*ca.* 50-60°C, 6 h)	1.6	2.5	2.8
1.0M HCl (*ca.* 80°C, 20 h)	4.5	4.5	2.9
0.1M NaOH (*ca.* 80°C, 4 h)	1.6	1.3	1.4
Remaining	7.4	7.9	8.3

Notes:

NC = Not conducted.

Table 58 Identification of TRR from wheat straw in various extracts and fractions from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]-fluazaindolizine (% TRR)[1]

Metabolite	30DAA		120DAA	300DAA
TRR (mg eq/kg)	3.547	Post hydrolysis	1.357	4.0373
Fluazaindolizine	4.3 (3.5)	-	1.4	-
IN-REG72	0.9 (0.9)	-	-	-
Glucose conjugate of IN-REG72	6.3 (5.8)	-	-	0.2
Total whole molecule metabolites	11.5 (10.2)	-	1.4	0.2
IN-RYC33	1.9 (0.8)	-	0.3	1.5
Malic acid conjugate of IN-QEK31 (IN-UJU44)	0.4 (0.4)	-	1.1	-
Conjugate of IN-QEK31 (*ca.* 20 min)[A]	2.2 (1.8)	-	3.0	0.3
Conjugate of IN-QEK31 (*ca.* 21 min)[A]	3.0 (2.4)	-	2.4	0.3
Conjugate of IN-QEK31 (*ca.* 27 min)[A]	3.5 (3.1)	-	1.8	-
Inositol conjugate of IN-QEK31 (IN-UHD13)	-		-	2.3
Glucose conjugate of IN-QEK31 (IN-UGA20)	3.1 (1.1)	1.6	1.6	6.0
IN-QEK31	12.3 (5.4)	34.1	16.1	24.9
Methyl ester of IN-QEK31 (IN-R2W56)	3.8 (2.4)	2.1	-	4.7
Total IN-QEK31 metabolites	30.2 (17.4)	37.8	26.3	40
Volatile	0.5		-	-
Unidentified	49.4[C]/20.4[B] 29.8[F]/13.4[B]	24.3[G]/17.9[B]	61.0[D]/34.0[B]	49.7[E]/23.2[B]

Notes:

[1] the numbers in parentheses are results, where different, of repeat analysis just prior to acid hydrolysis

[A] Glucose-polyol and other conjugates of IN-QEK31 as determined by LC-MS.

[B] indicate the summed residue of multiple unidentified polar metabolites in the 0-5 min region of the chromatogram, and was demonstrated to be incorporated of multiple components.

[C] Unidentified consisting of 60 components none >6.4 percent TRR, 0.228 mg eq/kg.

[D] Unidentified consisting of 33 components none >7.6 percent TRR, 0.103 mg eq/kg.

[E] Unidentified consisting of 63 components none >3.9 percent TRR, 0.157 mg eq/kg.

[F] Unidentified, including unretained components, consisting of 30 components none >6.4 percent TRR, 0.228 mg eq/kg.

[G] Unidentified, including unretained components, consisting of 7 components none >8.5 percent TRR, 0.300 mg eq/kg.

Grain

Extractability of ^{14}C in [IP-5,8a-^{14}C] grain using methanol:water was poor (53.8-62.8 percent TRR Table 49). The unextracted residues were subject to further investigations; these further treatments released an additional 36.5-46.2 percent TRR with terminal residues remaining in solids accounting for 0.7 percent TRR (Table 49).

Fluazaindolizine was not found in the grain samples. The principal extractable residue identified in grain was IN-QEK31 accounting for a total of 58.7–64.6 percent TRR (0.337–0.889 mg eq/kg) although

these values incorporate the residues identified following further extracts, which may have become deconjugated during the extraction procedure (Table 50).

Other identified metabolites were the inositol conjugate of IN-QEK31 (IN-UHD13), glucose conjugate of IN-QEK31 (IN-UGA20), IN-RYC33, and methyl ester of IN-QEK31 (IN-R2W56) (all ≤ 2.9 percent TRR, ≤ 0.023 mg eq/kg; Table 50). Radioactivity in the chromatograms with a similar retention time to IN-VM862 was found only in the 30DAA grain sample and accounted for 1.1 percent TRR (0.016 mg eq/kg) but could not be confirmed due to its low concentration. Additional metabolites included a glucose conjugate of IN-REG72 (≤ 2.6 percent TRR, ≤ 0.040 mg eq/kg) and multiple conjugates of IN-QEK31 (≤ 1.9 percent TRR, ≤ 0.025 mg eq/kg).

Multiple unidentified metabolites, including unretained components, were also detected accounting for an aggregate total of 15.3–26.7 percent TRR in each sample but each individually was ≤ 7.8 percent TRR (≤ 0.041 mg eq/kg). Most of these components appeared to be conjugates of IN–QEK31 based on their formation of IN-QEK31 after hydrolysis (Table 50).

Table 49 Extraction of residues in wheat grain from crops sown 30, 120, and 300 days after application of [IP–5,8a–^{14}C] fluazaindolizine (% TRR)

	30DAA TRR=1.517 mg eq/kg	120DAA TRR=0.521 mg eq/kg	300DAA TRR=1.296 mg eq/kg
Extracted (MeOH/water)	59.8	53.8	62.8
Unextracted (MeOH/water)	40.2	46.2	37.2
Aqueous soak 1 (overnight)	13.8	12.4	10.2
Acetonitrile:water	5.9	9.1	6.6
Aqueous soak 2 (*ca.* 5 h, ambient)	<LOD	<LOD	<LOD
Enzyme (Driselase)	9.2	9.9	5.2
0.1M HCl (*ca.* 50-60°C, 6 h)	4.9	6.1	6.8
1.0M HCl (*ca.* 80°C, 20 h)	5.5	7.5	6.6
0.1M NaOH (*ca.* 80°C, 4 h)	1.0	1.2	1.0
Remaining	NC	NC	0.7

Notes:
NC = Not conducted.

Table 50 Identification of TRR from wheat grain in various extracts and fractions from crops sown 30, 120, and 300 days after application of [IP-5,8a-^{14}C]-fluazaindolizine (% TRR)[1]

Metabolite	30DAA	120DAA	300DAA	
TRR (mg eq/kg)	1.517	0.521	1.296	Post hydrolysis
Glucose conjugate of IN-REG72	2.6	-	2.2	-
Total whole molecule metabolites	2.6	-	2.2	-
IN-RYC33	-	-	0.7	-
Conjugate of IN-QEK31 (*ca.* 18 min)[A]	-	-	1.9	-
Conjugate of IN-QEK31 (*ca.* 20 min)[A]	-	1.5	0.9	-
Inositol conjugate of IN-QEK31 (IN-UHD13)	-	1.5	-	-
Glucose conjugate of IN-QEK31 (IN-UGA20	1.4	2.1	2.6	-
IN-QEK31)	72.8	64.6	62.1 (41.8)	58.8
methyl ester of IN-QEK31 (IN-R2W56)	1.5	2.9	1.1 (0.5)	-
Total IN-QEK31 metabolites	75.7	72.6	69.3 (46.5)	58.8
IN-VM862	1.1	-	-	-
Unidentified [B]	15.2[C]/8.4	26.1[D]/18.8	26.7[E]/9.9 14.1[F]/3.2	4.0[G]/3.1

Notes:
[1] the numbers in parentheses are results, where different, of repeat analysis just prior to acid hydrolysis

[A] Glucose-polyol and other conjugates of IN-QEK31 as determined by LC-MS.
[B] Values in parenthesis, included in the unidentified value, indicate the total residue of multiple unidentified polar metabolites in the 0-5 min region of the chromatogram, which were further analysed in the IP-5,8a straw extracts and demonstrated to incorporate multiple components.
[C] Unidentified, including unretained components, consisting of 14 components none >2.3 percent TRR and 0.035 mg eq/kg. The unretained residue was demonstrated to incorporate multiple components in the IP-5,8a straw extracts.
[D] Unidentified, including unretained components, consisting of 14 components none >7.8 percent TRR and 0.041 mg eq/kg. The unretained residue was demonstrated to incorporate multiple components in the IP-5,8a straw extracts.
[E] Unidentified, including unretained components, consisting of 26 components none >2.4 percent TRR and 0.032 mg eq/kg. The unretained residue was demonstrated to incorporate multiple components in the IP-5,8a straw extracts.
[F] Unidentified, including unretained components, consisting of 13 components none >2.2 percent TRR, 0.028 mg eq/kg.
[G] Unidentified, including unretained components, consisting of 6 components none >2.0 percent TRR, 0.025 mg eq/kg.

Characterisation of residues in soil

The total radioactive residues for soil sampled at various points, determined after extraction and analysis of unextracted residues are shown in Table 51 and Table 52.

The majority of the ^{14}C in the [Ph-^{14}C] soil was released by initial extractions, 77.3–99.5 percent TRR. Fluazaindolizine was identified in all samples. Other metabolites identified included IN-REG72, IN-F4106, IN-A5760, IN-QEK31, and IN-RYC33.

Similarly, in the [IP-5,8a-^{14}C] experiment soil the initial extractions released 77.1–99.6 percent TRR. Fluazaindolizine was identified in all samples. Other metabolites identified included IN-REG72, IN-QEK31, and IN-RYC33. A volatile metabolite was captured during the concentration of the [IP-5,8a-^{14}C] labelled soil extracts. A volatile metabolite, trapped using the same methods, was identified as IN-VM862 in other studies with fluazaindolizine. Due to the low levels of the volatile metabolite observed in this study (<4 percent TRR), no further characterization or confirmation was attempted.

Table 51 TRR (mg eq/kg) in soil after a soil drench application of [Ph-^{14}C]-fluazaindolizine (% TRR)

	0DAA TRR=3.058 mg eq/kg	30DAA TRR=1.601 mg eq/kg	120DAA TRR=0.606 mg eq/kg	300DAA TRR=0.379 mg eq/kg
Total Extracted [A]	99.5	91.7	76.4	77.3
Fluazaindolizine	99.5	61.4	27.3	19.6
IN-REG72	ND	4.3	ND	ND
IN-F4106	ND	26.0	49.1	51.5
IN-A5760	ND	ND	ND	6.2
Unextracted	0.5	8.3	23.6	22.7
Difference rounding	<0.1	ND	<0.1	<0.1
Total	100	100	100	100

Notes:
[A] Samples were extracted sequentially; once with acetonitrile:0.2 percent formic acid (aq) (9:1), once with acetonitrile:0.2 percent formic acid (aq) (4:1) and twice with acetonitrile:0.2 percent formic acid (aq) (1:1).

Table 52 TRR (% TRR) in soil after a soil drench application of [IP-5,8a-^{14}C]-fluazaindolizine

Fraction	0DAA TRR=4.655	30DAA TRR=3.628	71DAA TRR=0.456	120DAA TRR=0.662	300DAA TRR=1.307
Total Extracted[A]	99.6	91.6	94.4	86.9	77.1
Fluazaindolizine	99.5	48.8	41.8	23.0	32.0
IN-REG72	ND	2.9	4.0	2.3	3.5
IN-QEK31		28.3	40.1	51.8	33.4
IN-RYC33	ND	11.7	4.5	5.0	5.5
Volatile[B]	<0.1	0.6	4.0	1.4	1.2

Fraction	0DAA TRR=4.655	30DAA TRR=3.628	71DAA TRR=0.456	120DAA TRR=0.662	300DAA TRR=1.307
Unextracted	0.3	7.9	5.6	13.1	22.9
Difference rounding	0.1				1.5[C]
Total	100	100	100	100	100

Notes:

[A] Samples were extracted sequentially; once with acetonitrile:0.2 percent formic acid (aq) (9:1), once with acetonitrile:0.2 percent formic acid (aq) (4:1) and twice with acetonitrile:0.2 percent formic acid (aq) (1:1). The percent TRR values and mg/kg values have been calculated from the total TRR. The limit of detection was determined by the Currie method.

[B] During concentration process, a PU plug was placed in the flask to trap any volatile metabolites, this was rinsed with methanol and quantified by LSC.

[C] Unknown metabolite.

Uptake and metabolism of fluazaindolizine in rotational crops

The metabolic pathway for fluazaindolizine in rotational crops following soil application is presented in Figures 6 to 8 and is proposed based on the metabolites identified in wheat, spinach, and radish.

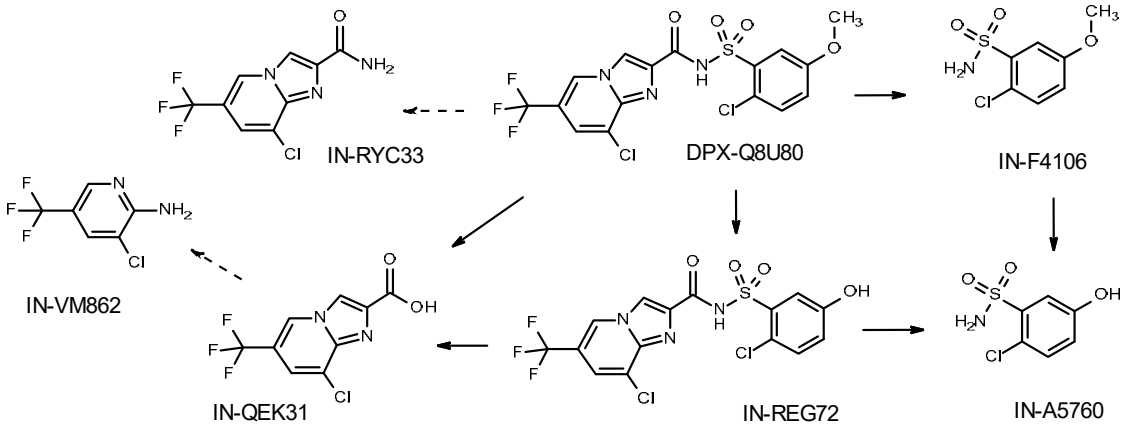


Figure 6 Proposed metabolic pathway of fluazaindolizine in soil from rotational crops

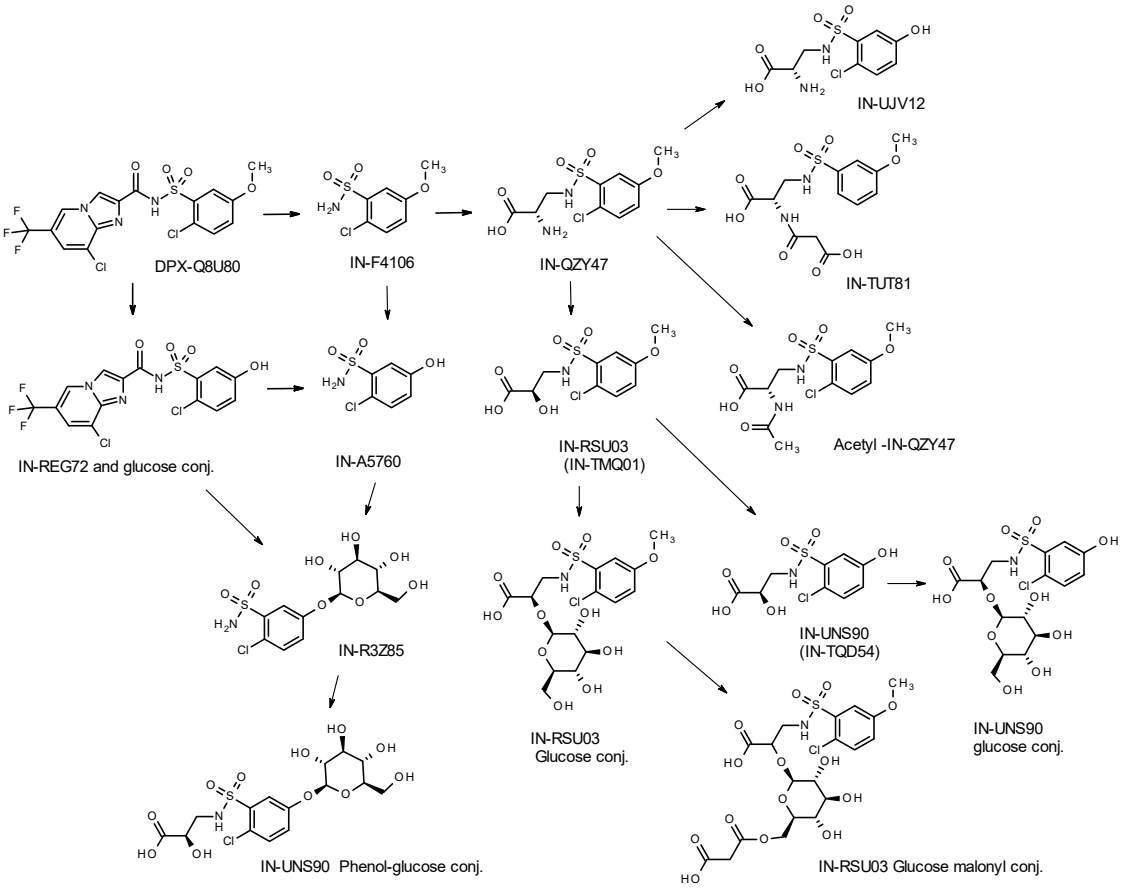


Figure 7 Proposed metabolic pathway of fluazaindolizine in rotational crops (metabolites containing [Ph-^{14}C] radiolabel

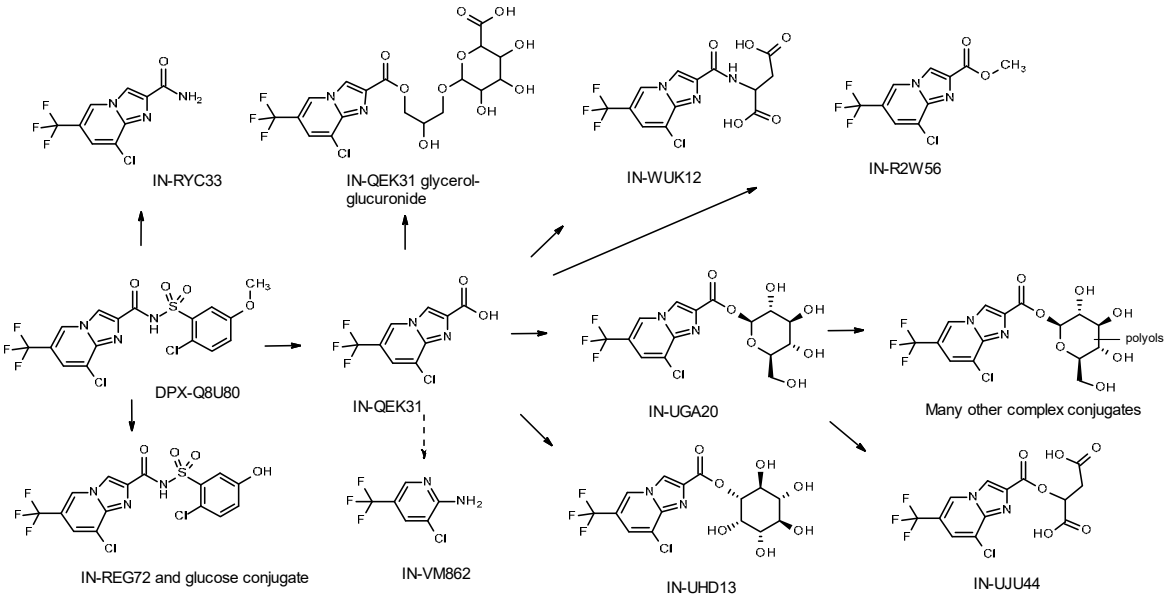


Figure 8 Proposed metabolic pathway of fluazaindolizine in rotational crops (metabolites containing [IP-5,8a-^{14}C] radiolabel)

Metabolic pathways of fluazaindolizine and its soil metabolites were similar across all the rotational crops and rotational intervals. Differences observed in the various crops were mainly in the degree and type of more complex conjugation with endogenous constituents that occurred.

Fluazaindolizine, IN-F4106, and IN-QEK31 were the major residues found in the soil which were available for uptake by plants. IN-F4106 and IN-QEK31 exceeded 50 percent of the residue by the 120-300 day plant-back intervals, while IN-RYC33, IN-REG72, and IN-A5760 were found between 2.3 and 11.7 percent TRR at various sampling intervals. IN-VM862, a soil metabolite containing only the pyridinyl ring was not observed in significant amounts in the soil (<4 percent TRR determined *via* capture of volatile residues). The major metabolic route in the soil was the hydrolysis of fluazaindolizine at the amide bond, resulting in IN-F4106 and IN-QEK31. Fluazaindolizine was also *O*-demethylated to form IN-REG72, which also was hydrolysed to IN-A5760 and IN-QEK31. A less prominent pathway was hydrolysis of fluazaindolizine at the sulfonamide bond, resulting in IN-RYC33 or the further degradation of IN-QEK31 to IN-VM862.

Fluazaindolizine was taken up and metabolised in the various rotational crops primarily by hydrolysis of the amide bond to IN-F4106 (observed in the [Ph–^{14}C]fluazaindolizine experiment) and to IN-QEK31 (observed in the [IP-5,8a-^{14}C]fluazaindolizine experiment). IN-F4106 and IN-QEK31 were the major metabolites found in soil at the various plant-backs and were likely taken up from soil directly. Hydrolysis of fluazaindolizine at the sulfonamide bond occurred to a lesser extent and resulted in forming IN-RYC33 from the [IP-5,8a-^{14}C]fluazaindolizine which could also be taken up directly from the soil into the various crops. Fluazaindolizine was metabolised to IN-REG72 *via O*-demethylation and found mostly in crops as its glucose conjugate. IN-F4106 and IN-QEK31 residues, whether formed in the plant or taken up from the soil were metabolised and conjugated to a significant degree. IN-F4106 was conjugated to serine to form IN-QZY47. The free amino moiety of IN-QZY47 was conjugated with malonic acid to form IN-TUT81 or acetylated, as found in spinach. The amino moiety of IN-QZY47 was also oxidatively deaminated to the lactic acid derivative, IN-RSU03, which was found only as the *R*-enantiomer, IN-TMQ01. IN-RSU03 (as IN-TMQ01) was conjugated to glucose and subsequently to malonic acid. IN-RSU03 underwent *O*-demethylation to form IN-UNS90 (as *R*-enantiomer IN-TQD54) and was found mostly conjugated to glucose at the 2-hydroxy propionic and/or phenolic positions, which were the major metabolites in wheat commodities. IN-REG72 and its glucose conjugate were metabolised to IN-A5760 and to IN-R3Z85 (IN-A5760 glucose conjugate). IN-UJV12 was formed from the *O*-demethylation of IN-QZY47 and was further conjugated. Several more complex conjugates originating from the [Ph Proposed metabolic pathway of fluazaindolizine in rotational crops (metabolites containing [Ph-^{14}C] radiolabel–^{14}C]fluazaindolizine were hydrolysed with 1N HCl to IN-F4106, IN-A5760, IN-QZY47, IN-RSU03, IN-UNS90, and IN-UJV12.

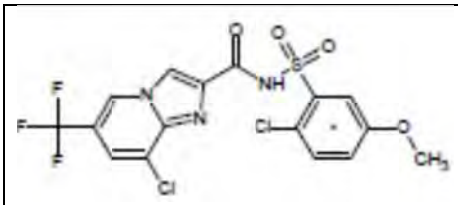
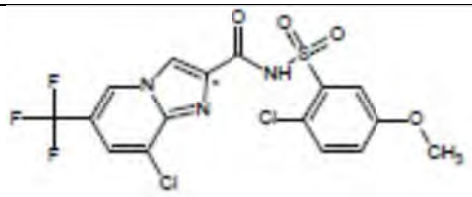
IN-QEK31 unconjugated and/or conjugated with various endogenous constituents, were the predominant metabolites identified from the [IP-5,8a-^{14}C]fluazaindolizine. IN-QEK31 was the major metabolite in wheat forage, hay, grain, and spinach. Glucose conjugation of IN-QEK31 through the acyl moiety to form IN-UGA20 appeared to be the initial conjugation reaction, while conjugation to inositol sugar derivative (IN-UHD13) occurred at lower levels in the various crops. The simple glucose conjugates of IN-QEK31 underwent acyl migration forming multiple isomers. In wheat, more complex glucose-polyols derivatives were also proposed to be formed based on mass spectral analysis. The glucose conjugate of IN-QEK31 in radish was largely displaced with malic acid, to form IN-UJU44. Other IN-QEK31 conjugates identified included a glycerol glucuronide found primarily in spinach, and a glutamic acid conjugate (IN-WUK12) found in radish. IN-RYC33, IN-REG72 and/or its glucose conjugate were also found in all rotated crops. The methyl ester of IN-QEK31 (IN-R2W56) was formed to a small extent and could be an artefact of the extraction methodology or a metabolite. Most residues found in rotated crops from the [IP-5,8a-^{14}C]fluazaindolizine were hydrolysed to IN-QEK31 with 1N HCl. Certain highly polar residues composed of multiple components were present in various wheat straw extracts and accounted for a sum of 15.3 percent TRR. These polar components were resistant to HCl hydrolysis and, where analysed, no chromatographically separated polar metabolites from this polar region exceeded 4.3 percent TRR upon further HPLC separation. These components may be formed from the further degradation of IN-QEK31 or

may be the result of $^{14}CO_2$ incorporation into endogenous constituents. The soil metabolite IN-VM862 was found as a minor component in wheat straw < 1 percent TRR and may account for 1.1 percent TRR in wheat grain, based on the radioactive region co-eluting at its HPLC retention time, but appeared to be insignificant in other crop matrices (<LOD).

Identified residues found at greater than 10 percent TRR in any crop commodity at any PBI (30, 120, or 300DAA) are presented as sum total of metabolites and their corresponding conjugates (e.g., IN-UNS90 and glucose conjugates expressed as IN-UNS90, IN-QZY47 and its malonyl conjugate IN-TUT81 expressed as IN-QZY47, etc.). Those residues for wheat are IN-UNS90 (IN-TQD54), IN-RSU03 (IN-TMQ01), and IN-QEK31, but in spinach are fluazaindolizine, IN-REG72, IN-QZY47, IN-QEK31, and on acid hydrolysis IN-F4106 (identified residues in rotated crops >10 percent TRR). In radish the residues are fluazaindolizine, IN-RSU03, IN-QZY47, IN-UNS90, IN-QEK31, and on acid hydrolysis IN-F4106 (identified residues in rotated crops >10 percent TRR). This also includes any degradates greater than 10 percent TRR released by hydrolysis.

Animal metabolism

Metabolism studies with livestock were carried out with [Ph-^{14}C (U)]-fluazaindolizine or [imidazo-(1,2-α)-pyridine-2-^{14}C]-fluazaindolizine.

	
[phenyl-^{14}C (U)]fluazaindolizine	[imidazo[1,2-a]pyridine-2-^{14}C]fluazaindolizine

Livestock (goat) studies

Wicksted (2014 DuPont-33573, Revision No. 1) investigated the metabolism of fluazaindolizine in lactating goats administered either as [Ph-^{14}C]fluazaindolizine or [IP-2-^{14}C]fluazaindolizine.

Two goats (Saanen/Toggenburg cross breed, 2-4 years, 42-44 kg bw) were dosed orally *via* capsules, once a day for seven consecutive days, with [Ph-^{14}C]-fluazaindolizine or [IP-2-^{14}C]-fluazaindolizine, at doses equivalent to 12.2 and 11.8 ppm of fluazaindolizine in the diet. The average feed consumption of 1.393-1.819 kg/day during the dosing period. These doses were equivalent to an average dose of 0.420 and 0.479 mg fluazaindolizine/kg bw, respectively. Milk production was 2.2 kg/day. Milk samples were collected twice a day while urine and faeces samples were collected daily. Goats were sacrificed 6 hours after the final dose, samples of whole liver, both kidneys, muscle (composite of loin, hind, and fore quarter muscle in approximately equal proportions) and fat (individual omental, renal, and subcutaneous fat samples) were collected from each goat. Composite milk (Day 4-6), urine and faeces (Day 1-7) samples were prepared for analysis for both dose groups. Milk, tissues, and excreta were extracted and analysed within 189 days (6 months) of sampling.

Analytical procedures

Tissue and fat samples were homogenised in dry ice. Equal amounts of milk from Days 4–6 were pooled by dose group. Aliquots of the composited whole milk samples were also centrifuged (3000 rpm; 10 min) to determine the radioactive distribution in the cream and skimmed milk fractions prior to storage at ca. -

20 °C and assayed by LSC. Subsamples of tissues and milk were extracted with acetonitrile:0.1 M ammonium formate, pH 7 (9:1). The resulting extracts were combined by matrix, concentrated, and analysed by LSC and HPLC. The liver PES from both goats were further treated with protease (37 °C; 72 hours) to release unextracted residues. All extracts were stored at -20 °C prior to HPLC.

The total recovery was 96.7-108 percent of the administered dose for both goats. For both [^{14}C]-labels, 79.9 and 89.7 percent of the dose was recovered from the urine, faeces, bile, and cage wash with a further 15.9 and 17.4 percent found in the gastrointestinal tract (Table 53). About 0.9 percent of the dose was recovered in edible tissues and another 0.1 percent of the dose was recovered in the milk. Radioactivity plateaued in milk within 3 days at 0.05–0.06 mg eq/kg for the [Ph-^{14}C]fluazaindolizine and 0.04-0.05 mg eq/kg for the [IP-2-^{14}C]fluazaindolizine (Table 53). TRR in the composite milk (Day 4–6) from goats dosed with [Ph-^{14}C]fluazaindolizine partitioned equally in cream (0.050 mg eq/kg) in skim milk (0.054 mg eq/kg). TRR in the composite milk from goats dosed with [IP-2-^{14}C]fluazaindolizine partitioned slightly higher in cream (0.063 mg eq/kg) *vs.* skim milk (0.046 mg eq/kg), however, overall, this data indicated no selective partitioning as residues (less than 2-fold) in the milk fractions from goats dose with either radiolabel.

TRR levels in edible tissues were 0.223 mg eq/kg in liver, 0.358 in kidney, 0.011 in muscle, 0.015 in omental fat, 0.028 in renal fat and 0.024 mg eq/kg in subcutaneous fat from the [Ph-^{14}C]fluazaindolizine dosed goat, and 0.275 mg eq/kg in liver, 0.357 in kidney, 0.010 in muscle, 0.008 in omental fat, 0.014 in renal fat and 0.013 mg eq/kg in subcutaneous fat from the [IP-2-^{14}C]fluazaindolizine dosed goat. There was not a selective partitioning into skim milk or cream or into the various fat types (Tables 53 and 54). The residues in whole milk from day 1 to 6 os shown in Figure 9.

Extractability from tissues with acetonitrile:0.1 M ammonium formate, pH 7 (9:1) was good for liver, kidney, muscle, and fat (>85 percent TRR) and Day 4-6 milk (>96.9 percent TRR). Digestion of liver PES with protease released ^{14}C, overall ≥96 percent TRR. Extracted [^{14}C]-residues were analysed and quantified by reverse phase HPLC, and metabolite identities were confirmed by co-chromatography with reference standards and by LC-MS.

Table 53 Percent administered dose recovered in milk, tissues and excreta from lactating goats following seven consecutive daily oral doses of [Ph-^{14}C]fluazaindolizine or [IP-2-^{14}C]fluazaindolizine

Sample	[Ph-^{14}C]fluazaindolizine	[IP-2-^{14}C]fluazaindolizine
Tissues	0.8	0.9
Liver	0.3	0.4
Kidney	0.5	0.5
Muscle[A]	<0.1	<0.1
Omental fat[A]	<0.1	<0.1
Renal fat[A]	<0.1	<0.1
Subcutaneous fat[A]	<0.1	<0.1
Milk (Total Day 1-7)	0.1	<0.1
Faeces	50.6	52.3
Urine	32.9	21.3
Cage wash	3.3	1.5
Bile	2.9	4.8
GI tract contents	14.5	13.2
GI tract	2.9	2.7
Total	108	96.7

Notes:

[A] Total muscle mass was assumed to be approximately 25 percent of body weight, total fat approximately 15 percent of body weight. Each fat type accounted for the following percentages of total bodyweight, renal fat *ca.* 0.9 percent, omental fat *ca.* 4.1 percent and subcutaneous fat *ca.* 9.4 percent.

Table 54 Daily TRR in whole milk following oral administration of [Ph-^{14}C] or [IP-2-^{14}C]fluazaindolizine to lactating goats for 7 consecutive days

Sampling		[Ph-^{14}C]fluazaindolizine	[IP-2-^{14}C]fluazaindolizine
Day	Hour	Concentration (mg eq/kg)	Concentration (mg eq/kg)
1	24	0.037	0.031
2	48	0.046	0.040
3	72	0.049	0.043
4	96	0.047	0.042
5	120	0.059	0.047
6	144	0.055	0.043
7 [A]	150	0.064	0.068

Notes:

[A] Only partial day milk collection.

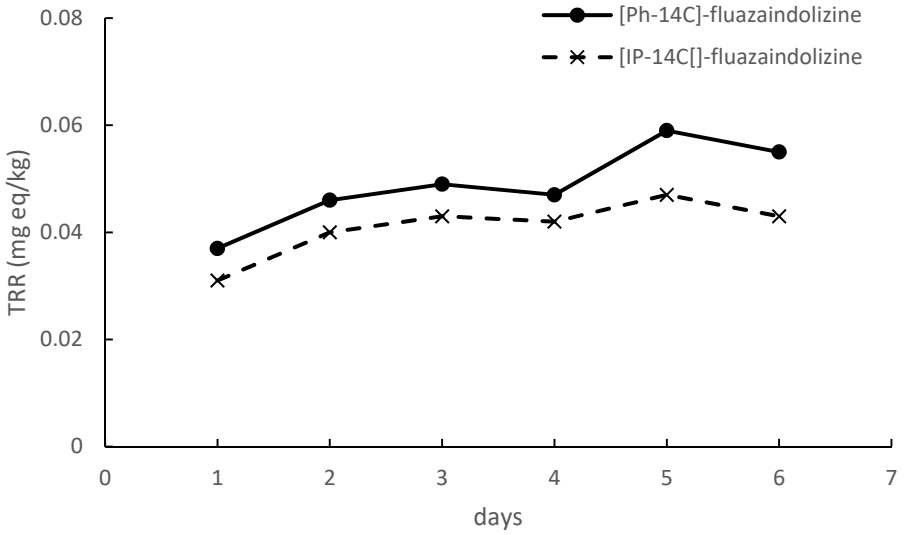


Figure 9 Residues in milk following dosing with ^{14}C-fluazaindolizine (note last milk sample not plotted as does not represent a 24-hour sample, i.e. residues in milk are higher in the first milking collected after dosing).

Identification and characterisation of radioactive residues

The majority of radioactive residues (73.3–92.3 percent TRR) was identified and/or characterised in tissues from the [Ph-^{14}C]fluazaindolizine dosed goat (Table 55). Fluazaindolizine accounted for 17.5-84.6 percent TRR in tissues and milk. The phenyl-derived metabolites included IN-A5760 (maximum 4.1 percent TRR), IN-F4106 (maximum 38.4 percent TRR) and IN-REG72 (maximum 7.0 percent TRR). Several minor unidentified metabolites were also detected, none of which individually were greater than 7.3 percent TRR, which combined accounted for 5.5–12.8 percent TRR in milk and tissues.

The majority of radioactive residues (83.1-100 percent TRR) was identified and/or characterised in tissues from the [IP-2-^{14}C]fluazaindolizine dosed goat (Table 56). Fluazaindolizine (parent) accounted for 25.0-83.2 percent TRR in tissues and milk. The imidazopyridine-derived metabolites included IN-QEK31 (maximum 42.8 percent TRR), IN-REG72 (maximum 11.6 percent TRR) and methyl ester of IN-QEK31 (IN-R2W56) (maximum 0.6 percent TRR). Several minor unidentified metabolites were also

detected, none of which individually were greater than 4.0 percent TRR, which combined accounted for 4.0–8.3 percent TRR in milk and tissues.

Table 55 Identification/characterization of radioactivity in milk and tissues from [Ph-^{14}C]-fluazaindolizine dosed lactating goat (% TRR)

	Milk (day 6)	Liver	Kidney	Muscle	Omental fat	Renal fat	Subcutaneous fat
TRR (mg eq/kg)	0.056	0.222	0.358	0.011	0.014	0.028	0.025
Extracted MeCN/ammonium formate	90.2	80.4	91.5	91.2	89.7	89.8	92.2
IN-A5760	ND	3.4	0.8	3.4	ND	ND	ND
IN-F4106	ND	37.9	7.4	25.6	9.8	6.7	7.1
IN-REG72	ND	6.5	2.6	ND	ND	ND	ND
Fluazaindolizine	84.6	17.5	65.3	62.1	67.0	71.7	76.2
Unidentified metabolites	5.5[A]	2.1[B]	8.6[D]	NA	12.8[E]	11.5[F]	9.0[G]
Losses		13.1	6.6				
Unextracted MeCN/ammonium formate	0.7	17.7	6.2	7.7	9.5	5.7	2.4
Protease		13.6					
IN-A5760		0.7					
IN-F4106		0.6					
IN-REG72		0.5					
Unidentified metabolites		4.3[C]					
Remaining		4.1					

Notes:
ND Not detected.
NA Not applicable.
[An] individually ≤ 0.002 mg eq/kg, ≤3 percent TRR.
[B] individually ≤ 0.003 mg eq/kg, ≤ 1.2 percent TRR.
[C] individually ≤ 0.005 mg eq/kg, ≤ 2.2 percent TRR.
[D] individually ≤ 0.023 mg eq/kg, ≤6.3 percent TRR.
[E] individually ≤ 0.001 mg eq/kg, ≤6.7 percent TRR.
[F] individually ≤ 0.001 mg eq/kg, ≤ 3.7 percent TRR.
[G] individually ≤ 0.001 mg eq/kg, ≤4.3 percent TRR.

Table 56 Identification/characterisation of radioactivity in milk and tissues from [IP-2-^{14}C]-fluazaindolizine dosed lactating goat (% TRR)

	Milk (day 4-6)	Liver	Kidney	Muscle	Omental fat	Renal fat	Subcutaneous fat
TRR (mg eq/kg)	0.044	0.275	0.357	0.010	0.008	0.014	0.012
Extracted MeCN/ammonium formate	88.9	90.5	96.5	88.7	83.1	86.8	87.8
Methyl ester of IN-QEK31 (IN-R2W56)	ND		ND	ND	ND	ND	ND
IN-QEK31	7.6	38.8	11.0	1.9	ND	ND	1.7
IN-REG72	5.0	10.7	3.3	ND	5.0	ND	2.9
Fluazaindolizine	72.3	25.0	78.7	79.3	78.1	78.5	83.2
Unidentified metabolites	4.0	5.8[A]	NA	7.5[C]	NA	8.3[D]	NA
Losses		10.3	3.5				
Unextracted MeCN/ammonium formate	3.1	9.6	2.1	8.3	15.0	5.7	3.2
Protease		9.6					
methyl ester of IN-QEK31 (IN-R2W56)		0.6					
IN-QEK31		4.0					
IN-REG72		0.9					
Unidentified metabolites		1.5[B]					
Losses		2.6					
Remaining		<0.1					

Notes:

ND Not detected.
NA Not applicable.
[A] individually ≤ 0.005 mg eq/kg, ≤ 1.7 percent TRR.
[B] individually ≤ 0.002 mg eq/kg, ≤ 0.7 percent TRR.
[C] individually ≤ 0.001 mg eq/kg, ≤ 3.2 percent TRR.
[D] individually ≤ 0.001 mg eq/kg, ≤ 2.4 percent TRR.

Identification/characterisation of urine, faeces, and bile metabolites

In Day 1-7 urine composite from a goat dosed with [Ph-^{14}C]-fluazaindolizine, unchanged fluazaindolizine accounted for 25.2 percent of the total administered dose. The metabolites IN-A5760 (4.1 percent dose), IN-F4106 (0.2 percent dose) and IN-REG72 (1.0 percent dose) were identified. Several minor unknown components were detected none of which individually accounted for >1.0 percent dose.

In Day 1–7 faeces composite from a goat dosed with [Ph-^{14}C]-fluazaindolizine, unchanged fluazaindolizine accounted for 30.8 percent of the total administered dose. The metabolite IN-REG72 (7.9 percent dose) was identified. Several minor unknown components were detected, however, none of which individually accounted for >0.9 percent dose.

Bile from a goat dosed with [Ph-^{14}C]fluazaindolizine contained metabolites IN-A5760 (<0.1 percent dose), IN-F4106 (0.1 percent dose) and IN-REG72 (2.7 percent dose). Two minor unknown components were detected, neither of which individually accounted for >1.0 percent dose.

In Day 1-7 urine composite from a goat dosed with [IP-2-^{14}C]fluazaindolizine, unchanged fluazaindolizine accounted for 19.0 percent of the total administered dose. The metabolites IN-QEK31 (1.5 percent dose) and IN-REG72 (0.7 percent dose) were identified. Two minor unknown components were detected, neither of which individually accounted for >0.1 percent dose.

For Day 1-7 faeces composite from a goat dosed with [IP-2-^{14}C]fluazaindolizine, unchanged fluazaindolizine accounted for 27.7 percent of the total administered dose. The metabolites, IN-QEK31 (0.8 percent dose), IN-REG72 (6.5 percent dose) and IN-RYC33 (0.1 percent dose) were identified. Several minor unknown components were also detected, none of which individually accounted for >3.9 percent dose.

Bile from a goat dosed with [IP-2-^{14}C]fluazaindolizine contained unchanged fluazaindolizine which accounted for <0.1 percent of the total administered dose. The metabolites IN-QEK31 (<0.5 percent dose), IN-REG72 (3.9 percent dose) and IN-R2W56 (0.1 percent dose) were identified. Two minor unknown components were detected, neither of which individually accounted for >0.3 percent dose.

Proposed metabolic pathway of fluazaindolizine in the goat

The proposed metabolic pathway of [^{14}C]fluazaindolizine was shown in Figure 10. The metabolism of fluazaindolizine was complex and was based on metabolites identified in the tissues and in the excreta. The following metabolic pathway was proposed: biotransformation of fluazaindolizine in the goat occurred primarily through O-demethylation and hydrolysis of the amide bond to form the cleaved metabolites IN-QEK31 and IN-A5760 via IN-REG72. Direct hydrolysis of fluazaindolizine gave metabolites IN-F4106 and IN-QEK31. IN-RYC33 was formed from the hydrolysis of the sulphonamide bond of fluazaindolizine. The metabolite IN-R2W56 was formed via methylation of the carboxylic acid group on IN-QEK31.

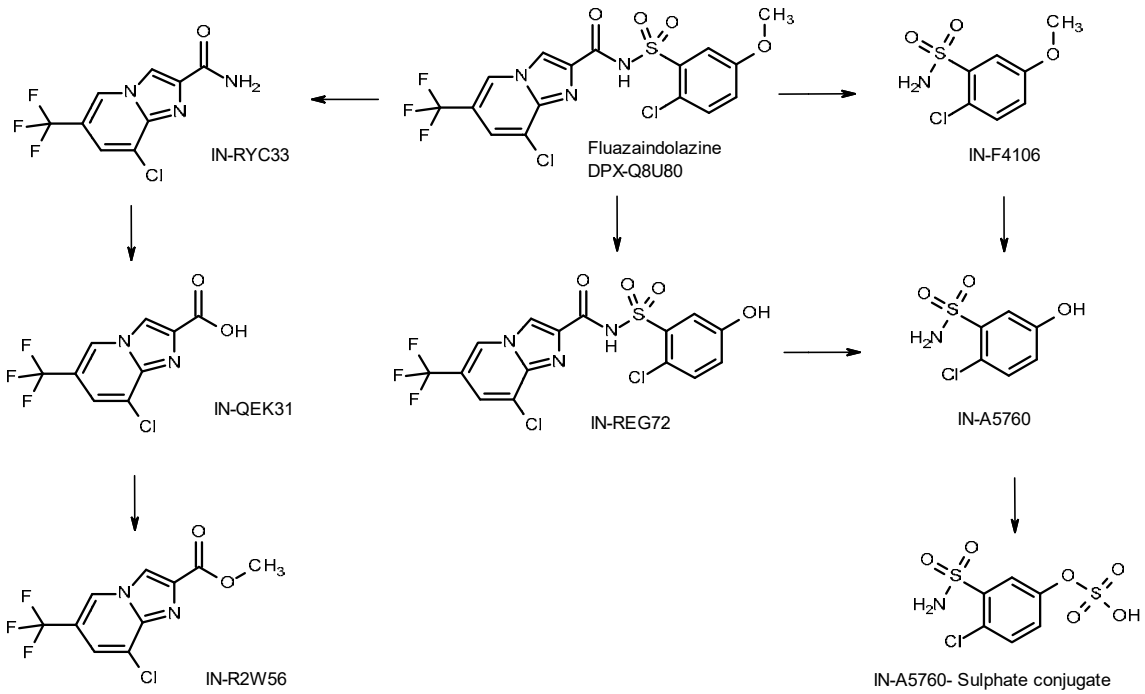
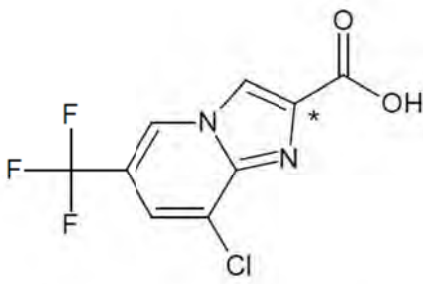


Figure 10 Proposed metabolic pathway of fluazaindolizine in lactating goats

Metabolism of [^{14}C]IN-QEK31 in lactating goats

Cochrane (2017a DuPont-41870) studied the metabolism of [^{14}C]-IN-QEK31 in the lactating goat.



* – Denctes position of radiolabel

[IP-2-^{14}C]-IN-QEK31 was administered *via* capsules to a lactating goat (Saanen/Alpine cross, 4 years old, 44–45 kg bw) as a single daily oral dose for five consecutive days. The average daily dose administered to the goat was 12.469 ppm [IP-two-^{14}C]-IN-QEK31 in the diet (dw equivalent). The dose level was equivalent to an average dose of 0.348 mg IN-QEK31/kg bw/day. Milk production was 2.2 kg/day. Feed consumption was 1.26 kg/day.

Faeces and urine were collected once daily and milk collected twice daily. The goat was sacrificed approximately 6 h after the last dose and liver, kidney, muscle, omental fat, renal fat, subcutaneous fat, bile, gastrointestinal tract, and contents were collected.

All samples were stored at *ca.* -20 °C until taken for analysis. All samples were extracted within 13 days and extracts initially analysed within 52 days following the extraction procedure. The longest storage interval (between sample collection and analysis of sample extracts) recorded for any individual sample (liver, kidney, and renal fat) was 63 days (*ca.* 2 months).

Analytical procedures

Tissue samples were homogenised with the aid of dry ice. Composite samples (Days 1-5) of milk, faeces, and urine were prepared by combining equal amounts of each daily sample by matrix. Subsamples of tissues, milk (Days 1-5 composite) and faeces (Days 1-5 composite) were prepared for metabolite extraction and analysis. Samples were extracted with acetonitrile:0.1 M ammonium formate, pH 7 (9:1). The liver PES from the goat were further treated with dilute acid (acetonitrile:0.1M hydrochloric acid (1:4)) to release unextracted residues. All extracts were stored at -20°C prior to HPLC.

The total recovery was 87.1 percent of the administered dose, of which 73.1 percent of the dose was recovered from the urine, faeces, and cage wash with a further 11.8 percent found in the gastrointestinal tract (Table 57), 2.1 percent of the dose was recovered in the milk. Radioactivity in milk reached plateau within 3 days post first dose *ca.* 0.169 mg eq/kg (Table 58). TRR in the composite milk (Day 1-5) were separated into cream (0.105 mg eq/kg) and skim milk (0.170 mg eq/kg) fractions by LSC analysis following physical separation (centrifugation). This data indicated no selective partitioning as residues (less than 2-fold) in the milk fractions from goats dosed with IN-QEK31. Negligible amounts of radioactivity were recovered in the edible tissues (< 0.1 percent of the administered total dose).

TRR in edible tissues were 0.035 mg eq/kg in liver, 0.282 in kidney, <0.001 in muscle, 0.005 in omental fat, 0.046 in renal fat and 0.002 mg eq/kg in subcutaneous fat (Table 57). Residues in milk from day 1 to 4 is shown in Figure 11.

Table 57 Percent administered dose recovered in milk, tissues and excreta from a lactating goat following five consecutive daily oral doses of [IP-2-^{14}C]-IN-QEK31

Sample	% dose
Tissues	<0.1
Liver	<0.1
Kidney	<0.1
Muscle	<0.1
Omental Fat	<0.1
Renal Fat	<0.1
Subcutaneous Fat	<0.1
Milk[A]	2.1
Faeces	14.4
Urine	57.1
Cage Wash	1.7
GI Tract Contents	11.8
Total	87.1

Notes:

[A] TRR in the composite milk (Day 1-5) were separated into cream (0.105 mg eq/kg) and skim milk (0.170 mg eq/kg) fractions by LSC analysis following physical separation (centrifugation).

Table 58 TRR in milk following daily oral administration of [IP-2-^{14}C]-IN-QEK31 to a goat for five consecutive days

Sampling Time (day)	Sampling Time (hours)	Total mg excreted/day	Concentration (mg eq/kg)
1	24	0.296	0.148

Sampling Time (day)	Sampling Time (hours)	Total mg excreted/day	Concentration (mg eq/kg)
2	48	0.316	0.136
3	72	0.388	0.169
4	96	0.369	0.172
5[A]	104[B]	0.209	0.342

Notes:

[A] Partial day milk sample (0-6 hr) after last dose.

[B] 104 h refers to the sample ID only, the goat was milked directly before sacrifice which was *ca.* 102 hour (6 hour after last dose).

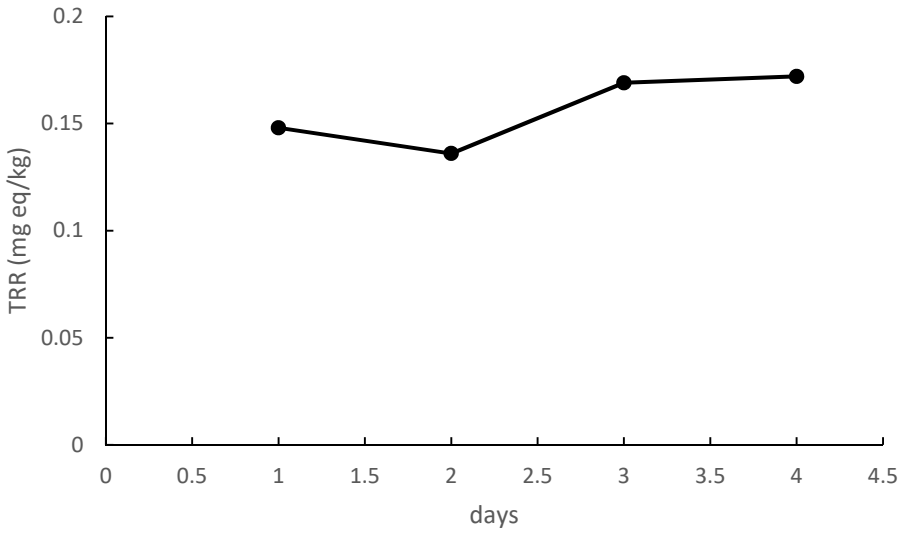


Figure 11 Residues in milk following dosing with ^{14}C-IN-QEK31 (note last milk sample not plotted as does not represent a 24-hour sample, i.e. residues in milk are higher in the first milking collected after dosing).

Identification and characterization of radioactive residues

Extractability of milk and tissues with acetonitrile:(100 mM ammonium formate) (9:1) was good with > 90 percent TRR released, the exception being liver for which 79.7 percent TRR was released. Extraction of the bound liver residues with more polar and acidic extraction methods did not release any further radioactivity; however, the levels of bound residue were low 0.007 mg eq/kg.

Identification/characterization of tissue residues

The majority of radioactive residues (74.1–95.5 percent TRR) was identified and/or characterised in tissues. Unchanged IN-QEK31 accounted for 74.3–95.4 percent TRR in tissues and milk. A single metabolite was present at greater than 10 percent TRR which was identified as IN-R2W56 and detected only in renal fat (Table 59). Several minor unidentified metabolites were also detected, none of which individually were greater than 5.0 percent TRR, which combined accounted for 6.2 percent TRR in milk and tissues.

Table 59 Identification of radioactivity in milk and tissues from [IP-2-^{14}C]-IN-QEK31 dosed lactating goat (% TRR)

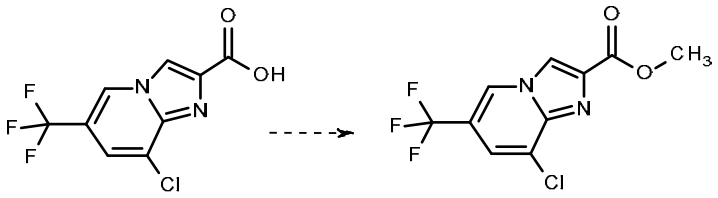
	Milk, TRR= 0.168 mg eq/kg	Liver, TRR= 0.035 mg eq/kg	Kidney, TRR= 0.282 mg eq/kg	Renal fat. TRR= 0.046 mg eq/kg

	Milk, TRR= 0.168 mg eq/kg	Liver, TRR= 0.035 mg eq/kg	Kidney, TRR= 0.282 mg eq/kg	Renal fat. TRR= 0.046 mg eq/kg
Extracted MeCN/ammonium formate	95.4	74.1	95.5	90.9
IN-QEK31	95.4	69.1	94.3	74.3
methyl ester of IN-QEK31 (IN-R2W56)	ND	ND	ND	16.6
Unidentified metabolites[A]	NA	5.0	1.2	NA
Fractions not analysed	3.8	5.6	NA	NA
Unextracted MeCN/ammonium formate	0.8	20.3	4.5	9.1

Notes:
NA = Not applicable.
ND = Not detected.
[A] Sum of all unidentified radioactivity in chromatograms, no single metabolite greater than 5.0 percent TRR or 0.004 mg eq/kg

Unchanged IN-QEK31 was identified in urine (57.1 percent dose) and faeces (11.8 percent dose).

IN-QEK31 was eliminated from the goat and remained unchanged in milk, tissues, urine, and faeces with no significant metabolism observed. IN-R2W56 was formed via methylation of the carboxylic acid group on IN-QEK31 and was present as a metabolite only in renal fat at 16.6 percent TRR but at a low concentration of 0.008 mg/kg (Figure 12).

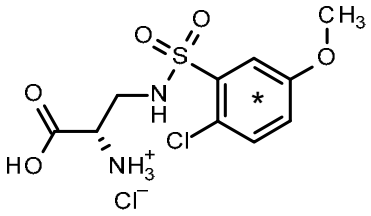


IN-QEK31 IN-R2W56 (renal fat only)

Figure 12 Proposed metabolic pathway for IN-QEK31 in lactating goat

Metabolism of [^{14}C]-IN-QZY47 in lactating goats

Cochrane (2017b DuPont-44398) also studied the metabolism of [^{14}C]-IN-QZY47 in the lactating goat (Saanen/Toggenburg breed, 5 years old, 59 kg bw). [Phenyl-^{14}C (U)]-IN-QZY47 was administered via capsules to a lactating goat as a single daily oral dose containing on average 20 mg [^{14}C]-IN-QZY47 for five consecutive days. The average daily dose administered to the goat was 10.06 ppm [^{14}C]-IN-QZY47 in the diet (dry weight equivalent). Feed consumption was 1.999 kg/day. The dose level was equivalent to an average dose of 0.351 mg IN-QZY47/kg bw/day. Milk production was 2.3 kg/day.



Faeces and urine were collected once daily, and milk collected twice daily. The goat was sacrificed approximately 6 hours after the last dose and liver, kidney, muscle, omental fat, renal fat, subcutaneous fat, bile, gastrointestinal tract, and contents were collected.

Analytical procedures

Tissue samples were homogenised with the aid of dry ice. Composite samples (Days 1–5) of milk, faeces and urine were prepared by combining equal amounts of each daily sample by matrix. Subsamples of tissues and milk (Days 1–5 composite) were prepared for metabolite extraction and analysis. Samples were extracted with acetonitrile:0.1 M ammonium formate, pH 7 (9:1). The extraction mixtures were homogenised followed by centrifugation and the resulting supernatants analysed by LSC. The liver and kidney PES were further treated with protease enzyme to release unextracted residues. To confirm the presence of certain conjugates, a urine extract was also incubated with β-glucuronidase from Helix pomatia, in sodium acetate buffer for ca. 18 hours at ca. 37 ºC. The reaction was stopped by addition of acetonitrile and the radioactive content was determined by LSC analysis of triplicate aliquots prior to HPLC analysis. All extracts were stored at -20 °C prior to analysis.

The total recovery was 86.4 percent of the administered dose, of which 83.7 percent of the dose was recovered from the urine, faeces, and cage wash with a further 2.5 percent found in the gastrointestinal tract (Table 60). Negligible amounts of radioactivity were recovered in the edible tissues and milk (*ca.* 0.2 percent of the administered total dose). Radioactivity reached plateau in milk after 1 day at *ca.* 0.016 mg eq/kg (Table 61 and Figure 13). A Day 1-5 composite milk sample (0.018 mg eq/kg) was prepared and separated into cream (0.021 mg eq/kg) and skimmed milk (0.018 mg eq/kg). This data indicated no selective partitioning of residues (less than 2-fold) in the milk fractions from goats dosed with IN-QZY47.

TRR in edible tissues were 0.344 mg eq/kg in liver, 0.824 mg eq/kg in kidney, 0.057 mg eq/kg in muscle, 0.034 mg eq/kg in omental fat, 0.044 mg eq/kg in renal fat and 0.050 mg eq/kg in subcutaneous fat (Table 60).

Table 60 Percent administered dose recovered in milk, tissues and excreta from the lactating goat following five consecutive daily oral doses of [^{14}C]-IN-QZY47

Sample	% dose
Tissues	<0.1
Liver	<0.1
Kidney	<0.1
Muscle	<0.1
Omental Fat	<0.1
Renal Fat	<0.1
Subcutaneous Fat	<0.1
Milk[A,B]	0.2
Faeces	7.2
Urine	75.1
Cage Wash	1.4
GI Tract Contents	2.5
Total	86.4

Notes:

[A] TRR values correspond to the concentration determined for the Day 1-5 composite samples.

[B] Total radioactive residues in the composite milk (Day 1-5) were separated into cream (0.021 mg eq/kg) and skim milk (0.018 mg eq/kg) fractions by LSC analysis following physical separation (centrifugation).

Table 61 Total radioactive residues (TRR) in milk following daily oral administration of [^{14}C]-IN-QZY47to a goat for five consecutive days

Sampling Time (Day)	Sampling Time (hours)	Total mg excreted/day	Concentration (mg eq/kg)
1	24	0.038	0.016

Sampling Time (Day)	Sampling Time (hours)	Total mg excreted/day	Concentration (mg eq/kg)
2	48	0.034	0.016
3	72	0.034	0.014
4	96	0.037	0.017
5[A]	104[B]	0.023	0.044

Notes:

[A] Partial day milk sample (0-6 hr) after last dose.

[B] 104 h refers to the sample ID only, the goat was milked directly before sacrifice which was *ca.* 102 hour (6 hour after last dose).

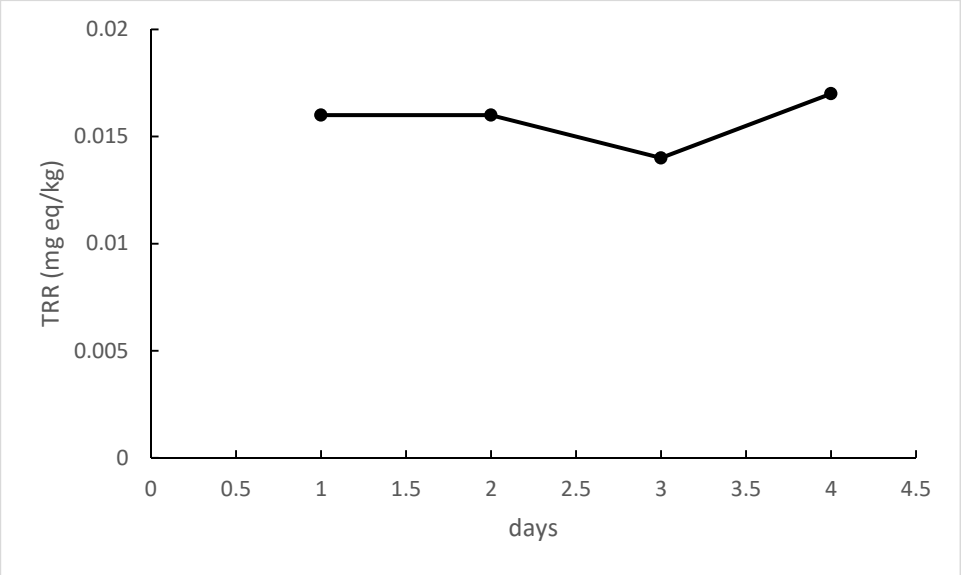


Figure 13 Residues in milk following dosing with ^{14}C-IN-QZY47 (note last milk sample not plotted as does not represent a 24-hour sample, i.e. residues in milk are higher in the first milking collected after dosing).

Extractability of milk and tissues with acetonitrile:(100 mM ammonium formate) (9:1) was good with > 83 percent TRR released.

The majority of radioactive residues (74.6–98.6 percent TRR) were identified and/or characterised in various tissues from the [^{14}C]-IN-QZY47 dosed goat. In milk and tissues, metabolites included IN-F4106 (maximum 81.4 percent TRR), IN-A5760 (maximum 11.4 percent TRR) and IN-A5760 conjugates (maximum 41.0 percent TRR). Several minor unidentified metabolites were also detected, none of which individually were greater than 8.3 percent TRR or 0.017 mg eq/kg (Table 62).

Table 62 Characterization of radioactivity in milk and tissues from [^{14}C]-IN-QZY47 dosed lactating goat (% TRR)

	Milk	Liver	Kidney	Muscle	Omental fat	Renal fat	Subcutaneous fat
TRR, mg eq/kg	0.017	0.354	0.824	0.057	0.034	0.044	0.050
Extracted MeCN/ammonium formate	99.4	83.9	92.2	91.4	89.7	84.5	93.7
IN-A5760 Glucuronide	ND	5.8	64.4	ND	ND	20.2[B]	18.0
IN-A5760 Glutathione	ND	21.1	ND	ND	ND	ND	ND
IN-A5760 Sulphate	41.0	ND	6.2	ND	ND	ND	ND
IN-A5760	2.3	10.3	5.4	5.0	ND	4.0	5.3

	Milk	Liver	Kidney	Muscle	Omental fat	Renal fat	Subcutaneous fat
TRR, mg eq/kg	0.017	0.354	0.824	0.057	0.034	0.044	0.050
IN-F4106	22.7	40.9	15.0	81.1	81.4	60.2	70.3
IN-QZY47	3.1	ND	ND	ND	ND	ND	ND
Unidentified metabolites	5.5	5.7[A]	1.2[D]	5.3[E]	8.3	ND	ND
Losses	24.9	2.2					
Fractions not analysed	NA	0.6	0.4	1.8	NA	NA	NA
Unextracted MeCN/ammonium formate	0.6	15.5	7.4	6.8	10.3	15.5	6.3
Protease		13.6	7.4				
IN-AS570 glucuronide		ND	3.2				
IN-AS760		1.1	3.2				
IN-F4106		1.6	ND				
Unidentified metabolites		8.6[C]	ND				
Losses		2.2	0.9				
Remaining		4.6	<0.1				

Notes:

NA=Not applicable.

ND=Not detected (detection limit typically 0.001 mg/kg).

[A] Sum of all other unidentified radioactivity in chromatograms, no single metabolite greater than 3.2 percent TRR (0.011 mg eq/kg).

[B] Sum of two components identified as IN-A5760 glucuronide.

[C] Sum of all other unidentified radioactivity in chromatograms, no single metabolite greater than 5.0 percent TRR (0.017 mg eq/kg).

[D] Sum of all other unidentified radioactivity in chromatograms, no single metabolite greater than 5.0 percent TRR (0.017 mg eq/kg).

[E] Sum of all other unidentified radioactivity in chromatograms, no single metabolite greater than 5.0 percent TRR (0.003 mg eq/kg).

HPLC analysis of composite urine identified IN-A5760 (14.4 percent dose) and IN-F4106 (0.8 percent dose) and to unknown components tentatively identified as glucuronide and sulfate conjugates of IN-A5760 accounting for 53.6 and 5.6 percent of the total administered dose, respectively. Qualitative analysis of urine incubated in a β-glucuronidase solution supported the presence of IN-A5760 conjugates. Faeces contained IN-A5760 (6.0 percent dose) and IN-F4106 (0.8 percent dose), a sulfate conjugate of IN A5760 (0.1 percent dose).

Proposed metabolic pathway of IN-QZY47 in the lactating goat

The proposed metabolic pathway of [^{14}C]-IN-QZY47 is shown in Figure 14. The primary biotransformation pathway of IN-QZY47 involved hydrolysis of the amide bond to form the cleaved metabolite IN-F4106. The metabolite IN-A5760 formed via O-demethylation of IN-F4106. Glucuronide and sulphate conjugates of IN-A5760 were formed and eliminated in the excreta. A glutathione conjugate of IN-A5760 was found only in liver extracts.

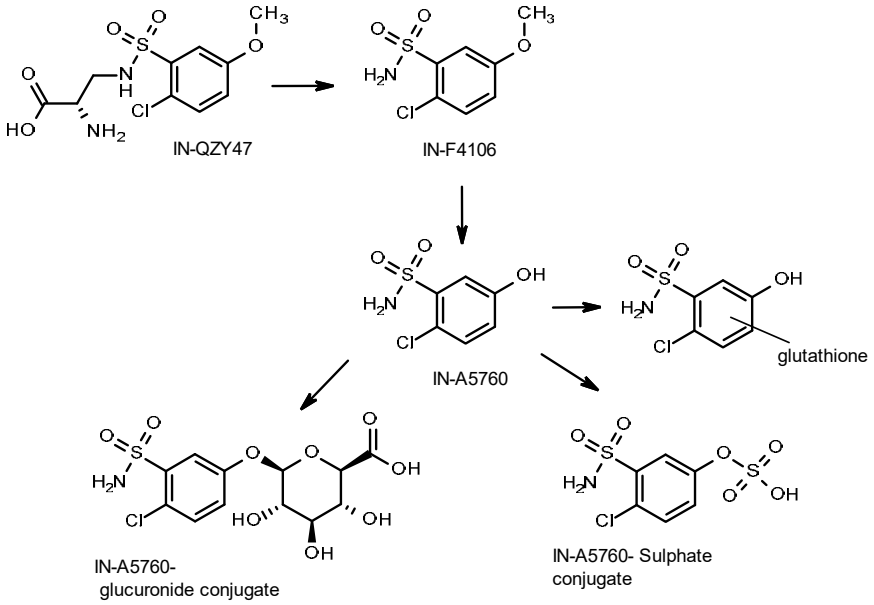
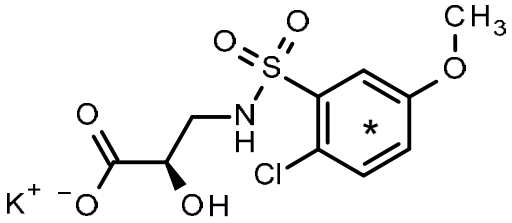


Figure 14 Proposed metabolic pathway for IN-QZY47 in the lactating goat

Note: All structures are depicted in their free acid or base forms.

Metabolism of [^{14}C]-IN-TMQ01 in lactating goats

Cochrane (2017c DuPont-42614) also investigated the metabolism of [^{14}C]-IN-TMQ01 in the lactating goat.



[Phenyl-^{14}C(U)]IN-TMQ01 was administered *via* capsules to a lactating goat (Saanen/Toggenburg breed, 4 years old, 57.5 kg bw) as a single daily oral dose for five consecutive days. The average daily dose administered to the goat was 10.86 ppm [^{14}C]-IN-TMQ01 diet (dry weight equivalent). The dose level was equivalent to an average dose of 0.278 mg IN-TMQO1 mg/kg bw/day. Milk production was 2.0 kg/day. Feed consumption was 1.478 kg/day.

Faeces and urine were collected once daily, and milk collected twice daily. The goat was sacrificed approximately 6 hours after the last dose and liver, kidney, muscle, omental fat, renal fat, subcutaneous fat, bile, gastrointestinal tract, and contents were collected.

All samples were stored at *ca.* -20 °C until taken for analysis. All samples were extracted within 47 days frozen storage and extracts initially analysed within 96 days from collection with the exception of analysis of subcutaneous fat, which occurred within 222 days (7 months).

Tissue samples were homogenised with the aid of dry ice. Composite samples (Days 1-5) of milk, faeces and urine were prepared by combining equal amounts of each daily sample by matrix. Subsamples of tissues, milk (Days 1-5 composite) and faeces (Days 1-5 composite) were prepared for metabolite extraction and analysis. Samples were extracted with acetonitrile:0.1 M ammonium formate, pH 7 (9:1).

Fat samples were first extracted with dichloromethane which removed negligible amount of radioactivity. The extraction mixtures were homogenised followed by centrifugation and the resulting supernatants analysed by LSC. Extracts warranting analysis were combined by matrix (where applicable), concentrated, and analysed by LSC and HPLC. The liver PES was further treated with protease enzyme to release unextracted residues, and the resulting extract analysed by LSC. All extracts were stored at -20 °C prior to HPLC.

The total recovery was 98.3 percent of the administered dose, of which 80.4 percent of the dose was recovered from the urine, faeces, and cage wash with a further 17.9 percent found in the gastrointestinal tract (Table 63). Negligible amounts of radioactivity were recovered in the edible tissues and milk (<0.1 percent of the administered total dose). Radioactivity reached plateau in milk within 3 days at 0.008 mg eq/kg (Table 64, Figure 15).

TRR in edible tissues were 0.021 mg eq/kg in liver, 0.220 in kidney, 0.002 in muscle, 0.001 in omental fat, 0.003 in renal fat and 0.003 mg eq/kg in subcutaneous fat (Table 63).

Table 63 Percent administered dose recovered in milk, tissues and excreta from a lactating goat following five consecutive daily oral doses of [^{14}C]-IN-TMQ01

Sample	% dose [A]
Tissues	<0.1
Liver	<0.1
Kidney	<0.1
Muscle	<0.1
Omental Fat	<0.1
Renal Fat	<0.1
Subcutaneous Fat	<0.1
Milk[A,B]	<0.1
Faeces	43.7
Urine	34.5
Cage Wash	2.2
GI Tract Contents	17.9
Total	98.3

Notes:

[A] TRR values correspond to the concentration determined for the Day 1-5 composite samples.

[B] Total radioactive residues in the composite milk (Day 1-5) were separated into cream (0.006 mg equiv/kg) and skim milk (0.007 mg equiv/kg) fractions and measured by LSC analysis following physical separation (centrifugation).

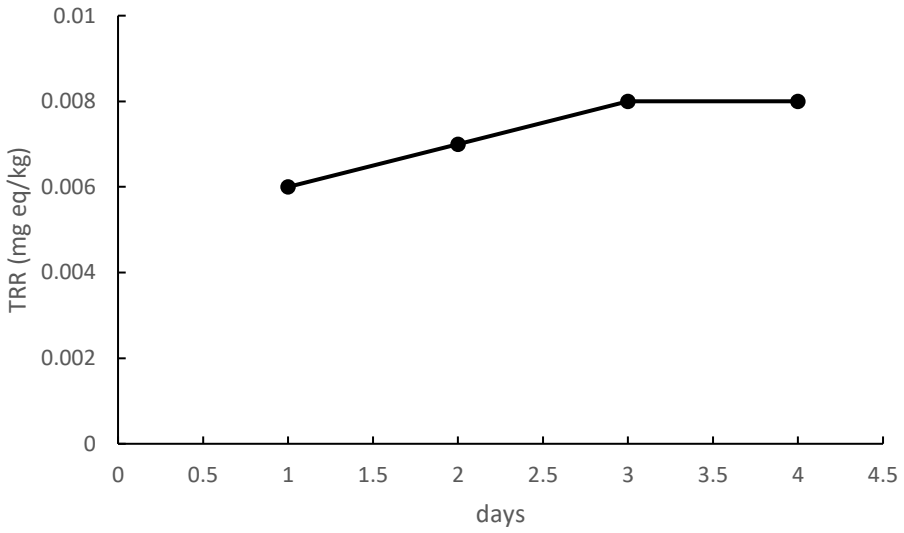


Figure 15 Residues in milk following dosing with ^{14}C-IN-TMQ01 (note last milk sample not plotted as does not represent a 24-hour sample, i.e. residues in milk are higher in the first milking collected after dosing).

In milk and tissues containing residues greater than 0.01 mg eq/kg, nearly all of the radioactive residues (66.1 percent to 100.0 percent TRR) were extracted with acetonitrile:(100 mM ammonium formate) (9:1). Digestion with protease released the remaining radioactive residues in liver increasing the total extracted in this tissue to 100 percent TRR (Table 65).

Table 64 TRR in milk following daily oral administration of [^{14}C]-IN-TMQ01 to a goat for five consecutive days

Sampling Time (Day)	Sampling Time (Hours)	Total mg excreted	Concentration (mg eq/kg)
1	24	0.010	0.006
2	48	0.012	0.007
3	72	0.017	0.008
4	96	0.017	0.008
5[A]	104[B]	0.006	0.009

Notes:

[A] Partial day milk sample (0-6 hour) after last dose

[B] 104 h refers to the sample ID only, the goat was milked directly before sacrifice which was *ca.* 102 hour (6 hour after last dose)

The majority of radioactive residues (66.1–100 percent TRR) were identified and/or characterised in the various tissues. Unchanged IN-TMQ01 accounted for 42.7–86.7 percent TRR in tissues. IN-F4106 accounted for 1.0–43.6 percent TRR in tissues and essentially all of the residue in milk (97.5 percent TRR; 0.007 mg eq/kg). Several minor unidentified metabolites were also detected, none of which individually exceeded 4.8 percent TRR, with the exception of muscle and subcutaneous fat in which a single unknown component accounting for 13.7–22.7 percent TRR was detected, however, these unknowns only equated to a low concentration of ≤ 0.001 mg eq/kg.

The major components in kidney and faeces were IN-TMQ01 and IN-F4106. The metabolite IN-UNS90 (expected to be as the *R*-enantiomer IN-TQD54) was also tentatively identified in the faeces.

Table 65 Characterization of radioactivity in milk and tissues from [^{14}C]-IN-TMQ01 dosed lactating goats (% TRR)

	Milk	Liver	Kidney	Muscle	Subcutaneous fat
TRR (mg eq/kg)	0.007	0.021	0.220	0.002	0.003
Extracted MeCN/ammonium formate	97.5	66.1	92.5	100	100[E]
IN-F4106	97.5	9.6	1.0	43.6	10.7
IN-TMQ01	ND	47.6	86.7	42.7	49.5
Unidentified metabolites	NA	8.9[A]	4.8[B]	13.7[C]	22.7[D]
Losses	-	-	-	-	17.1
Fractions not analysed	<LOQ	NA	2.4	<LOQ	<LOQ
Unextracted MeCN/ammonium formate	2.5	33.9	5.1	<0.1	<0.1
Protease		33.9			
Remaining		<0.1			

Notes:

NA = Not applicable.

ND = Not detected (detection limit typically 0.001 mg eq/kg).

[A] 2 unknowns (individually ≤ 0.001 mg eq/kg, ≤4.8 percent TRR) totalling 0.002 mg eq/kg (8.9 percent TRR).

[B] 2 unknowns (individually ≤ 0.006 mg eq/kg, ≤ 2.8 percent TRR) totalling 0.011 mg eq/kg (4.8 percent TRR).

[C] 1 unknown ≤ 0.001 mg eq/kg, 13.7 percent TRR.

[D] One unknown 0.001 mg eq/kg, 22.7 percent TRR.

[E] Fat samples were first extracted with dichloromethane which removed negligible amount of radioactivity (<LOQ) before proceeding with acetonitrile/buffer.

Proposed metabolic pathway of IN-TMQ01 in the lactating goat

The proposed metabolic pathway of [^{14}C]-IN-TMQ01 in the lactating goat is shown in Figure 16. IN-TMQ01 was eliminated from the goat and remained unchanged in tissues, urine and faeces with minor metabolism observed. The primary biotransformation pathway of IN-TMQ01 involved hydrolysis of the amide bond to form the cleaved metabolite IN-F4106. O-demethylation of IN-TMQ01 to IN-UNS90 (expected to be as the R-enantiomer IN-TQD54) occurred to a small extent.

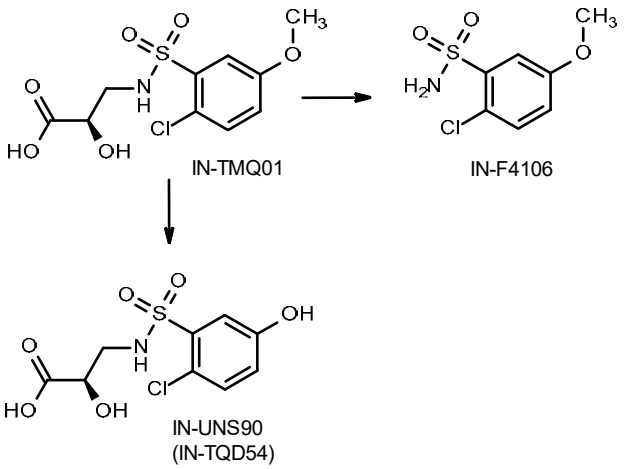


Figure 16 Proposed metabolic pathway of IN-TMQ01 in the lactating goat

Note: All structures depicted in the free acid forms

Metabolism of [^{14}C]fluazaindolizine in laying hens

Wicksted (2013 DuPont-33572, Revision No. 1) studied the metabolism of [^{14}C]-fluazaindolizine in laying hens.

Two groups of Hy-Line Layer hens (5 per group, 1.72–2.08 kg bw) were dosed orally *via* capsules, once a day for 14 consecutive days, with [Ph-^{14}C]-fluazaindolizine or [IP-2-^{14}C]-fluazaindolizine at doses averaging 13.1 and 13.6 ppm of fluazaindolizine in the diet (dry weight basis). These dose rates were equivalent to 0.875 and 0.880 mg fluazaindolizine/kg bw) for the ([Ph-^{14}C] and ([IP-2-^{14}C]fluazaindolizine) dosed hens, respectively. Average feed consumption of 0.123–0.130 g/day during the dosing period. Egg samples were collected twice a day while excreta samples were collected daily. Hens were sacrificed 6 hours after the final dose, and samples of liver, muscle, abdominal fat, and subcutaneous fat with skin was collected from each hen. All whole egg, tissue and excreta samples were pooled by dose group. Composite whole egg samples (Day 9–13), and excreta (Day 1–14) were prepared for analysis for both dose groups.

Tissue samples were homogenised in dry ice. Equal amounts (10 percent) of daily excreta from Days 1-14 were pooled by dose group and equal amounts (10 percent) of whole egg from Day 9–13 were pooled by matrix and dose group. Subsamples (*ca.* 25–30 g) of composite samples of tissues, whole egg and excreta were prepared for metabolite extraction and analysis. Samples were extracted using acetonitrile:0.1 M ammonium formate, pH 7 (9:1). The resulting extracts were combined by matrix, concentrated, and analysed by LSC and HPLC. All extracts were stored at -20 °C prior to HPLC analysis and HPLC profiles were conducted within 6 months of sacrifice.

The recovery of the administered dose was 94.6 percent for [Ph-^{14}C]-fluazaindolizine and 93.5 percent for the [IP-2-^{14}C]fluazaindolizine. For both [^{14}C]-labels, 92.9–94 percent of the dose was recovered from the excreta and cage wash. For both [^{14}C]-labels, <0.1 percent of the dose was recovered in eggs, muscle, and fat with 0.6 percent of the dose recovered in liver (Table 66). Total radioactive residue levels in edible tissues were 0.732 mg eq/kg in liver, 0.043 in muscle and 0.020 mg eq/kg in abdominal fat from the [Ph-^{14}C]-fluazaindolizine dosed hens, and 0.701 mg eq/kg in liver, 0.047 in muscle and 0.027 mg eq/kg in abdominal fat from the [IP-2-^{14}C]-fluazaindolizine dosed hens. Radioactivity plateaued in whole eggs within 10 days from the start of dosing at *ca.* 0.017 mg eq/kg in the [Ph-^{14}C]-fluazaindolizine and within 8 days at *ca.* 0.018 mg eq/kg in the [IP-2-^{14}C]-fluazaindolizine (Table 67, Figure 17).

Table 66 Percent administered dose recovered in eggs, tissues and excreta of laying hens following 14 consecutive daily oral doses of [Ph-^{14}C]-fluazaindolizine or [IP-2-^{14}C]-fluazaindolizine (% dose)

Sample	[Ph-^{14}C]-fluazaindolizine	[IP-2-^{14}C]-fluazaindolizine
Tissues	0.6	0.6
Liver	0.6	0.6
Muscle[A]	<0.1	<0.1
Abdominal fat[A]	<0.1	<0.1
Whole egg (Total Day 1-14)	<0.1	<0.1
Excreta (Total Day 1-14)	85.9	83.8
Cage wash	8.1	9.1
Total	94.6	93.5

Notes:

[A] Total muscle mass was assumed to be approximately 25 percent of body weight, total fat approximately 12 percent of body weight.

Table 67 Daily Total Radioactive Residues (TRR) in whole eggs following oral administration of [Ph-^{14}C]- or [IP-2-^{14}C]-fluazaindolizine to laying hens for 14 consecutive days

Radiolabel		[Ph-^{14}C]-fluazaindolizine	[IP-2-^{14}C]-fluazaindolizine
Sampling Time (Day)	Sampling Time (H)	Concentration (mg eq/kg)	Concentration (mg eq/kg)
1	24	0.006	0.001
2	48	0.011	0.006
3	72	0.011	0.008
4	96	0.015	0.010
5	120	0.014	0.013
6	144	0.015	0.014
7	168	0.017	0.015
8	192	0.018	0.015
9	216	0.017	0.017
10	240	0.017	0.017
11	264	0.017	0.016
12	288	0.022	0.018
13	312	0.018	0.018
14	318	0.014	0.019

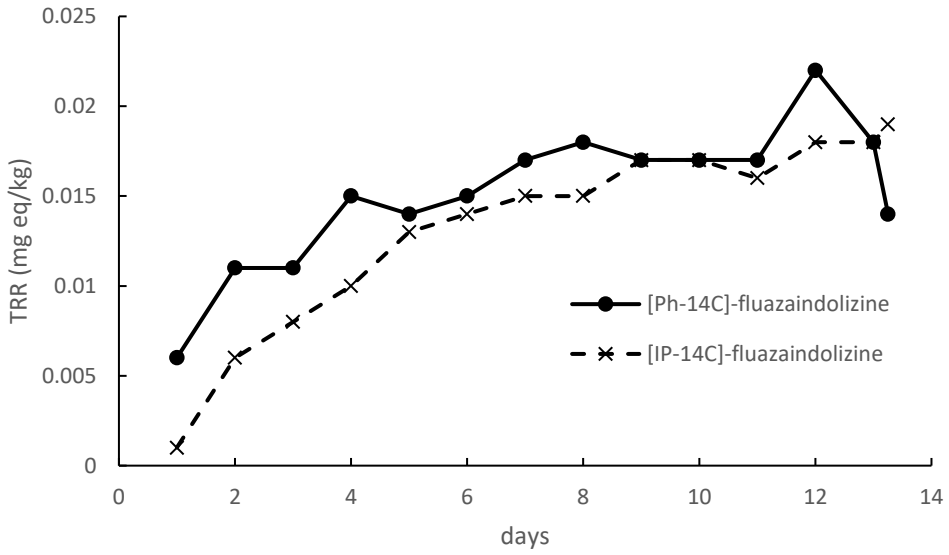


Figure 17 Residues in eggs following dosing with ^{14}C-fluazaindolizine.

Identification and characterisation of radioactive residues

The initial acetonitrile/buffer extractions released 87.3–99.2 percent TRR [Ph-^{14}C] and 89.8–99.0 percent TRR IP-2-^{14}C] from tissues and whole eggs (Table 68).

The majority of radioactive residues (82.4–98.0 percent TRR) was identified and/or characterised in liver and eggs from the [Ph-^{14}C]fluazaindolizine dosed hens (Table 68). Fluazaindolizine was a major [^{14}C] residue, accounting for 67.5–96.5 percent TRR in liver and eggs. The concentration of fluazaindolizine was 0.013 mg/kg in eggs, 0.680 mg/kg in liver, 0.041 mg/kg in muscle and 0.014 mg/kg in fat.

In eggs and tissues, the phenyl-derived metabolites included IN-F4106 (maximum 5.7 percent TRR) and IN-REG72 (maximum 1.1 percent TRR). Several minor unidentified metabolites were also

detected, none of which individually were greater than 11.3 percent TRR, which combined accounted for 0.9–11.3 percent TRR in eggs and tissues.

The majority of radioactive residues (73.1-97.9 percent TRR) was identified and/or characterised in tissues and eggs from the [IP-2-^{14}C]fluazaindolizine dosed hens (Table 69). Fluazaindolizine was a major [^{14}C]-residue, accounting for 66.2–97.1 percent TRR in all tissues and eggs. The concentration of fluazaindolizine was 0.012 mg/kg in eggs, 0.639 mg/kg in liver, 0.046 mg/kg in muscle and 0.018 mg/kg in fat.

In eggs and tissues, the imidazopyridine-derived metabolites included IN-QEK31 (maximum 4.9 percent TRR), IN-REG72 (maximum 1.1 percent TRR), IN-RYC33 (maximum 11.0 percent TRR) and methyl ester of IN-QEK31 (IN-R2W56) (maximum 1.9 percent TRR). An unidentified metabolite was detected in abdominal fat which accounted for 2.5 percent TRR (0.001 mg/kg).

Table 68 Identification of radioactivity in egg and tissues from [Ph-^{14}C]-fluazaindolizine dosed laying hens (% TRR)

	Whole egg	Liver	Muscle	Abdominal fat
TRR (mg eq/kg)	0.017	0.732	0.043	0.020
Total extracted acetonitrile/ammonium formate	99.2	98.9	98.1	87.3
Extract analysed	91.7	98.0	96.5	82.5
IN-F4106	5.7	3.0	ND	3.6
IN-REG72	ND	1.1	ND	ND
Fluazaindolizine	76.8	93.0	96.5	67.5
Unidentified metabolites[A]	9.2	0.9	ND	11.3
Extracts not analysed	7.5	0.9	1.6	4.8
Unextracted	0.8	1.1	1.8	12.8
Hexane			1.5	2.9
Dichloromethane				2.2
Remaining			0.3	7.7

Notes:

ND Not detected.

[A] Sum of all other unidentified radioactivity in chromatograms, no single metabolite greater than 11 percent TRR.

Table 69 Identification of radioactivity in egg and tissues from [IP-2-^{14}C]-fluazaindolizine dosed laying hens (% TRR)

	Whole egg	Liver	Muscle	Abdominal fat
TRR (mg eq/kg)	0.016	0.701	0.047	0.027
Total extracted acetonitrile/ammonium formate	92.6	98.8	99.0	89.8
Extract analysed	86.6	97.8	97.1	86.1
IN-REG72	ND	1.1	ND	ND
Fluazaindolizine	75.6	91.2	97.1	66.2
methyl ester of IN-QEK31 (IN-R2W56)	ND	ND	ND	1.9
IN-QEK31	ND	4.9	ND	ND
IN-RYC33	11.0	0.7	ND	2.5
Unidentified metabolite	ND	ND	ND	2.5
Losses	-	-	-	13.0
Extracts not analysed	6.0	1.0	1.9	3.7
Unextracted	7.5	1.1	1.0	10.2
Hexane			0.5	3.5
Remaining			0.5	6.7

Notes:

ND Not Detected.

Day 1–14 excreta composite from hens dosed with [Ph-^{14}C]-fluazaindolizine was extracted and analysed by HPLC. Fluazaindolizine was a major component representing 67.1 percent of the total administered dose. The metabolites IN-A5760 sulphate (3.1 percent dose), IN-A5760 (3.8 percent dose), IN-F4106 (0.4 percent dose) and IN-REG72 (6.1 percent dose) were detected. Two minor unknown components were also detected which accounted for 0.6 and 2.8 percent dose.

Day 1–14 excreta composite from hens dosed with [IP-2-^{14}C]-fluazaindolizine was extracted and analysed by HPLC. Fluazaindolizine was a major component representing 67.1 percent of the total administered dose. The metabolites IN-QEK31 (5.5 percent dose) and IN-REG72 (5.5 percent dose) were detected. Several minor unknown components were also detected none of which individually accounted for > 0.7 percent dose.

The proposed metabolic pathway of fluazaindolizine in laying hens is shown in Figure 18. The metabolism of fluazaindolizine was complex and is based on metabolites identified in the tissues and in the excreta. The following metabolic pathway is proposed: biotransformation of fluazaindolizine in the hen occurred primarily through *O*-demethylation to form IN-REG72 followed by hydrolysis of the amide bond to form the cleaved metabolites IN-QEK31 and IN-A5760. Direct hydrolysis of fluazaindolizine yielded the metabolite IN-F4106 which was *O*-demethylated to IN-A5760. A sulphate conjugate of IN-A5760 was formed and eliminated in the excreta. IN-RYC33 was formed from the hydrolysis of the sulfonamide bond of fluazaindolizine. The metabolite IN-R2W56 was formed *via* methylation of the carboxylic acid group of IN-QEK31.

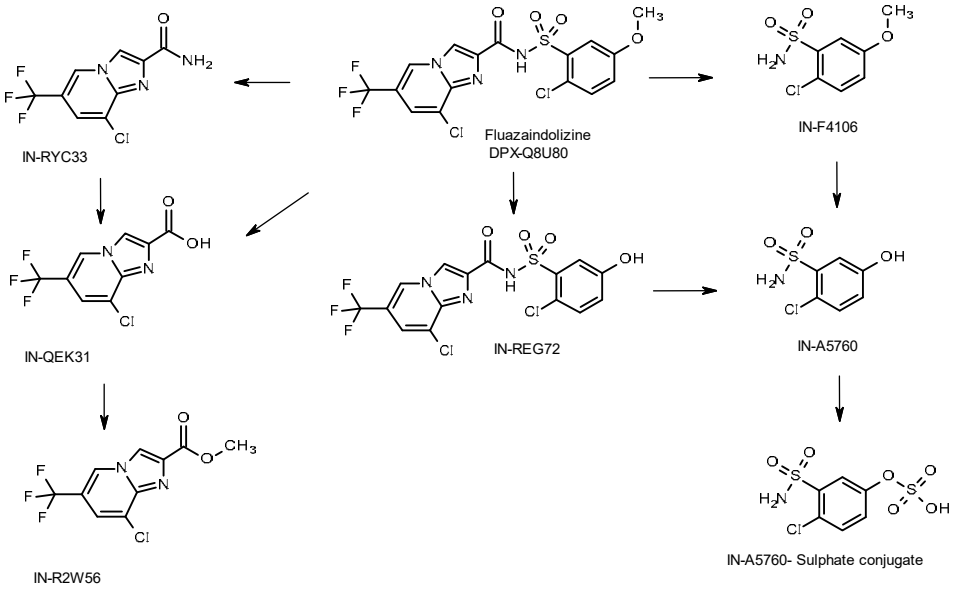
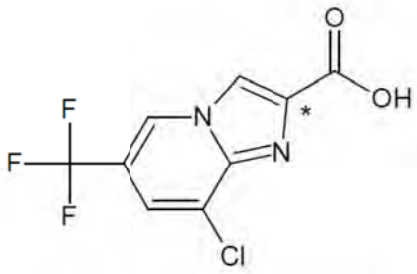


Figure 18 Proposed metabolic pathway of fluazaindolizine in the laying hen

Metabolism of [IP-2-^{14}C]-IN-QEK31 in laying hens

Cochrane (2018 DuPont-43221 Revision 1) studied the metabolism of [^{14}C]-IN-QEK31 in laying hens.



* – Denotes position of radiolabel

[Imidazo[1,2-a]pyridine-2-^{14}C]-IN-QEK31was administered *via* capsules to five laying hens (Novogen Brown, 56 weeks old, 1.726–2.154 kg bw) as a single daily oral dose for 14 consecutive days. The average daily dose administered to the five hens was 9.96 ppm [IP-2-^{14}C]-IN-QEK31 diet (dry weight equivalent) and 0.68 mg [IP-2-^{14}C]-IN-QEK31/kg bw. Average feed consumption of 0.129 g/day.

Excreta was collected once daily, and eggs collected twice daily. All hens were sacrificed approximately 6 h after the last dose and liver, muscle, abdominal fat, bile, gastrointestinal tract, and contents were collected. All samples were stored at *ca.* -20 °C until taken for analysis. All samples were extracted within 68 days and extracts initially analysed within 408 days following the extraction procedure. The longest storage interval (between sample collection and analysis of sample extracts) recorded for any individual sample (liver, kidney, and renal fat) was 476 days (*ca.* 16 months).

Tissue samples were homogenised with the aid of dry ice. Composite samples of whole egg (Days 9-13) and excreta (Days 1–14) were prepared by combining equal amounts of each daily sample by matrix. Subsamples (*ca.* 30 g) of tissues, whole egg (Days 9–13 composite) and excreta (Days 1–14 composite) were prepared for metabolite extraction and analysis. Samples were extracted with acetonitrile:0.1 M ammonium formate, pH 7 (9:1). The extraction mixtures were homogenised followed by centrifugation and the resulting supernatants analysed by LSC. Extracts warranting analysis were combined by matrix (where applicable), concentrated, and analysed by LSC and HPLC. All extracts were stored at -20 °C prior to HPLC.

IN-QEK31 was rapidly eliminated from hens into the excreta (approximately 93.2 percent of the dose). The total recovery was 100.9 percent of the administered dose, of which 100.7 percent of the dose was recovered from the excreta and cage wash with a further 0.2 percent found in the gastrointestinal tract (Table 70). Less than 0.1 percent of the dose was recovered in whole eggs.

TRR in edible tissues were 0.014 mg eq/kg in liver and 0.002 mg eq/kg in abdominal fat (Table 70). There was no measurable radioactivity found in muscle (< 0.001 mg eq/kg).

Radioactivity in whole eggs reached plateau within 5 days post first dose *ca.* 0.005 mg/kg (Table 71, Figure 19). Negligible amounts of radioactivity were recovered in the edible tissues (< 0.1 percent of the administered total dose).

Table 70 Percent administered dose recovered in whole egg, tissues and excreta from laying hens following 14 consecutive daily oral doses of [IP-2-^{14}C]-IN-QEK31 (% dose)

Sample	[IP-2-^{14}C]IN-QEK31
Tissues	<0.1
Liver	<0.1
Muscle	<0.1
Abdominal Fat	<0.1
Whole Egg	<0.1
Excreta	93.2
Cage Wash	7.5
GI Tract Contents	0.2
Total	100.9

Table 71 Daily Total Radioactive Residues (TRR) in whole eggs following oral administration of [IP-2-^{14}C]-IN-QEK31 to laying hens for 14 consecutive days

Sampling Time (Day)	Sampling Time (H)	Whole egg (TRR mg eq/kg)
1	24	<0.001
2	48	0.002
3	72	0.003
4	96	0.005
5	120	0.006
6	144	0.005
7	168	0.005
8	192	0.004
9	216	0.004
10	240	0.004
11	264	0.006
12	288	0.005
13	312	0.005
14	318	0.006

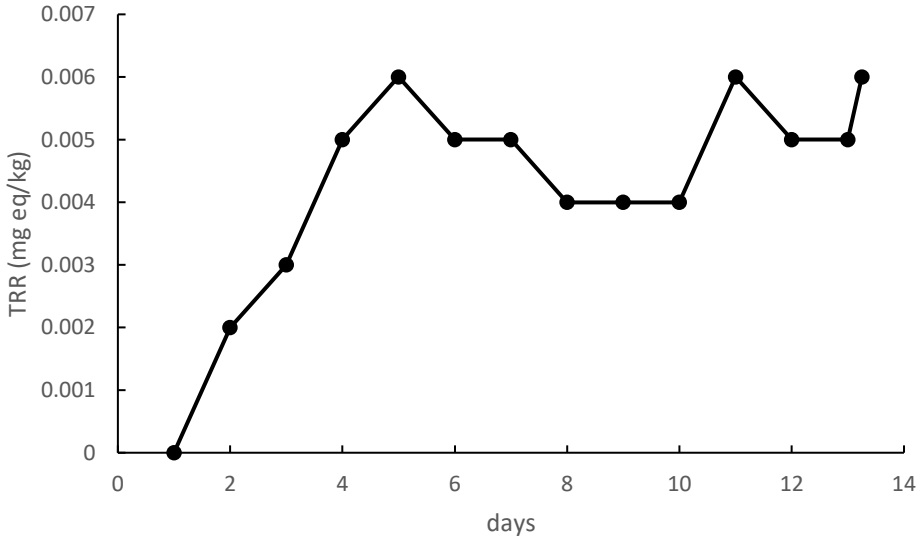


Figure 19 Residues in eggs following dosing with ^{14}C-IN-QEK31

Liver tissues contained residues greater than 0.01 mg eq/kg, and the majority of the radioactive residues (≥ 71.1 percent TRR) were extractable with acetonitrile:(100 mM ammonium formate) (9:1). A summary of the metabolites identified in liver is provided in Table 72. Unchanged IN-QEK31 accounted for 71.1 percent TRR in liver. As radioactive residues in composite whole egg, muscle and fat samples were very low (< 0.01 mg eq/kg) metabolite profiling of these tissues was not conducted. Unchanged IN-QEK31 (93.2 percent dose) was identified in faeces.

In summary, [IP-2-^{14}C]-IN-QEK31 was eliminated from the hens and remained unchanged in tissues and excreta with no significant metabolism observed.

Table 72 Summary of radioactive residues identified in egg and tissues from laying hens dosed with [IP-2-^{14}C]-IN-QEK31

	Whole egg	Liver	Muscle	Abdominal fat
TRR (mg eq/kg)	0.003	0.014	<LOQ	0.002
Extracted	58.2	71.1	<LOQ	100.0
Extracted acetonitrile ammonium acetate	58.2	71.1	-	<0.1
IN-QEK31	NA	71.1	NA	NA
Fraction not analysed	<0.1	<0.1		
Unextracted	41.8	28.9	<LOQ	100.0
Hexane				100.0
4M HCl				<0.1
Remaining				0

Notes:

NA = Not applicable.

ENVIRONMENTAL FATE

The FAO Manual on the Submission and Evaluation of Pesticide Residues Data for the Estimation of Maximum Residue Levels in Food and Feed (2009) explains the data requirements for studies of environmental fate. The focus should be on those aspects that are most relevant to MRL setting. For fluazaindolizine, supervised residue trials data following soil application are available for cucumbers, melons, squash, tomatoes, peppers, carrots and potatoes. Aerobic degradation in soil is relevant, as well as the normal requirements for hydrolysis, soil photolysis and rotational crop studies.

The Meeting received information on soil aerobic and anaerobic metabolism, aqueous hydrolysis and soil photolysis properties of fluazaindolizine.

Route of degradation in soil

Aerobic degradation of fluazaindolizine in soil

Wardrope and Anderson (2013 DuPont-35135) studied the aerobic soil metabolism of [^{14}C]fluazaindolizine in two soils, Sassafras and Nambsheim using [Ph-^{14}C] label or [IP-5,8a-^{14}C]-label. The radiolabelled test item was applied to the soil at a nominal rate of 1.0 μg/g oven dry soil which corresponded to a 1 kg ai/ha application assuming 10 cm incorporation and 1.0 g/cm3 soil density. The soil samples were continuously aerated throughout the 150-day incubation period. Traps for volatiles included ethanediol, and two 1M NaOH traps to collect organic volatiles and CO_2. Table 73 shows the soil characteristics used in the study

Table 73 Characteristic of soils used for aerobic degradation (DuPont-35135)

Soil	Texture	pH 0.01 M $CaCl_2$	% OM [Walkley-Black]	CEC (meq/100 g)	Biomass (mg C/kg soil)
Sassafras, United States	Sandy loam	5.7	2.0	5.3	383.9
Nambsheim, France	Sandy loam	7.5	2.7	9.0	430.5

Soil samples were sequentially extracted with acetonitrile:water (9:1 , with 2 percent formic acid) followed by acetonitrile:water (4:1, with 2 percent formic acid), acetonitrile:water (1:1, with 2 percent formic acid), both at 50 °C.

The material balance for both labels was good at 90.64 to 107.68 percent AR for the [Ph-^{14}C]-label and 74.49 to 107 percent AR for the [IP-5,8a-^{14}C]-label. The lower mass balances observed for the [IP-5,8a-^{14}C]-labelled samples at late sampling intervals were likely due to formation and loss a portion of the volatile degradate, IN-VM862.

Substantial degradation of fluazaindolizine was obvious in both soils, as noted by a significant amount of $^{14}CO_2$ generation as well as incorporation of radiocarbon into natural carbon pool.

The distribution of radioactivity for the two soils is shown in Figure 20. In the Sassafras soil treated with [^{14}C]-fluazaindolizine, the solvent extracted ^{14}C-residues in the soil decreased from mean values of 104–105 percent AR at Day 0 to 66.2 percent AR for the [Ph-^{14}C]-label and 44.2 percent AR for the [IP-5,8a-^{14}C]-label after 150 days of aerobic incubation. Unextracted residues increased to a maximum value of 19.6 percent AR at Day 80 in the [Ph-^{14}C]-label and 22.1 percent for the [IP-5,8a-^{14}C]-label on Day 120. There was some decline in the unextracted residue by Day 150 for both labels. At the same time, $^{14}CO_2$ accounted for 10.1 percent AR for the [Ph-^{14}C]-label and 14.6 percent for the [IP-5,8a-^{14}C]-label over the course of the study.

In the Nambsheim soil system treated with [^{14}C]-fluazaindolizine the solvent extracted ^{14}C-residues in the soil decreased to 38.1 percent AR in the [Ph-^{14}C]-label and 49.3 percent AR for the [IP-5,8a-^{14}C]-label after 150 days aerobic incubation. Unextracted residues increased to a maximum value of 37.41 percent AR and 22.4 percent AR for the same two labels by Day 150. Radiolabelled $^{14}CO_2$ cumulatively accounted for 16.2 percent AR for the [Ph-^{14}C]-label and 5.1 percent AR for the [IP-5,8a-^{14}C]-label over the course of the study.

Fluazaindolizine degraded extensively *via* the molecule splitting into two portions at the sulphonamide bridge between the two rings. Fluazaindolizine decreased to about 1.5-1.6 percent AR in for both labels by Day 150 for the Sassafras soil and 15.0–15.6 percent AR by Day 150 for the Nambsheim soil.

Three main degradation products (>5 percent of AR) were IN-F4106, IN-QEK31 and IN-VM862, and they all resulted from the cleavage of the molecule into two portions. IN-F4106 and IN-QEK31 were formed due to molecule splitting into two portions. IN-F4106 was detected in the [Ph-^{14}C]-label samples and IN-QEK31 with the [IP-5,8a-^{14}C]-label. IN-VM862, presumably formed from further degradation of IN-QEK31, was also detected in the [IP-5,8a-^{14}C]-label. IN-REG72, the only metabolite with most of the structure staying intact, was a minor degradation product in both soils (<5 percent AR). IN-A5760 was also a minor degradation product reaching a mean maximum of 2.6 percent AR at Day 60 in the Sassafras samples and a maximum of 2.7 percent AR at Day 80 in the Nambsheim samples. No unidentified metabolites exceeded 5 percent AR at any sampling interval.

Unextracted residues were characterised using organic matter fractionation into humin, fulvic acid and humic acid fractions. The largest portion of the unextracted radioactivity (≥ *ca* 9.4–34.7 percent

AR, day 150) was associated with the humin fraction of the soil organic matter, while the balance was found in humic (0.5–4.9 percent AR) and fulvic acid (2.5–7.3 percent AR) fractions indicating substantial incorporation of ^{14}C into natural constituents.

Parent only kinetics was assessed using the single first-order (SFO) and additional biphasic decline models, where appropriate. The Sassafras data set was also assessed using the Indeterminate Order Rate Equation (IORE) model. The kinetic analysis results are in Table 74.

Table 74 Summary of degradation kinetics for fluazaindolizine in Sassafras and Nambsheim soils, study DuPont-35135

Soil	DT_{50} (days)	DT_{90} (days)	χ^2	r^2	Model
Sassafras	11.8	47.0	4	0.991	DROP
	11.8	48.4	4	0.990	IORE
Nambsheim	43.9	145.7	6	0.976	SFO

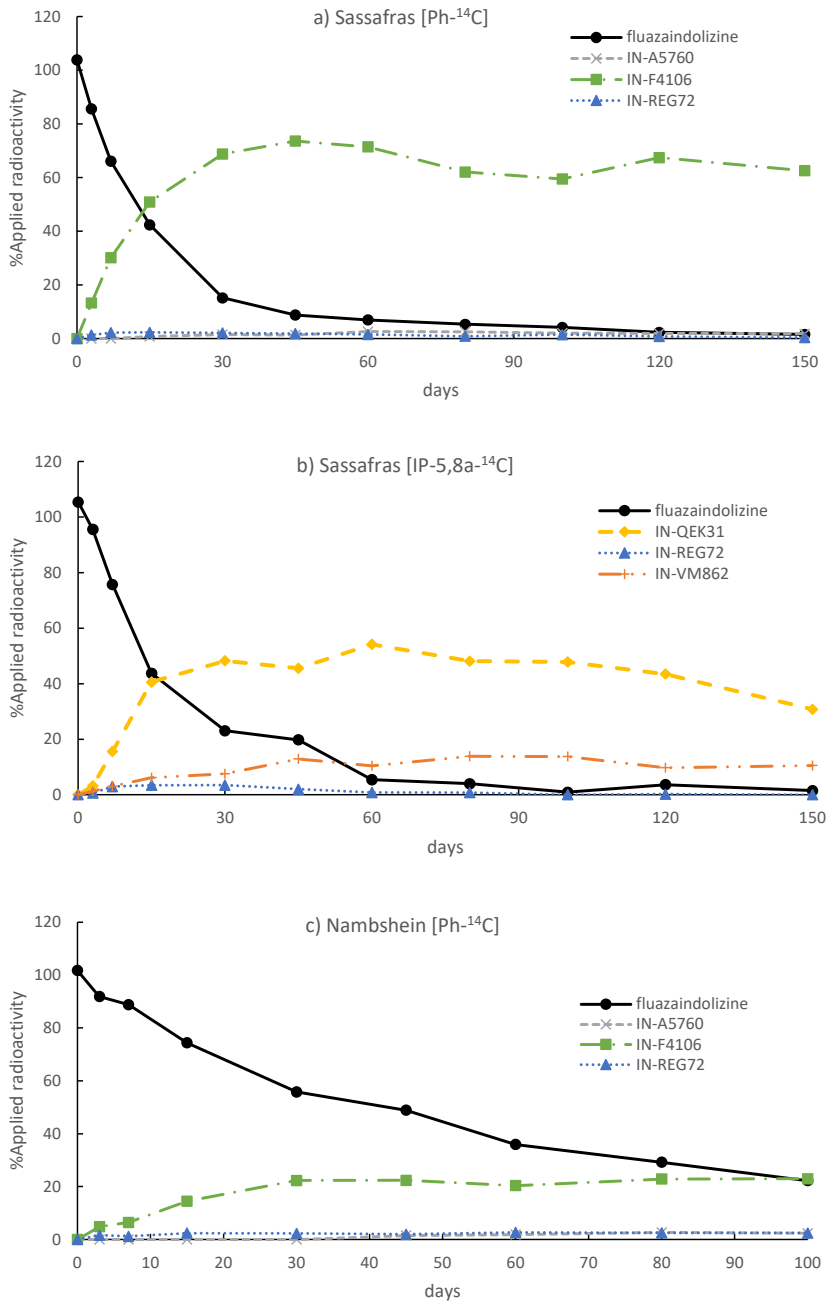


Figure 20 Aerobic soil degradation of ^{14}C-fluazaindolizine on Sassafras and Nambsheim sandy loam soils

The degradation pathway for fluazaindolizine in aerobic soil is shown in Figure 21.

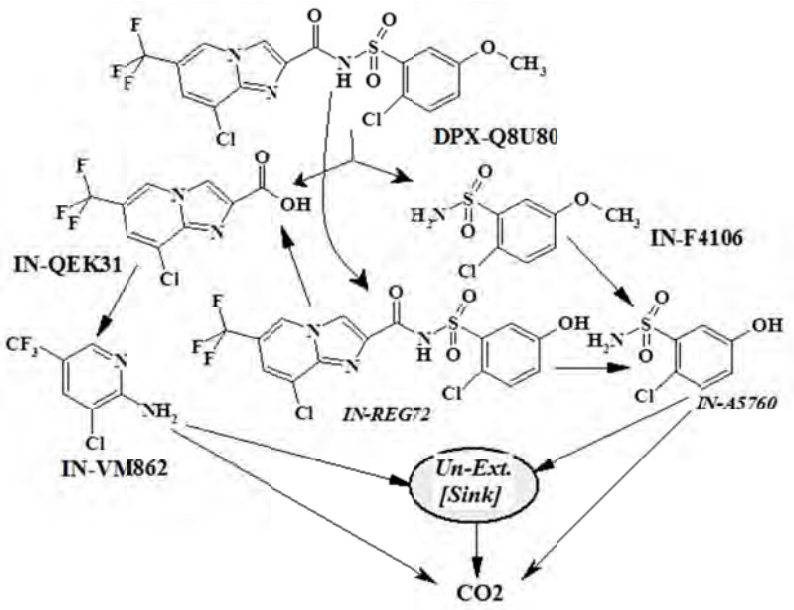


Figure 21 Degradation pathway for fluazaindolizine in aerobic soil

Grant and Wardrope (2015 DuPont-37393) investigated the aerobic soil metabolism of ^{14}C-fluazaindolizine in four soils in the dark at 20 ± 2 °C for up to 150 days. The soils used were Speyer 2.2, Thessaloniki, Graffignana and Lleida, collected from Hanofen, Germany; Thessaloniki, Greece; Graffignana, Italy and Lleida, Spain (Table 75). In addition, aerobic degradation in sterilized Speyer 2.2 was included to determine the effect of microbial activity on the rate of degradation.

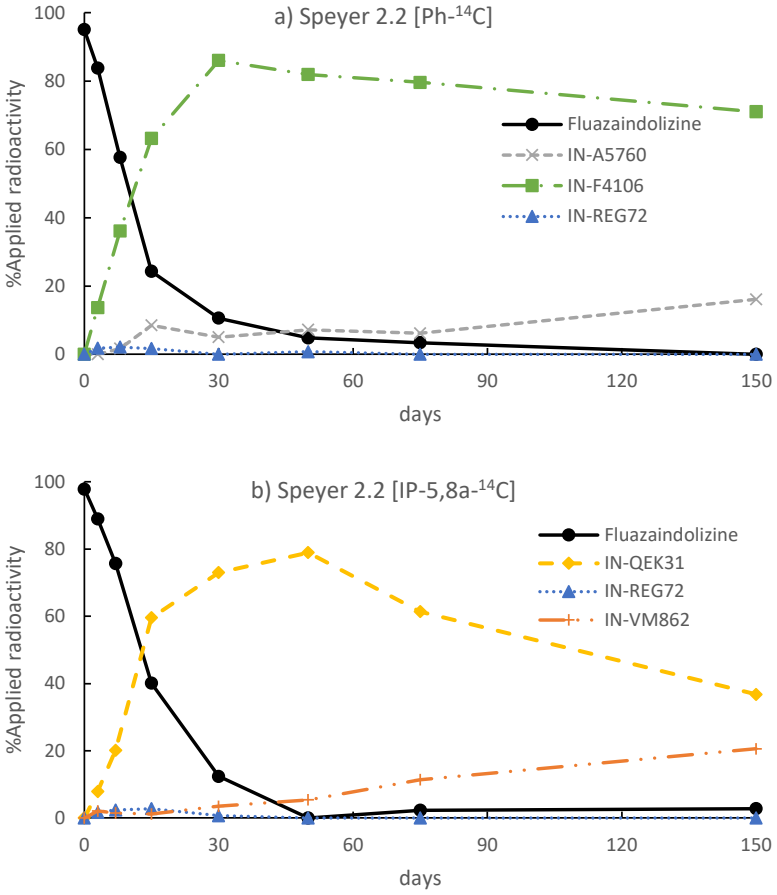
Table 75 Characteristic of soils used for aerobic degradation (DuPont-37393)

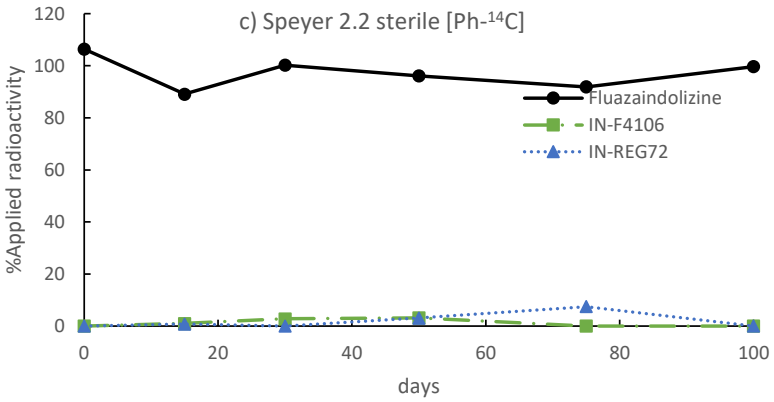
Soil	Texture	pH 0.01 M $CaCl_2$	% OM [Walkley-Black]	CEC (meq/100 g)	Biomass (mg C/kg dry soil)
Speyer 2.2, Germany	Loamy sand	5.2	2.9	7.6	387.33
Thessaloniki, Greece	Loam	6.7	2.4	17.4	1203.00
Graffignana, Italy	Loam	6.0	1.9	12.2	689.00
Lleida, Spain	Silty clay loam	7.7	2.8	16.6	816.93

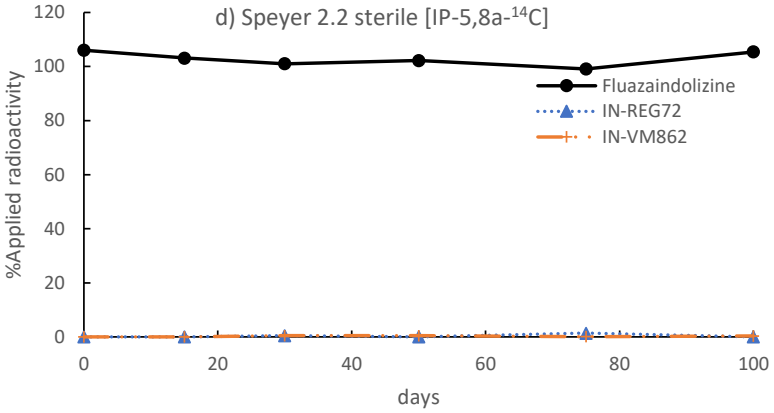
Two radiolabelled forms were used, [Ph-^{14}C] or [IP-5,8a-^{14}C]-fluazaindolizine with application at a nominal rate of 1.0 mg/kg oven dry soil, which corresponded a field application at 1000 g ai/ha assuming 10 cm incorporation and 1.0 g/cm^3 soil density. The soil samples were continuously aerated throughout the 150-day incubation period. Traps for volatiles included ethanediol, and two 1M NaOH traps to collect organic volatiles and CO_2. Soil samples were extracted sequentially with acetonitrile:2 percent aqueos formic acid (9:1), acetonitrile:2 percent aqueous formic acid (4:1); and twice with acetonitrile:2 percent aqueous formic acid (1:1). For each extraction soil/solvent slurries were sonicated at 50 °C for 45 minutes, and the soil and supernatant separated by centrifugation. Solvent unextracted ^{14}C-residues were quantified by combustion analysis and characterized *via* strong base extractions.

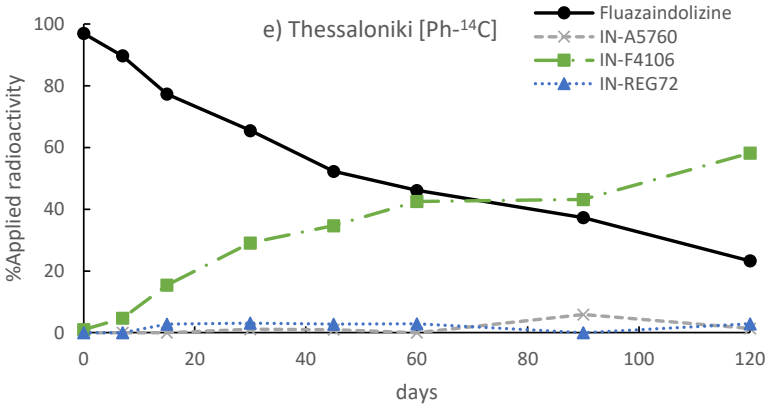
The material balance for all [Ph-^{14}C]-label samples ranged between, 91.8 to 106.8 percent AR and for the [IP-5,8a-^{14}C]-label samples between 88.7 to 109.5 percent AR. The <90 percent AR recovery was for day 150 analysis for [IP-5,8a-^{14}C]-label and was attributed to the formation of a substantial amount of a volatile metabolite IN-VM862.

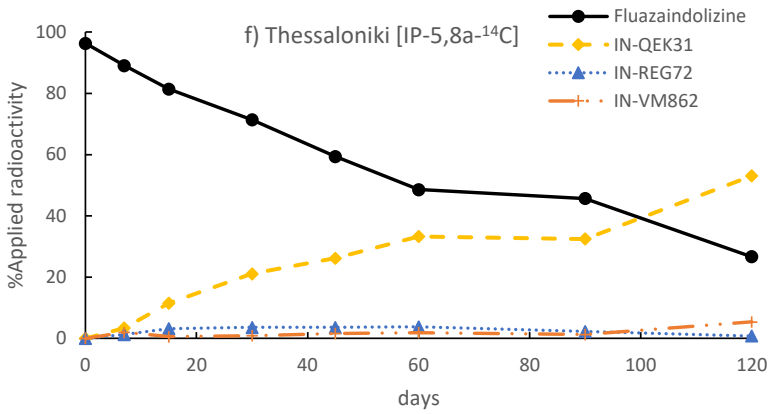
The distribution of radioactivity for the two soils is shown in Figure 22. The extracted ^{14}C decreased from close to 100 percent at zero-time to 44-87 percent AR by 150 days while unextracted ^{14}C increased to a maximum of 6.6 to 22.6 percent AR by the end of the study. Mineralisation to $^{14}CO_2$ was 0.2-12.5 percent AR and was greater for the [IP-5,8a-^{14}C]-label in all soils.

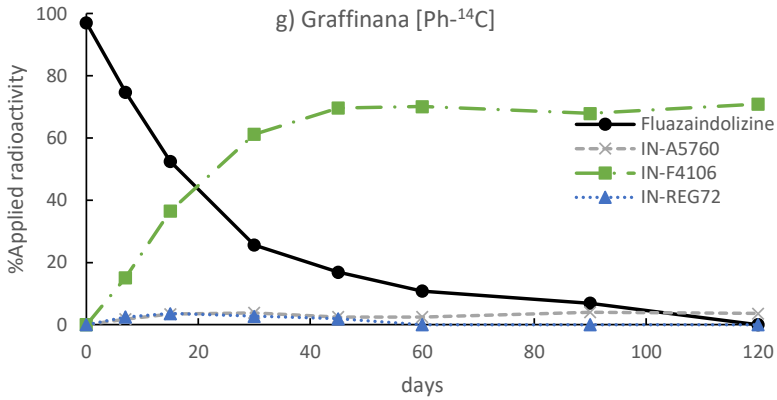


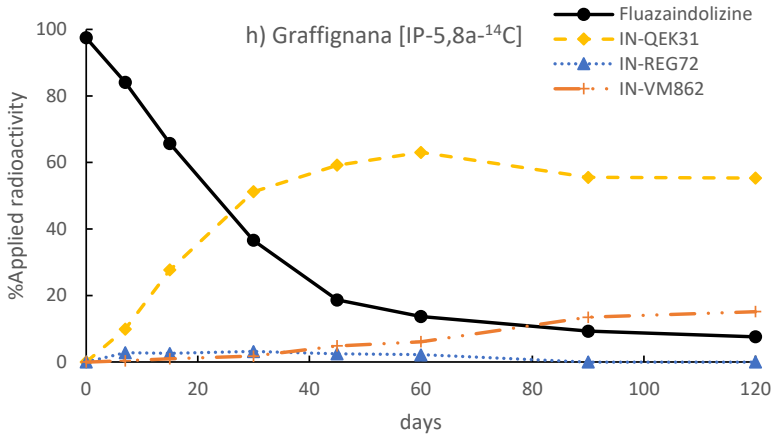

c) Speyer 2.2 sterile [Ph-14C]
%Applied radioactivity
days
Fluazaindolizine
IN-F4106
IN-REG72


d) Speyer 2.2 sterile [IP-5,8a-14C]
%Applied radioactivity
days
Fluazaindolizine
IN-REG72
IN-VM862


e) Thessaloniki [Ph-14C]
%Applied radioactivity
days
Fluazaindolizine
IN-A5760
IN-F4106
IN-REG72


f) Thessaloniki [IP-5,8a-14C]
%Applied radioactivity
days
Fluazaindolizine
IN-QEK31
IN-REG72
IN-VM862


g) Graffinana [Ph-14C]
%Applied radioactivity
days
Fluazaindolizine
IN-A5760
IN-F4106
IN-REG72


h) Graffignana [IP-5,8a-14C]
%Applied radioactivity
days
Fluazaindolizine
IN-QEK31
IN-REG72
IN-VM862

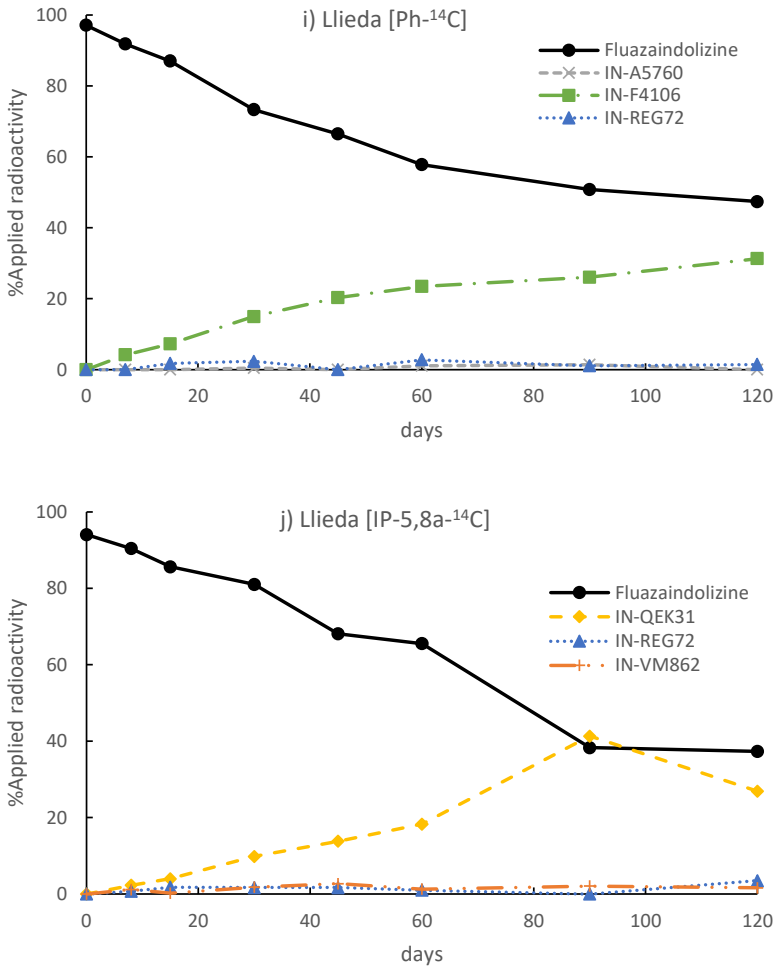


Figure 22 Aerobic degradation of ^{14}C-fluazaindolizine on Speyer 2.2, loamy sand, Thessaloniki loam, Graffignana loam and Lleida silty clay loam soils

Unextracted residues were characterised using organic matter fractionation into humin, fulvic acid and humic acid fractions. The largest portion of the unextracted radioactivity (≥ *ca* 5.1–12.5 percent AR, day 50–150) was associated with the humin fraction of the soil organic matter, while the balance was found in humic (0.4–2.9 percent AR) and fulvic acid (3.1–7.0 percent AR) fractions indicating substantial incorporation of ^{14}C into natural constituents.

Degradation in all soils proceeded primarily *via* cleavage of the fluazaindolizine molecule into two major metabolites, IN-QEK31 and IN-F4106. Peak levels of these two metabolites were found after 30–50 days in Speyer 2.2 soil and at later sampling intervals in other soils due to different rates of degradation of the parent compound. Smaller amounts of one other metabolite, IN-A5760 were observed in all soils from the [Ph-^{14}C]-label treatments and a volatile metabolite IN-VM862 was found in all [IP-5,8a-^{14}C]-label treatments. IN-REG72, accounted for <5 percent in all viable soils

The lack of any significant metabolites in the sterile Speyer 2.2 samples demonstrated that degradation observed in all soils was primarily due to microbial processes. The degradation rates (DT_{50} and DT_{90}) in each soil were determined using SFO kinetics (Table 76).

Table 76 SFO kinetics summary for fluazaindolizine in four soils

Soil	DT_{50} (days)	DT_{90} (days)	χ^2
Speyer 2.2	10.3	34.2	9.9
Speyer 2.2 (sterile)	2790	9280	2.9
Thessaloniki	63.8	212	3.2
Graffignana	19.5	64.6	4.9
Lleida	91.9	305	2.1

Manikandan (2014 DuPont-35133) studied the rate of degradation of [^{14}C]-fluazaindolizine in three soils, incubated in dark for 120 days under aerobic conditions at 20 ± 2 °C. The soils used in this study were shown in Table 77.

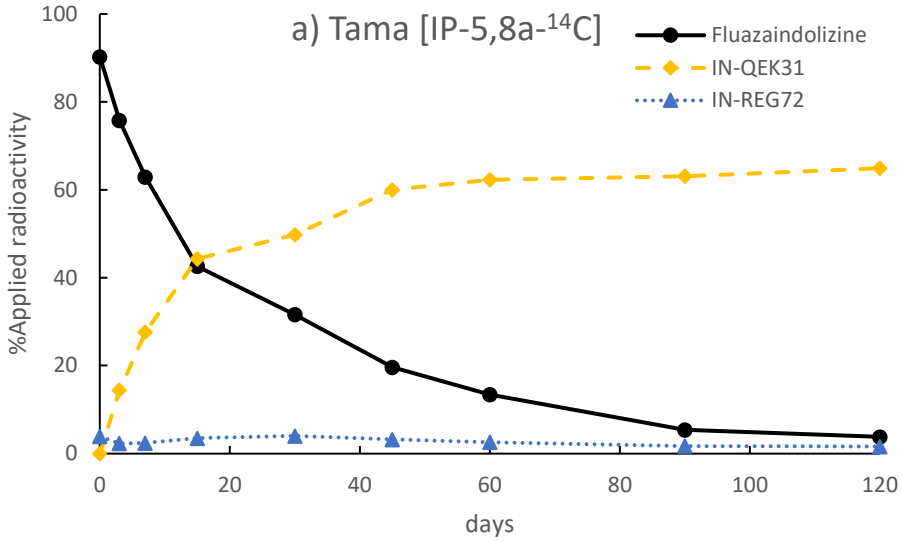
Table 77 Characteristic of soils used for aerobic degradation, study DuPont-35133

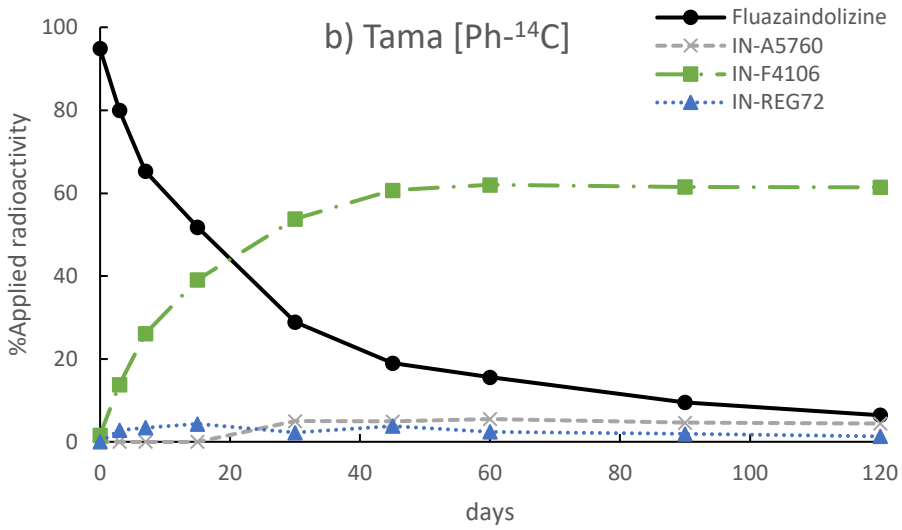
Soil	Texture	pH 0.01 M $CaCl_2$	% OM [Walkley-Black]	CEC (meq/100 g)	Biomass (mg C/kg soil)
Tama, United States	Silty clay loam	5.9	3.5	17.6	1437.49
Porterville, United States	Sandy loam	6.4	0.97	14.8	1340.97
Speyer 2.2, Germany	Loamy sand	5.4	3.0	9.4	1182.04

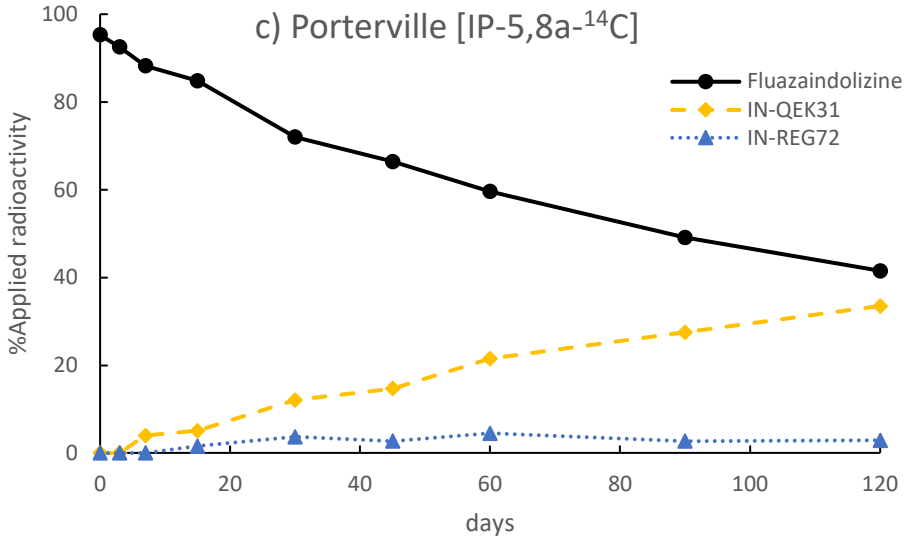
The moisture content of the soils was adjusted to 50 percent MWHC which corresponded to 35.8, 26.5 and 26.2 percent moisture for Tama, Porterville and Speyer soils, respectively. Application as at a nominal rate of 1 mg/kg dry soil. The soil samples were continuously aerated throughout the 120-day incubation period. Traps for volatiles included ethanediol, and two 1M KOH traps to collect organic volatiles and CO_2. Soil samples were sequentially extracted with acetonitrile:2 percent aqueous formic acid, 9:1; acetonitrile:2 percent aqueous formic acid, 4:1; followed by two extractions with acetonitrile:2 percent aqueous formic acid, 1:1. The distribution of radioactivity for the two soils is shown in Figure 23. IN-QEK31 and IN-F4106 were the main degradation products exceeding 5 percent AR.

The mean material balance was 91.2, 92.3 and 82.5 percent AR for Tama, Porterville and Speyer soils, respectively. Mass balance had declined to less than 90 percent towards the late stages of the study in two soils. Largest loss of ^{14}C was associated with Speyer soil, which also showed the fastest degradation rate and mineralization to $^{14}CO_2$. Due to inaccurate counting of the trap solutions, $^{14}CO_2$ was not appropriately accounted for and caused inaccurate material balance. It was quite certain that all losses were due to the low counting of $^{14}CO_2$. Porterville soil, which showed slower degradation and low CO_2 production displayed acceptable material balance. Therefore, the degradation rates, which are based on the amounts of parent and metabolites, are still valid.

Extracted ^{14}C recovered in soils treated with ^{14}C-fluazaindolizine and incubated at 20°C, was near quantitative at zero time with recoveries ranging from 94.1 and 98.2 percent AR. The amount of extracted radioactivity then decreased to a range of 48-86 percent of AR in soils incubated for 120 days, and unextracted ^{14}C increased to 4.9-15.2 percent AR. The ^{14}C associated with volatile traps at 120 days was ≤ 1.7 percent in all soils; however, it was recognized that the amount of $^{14}CO_2$ had been under-estimated.


a) Tama [IP-5,8a-14C]
Fluazaindolizine
IN-QEK31
IN-REG72
%Applied radioactivity
days
0
20
40
60
80
100
120


b) Tama [Ph-14C]
Fluazaindolizine
IN-A5760
IN-F4106
IN-REG72
%Applied radioactivity
days
0
20
40
60
80
100
120


c) Porterville [IP-5,8a-14C]
Fluazaindolizine
IN-QEK31
IN-REG72
%Applied radioactivity
days
0
20
40
60
80
100
120

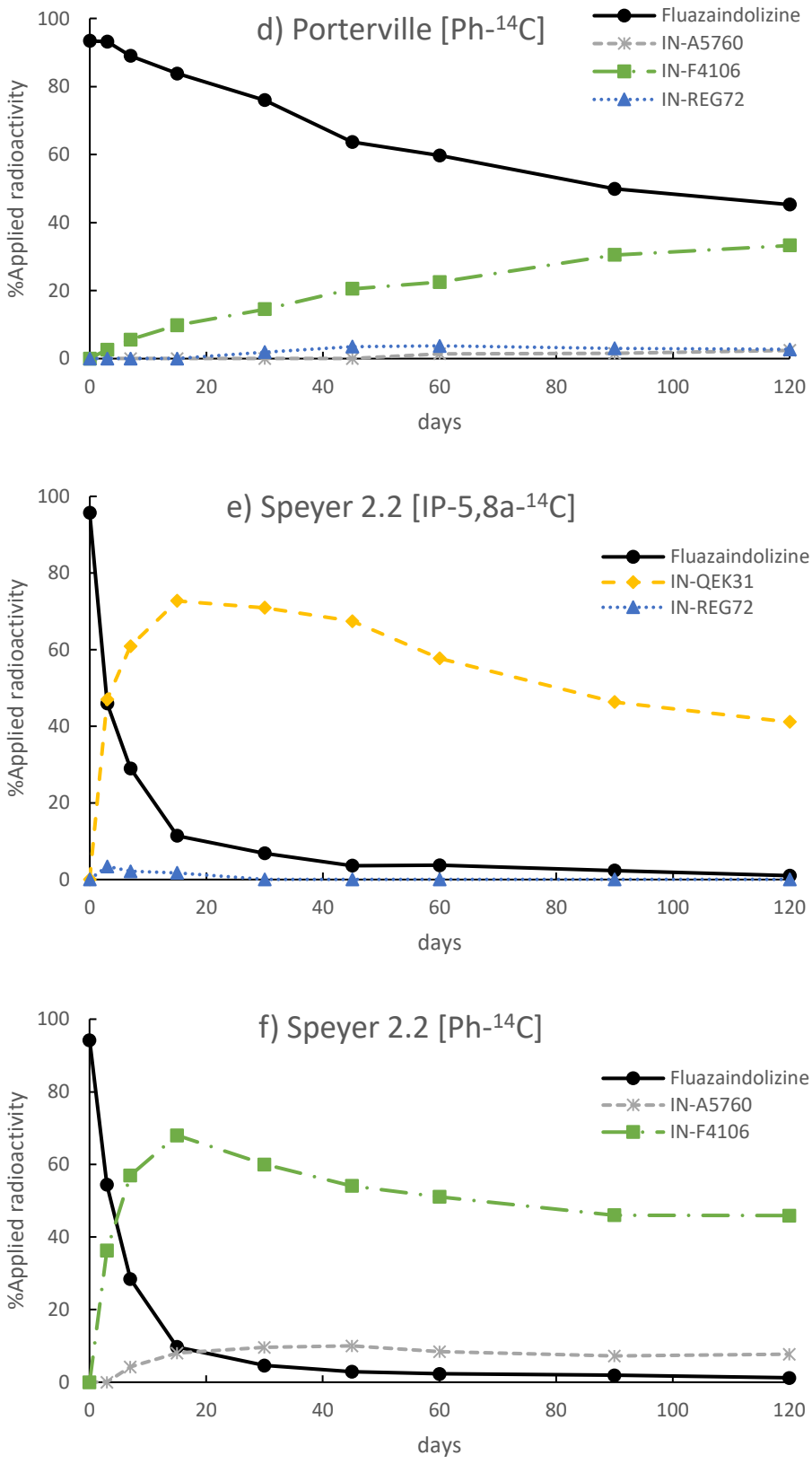


Figure 23 Aerobic degradation of ^{14}C-fluazaindolizine on Tama silty clay loam, Porterville sandy loam and Speyer 2.2 loamy sand soils

The non-normalized DT_{50} and DT_{90} values for fluazaindolizine are summarised in Table 78.

Table 78 Summary degradation kinetics for fluazaindolizine in three soils

Soil	DT_{50} (days)	DT_{90} (days)	χ^2	r^2	Best fit model
Tama	14.3	78.5	5	0.99	DFOP
Porterville	97.8	325	2	0.99	SFO
Speyer	3.4	18.1	8	0.998	DFOP

In a separate study, Manikandan (2017 DuPont-40810, Revision No. 1) investigated the rate of degradation of [^{14}C]-fluazaindolizine at a nominal rate of 1.0 mg/kg in four soils, incubated for 120 days in dark under aerobic conditions at 20 ± 2 °C. The moisture content of the soils was adjusted to pF 2.0 (moisture at 1/10 bar) which corresponded to 45.2, 28.7, 31.8 and 24.9 percent moisture for Tama, Hidalgo, Penn, and Woodland soils, respectively. The soils used in this study are shown in Table 79.

Table 79 Characteristic of soils used for aerobic degradation (DuPont-40810)

Soil	Texture	pH 0.01 M $CaCl_2$	% OM Walkley-Black	CEC (meq/100 g)	microbial (mg C/kg soil)
Tama, IL United States	Clay loam	6.7	6.4	20.2	207
Hidalgo, TX United States	Sandy clay loam	7.9	0.65	16.4	253
Penn NJ United States	Loam	6.0	2.1	8.3	213
Woodland CA United States	Loam	5.9	2.3	16.1	241

The soil samples were continuously aerated throughout the 120-day incubation period. Traps for volatiles included ethanediol, and two 1M KOH traps to collect organic volatiles and CO_2. Since the purpose of the study was to obtain aged sorption data as well as the degradation rate data, at each sampling occasion, a desorption step was carried out first by extracting the soil samples overnight (12 to 18 hours) with 0.01 M $CaCl_2$ using a 2:1 solvent to soil ratio. Subsequently, each soil sample was sequentially extracted for 45 minutes at 50 °C using acetonitrile:water, 9:1, (v/v), acetonitrile:water, 4:1, (v/v) and finally with acetonitrile:water, 1:1, (v/v).

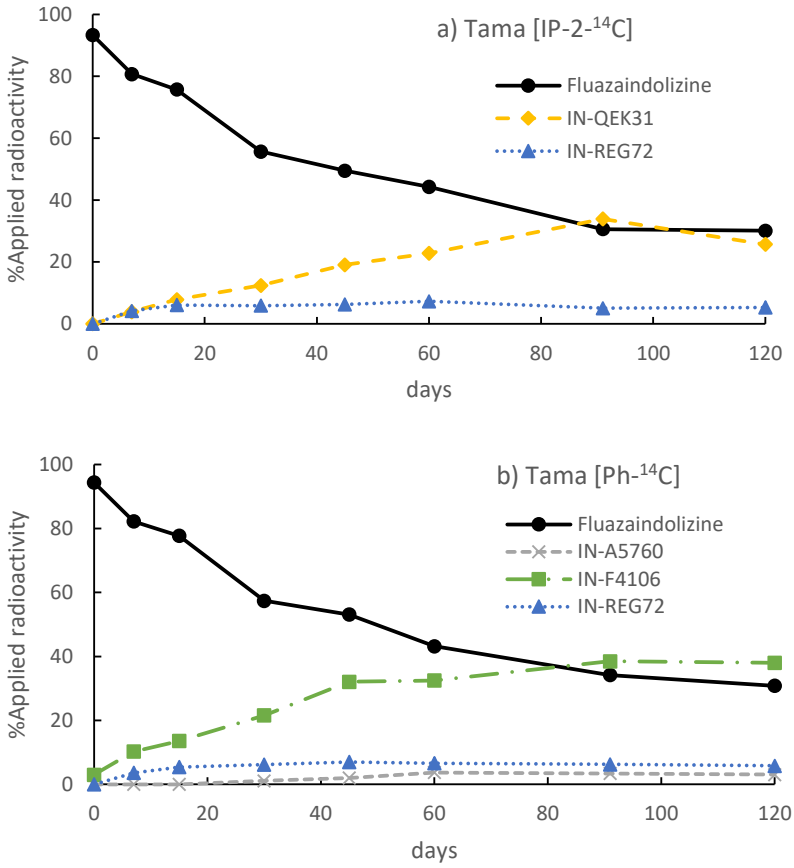
Material balances were quantitative for all samples (overall mean 98.2 ± 4.5 percent), with individual values in the range of 90.2–108.3 percent AR. The distribution of radioactivity for the two soils is shown in Figure 24.

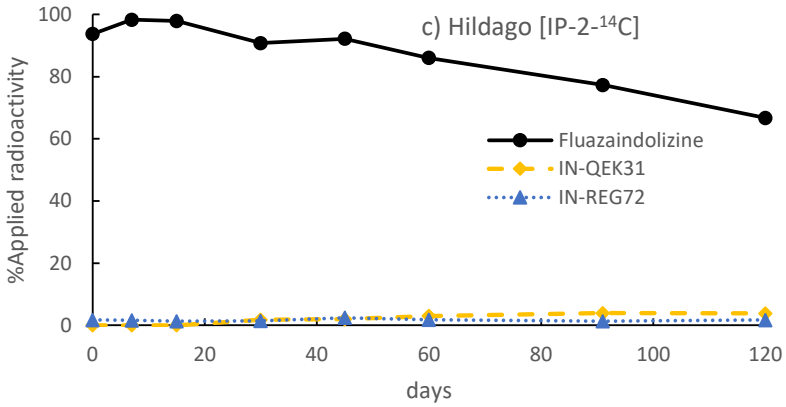
Total extracted ^{14}C in all soils treated with [IP-2-^{14}C]-label and [Ph-^{14}C]-label of [^{14}C]-fluazaindolizine, was near quantitative at zero time and thereafter declined as the incubation progressed. Between 55.5-72.7 and 74.9-89.3 percent AR of the ^{14}C was extracted with 11.0-28.3 and 8.1-17.0 percent AR of the ^{14}C unextracted at the end of the incubation in soil samples treated with [IP-2-^{14}C]-label and [Ph-^{14}C]-label of fluazaindolizine, respectively. Traps for volatiles recovered 9.3-13.1 and 0.7-3.1 percent AR by 120 days the incubation for the [IP-2-^{14}C]- and [Ph-^{14}C]-label experiments, respectively.

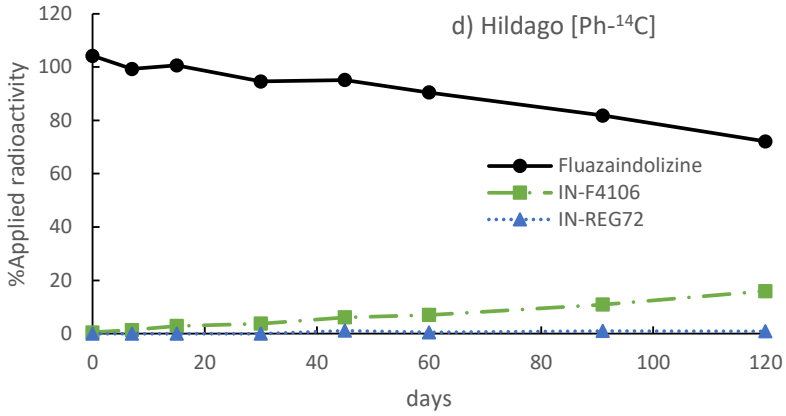
Degradation of [^{14}C]-fluazaindolizine proceeded *via* the molecular cleavage of sulphonamide linkage to generate IN-QEK31 and IN-F4106. Demethylation of the phenyl-ether linkage leading to IN-REG72 also provided a parallel degradation pathway. Further degradation of metabolites led to substantial amounts of $^{14}CO_2$ and incorporation of ^{14}C into soil organic matter.

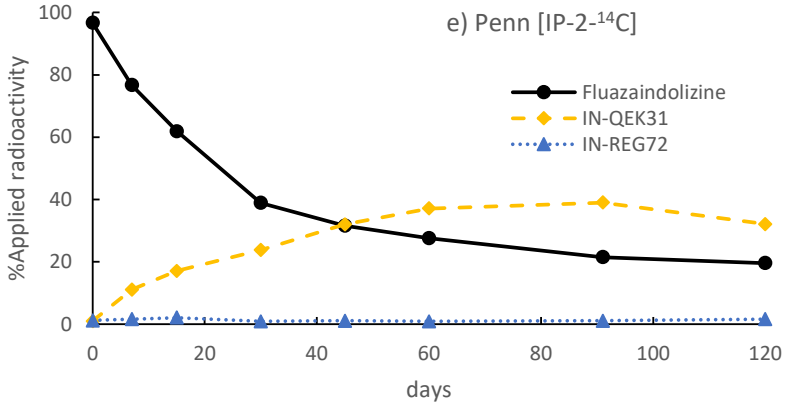
Unextracted residues were characterised using organic matter fractionation into humin, fulvic acid and humic acid fractions. Humin accounted for 3.1-9.4 percent AR, fulvic acid 4.3–17.3 percent AR and humic acid 0.5-1.9 percent AR indicating substantial incorporation of ^{14}C into natural constituents.

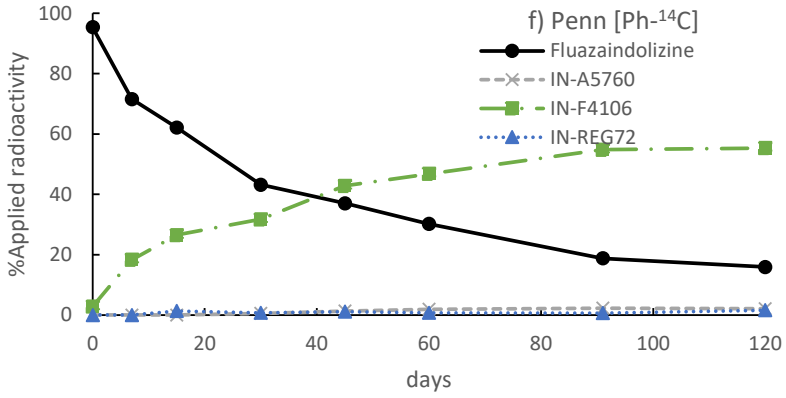
In all soils, for the [IP-2-^{14}C]-label IN-QEK31 was the main metabolite observed, while its counterpart, IN-F4106, was found in the [Ph-^{14}C] label experiment in amounts similar to IN-QEK31. Minor components (mostly <5 percent AR) were IN-REG72 and IN-A5760. There were no unidentified metabolites and no other component exceeding the LOQ




c) Hildago [IP-2-14C]
%Applied radioactivity
days
Fluazaindolizine
IN-QEK31
IN-REG72


d) Hildago [Ph-14C]
%Applied radioactivity
days
Fluazaindolizine
IN-F4106
IN-REG72


e) Penn [IP-2-14C]
%Applied radioactivity
days
Fluazaindolizine
IN-QEK31
IN-REG72


f) Penn [Ph-14C]
%Applied radioactivity
days
Fluazaindolizine
IN-A5760
IN-F4106
IN-REG72

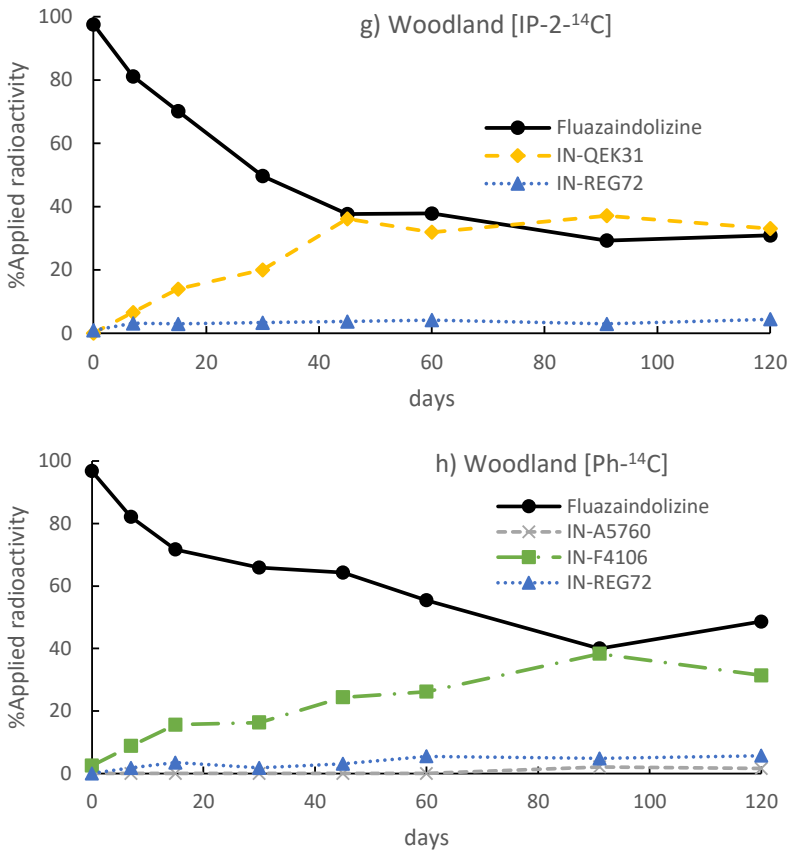


Figure 24 Aerobic degradation of ^{14}C-fluazaindolizine on Tama clay loam, Hidalgo sandy clay loam, Penn loam and Woodland loam soils

The DT_{50} and DT_{90} values for fluazaindolizine are summarised in Table 80

Table 80 Summary degradation kinetics for fluazaindolizine in four soils

Soil	DT_{50} (days)	DT_{90} (days)	χ^2	r^2	Model
Tama	49.5	521	2.97	0.9879	FOMC
Hidalgo	241	801	1.87	0.9063	SFO
Penn	23.1	236	2.34	0.9932	FOMC
Woodland	46.0	725	3.66	0.8774	DFOP

Aerobic rate of degradation for fluazaindolizine metabolites in soil

[^{14}C]IN-VM862: Rate of dissipation in five aerobic soils

Grant (2015 DuPont-40719) studied the aerobic transformation of [^{14}C]-IN-VM862 in five soils under aerobic conditions in the dark at 20 ± 2 °C for 60 days. A radiolabelled form of the test item with carbon 14 in the pyridine-2,6 position was used in the test substance. [^{14}C]-IN-VM862 was applied at a nominal rate of 1.0 mg/kg oven dry soil. Soil characteristics are shown in Table 81.

Table 81 Characteristic of soils used for aerobic degradation of [^{14}C]-IN-VM862 (DuPont-40719)

Soil Name	Texture	pH 0.01 M $CaCl_2$	% OM Walkley-Black	CEC (meq/100 g)	Microbial (mg C/kg soil)
Tama United States	Silty clay loam	6.7	4.9	20.9	673.13
Saasafras United States	Sandy loam	4.7	2.4	6.1	296.67
Nambsheim France	Sandy loam	7.4	2.5	9.5	698.33
Porterville United States	Loam	7.5	1.3	10.8	193.27
Speyer 2.2 Germany	Loamy sand	6.1	2.5	6.8	452.20

The soil samples were continuously aerated throughout the 60-day incubation period however volatilization was difficult to control, even with reduced rates of airflow. Traps for volatiles included ethanediol, and two 1M NaOH traps to collect organic volatiles and CO_2. Soil samples was sequentially with acetonitrile:2 percent formic acid (aq) (9:1); followed by acetonitrile:2 percent formic acid (aq) (4:1); and then acetonitrile:2 percent formic acid (aq, 1:1).

The material balance for all samples ranged between 90.2-122.9 percent AR. Samples with high material balance had a disproportionately high recovery from the polyurethane plug associated with the sample. In the Tama soil system treated with [^{14}C]IN-VM862 the solvent extracted ^{14}C in the soil decreased from mean values of 102.6 percent AR at Day 0 to approximately 51.0 percent AR after 60 days aerobic incubation while another 49.5 percent was found in the polyurethane plug extracts. A maximum of 13.6 percent was unextracted. A minor portion of the unextracted ^{14}C was mineralized to CO_2 ($^{14}CO_2$ cumulatively accounted for 0.4 percent AR).

Porterville soil system treated with [^{14}C]IN-VM862 also showed similar behaviour as Tama soil. Solvent extracted ^{14}C-residues in the soil decreased from mean values of 100.7 percent AR at Day 0 to 48.8 percent AR after 60 days aerobic incubation, with 49.9 percent of the parent compound appearing in the polyurethane plug extracts. Non-extracted residues increased to a mean maximum value of 4.4 percent AR at Day 40 before decreasing to 3.8 percent AR at Day 60. Radiolabelled $^{14}CO_2$ cumulatively accounted for 0.5 percent AR, indicating a minor portion of the applied test substance undergoing degradation.

The remaining three soils showed even higher degree of volatilization of [^{14}C]IN-VM862. In the Sassafras, Nambsheim and Speyer 2.2 soil systems treated with [^{14}C]IN-VM862, 80-105 percent of the applied test substance evaporated over 60 days and was found unchanged in the polyurethane plugs used to collect the volatile materials. The solvent extracted ^{14}C-residues in the soils decreased from mean values of approximately 100 percent AR at Day 0 to only 10 to 20 percent by Day 60. Unextracted ^{14}C resulting from degradation of IN-VM862 only accounted for 2.7 to 8.7 percent AR at Day 60 while $^{14}CO_2$ cumulatively accounted for 0.3 to 0.4 percent AR.

Volatilised ^{14}C collected in these polyurethane plugs was unchanged IN-VM862. Modelling of the data was used to separate volatilization from rate of degradation, using a model which accounted for both processes simultaneously. The volatilisation rate was directly correlated to the rate of air flow. Volatilization dominated the overall dissipation from soil. A summary of volatilization rate constants and degradation rate is shown in Table 82.

Table 82 Summary of volatilization and degradation rates for IN-VM862

Soil	K_evaporation (% $mL^{-1}day^{-1}$)	K_degradation (day^{1})	Degradation DT_{50} (days)
Tama	1.10×10^{-6}	0.0019	361
Sassafras	3.30×10^{-6}	0.0031	226
Nambsheim	5.43×10^{-6}	0.0006	>1000
Porterville	8.55×10^{-7}	0.0012	>500

Soil	K_evaporation (% $mL^{-1}day^{-1}$)	K_degradation (day^{1})	Degradation DT_{50} (days)
Speyer 2.2	3.05×10^{-6}	0.0016	>400

In a separate study, Hussain and McCorquodale (2017 DuPont-42493) studied the rate of degradation of [^{14}C]IN-VM862 in four soils. This study was conducted with minimum possible airflow needed for aerobic soil conditions to minimize volatilization. The soils used are shown in Table 83.

Table 83 Characteristic of soils used for aerobic degradation of [^{14}C]-IN-VM862, study DuPont-42493

Soil	Texture	pH 0.01 M $CaCl_2$	% OM Walkley-Black	CEC (meq/100 g)	M0icrobial (mg C/kg soil)
Lleida, Spain	Silty clay	7.6	1.3	16.1	431.2
Vimagano, Italy	Loam	5.9	1.4	11.2	291.5
Sindos, Greece	Sandy loam	6.9	1.4	12.7	566.3
Nambsheim, France	Silty loam	7.6	1.0	11.3	264.7

The test system was acclimatized for up to 17 days prior to treatment. [^{14}C]IN-VM862 labelled in the pyridine-2,6 position was used on this study and was applied to the soil at a nominal rate of *ca* 3.0 mg/kg dry soil weight. Treated vessels were incubated for up to 120 days under aerobic conditions in the dark at 20 $\pm$ 2 °C at pF 2 moisture. The soil samples were continuously aerated throughout the 120-day incubation period. Ethanediol and 2 1M NaOH traps. Each sample also utilised two polyurethane plugs to trap IN-VM862 and any volatile degradation products.

Soil samples were sequentially extracted with solvents acetonitrile:2 percent formic acid (9:1); followed by acetonitrile:2 percent formic acid (4:1) and then acetonitrile:2 percent formic acid (1:1) and finally THF:0.1 percent formic acid (aq) (9:1). Polyurethane plugs were replaced at each sampling interval and were extracted with 20 mL acetonitrile.

The mean material balance was quantitative for all samples and was in the range 94.7–99.6 percent AR, 94.8–99.3 percent AR, 94.3–99.0 percent AR, and 91.2–98.6 percent AR for Lleida, Vimagano, Sindos and Nambsheim soils, respectively.

Soil extraction of ^{14}C was essentially quantitative at Day 0 in all soils (> 97.3 percent) and decreased progressively to 21.0, 27.9, 21.0 and 20.0 percent AR at Day 120 in Lleida, Vimagano, Sindos and Nambsheim soils, respectively. This decrease was mainly attributable to volatilisation, although degradation, in the form of evolved $^{14}CO_2$, and potentially indicated by unextracted ^{14}C, also contributed. Volatilized IN-VM862 accounted for 67.8, 59.3, 64.7 and 68.3 percent AR at Day 120 in Lleida, Vimagano, Sindos and Nambsheim soils, respectively. Unextracted ^{14}C accounted for 5.3, 7.8, 8.6 and 4.7 percent AR in Lleida, Vimagano, Sindos and Nambsheim soils, respectively while cumulative $^{14}CO_2$ accounted for 0.6, 0.5, 0.9 and 0.5 percent AR at Day 120 in Lleida, Vimagano, Sindos and Nambsheim soils, respectively. Unidentified components accounted for a mean maximum of <3 percent AR in soil or polyurethane plug extracts in each soil.

The decline of parent IN-VM862 in soil extracts and the amount which volatilised, was analysed using a model to distinguish between degradation and volatilisation, and the degradation rate (DT_{50} degradation) and overall dissipation rate (DT_{50} dissipation) determined using SFO kinetics. Table 84 shows the DT_{50} values for each soil type.

Table 84 Summary of degradation rates for IN-VM862

Soil	Degradation DT_{50} (days)	Dissipation DT_{50} (days)
Lleida	409	45
Vimagano	352	57

Soil	Degradation DT_{50} (days)	Dissipation DT_{50} (days)
Sindos	347	49
Nambsheim	451	42

IN-VM862 degrades mainly to unextracted soil components *via* microbial degradation and dissipates from the environment by a combined process of degradation and volatilisation.

[^{14}C]IN-A5760: Rate of degradation

Yogeesha (2015b DuPont 40734) studied the rate of degradation of IN-A5760 in five soils, incubated in dark under aerobic conditions at a nominal temperature of 20 ± 2 °C for a study duration of 120 days. The soils used in this study were as follows.

Table 85 Characteristic of soils used for aerobic degradation of [^{14}C]-IN-A5760, study DuPont-40734

Soil	Texture	pH 0.01 M $CaCl_2$	% OM Walkley-Black	CEC (meq/100 g)	Microbial (mg C/kg soil)
Nambsheim France	Sandy loam	7.1	4.0	10.4	321
Tama United States	Silty clay loam	6.7	6.4	20.2	309
Penn	Loam	6.0	2.1	8.3	192
Woodland	Loam	5.9	2.3	16.1	190
Saasafras United States	Sandy loam	4.7	1.9	6.4	262

The moisture content of the soils was adjusted to pF 2.0 (moisture at 1/10 bar) which corresponded to 23.5, 45.2, 31.8, 24.9 and 23.6 percent moisture for Nambsheim, Tama, Penn, Woodland, and Sassafras soils, respectively. The soils were allowed to acclimate for 8 days prior to test item application. The test item was radiolabelled on phenyl-(U)-^{14}C and was applied at a nominal rate of 1.0 mg/kg dry soil. The soil samples were continuously aerated throughout the 120-day incubation period. A series of traps containing ethanediol and potassium hydroxide was used for the retention of non-specific ^{14}C-volatile organic compounds and $^{14}CO_2$, respectively. Soil samples were sequentially extracted by sonication at 50 ± 5 °C for 45 minutes with acetonitrile:water, 80:20 (v/v) and then acetonitrile:water, 50:50 (v/v).

Material balances were quantitative for all samples (overall mean 93.3 ± 1.7 percent AR), with individual values in 91-101 percent AR range. The distribution of radioactivity for the two soils is shown in Figure 25.

Total extracted radioactivity in all soils showed a rapid decrease soon after test substance application. As an example, extracted ^{14}C in Nambsheim (sandy loam) soil was 97.3 percent AR at zero time decreased to 12.8 percent AR after 120 days. Concurrently, the unextracted ^{14}C increased from 0.3 percent AR at zero time to 62.2 percent AR after 120 days. A similar trend was noted in all five soils, and the extracted ^{14}C ranged from a low 12.5 percent AR in Nambsheim soil to a high of 60.0 percent AR in Woodland soil. Higher unextracted ^{14}C correlated with the amount of $^{14}CO_2$ collected in the volatile traps. Percent $^{14}CO_2$ reached a peak level of 17.6 percent AR after 120 days in Nambsheim and ranged between 7–19 percent AR in other soils by the end of the study. Levels of $^{14}CO_2$ and unextracted ^{14}C indicated rapid and extensive degradation of IN-A5760 in all soils.

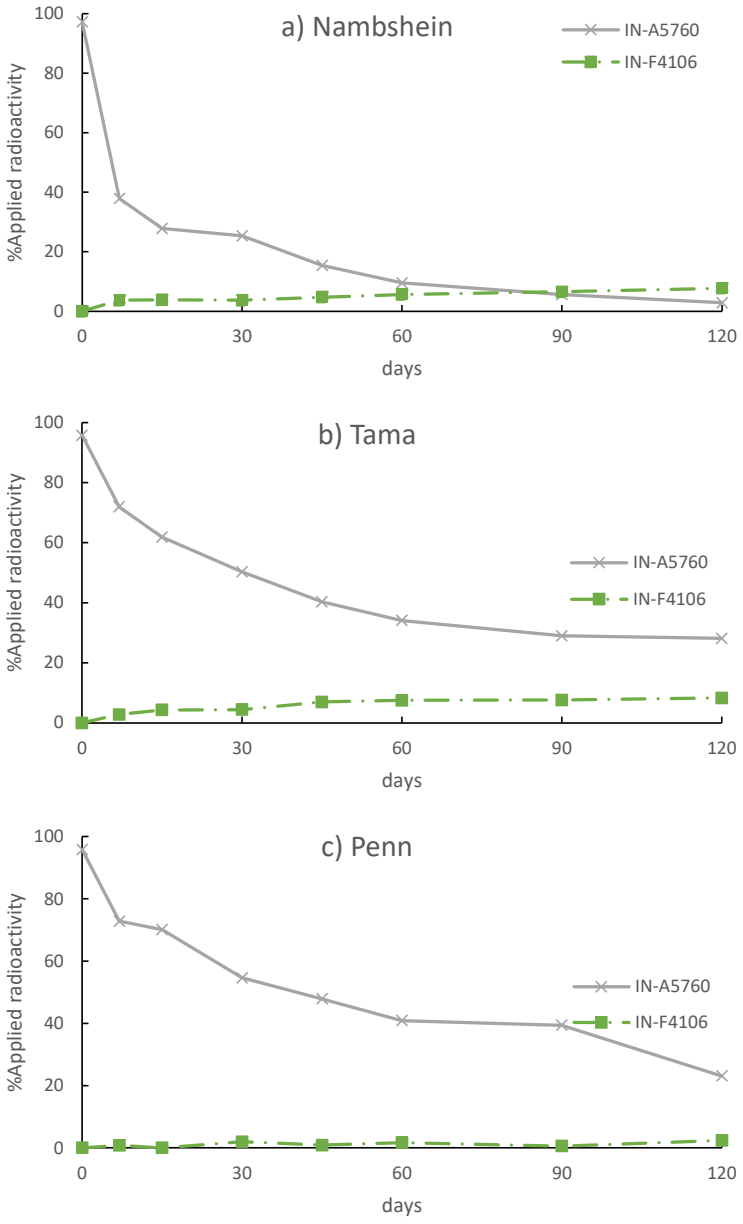
Surprisingly, a small amount (max. 7.7–8.3 percent AR) of IN-F4106 was observed in two soils (Nambsheim and Tama). It remained below 5 percent in the other three soils. Observation of IN-F4106 was surprising because IN-A5760 is itself formed from degradation of IN-F4106 (or from IN-REG72).

The calculated DT_{50} and DT_{90} values for IN-A5760 are summarised in Table 86.

Table 86 Summary of degradation rates for IN-A5760

Soil	DT_{50} (days)	DT_{90} (days)	χ^2	r^2	Model
Nambsheim	3.66	66.5	5.43	0.9632	DFOP
Tama	30.2	948	2.5	0.9466	FOMC
Penn	48.8	227	5.22	0.8825	DFOP
Woodland	88.6	728	2.67	0.8676	DFOP
Sassafras	29.9	931	3.72	0.964	DFOP

In soil, IN-A5760 degrades primarily by forming unextracted ^{14}C and CO_2. Kinetics modelling of the data suggests that unextracted ^{14}C continues to be mineralized and generate $^{14}CO_2$.



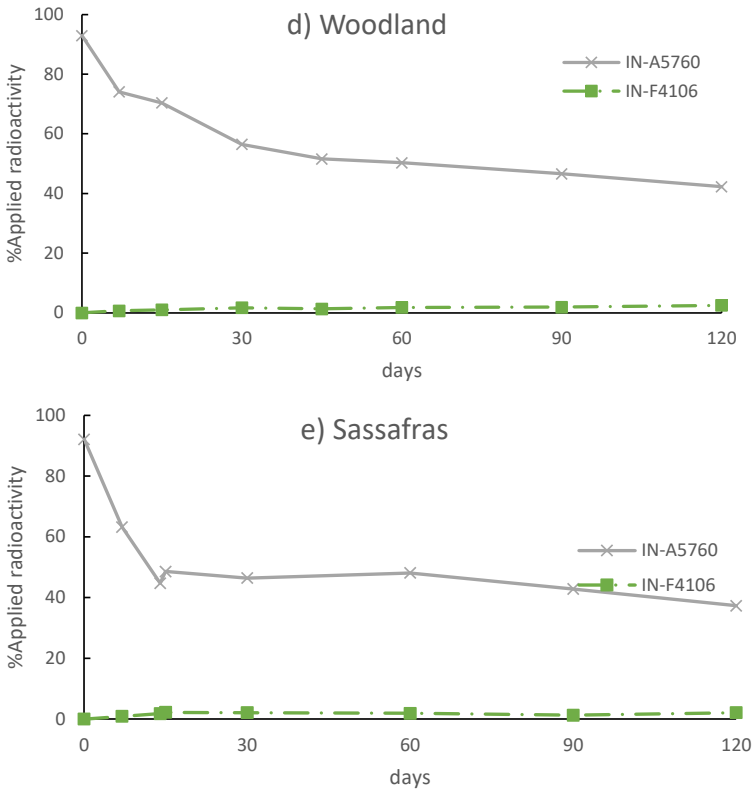


Figure 25 Aerobic degradation of ^{14}C-IN-A5760 on Nambsheim sandy loam, Tama silty clay loam, Penn loam, Woodland loam and Sassafras sandy loam soils

[^{14}C]IN-F4106: Rate of degradation

The rate of degradation of IN-F4106 was studied using [^{14}C]IN-F4106 at a nominal rate of 1.0 μg/g (dry soil) in five soils, incubated in dark under aerobic conditions at a nominal temperature of 20 ± 2 °C for a study duration of 150 days (Yogeesha 2015a DuPont-35485). The moisture content of the soils was adjusted to pF 2.0 (moisture at 1/10 bar) which corresponded to 20.8, 41.7, 33.4, 10.3 and 20.6 percent moisture for Nambsheim, Tama, Cajon (Porterville), Speyer and Sassafras soils, respectively. The soils used in this study are shown in Table 87.

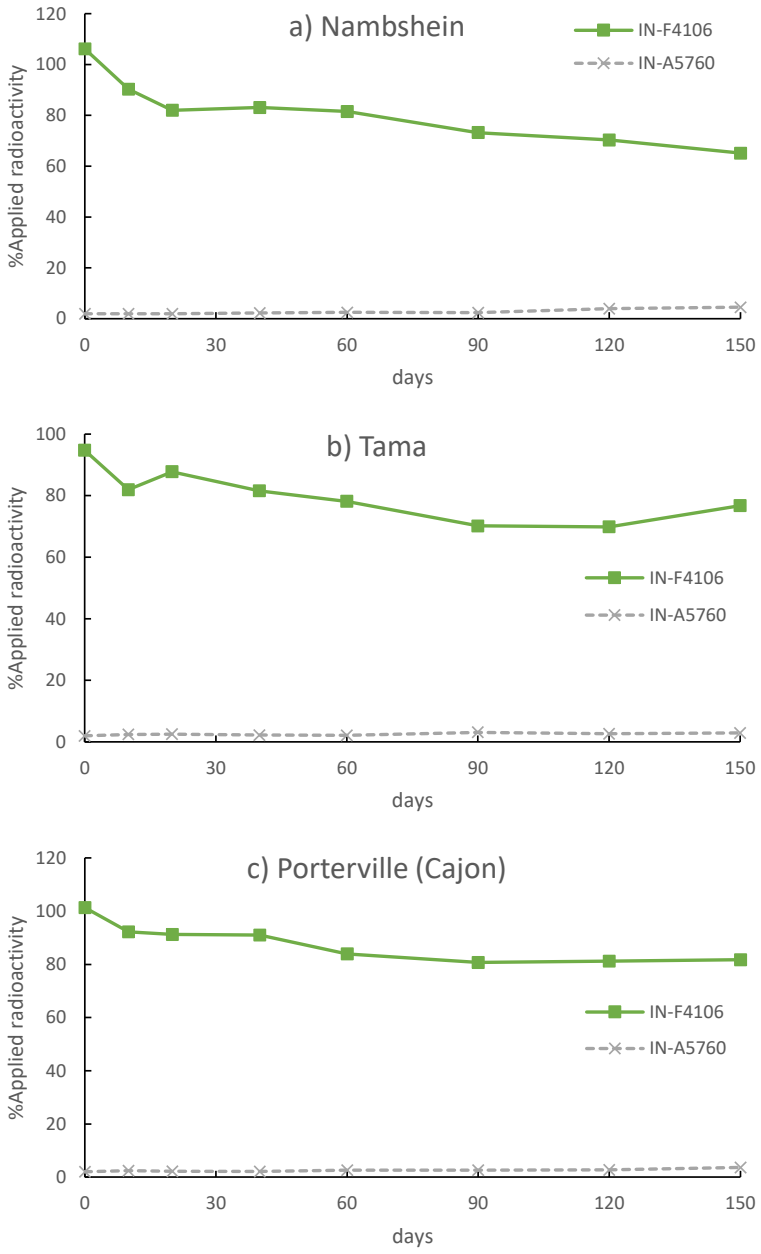
Table 87 Characteristic of soils used for aerobic degradation of [^{14}C]-IN-F4106, study DuPont-35485

Soil	Texture	pH 0.01 M $CaCl_2$	% Organic matter Walkley-Black	CEC (meq/100 g)	Microbial (mg C/kg)
Nambsheim France	Sandy loam	7.4	2.5	9.5	239
Tama United States	Silty clay loam	6.7	4.9	20.9	285
Cajon (Porterville) United States	Loam	7.5	1.3	10.8	213
Speyer 2.2 Germany	Loamy sand	6.1	2.5	6.8	211
Sassafras United States	Sandy loam	4.7	2.4	6.1	277

The soil samples were continuously aerated throughout the 150-day incubation period. Material balances were quantitative for all samples (overall mean 100.4 ± 5.2 percent), with individual values in the range of 92.6–111.1 percent of applied radioactivity (AR). The distribution of radioactivity for the two soils is shown in Figure 26.

Total extracted ^{14}C in all soils showed a moderate decline as the incubation progressed and it reflected partial degradation of IN-F4106 during the study. Between 69.6–87.1 percent AR of the ^{14}C was extracted at the end of the incubation with 6.8-20.7 percent AR unextracted. In general, higher amounts of unextracted ^{14}C corresponded with more degradation of the parent compound. Volatile traps accounted for 2.2–6.9 percent AR by the end of the incubation.

IN-A5760 was the primary degradation product in all soils, and it accounted for as much as 21.9 percent in one of the soils. Low levels of this metabolite were partly due to a slow degradation of the parent compound and presumably a faster degradation of the metabolite.



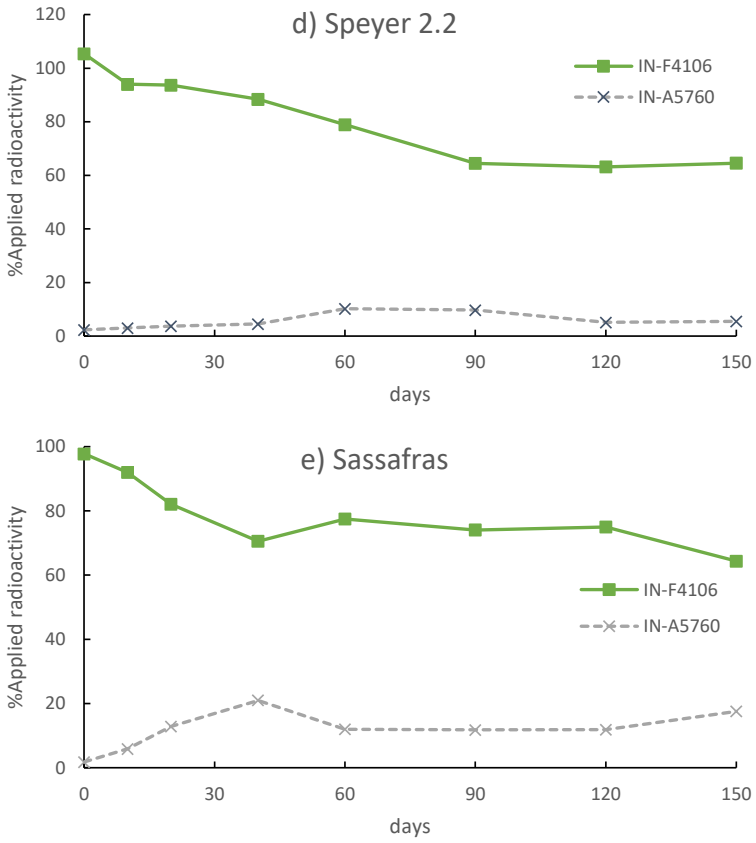


Figure 26 Aerobic degradation of ^{14}C-IN-F4106 on Nambsheim sandy loam, Tama silty clay loam, Porterville loam, Speyer 2.2 loamy sand and Sassafras sandy loam soils

In soil, IN-F4106 degrades by multiple processes forming IN-A5760, CO_2 and bound residues. DT50 and DT90 are shown in Table 88.

Table 88 The DT_{50} and DT_{90} values for IN-F4106 calculated for various soils.

Soil	DT_{50} (days)	DT_{90} (days)	Fit Model
Nambsheim	235	782	SFO
Tama	384	1280	SFO
Porterville (Cajon)	461	1530	SFO
Speyer 2.2	177	586	SFO
Sassafras	292	968	SFO

[^{14}C]IN-QEK31: Rate of degradation

Lowrie, C., Anderson, C. (2016 DuPont-35484, Revision No. 1) studied the rate of degradation and time dependent sorption of [^{14}C]IN-QEK31 was studied in five aerobic soils in the dark under aerobic conditions at 20 ± 2 °C for 119 days. The soils were Tama, Sassafras, Nambsheim, Porterville (Cajon) and Speyer 2.2. All soils (Table 89) were incubated under non-sterile conditions.

Table 89 Characteristic of soils used for aerobic degradation of [^{14}C]-IN-F4106 (DuPont-3548)

Soil	Texture	pH 0.01 M $CaCl_2$	% Organic matter Walkley-Black	CEC (meq/100 g)	Microbial (mg C/kg)
Nambsheim France	Sandy loam	7.4	2.5	9.5	239

Soil	Texture	pH 0.01 M $CaCl_2$	% Organic matter Walkley-Black	CEC (meq/100 g)	Microbial (mg C/kg)
Tama United States	Silty clay loam	6.7	4.9	20.9	285
Cajon (Porterville) United States	Loam	7.5	1.3	10.8	213
Speyer 2.2 Germany	Loamy sand	6.1	2.5	6.8	211
Sassafras United States	Sandy loam	4.7	2.4	6.1	277

Radiolabelled test item with ^{14}C in the imidazo[1,2-a]pyridine-5,8a ([IP-5,8a-^{14}C]) label, was applied to the soil at a nominal rate of 1.0 mg/kg oven dry soil. The soil samples were continuously aerated throughout the 119-day incubation period. Polyurethane plugs (inserted into the necks of soil flasks were used for retention of the volatile metabolite IN-VM862, in addition to ethanediol and sodium hydroxide traps for the collection of non-specific volatile organic compounds and $^{14}CO_2$, respectively.

Soil samples were sequentially extracted using acetonitrile:2 percent formic acid (aq) (9:1); followed by acetonitrile:2 percent formic acid (aq) (4:1); and then with acetonitrile:2 percent formic acid (aq) (1:1), all at 50°C in an ultrasonic bath.

The material balance for all samples was quantitative with the exception of Sassafras Day 40 (Replicate 1, 86 percent AR), Sassafras Day 90 [87 percent AR] and Porterville Day 119 (Replicate 2, 111 percent AR) samples. The distribution of radioactivity for the two soils is shown in Figure 27.

The amount of extracted ^{14}C declined gradually over the study period due to degradation of the applied material, IN-QEK31, and its partial incorporation into the soil organic matter. Unextracted ^{14}C increased to nearly 20 percent AR in three of the five soils (Sassafras, Nambsheim and Speyer 2.2). Percentages of 19.3, 7.4, 10.9 percent AR as $^{14}CO_2$ and 14.3, 23.4, 15.1 percent AR captured as volatilised IN-VM862 in the Sassafras, Nambsheim and Speyer 2.2 soils, respectively, were noted at the final sampling interval in these soils.

The degradation of IN-QEK31 in all soils resulted in the formation of one major metabolite, IN-VM862. A substantial amount of IN-VM862 had volatilised from the soil. The formation of IN-VM862 increased throughout the duration of the study in all five soils reaching mean maximum values of 21.5, 27.4, 48.8, 11.4 and 32.1 percent AR in the Tama, Sassafras, Nambsheim, Porterville and Speyer 2.2 soils at Day 119 (Tama Day 90), respectively.

Unextracted ^{14}C in all soils were characterised using organic matter fractionation into humin, fulvic acid and humic acid fractions. In four of the soils (Tama, Sassafras, Porterville, and Speyer 2.2) the majority of the radioactivity was associated with the humin and fulvic acid fractions of the soil organic matter, in nearly equal quantities, while the balance was found in the humic acid fractions.

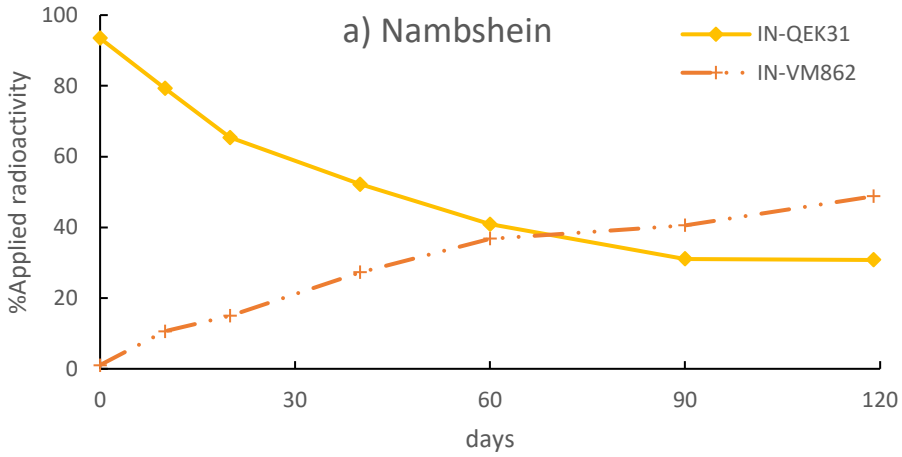
The decline of IN-QEK31 was plotted against sampling intervals and the degradation rates (DT_{50} and DT_{90}) determined using SFO kinetics. Table 90 shows the SFO DT_{50}, DT_{90} and χ^2 values for each soil type. Porterville soil failed to display meaningful decline of parent past the 10-day sampling interval, therefore, DT_{50} was not calculated.

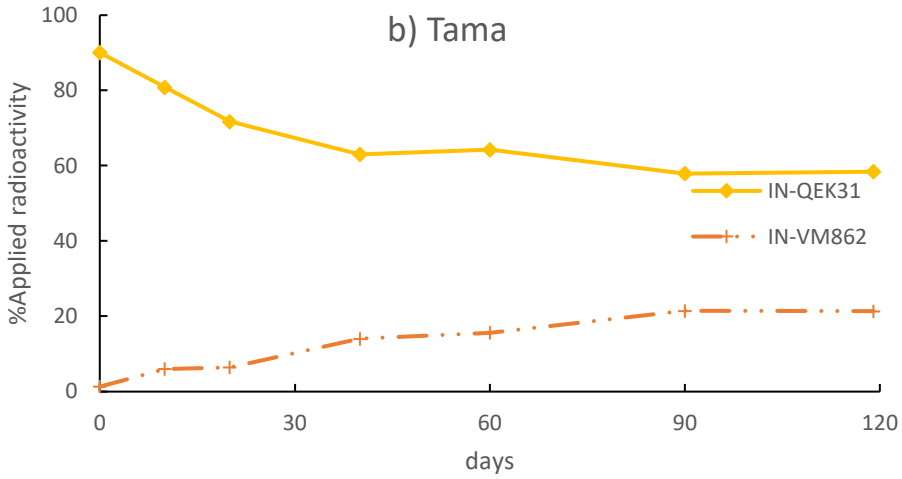
Table 90 The DT_{50} and DT_{90} values for IN-QEK31

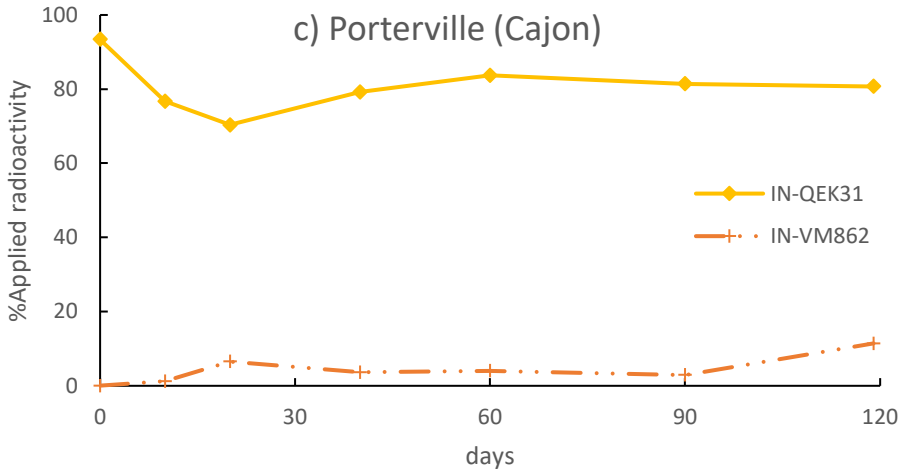
Soil [A]	Model	k-parent	r^2	χ^2	DT_{50} (days)	DT_{90} (days)
Tama	SFO	0.0038	0.76	5.83	182	605
Sassafras	SFO	0.0121	0.87	10.3	57.2	190
Nambsheim	SFO	0.0116	0.95	6.39	59.7	198
Speyer 2.2	SFO	0.0070	0.91	5.59	99.1	329

Notes:

[A] Porterville degradation data did not display a consistent pattern and was therefore not modelled.


a) Nambshein
IN-QEK31
IN-VM862
%Applied radioactivity
100
80
60
40
20
0
0
30
60
90
120
days


b) Tama
IN-QEK31
IN-VM862
%Applied radioactivity
100
80
60
40
20
0
0
30
60
90
120
days


c) Porterville (Cajon)
IN-QEK31
IN-VM862
%Applied radioactivity
100
80
60
40
20
0
0
30
60
90
120
days

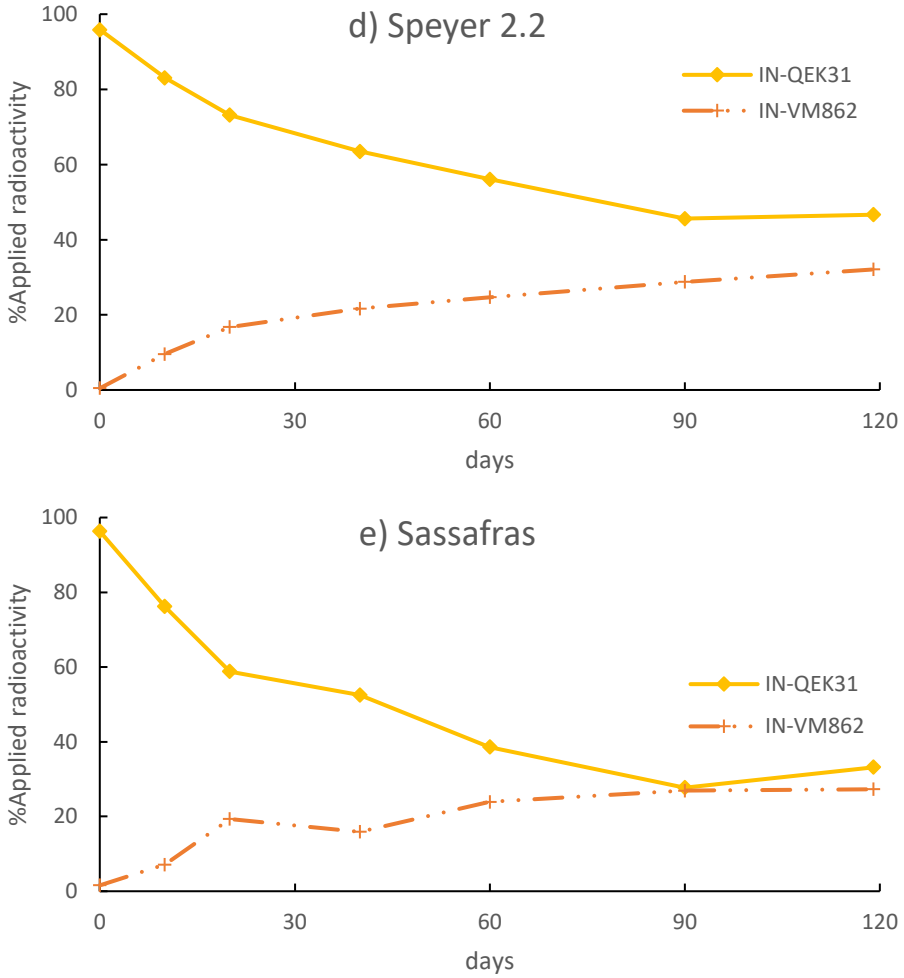


Figure 27 Aerobic degradation of ^{14}C-IN-QEK31 on Tama silty clay loam, Sassafras sandy loam, Namsheim sandy loam, Porterville loam and Speyer 2.2 loamy sand

[^{14}C]IN-REG72: Rate of degradation

The rate of degradation and aged desorption of IN-REG72 was studied using [^{14}C]IN-REG72 (uniformly labelled in the phenyl ring) at a nominal rate of 1.0 μg/g (dry soil) in five soils, incubated in dark under aerobic conditions at a nominal temperature of 20 ± 2 °C for a study duration of 120 days (Sannappa, H, 2015; Report No.: DuPont-39229).

The moisture content of the soils was adjusted to pF 2.0 (moisture at 1/10 bar) which corresponded to 20.8, 41.7, 33.4, 10.3 and 20.6 percent moisture for Nambsheim, Tama, Cajon (Porterville), Speyer and Sassafras soils, respectively. The soils used in this study are shown in Table 91.

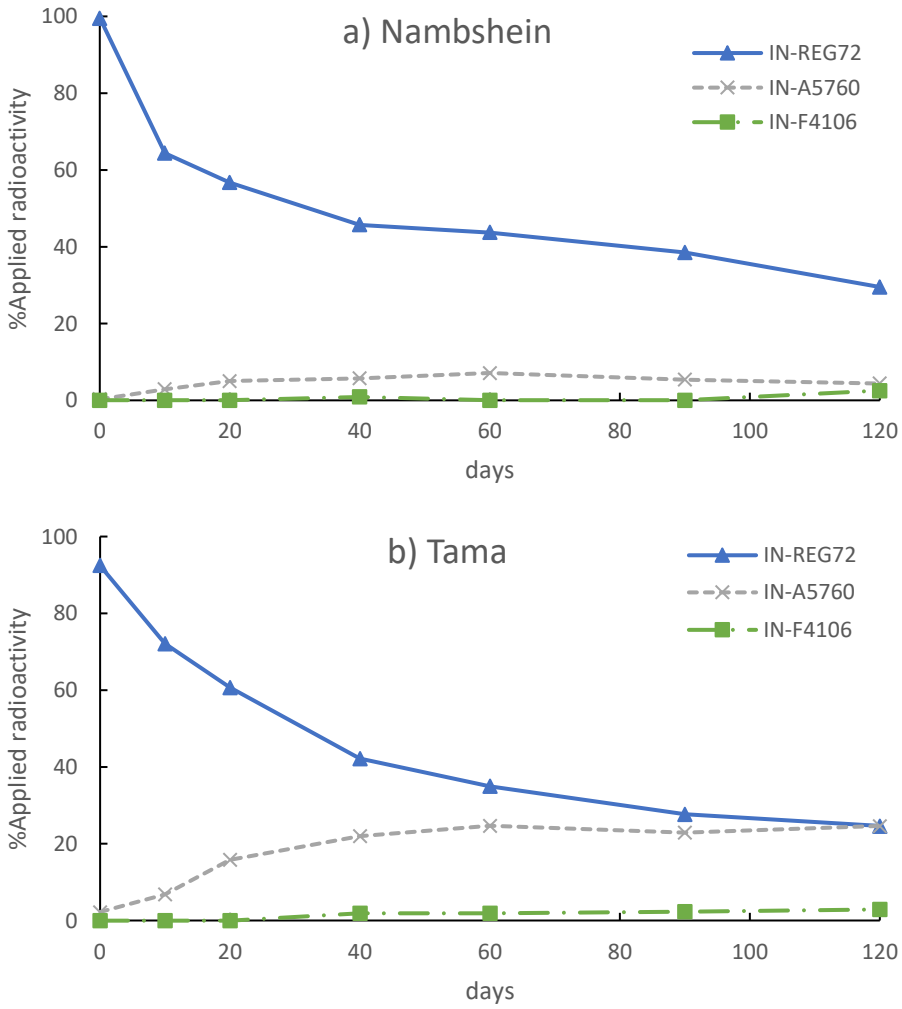
Table 91 Characteristic of soils used for aerobic degradation of [^{14}C]-IN-REG72, study DuPont-39229

Soil	Texture	pH 0.01 M $CaCl_2$	% OM Walkley-Black	CEC (meq/100 g)	Microbial (mg C/kg soil)
Tama United States	Silty clay loam	6.7	4.9	20.9	254
Saasafras United States	Sandy loam	4.7	2.4	6.1	217
Nambsheim France	Sandy loam	7.4	2.5	9.5	235
Porterville United States	Loam	7.5	1.3	10.8	217
Speyer 2.2 Germany	Loamy sand	6.1	2.5	6.8	152

The soil samples were continuously aerated throughout the 119-day incubation period. Soils were sequentially extracted with acetonitrile:water (4:1), and finally with acetonitrile:water (1:1).

Material balances were quantitative for all samples (overall mean 96.0 ± 3.7 percent), with individual values in the range of 91.7–105.2 percent AR. Total extracted ^{14}C in all soils showed a decline as the incubation progressed and it reflected partial degradation of IN-REG72 during the study. For the five soils used, 42.0–80.1 percent AR of the ^{14}C remained extracted at the end of the incubation and 12.6–43.3 percent AR was unextracted. Recoveries of cumulative radioactivity associated with volatile traps reached 2.0–7.2 percent AR by the end of incubation.

IN-A5760 was the primary degradation product noted in all soils and it accounted for as much as 30.5 percent AR in one of the soils. The degradation profiles are shown in Figure 28 and calculated DT_{50} and DT_{90} in Table 92.



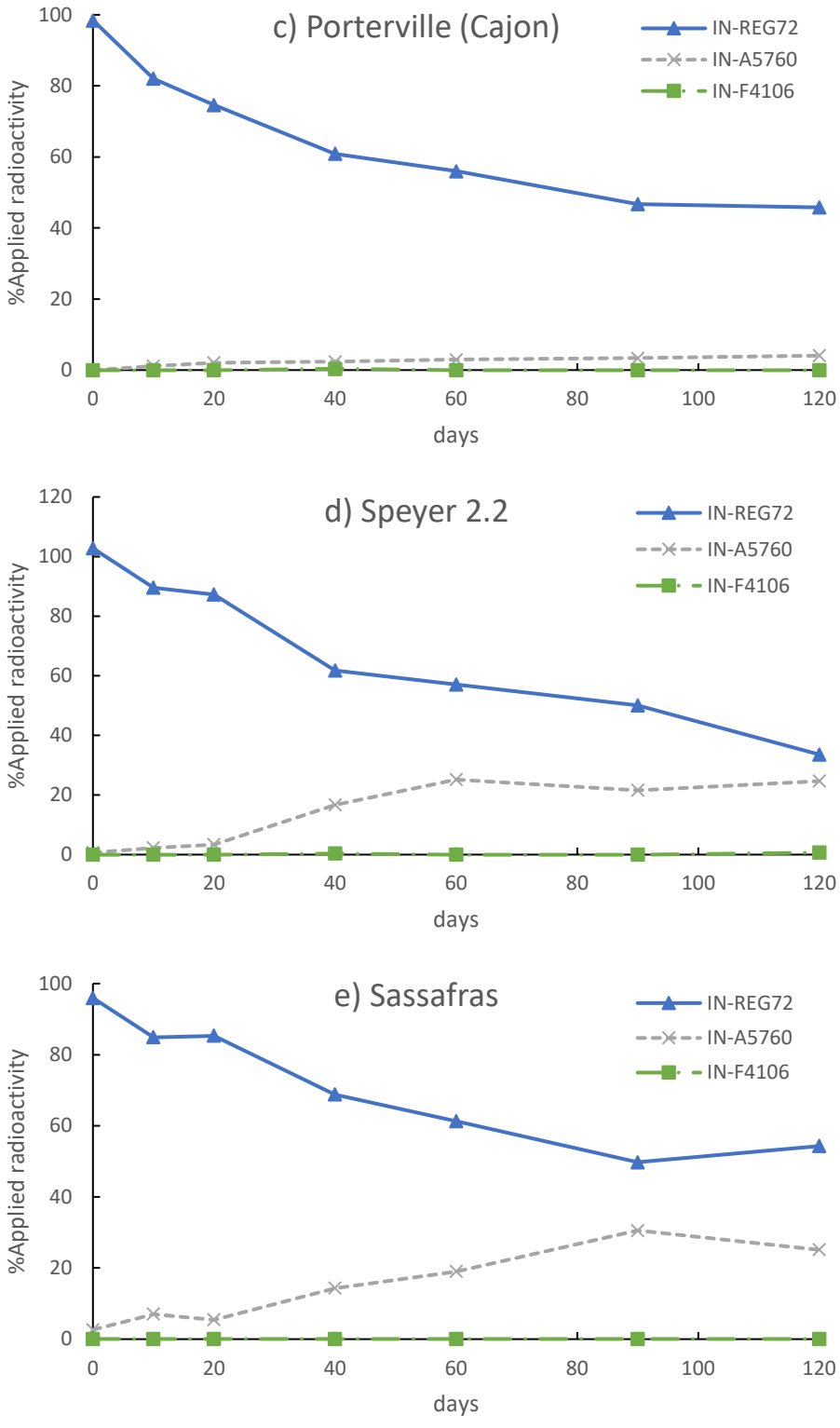


Figure 28 Aerobic degradation of ^{14}C-IN-REG72 on Tama silty clay loam, Sassafras sandy loam, Namsheim sandy loam, Porterville loam and Speyer 2.2 loamy sand

Table 92 The DT_{50} and DT_{90} values for IN-REG72

Soil	DT_{50} (days)	DT_{90} (days)	Fit Model
Nambsheim	28.4	319	DFOP
Tama	32.4	346	DFOP
Cajon (Porterville)	82.6	621	DFOP
Speyer	75.6	251	SFO
Sassafras	117	388	SFO

Soil photolysis

The rate and route of photo-degradation of [^{14}C]fluazaindolizine ([Ph-^{14}C]- or [IP-5,8a-^{14}C]-) were determined by Bell (2014 DuPont 35079) on the surface of Sassafras soil, a sandy loam soil from Maryland, United States. The test was conducted under continuous irradiation using a xenon arc lamp for approximately 15 days at ca 20 $\pm$ 2 °C. [Ph-^{14}C]-) or [IP-5,8a-^{14}C]-fluazaindolizine was applied, at a nominal rate of 1.0 mg ai/kg to thinly-layered soil (moist soil samples at ca 9.1 percent moisture, 75 percent field capacity) and irradiated using a lamp equipped with filters to eliminate wavelengths of $<$ 290 nm and reduce wavelengths greater than 800 nm to give a spectral distribution similar to natural sunlight. Non-irradiated (dark control) samples were also prepared for each radiolabel and maintained in the dark at ca 20 $\pm$ 2 °C.

Soil samples were extracted with acetonitrile:2 percent formic acid (aq) (9:1) followed by acetonitrile:2 percent formic acid (aq) (4:1) and then acetonitrile:2 percent formic acid (aq) (1:1).

[Ph-^{14}C]fluazaindolizine declined from quantitative levels at Day 0 to *ca* 48–53 percent AR following 15 days irradiation. IN-F4106 increased with irradiation duration. reaching 24.15 percent AR following 15 days of irradiation. Multiple minor metabolites were detected but not identified as none of these individually accounted for greater than 5.5 percent AR at two consecutive sampling intervals. Corresponding dark control soil extracts indicated that [Ph-^{14}C]fluazaindolizine also declined from quantitative levels at Day 0 to *ca* 44–58 percent AR following 15 days in dark conditions and contained one main degradation product identified as IN-F4106 reaching 36.53 percent AR after 15 days of incubation.

The [IP-5,8a-^{14}C]-labelled samples displayed a similar decline of fluazaindolizine as the [Ph-^{14}C] label, in dark control as well as irradiated soil extracts. One main degradation product (IN-QEK31) was identified in the dark control samples and this product was observed in minimal amounts in irradiated samples. This degradation product co-eluted with an authentic reference standard of IN-QEK31 and it was detected in the [IP-5,8a-^{14}C] label samples only. It reached a maximum value of 28.97 percent AR following 12 days of incubation, before declining slightly to 26.45 percent AR after 15 days incubation in the dark control soils. However, this degradate was not detected in substantial amounts in irradiated soils. Absence of IN-QEK31 in irradiated soils suggested that it was degraded by light almost as readily as it was generated. Multiple minor metabolites were detected in the irradiated [IP-5,8a-^{14}C] label samples as well, but not identified as none of these individually accounted for greater than 5 percent AR at any sampling interval.

Degradation of [^{14}C]fluazaindolizine in irradiated soil samples occurred primarily *via* non-photolytic degradation because the rate of degradation in irradiated and dark control soils did not differ appreciably. The main degradation products IN-F4106 ([Ph-^{14}C]-label) and IN-QEK31 ([IP-5,8a-^{14}C]-label) form on cleavage of parent molecule. Under irradiated conditions, IN-F4106 appeared to be stable and was therefore observed in similar amounts in light as well as dark soil. On the other hand, IN-QEK31 degraded quickly under irradiated conditions and was therefore, only observed in dark soil.

The DT_{50} and DT_{90} values of [^{14}C]fluazaindolizine were calculated using a simple first-order (SFO) model. Kinetics analysis for decline of the parent compound is summarised in Table 93.

Table 93 Summary of soil photolysis degradation rates for fluazaindolizine

System	k for fluazaindolizine (days^{-1})	DT_{50} (days)	DT_{90} (days)
Irradiated	0.0418 ± 0.005	16.6	55.1
Dark control	0.0368 ± 0.006	18.9	62.6
Corrected for dark control	0.0050	138	457

Fluazaindolizine does not undergo significant photolytic degradation on moist soil when exposed to artificial sunlight. Soil metabolite IN-QEK31 degrades readily on moist soil surface in the presence of light.

Field studies

Soil dissipation studies

According to the degradation pathway shown, monitoring of fluazaindolizine and five degradation products (IN-A5760, IN-F4106, IN-QEK31, IN-REG72, and IN-VM862) is adequate to characterize the behaviour of fluazaindolizine in soil environment. The metabolites analysed represented all significant metabolites in soil. IN-RYC33, an additional metabolite observed only in the confined rotation study as a minor metabolite in soil was also included for analysis in the dissipation studies. The data were used to determine the dissipation kinetics.

Ten field studies were conducted during 2013 through 2016 seasons in Canada, the European Union and the United States of America with SC formulations applied to bare soil. All studies in Europe were conducted with a use rate of 1 kg ai/ha while the studies in the North American region were conducted with use rates from 1.26 to 4 kg ai/ha. Five of the studied plots received two sequential applications at intervals ranging from 16 to 90 days, and the remaining plots received a single application.

While all major degradates were detected in the field studies, IN-QEK31, IN-F4106, and IN-VM862 made up the largest portion of observed residues. The remaining degradates (IN-A5760 and IN-REG72) were detected at levels below 3 percent of the applied material. Most fluazaindolizine and degradate residues in terrestrial field dissipation studies remained in the top 30–50 cm of soil, however residues were detected in the lowest sampled core depth (70–90 cm) in all studies.

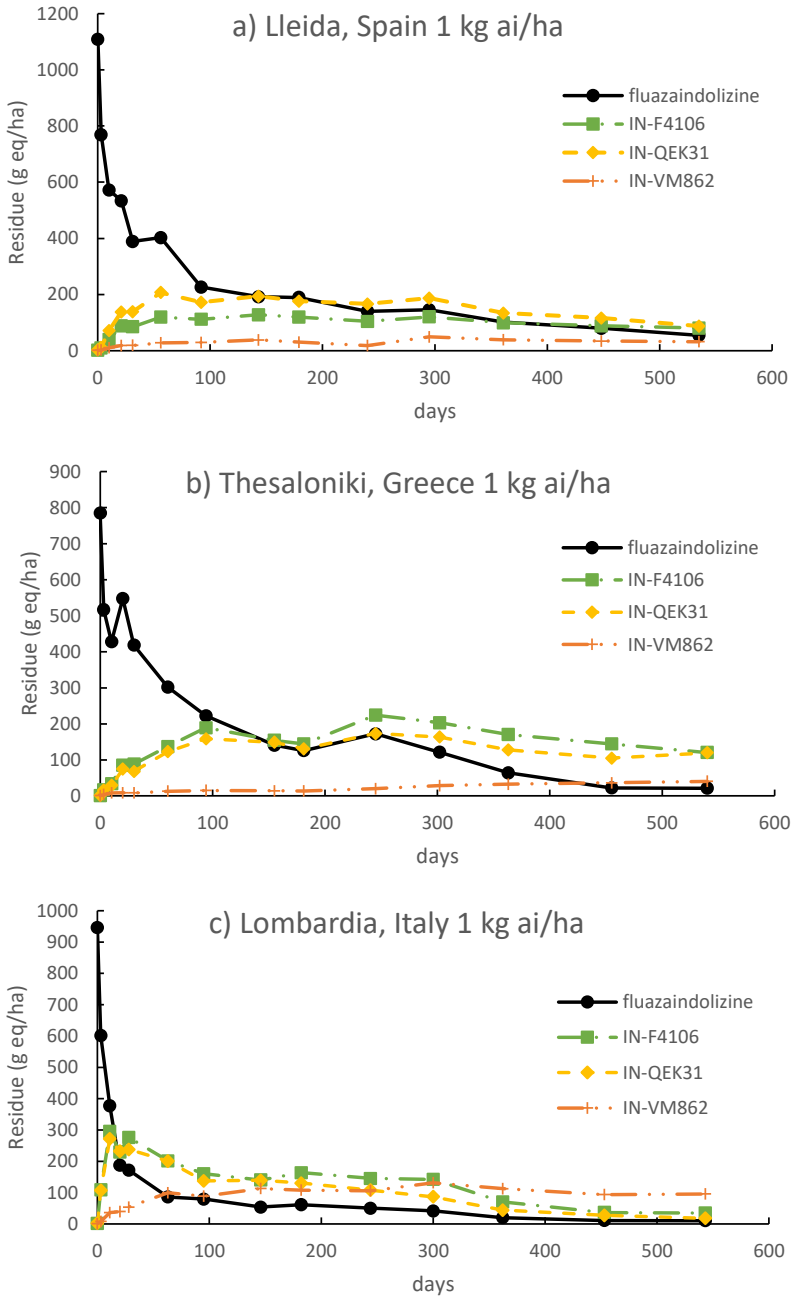
The field dissipation half-lives of fluazaindolizine ranged from 5.0–171 days which is similar to the aerobic soil metabolism degradation half-lives of 3.4–241 days. A summary of terrestrial field dissipation data is provided in Table 94.

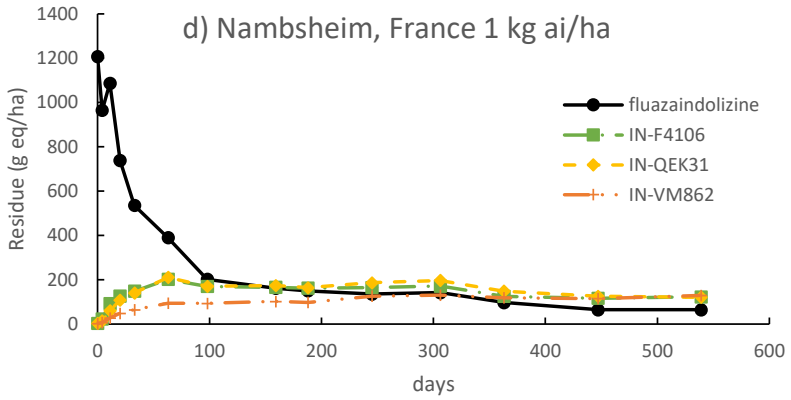
The dissipation of fluazaindolizine and metabolites (IN-QEK31, IN-F4106, IN-A5760, IN-REG72, IN-VM862 and IN-RYC33) under relevant growing conditions at four sites in Europe, one in Nambsheim France (Doig 2016 DuPont 36981), Thessaloniki Greece (Doig 2016 DuPont 36979), Lodigiano Italy (Doig 2016 DuPont 36980) and Lleida Spain (Doig 2016 DuPont 36978). Fluazaindolizine was applied to bare soil at a nominal rate of 1.0 kg ai/ha with 10-30 mm irrigation applied within 24 hours of application. Soil samples were collected to a depth of 0.9 m at predetermined intervals over an 18-month period. Residues of fluazaindolizine and metabolites were extracted using acetonitrile: 2 percent formic acid (aq) (1:1) at ambient temperature and with quantification by LC-MS/MS.

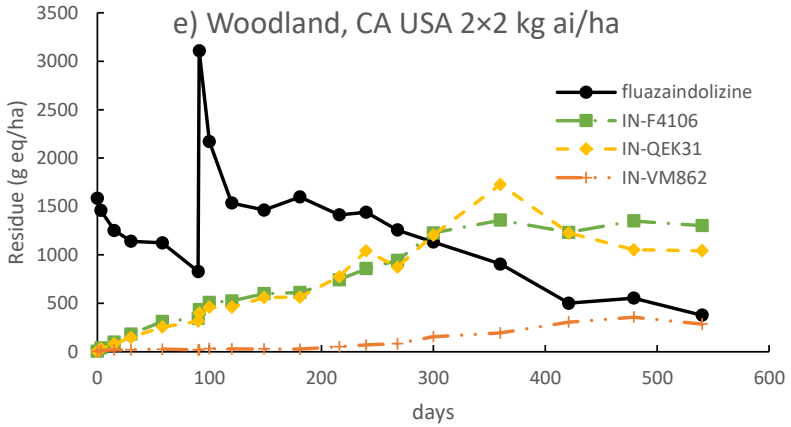
Additional dissipation studies were conducted at six sites in North America, Woodland California (2×2.0 kg ai/ha, 90 day interval, Shepard 2016 DuPont 40812), Porterville California (2 kg ai/ha and also 2×1 kg ai/ha at 90 day interval Theil 2016 DuPont-36687), Frenchtown New Jersey (0.76 and 0.5 kg ai/ha

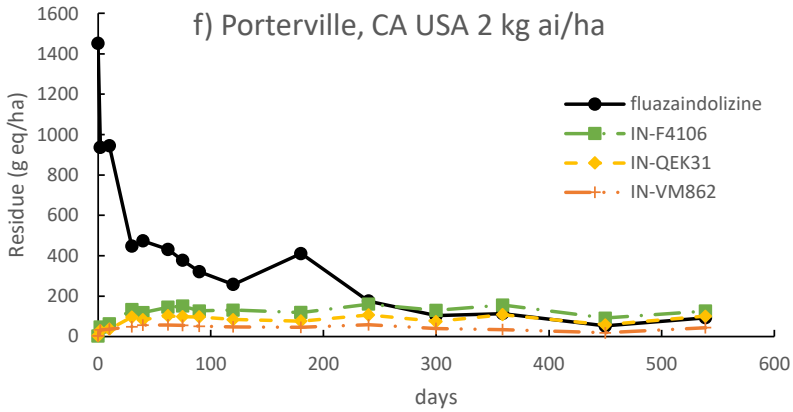
at 16 day interval Theil 2016 DuPont-36688), Branchton Ontario Canada (2 kg ai/ha Shepard 2017 DuPont-40811), Raymonville Texas (2 kg ai/ha and also 2×1 kg ai/ha at 90 day interval Theil 2016 DuPont 36689) and Oviedo Florida (2×1 kg ai/ha at 35 day interval Theil DuPont-36690).

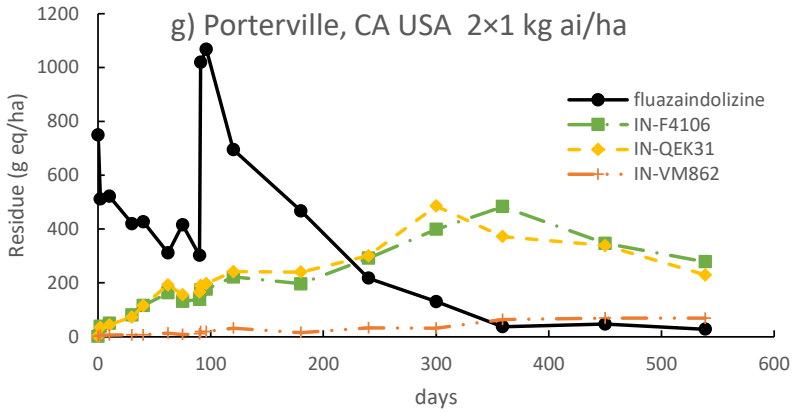
All six of the metabolites monitored were detected at some sampling intervals during these studies. Five metabolites, all except IN-A5760, were found immediately after the application. IN-QEK31 and IN-F4106 accounted for the largest proportion of the metabolites observed. With the exception of IN-VM862 the remaining components generally did not exceed 5 percent. The profiles of soil dissipation of fluazaindolizine at different locations in Europe and North America are shown in Figure 29.

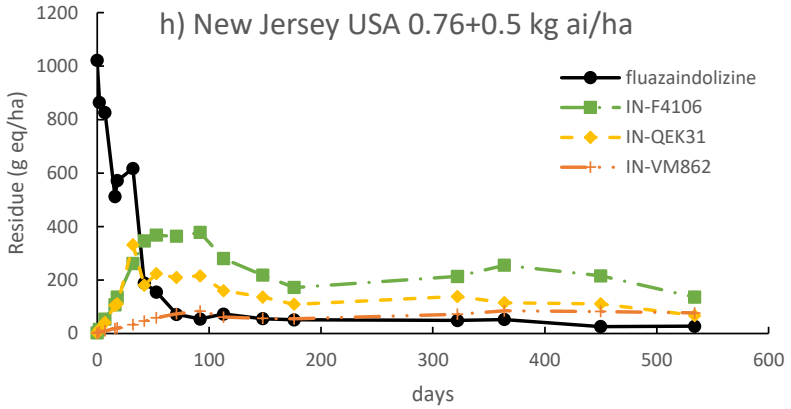


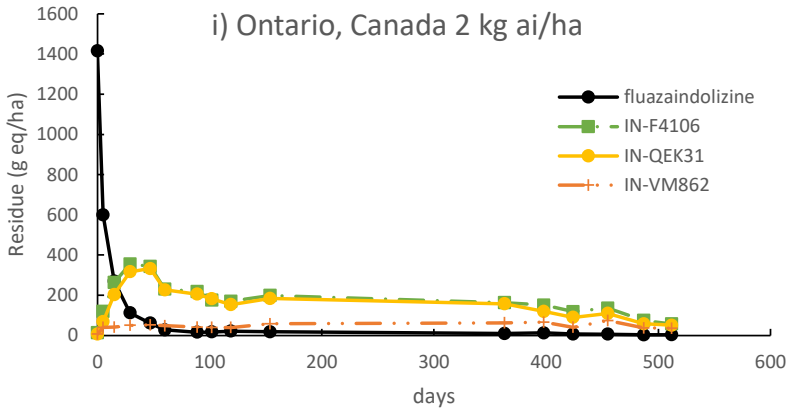

d) Nambsheim, France 1 kg ai/ha
Residue (g eq/ha)
days
fluazaindolizine
IN-F4106
IN-QEK31
IN-VM862

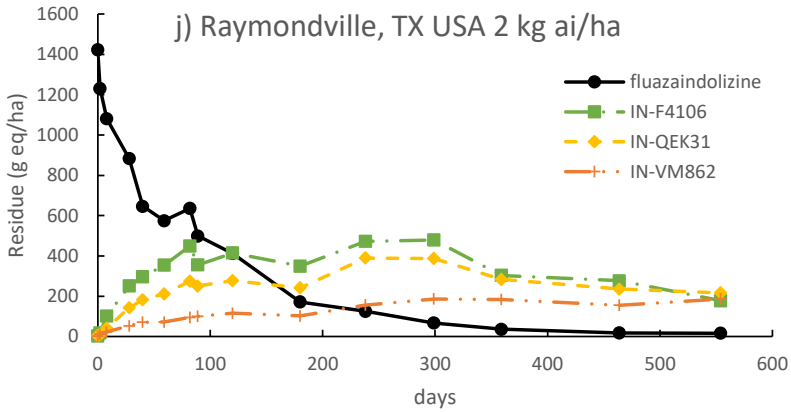

e) Woodland, CA USA 2×2 kg ai/ha
Residue (g eq/ha)
days
fluazaindolizine
IN-F4106
IN-QEK31
IN-VM862

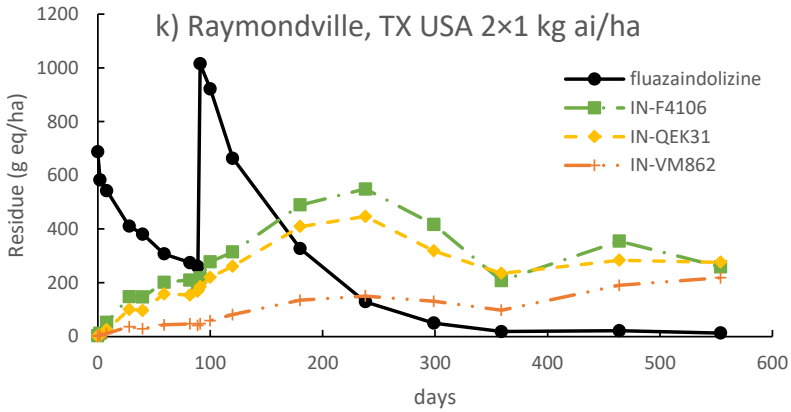

f) Porterville, CA USA 2 kg ai/ha
Residue (g eq/ha)
days
fluazaindolizine
IN-F4106
IN-QEK31
IN-VM862


g) Porterville, CA USA 2×1 kg ai/ha
Residue (g eq/ha)
days
fluazaindolizine
IN-F4106
IN-QEK31
IN-VM862


h) New Jersey USA 0.76+0.5 kg ai/ha
Residue (g eq/ha)
days
fluazaindolizine
IN-F4106
IN-QEK31
IN-VM862


i) Ontario, Canada 2 kg ai/ha
Residue (g eq/ha)
days
fluazaindolizine
IN-F4106
IN-QEK31
IN-VM862


j) Raymondville, TX USA 2 kg ai/ha
Residue (g eq/ha)
days
fluazaindolizine
IN-F4106
IN-QEK31
IN-VM862


k) Raymondville, TX USA 2×1 kg ai/ha
Residue (g eq/ha)
days
fluazaindolizine
IN-F4106
IN-QEK31
IN-VM862

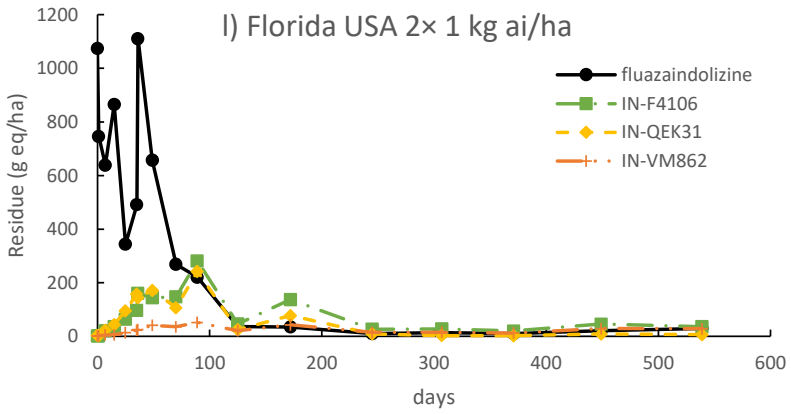


Figure 29 Field soil dissipation of fluazaindolizine at different locations in Europe and North America

Fluazaindolizine DT_{50} ranged from 5.0–171 days and DT_{90} estimates varied from 61.6 to 568 days (Table 94).

Table 94 Summary of fluazaindolizine field dissipation data[a]

System	Application kg ai/ha (interval)	DT_{50} (days)	DT_{90} (days)	Model	χ^2	r^2	Ref.
Lleida, Spain clay	1.0	2.86 (k1) 123 (k2) 9.56 (overall)	275	DFOP	9.74	0.9549	Doig 2016 DuPont 36978
Thessaloniki, Greece loam	1.0	0.802 (k1) 105 (k2) 38.4 (overall)	282	DFOP	14	0.9294	Doig 2016 DuPont 36979
Lombardian, Italy loam	1.0	5.69	67.1	FOMC	8.18	0.9832	Doig 2016 DuPont 36980
Nambsheim, France loam	1.0	25.5 (k1) 356 (k2) 32.8 (overall)	276	DFOP	12	0.9213	Doig 2016 DuPont 36981
Woodland CA United States, sandy loam	2×2.0 (90 d)	171	568	SFO	17.8	0.7732	Shepard 2016 DuPont 40812
Porterville CA United States, sandy loam Tx 02	2.0	16 (45.4)	531 (151)	FOMC (SFO)	18.6 (30.2)	0.8654 (0.7869)	Theil 2016 DuPont-36687
Porterville CA United States, sandy loam Tx 03	2×1.0 (90 d)	68 (after 2nd appl)	226	SFO	8.53	0.9588	Theil 2016 DuPont-36687
Frenchtown NJ United States, loam	0.76, 0.5 (16 d)	20.2 (after 1st and 2nd appl)	67.0	SFO		0.942	Theil 2016 DuPont-36688
Branchton, Ontario, Canada loam	2.0	5.01	16.6	SFO	22.8	0.9541	Shepard 2017 DuPont-40811
Raymondville TX United States , sandy clay loam, Tx 2	2.0	54 (63)	255 (210)	FOMC (SFO)	11.4 (12.3)	0.9406 (0.9354)	Theil 2016 DuPont 36689
Raymondville TX United States , sandy clay loam, Tx 3	2×1.0 (90 d)	51 (after 2nd appl)	169	SFO	3.47	0.9955	Theil 2016 DuPont 36689
Oviedo, FL United	2×1.0 (35 d)	18.5 (after 2nd	61.6	SFO		0.9021	DuPont-36690

System	Application kg ai/ha (interval)	DT_{50} (days)	DT_{90} (days)	Model	χ^2	r^2	Ref.
States, sand		appl)					

Notes:

[a] The numbers in parentheses are results, where different, of repeat analysis just prior to acid hydrolysis.

Environmental fate in water

Hydrolysis

Fluazaindolizine hydrolysis

Anand (2013 DuPont-35131) studied the hydrolysis of fluazaindolizine at 50°C and at pH 4, 7 and 9. The study duration was 30 days. Fluazaindolizine was stable with DT_{50} estimated as >429 days.

Based on the results of this study, hydrolysis is not expected to be a route of degradation of fluazaindolizine in the environment.

IN-F4106 hydrolysis

Manikandan (2015 DuPont-42585) studied the hydrolysis of ^{14}C-IN-F4106 at 50 °C for 5 days and at pH 4, 7 and 9. IN-F4016 was essetially stable (<6 percent degradtation) with an estimated DT_{50} at 25 °C of >1 year.

Based on the results of this study, hydrolysis is not expected to be a route of degradation of IN-F4106 in the environment.

IN-QEK31 hydrolysis

Yogeesha (2015 DuPont-40399) studied the hydrolysis of ^{14}C-IN-QEK31 at 50 °C for 5 days and at pH 4, 7 and 9. The hydrolysis of IN-QEK31 at 50 $\pm$ 0.5 °C after 5 days of incubation was <3 percent in pH 4, 7 and 9 buffer solutions (DT_{50} at 25 °C >1 year).

Based on the results of this study, hydrolysis is not expected to be a route of degradation of IN-QEK31 in the environment.

Photochemical degradation of fluazaindolizine and its photolysis products

Bell and Jewkes (2015 DuPont-37450) studied the aqueous photolysis of fluazaindolizine in sterile buffers and natural waters. Sterile pH 4 and 9 buffers and sterile natural water containing [Ph-^{14}C] or [IP-5,8a-14C] at a nominal concentration of 2 μg/mL were continuously irradiated using a xenon arc lamp for 10 to 15 days. Mass balance was satisfactory during the first 3-4 days of irradiation in all cases and for all pH 9 phenyl incubates. However, a decline in mass balance at the final timepoint was observed for pH 4 and natural water samples, accounting for recovery of 72.91 and 86.46 percent AR, respectively. In all of the test systems, no significant quantity of radioactivity was detected in the volatile trapping agents.

[^{14}C]fluazaindolizine degraded extensively and quite rapidly due to light in all irradiation experiments, such that the parent compound degraded to <10 percent AR within *ca.* 5 days.

In the pH 4 [Ph-^{14}C]fluazaindolizine samples, photodegradation products were identified as 2-chloro-5-methoxy benzene sulfonic acid, IN-UHC58, IN-UGA26, IN-F4106, IN-UGA22, IN-URA40 and fluazaindolizine hydroxylated in the phenyl ring, detected in proportions ranging from *ca.* <1-24 percent at various intervals. In addition, there was a cluster of polar unidentified components which consisted of

multiple degradation products, as well as minor components throughout the column elution. The minor components did not individually exceed 5 percent AR (except at one timepoint; 8.7 percent AR at Day 12). The maximum combined radioactivity attributed to all unknown minor degradation products at any one sampling interval was 20.5 percent AR at Day 12. The only degradates which exceeded 10 percent AR at multiple sampling intervals were IN-UGA22, IN-F4106 and 2-chloro-5-methoxy benzene sulfonic acid.

In pH 4 buffer samples treated with [IP-5,8a-^{14}C]-fluazaindolizine, the test item also showed degradation similar to that observed in [Ph-^{14}C]-fluazaindolizine, except for the observation of additional label specific degradates. Identified degradates included, IN-R6P21, IN-UGA22, IN-URA40 and hydroxylated fluazaindolizine, IN-UHC58, IN-UGA26, IN-VM862, hydroxylated IN-RYC33 and IN-RYC33. In addition, there was a cluster of polar unidentified components, as well as minor components throughout the column elution. The minor components did not individually exceed 5 percent. The maximum combined radioactivity attributed to the multiple unknown minor degradation products at any one sampling interval was 13.7 percent AR at Day 12.

Photodegradation at pH 9 and natural water samples was equally facile and showed little differences from the behaviour noted in pH 4 buffered solutions. More than 90 percent of the parent compound degraded by Day 4 or shortly afterwards. The degradation products found in the pH 4 buffers were also noted in pH 9 buffer and in natural water, except IN-QEK31 was also detected in pH 9 and natural water samples.

Fluazaindolizine remained unchanged in the pH 4 buffer, pH 9 buffer and natural water dark control samples. DT_{50} and DT_{90} are shown in Table 95.

Table 95 Calculated kinetics parameters for fluazaindolizine under constant irradiation of simulated sunlight

System	Kinetic model	k (days^{-1})	r^2	DT_{50} (days)	DT_{90} (days)
Irradiated pH 4 buffer	SFO	0.6281	0.999	1.1	3.7
Irradiated pH 9 buffer	SFO	0.5608	0.986	1.2	4.1
Irradiated natural water	SFO	0.4327	0.993	1.6	5.3

Fluazaindolizine is susceptible to rapid photolysis in sterile pH 4 and 9 buffer and natural water. The calculated quantum yield for the actinometer was 3.972×10^{-3} molecules degraded/photon. The quantum yield for [^{14}C]fluazaindolizine was calculated as 5.340×10^{-4} molecules degraded/photon.

Lin *et al*. (2019, Chemosphere 214:543-552) studied the photodegradation of fluazaindolizine in water and the effects of solution pH, humic acids (HA), nitrates (NO_3^-) and Fe(III) ions on photolysis of fluazaindolizine were studied. The results indicated that pH did not significantly affect its photodegradation. At low concentration (up to 5 mg/L), HA slightly facilitated the photodegradation of fluazaindolizine, while at high concentration (10–20 mg/L), HA inhibited its photodegradation. The presence of NO_3^- (0-10 mg/L) and Fe(III) (0–5 mg/L) noticeably accelerated the photodegradation of fluazaindolizine. Eleven transformation products were isolated and identified by LC-TOF-MS. The predominant photoproduct came from ring opening of imidazole-ring and dechlorination. Other transformation products resulted from a series of photochemical reactions involving hydroxyl substitution, ring-opening, cleavage, oxidation, and decarboxylation. The half-life in water at pH 4, 7.2 and 9.0 were 20.2, 19.0 and 18.6 hours, respectively.

A proposed patway for fluazaindolizine photolysis in water in shown in Figure 30.

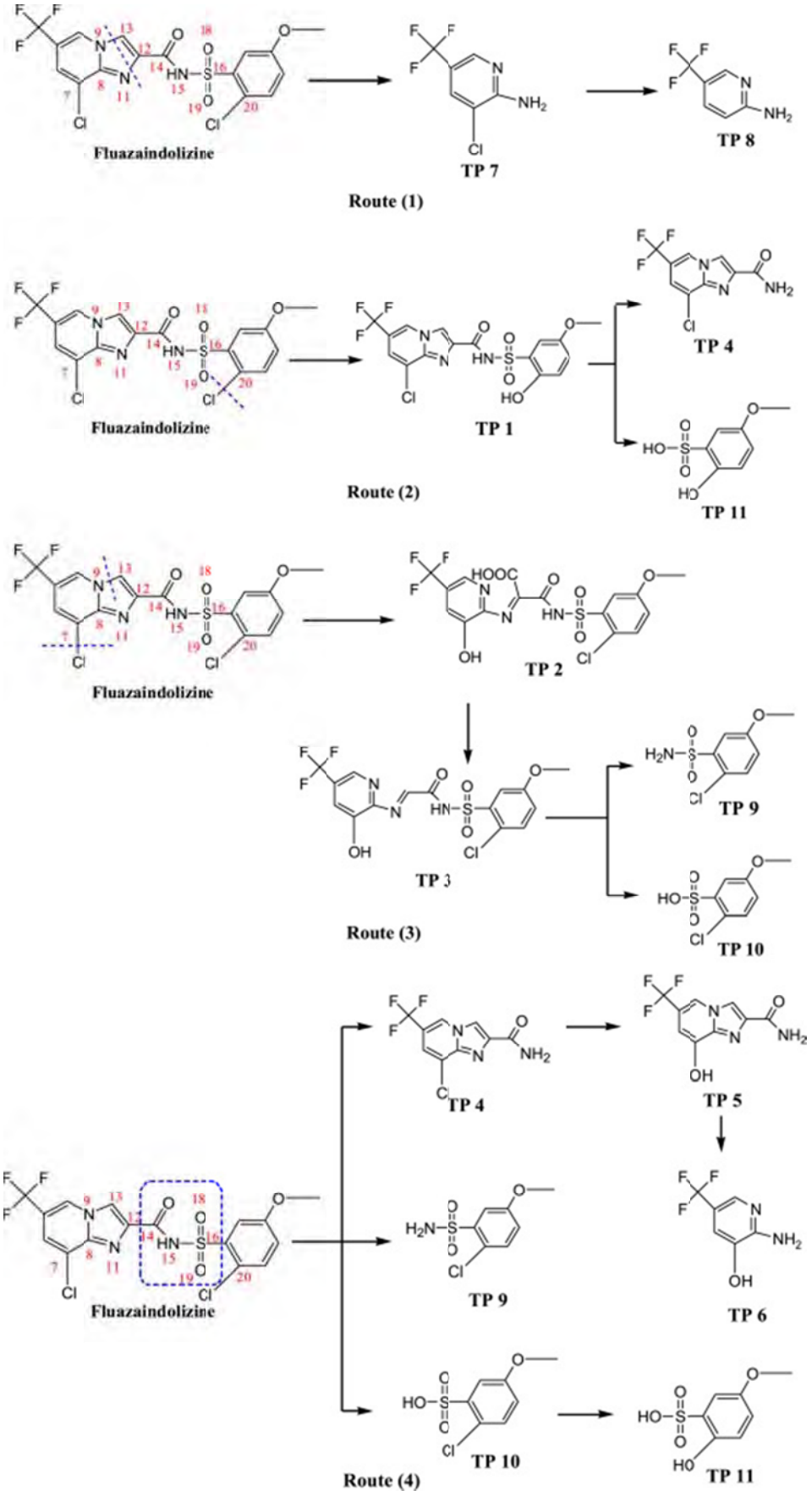
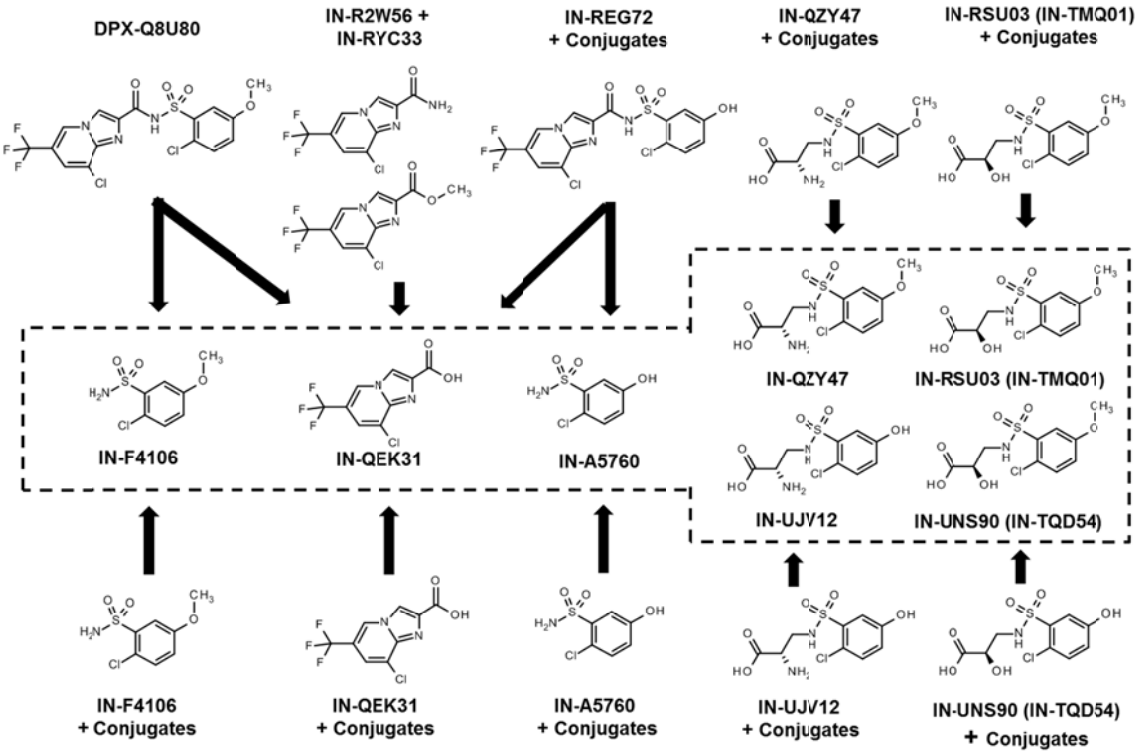


Figure 30 Proposed photolysis of fluazaindolizine in water

METHODS OF RESIDUE ANALYSIS

Plant matrices

The metabolism of fluazaindolizine in crops results in a complex mixture of metabolites that arise from hydrolysis of the amide bond resulting in cleavage of the molecule to produce IN-F4106 and IN-QEK31 which are subject to further metabolism including conjugation. A number of minor metabolites are formed by hydrolysis at the sulphonamide bond to form IN-RYC33, and O-demethylation to form IN-REG72 which may also form conjugates. Methods have been developed to measure fluazaindolizine as well as major metabolites. The methods involve analysis without (fluazaindolizine and free IN-F4106, IN-QEK31, IN-QZY47, IN-R2W56, IN-REG72, IN-TEQ01, IN-RYC33) and also with hydrolysis of conjugates (IN-AS5760, IN-F4106, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12, IN-UNS90). Hydrolysis of fluazaindolizine results in cleavage of the amide bond and formation of IN-F4106 and IN-QEK31.



Residues are reported in terms of the analytes but may be converted to parent equivalents using molecular weight conversion factors: 2.26 for IN-A5760, 2.11 for IN-F4106, 1.77 for IN-QEK31, 1.52 for INQZY47, 1.51 for IN-TMQ01, 1.59 for IN-UJV12 and 1.58 for IN-UNS90.

Method DuPont-47054 utilises different hydrolysis conditions to allow for higher throughput of samples as well as modified clean-up, and recovery data was supplied in support of this method. A summary of the LC-MS/MS methods to analyse fluazaindolizine and metabolites in plants is shown in Tables 96.

Table 96 Overview of the methods for determination of fluazaindolizine in crops

Method	DuPont-33681 (Revision 3)
Analytes	Pre-hydrolysis Fluazaindolizine, IN-F4106, IN-QEK31, IN-QZY47, IN-R2W56, IN-REG72, IN-RSU03, IN-RYC33 Post-hydrolysis

	IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-RSU03, IN-UJV12, IN-UNS90
Matrix	Watery, acidic, oily, and dry crops
Extraction	Part 1 (pre-hydrolysis) Fluazaindolizine and metabolites were extracted from watery, acid, and oily matrices with methanol/water (70/30) and centrifuged to separate supernatant and solids. For oily crops, the extract is partitioned with hexane and the hexane discarded. For all crops, an aliquot of the extract is diluted with 0.1 M aqueous formic acid and filtered for analysis by LC-MS/MS. . Dry crop matrices are extracted with 10 mM ammonium formate:methanol (1:2 v:v) with the ammonium formate added first and allowed to sit for 5 minutes before adding the methanol, then re-extracted 2× with methanol:10 mM ammonium formate (70:30 v:v), centrifuged and the combined extracts concentrated under nitrogen and diluted with water:methanol ((75:25 v:v containing 0.1 percent formic acid) for analysis by LC-MS/MS. Part 2 (hydrolysis) Watery, acidic, oily crops: An aliquot of the combined extracts concentrated to <0.5 mL under N_2, mixed with 1.2 M HCl and heated at 80°C for 16 hours. Dry crops: An aliquot of the part 1 extract is concentrated to <0.5 mL under N_2, mixed with 2 M HCl and heated at 80°C for 16 hours.
Column/sorbent	Part 1 (pre-hydrolysis) Watery, acidic, oily crops: Optional. The extract is diluted with 0.1 M aqueous formic acid and loaded onto a Bond-Elut LRC-CN-U SPE cartridge, the eluate collected and the cartridge rinsed with 0.1 M aqueous formic acid methanol (50:50 v:v). The combined eluate/reinstate is diluted with 0.1 M Aqueous formic acid for analysis by LC-MS/MS Part 2 (hydrolysis) Watery, acidic, oily, dry crops: The hydrolysed extract is applied to a Bond-Elut SAX SPE cartridge, the eluate collected and the cartridge rinsed with acetate buffer. The combined eluate, rinsate is adjusted to volume (acetate buffer for watery and acidic crops or methanol:water 25:75 v:v, containing 0.1 percent formic acid for oily and dry crops) for analysis by LC-MS/MS.
Column	Phenomenex Kinetex biphenyl, 4.6×100 mm, 2.6 micron particle size
Mobile phase	A: water methanol (75:25), 0.01 percent formic acid in methanol
Quantitative detection	ESI in positive mode for: IN-QEK31, IN-R2W56, IN-RYC33, and negative mode for all other analytes fluazaindolizine: 466 → 157 (quantification) and 466 → 142 (confirmation) IN-AS5760: 206 → 142 (quantification) and 206 → 122 (confirmation) IN-F4106 : 220 → 156 (quantification) and 220 → 141 (confirmation) IN-QEK31: 265 → 247, 265 → 192, (quantification) and 265 → 219, 265 → 184 (confirmation) IN-QZY47: 307 → 220 (quantification) and 307 → 156, 307 → 78 (confirmation) IN-R2W56: 279 → 247 (quantification) and 279 → 219 (confirmation) IN-REG72: 452 → 143 (quantification) and 452 → 123 (confirmation) IN-RSU03: 308 → 220 (quantification) and 308 → 156, 308 → 78 (confirmation) IN-RYC33: 264 → 157 (quantification) and 264 → 219, 264 → 184 (confirmation) IN-UJV12: 293 → 206 (quantification), 293 → 142, 293 → 78 (confirmation) IN-UNS90: 294 → 206 (quantification), 294 → 142, 294 → 78 (confirmation)
LOQ	0.01 mg/kg per analyte before adjusting for parent equivalents (LOD 0.003 mg/kg)
Whole method linearity (r^2)	0.02-10 ng/mL $r^2 \geq 0.9951$ (1/*x* weighting)

Method	Method DuPont-47054
Analytes	Fluazaindolizine, IN-A5760, IN-F4106, IN-QEK31, IN-RYC33, IN-QZY47, IN-TMQ01 (IN-RSU03), IN-R2W56, IN-UJV12, and IN-TQD54 (IN-UNS90)
Extraction	Part 1: same as DuPont 33681. Part 2: Fluazaindolizine and metabolites were extracted from watery, acid, oily and dry matrices with methanol/water (70/30 v/v) and centrifuged to separate supernatant and solids. The extracts are reduced in volume under a flow of N_2 at 60 °C and 4 M HCl added and heated at 100 °C for 1.25 hours.
Column	Part 1: same as DuPont 33681. Part 2: For watery, acid, oily, an aliquot of the hydrolysed extract is diluted and applied to an Oasis® MCX SPE cartridge to isolate IN-QEK31, IN-TMQ01 (IN-RUS03) and IN-A5760. A separate aliquot was applied to an ENVI™ - Carb SPE cartridge to isolate IN-UJV12, INQZY47, IN-TQD54 (IN-UNS90) and IN-F4106. For dry crops, an aliquot

Method	Method DuPont-47054
	of the hydrolysed extract is diluted and applied to an Oasis® MCX SPE cartridge to isolate IN-QEK31, IN-TMQ01 (IN-RUS03) and IN-A5760. A separate aliquot was applied to an ENVI™ -Carb followed by an Oasis®-MCX SPE cartridge to isolate IN-UJV12, INQZY47, IN-TQD54 (IN-UNS90) and IN-F4106.
Eluent	Part 1: same as DuPont 33681. Part 2: Watery, acid, oily matrices: Oasis® MCX - 1 percent formic acid in methanol water (60:40 v:v). The eluate is concentrated under a stream of N_2 and bought to volume with 15 mM aqueous citric acid for LC-MS/MS. ENVI-Carb – 15 mM citric acid in methanol. The eluate is concentrated under a stream of N_2 and bought to volume with water for LC-MS/MS. Dry matrices: Oasis® MCX - 1 percent formic acid in methanol water (60:40 v:v). The eluate is concentrated under a stream of N_2 and bought to volume with 15 mM aqueous citric acid for LC-MS/MS. ENVI-Carb – 15 mM citric acid in methanol, the eluate added to an Oasis(r) MCX cartridge and eluted with 5 percent ammonium hydroxide in methanol. Water is added prior to concentration under a stream of N_2, 150 mM aqueous citric acid is added and the extract bought to volume with water for LC-MS/MS.
Quantitation	ESI in positive mode for: IN-QEK31, IN-R2W56, IN-RYC33, and negative mode for all other analytes fluazaindolizine: 466 → 157 (quantification) and 466 → 142 (confirmation) IN-AS5760: 206 → 142 (quantification) and 206 → 122 (confirmation) IN-F4106 : 220 → 156 (quantification) and 220 → 141 (confirmation) IN-QEK31: 265 → 247, 265 → 192, 265 → 184 (quantification/confirmation) and 265 → 219 (confirmation) IN-QZY47: 307 → 220, 307 → 156 (quantification/confirmation) and 307 → 78 (confirmation) IN-R2W56: 279 → 247 (quantification) and 279 → 219 (confirmation) IN-REG72: 452 → 143 (quantification) and 452 → 123 (confirmation) IN-RSU03: 308 → 220, 308 → 156 (quantification/confirmation) and 308 → 78 (confirmation) IN-RYC33: 264 → 157 (quantification) and 264 → 219, 264 → 184 (confirmation) IN-TMQ01: 308 → 220 (quantification) and 466 → 142 (confirmation) IN-UJV12: 293 → 206 (quantification), 293 → 142 (quantification/confirmation), 293 → 78 (confirmation) IN-UNS90: 294 → 206, 294 → 142, 294 → 78 (quantification/confirmation)
LOQ	0.01 mg/kg per analyte before adjusting for parent equivalents (LOD 0.003 mg/kg)
Whole method linearity (r^2)	0.1-50 ng/mL for IN-QEK31, IN-A5760 and IN-TMQ01 (IN RSU03) and 0.2-50 ng/mL for IN-F4106, IN UJV12, IN QZY47 and IN-TQD54 (IN-UNS90) $r^2 \geq 0.9995$
Method	**DuPont 39990**
Analytes	Fluazaindolizine, IN-A5760, IN-F4106, IN-QEK31, IN-RYC33, IN-QZY47, IN-TMQ01 (IN-RSU03), IN-R2W56, IN-UJV12, and IN-TQD54 (IN-UNS90).
Matrix	AP.22468A: tomato, wheat grain, grapes, dried bean AP.22468B: wheat straw, soya bean seed
Extraction	AP.22468A: Samples are extracted with methanol:water 70:30 Part 1: Aliquots of extracts are diluted with 0.01 percent formic acid and analysed by LC-MS/MS Part 2: Aliquots of extracts are evaporated and acidified with 1.2M HCl at 80 °C overnight. AP.22468B: Samples are extracted with methanol:water (10 mM ammonium formate) 70:30 Part 1: Aliquots are diluted with 0.01 percent formic acid in 75:25 methanol:water and analysed by LC-MS/MS Part 2: Aliquots are evaporated and acidified with 2M HCl at 80 °C overnight.
Column	AP.22468A and B: Part 2: Post-hydrolysis samples are applied to an SAX SPE cartridge.
LOQ	0.01 mg/kg per analyte before adjusting for parent equivalents (LOD 0.003 mg/kg)

Method DuPont 33861 (Klems 2017 DuPont 33861, Revision No. 3, Rebstock 2021 DuPont-37832 Revision No. 1)

The residue method for the determination of fluazaindolizine residues in tomatoes, dried peas, soya beans, corn stover, oranges, grapes, wheat straw, and wheat grain involves simple extraction, clean-up,

and analytical determination by LC-MS/MS detection. Samples were also analysed after hydrolysis to determine if conjugated residue was present.

The recoveries of IN-QEK31, IN-F4106, IN-A5760, IN-TQD54 (IN-UNS90), IN-TMQ01 (IN-RSU03), IN-QZY47 and IN-UJV12 from crop samples fortified at 0.01 (LOQ) and 0.10 mg/kg and carried through the subsequent hydrolysis and clean-up procedures support the satisfactory performance of this method.

The solvent mixture used for extracting fluazaindolizine and its metabolites from crop samples (70/30 methanol/water) are based on the same solvent mixture/sample ratio used in the primary crop metabolism studies conducted with radiolabelled fluazaindolizine (DuPont-34947, DuPont-34948, DuPont-34946, DuPont-43220 and DuPont-41849). The extraction efficiency data ranged from 72.2 to 93.9 percent and averaged 84.6 percent of the total radioactivity in the various commodities.

The fortification data reported in the method for determination of residues in grape (high water), soya bean seed (high oil/high protein), tomato (high acid) and wheat straw (dry) are summarised in Tables 97 to 100. The average recovery were within the range 70 to 120 percent (pre-hydrolysis 72–113 percent, percent RSD 1.3–15 percent; post-hydrolysis 77–114 percent, percent RSD 1.1-13 percent).

Recovery and repeatability data for the determination of IN-QEK31, IN-F4106, IN-A5760, IN-TQD54 (IN-UNS90), IN-TMQ01 (IN-RSU03), IN-QZY47 and IN-UJV12 in crops using method DuPont-47054 are presented in Tables 101 and 103. Fluazaindolizine and its metabolites were extracted from plant matrices using a mixture of methanol and water. The final determination of fluazaindolizine and its metabolites was performed by LC-MS/MS.

The LOQ was defined by the lowest fortification level successfully tested, which was 0.01 mg/kg in grape (high water), soya bean seed (high oil/high protein), tomato (high acid) and wheat straw (dry). Good linearity was observed in the range of 0.2-10 ng/mL (nominal) for fluazaindolizine and its metabolites IN-REG72, IN-RYC33, IN-R2W56, IN-QEK31, IN-A5760, IN-F4106, IN-UJV12, IN-QZY47, IN-TQD54 (IN-UNS90) and IN-TMQ01 (IN-RSU03). These ranges corresponded to residue values of approximately 0.004-0.2 mg/kg for watery, oily, and acidic crops and 0.006–0.3 mg/kg for dry crops. At least five-point standard curves were prepared utilizing matrix-matched standards with r2 values ≥ 0.9989 (pre-hydrolysis) and ≥ 0.9954 (post-hydolysis).

Method DuPont-47054 is suitable to determine residues of fluazaindolizine and metabolites in the matrices investigated. The specificity of the method is provided using a mass selective detector. The method was successfully validated for fluazaindolizine metabolites.

Table 97 Recovery data for the analytical method for the determination of fluazaindolizine residues in food of plant origin (Klems, 2017 DuPont-33861, Revision No. 3)

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
		Fluazaindolizine: 466 → 157 m/z		IN-R2W56: 279 → 247 m/z	
Tomatoes	0.01	82	6.2	98	2.0
(pre-hydrolysis)	0.1	94	5.1	101	2.1
Soya beans	0.01	86	4.3	109	1.7
(pre-hydrolysis)	0.1	83	2.7	104	2.2
Grapes	0.01	103	3.3	107	3.0
(pre-hydrolysis)	0.1	113	8.6	101	5.1
Wheat Straw	0.01	94	8.8	91	4.1
(pre-hydrolysis)	0.1	92	7.4	92	7.6
		IN-REG72: 452 → 143 m/z		IN-RYC33: 264 → 157 m/z	
Tomatoes	0.01	95	9.6	97	2.9
(pre-hydrolysis)	0.1	104	3.3	103	1.5
Soya beans	0.01	76	5.5	113	3.1

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
(pre-hydrolysis)	0.1	72	2.0	110	2.7
Grapes	0.01	102	2.8	106	4.5
(pre-hydrolysis)	0.1	113	8.0	97	4.6
Wheat Straw	0.01	104	4.1	88	8.5
(pre-hydrolysis)	0.1	101	3.3	87	6.6
		IN-F4106: 220 → 156 m/z		IN-QEK31: 265 → 184 m/z	
Tomatoes	0.01	95	8.0	84	8.4
(pre-hydrolysis)	0.1	100	1.8	104	2.5
Tomatoes	0.01	92	9.8	83	4.4
(post-hydrolysis)	0.1	87	4.7	76	5.2
Soya beans	0.01	109	4.2	106	7.7
(pre-hydrolysis)	0.1	108	1.3	99	2.6
Soya beans	0.01	98	5.0	82	1.5
(post-hydrolysis)	0.1	93	4.7	84	2.5
Grapes	0.01	106	3.3	108	4.3
(pre-hydrolysis)	0.1	103	2.9	111	5.0
Grapes	0.01	92	13	90	4.7
(post-hydrolysis)	0.1	90	2.8	92	2.4
Wheat Straw	0.01	95	4.9	91	7.6
(pre-hydrolysis)	0.1	91	6.9	90	7.6
Wheat Straw	0.01	113	13	103	12
(post-hydrolysis)	0.1	87	3.8	90	4.1
		IN-QZY47: 307 → 220 m/z		IN-TMQ01 (IN-RSU03)[C]: 308 → 220 m/z	
Tomatoes	0.01	90	2.4	88	3.2
(pre-hydrolysis)	0.1	104	3.1	106	0.8
Tomatoes	0.01	103	3.0	83	5.4
(post-hydrolysis)	0.1	101	2.7	93	5.2
Soya beans	0.01	104	2.5	104	2.5
(pre-hydrolysis)	0.1	108	2	105	2
Soya beans	0.01	103	5.5	91	3.3
(post-hydrolysis)	0.1	109	2.7	89	3.7
Grapes	0.01	105	5.4	105	2.1
(pre-hydrolysis)	0.1	104	4.5	104	2.9
Grapes[A]	0.01	109	7.4	99	2.5
(post-hydrolysis)	0.1	114	2.3	102	3.9
Wheat Straw	0.01	84	15	90	4.2
(pre-hydrolysis)	0.1	89	12	93	12
Wheat Straw	0.01	106	5.9	94	5.8
(post-hydrolysis)	0.1	114	2.9	102	3.5
		IN-TQD54 (IN-UNS90)[D]: 294 m/z → 206 m/z		IN-UJV12: 293 → 206 m/z	
Tomatoes	0.01	77	6.0	85	12.0
(post-hydrolysis)	0.1	91	6.6	88	4.7
Soya beans	0.01	88	5.1	89	1.3
(post-hydrolysis)	0.1	98	6.9	93	2.8
Grapes[A]	0.01	105	6.6	93	12.0
(post-hydrolysis)	0.1	105	6.5	108	3.5
Wheat Straw[B]	0.01	91	6.0	80	1.9
(post-hydrolysis)	0.1	100	3.9	99	1.4
		IN-A5760: 206 → 142 m/z			
Tomatoes	0.01	87	4.1		
(post-hydrolysis)	0.1	90	5.0		
Soya beans	0.01	92	6.7		
(post-hydrolysis)	0.1	90	4.7		
Grapes	0.01	99	5.2		
(post-hydrolysis)	0.1	105	2.7		

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
Wheat Straw [B]	0.01	88	4.3		
(post-hydrolysis)	0.1	92	1.1		

Notes:

[A] Post-hydrolysis grapes: 307 m/z 156 m/z used for quantitation of IN-QZY47, 308 m/z 156 m/z used for quantitation of IN-TMQ01 (IN-RSU03), 294 m/z 142 m/z used for quantitation of IN-TQD54 (IN-UNS90).

[B] Post-hydrolysis wheat straw: 294 m/z 78 m/z used to quantify IN-TQD54 (IN-UNS90), 293 m/z 142 m/z used to quantify IN-UJV12.

CIN-TQD54 is the R enantiomer contained in IN-UNS90 (racemic mixture).

DIN-TMQ01 is the R enantiomer contained in IN-RSU03 (racemic mixture).

The method of Klems (2017) was also validated by Rebstock (2021). The recovery data obtained are summarised in Table 98. The successful validation demonstrates the reproducibility of this method.

The fortification data reported in the method for determination of residues in tomato (high acid/high water), wheat grain (high starch), oranges (high acid), corn stover (dry), soya bean seed (high oil/high protein) and dried peas (high protein) are summarised in Table 98. The average recovery were generally within the range 70 to 120 percent (pre-hydrolysis 69-102 percent (69 percent was for confirmation ion for IN-QZY47 in field stover at the LOQ, percentRSD ≤17 percent except one percentRSD 23 percent (confirmation ion for IN-F4106 at LOQ in dry pea seed); post-hydrolysis 73-119 percent, percentRSD generally ≤19 percent except two RSDs of 21 percent and 27 percent occurred for the quantitation ion transition for IN-F4106 in field corn stover and soya bean seed, respectively, each at the LOQ. For field corn stover, the high RSD is due to 1 high recovery (125 percent) out of five samples. For soya bean seed, the high RSD is due to two recoveries of 57 percent and 123 percent, out of 5 samples.

Recovery and repeatability data for the determination of IN-QEK31, IN-F4106, IN-A5760, IN-TQD54 (IN-UNS90), IN-TMQ01 (IN-RSU03), IN-QZY47 and IN-UJV12 in crops using method DuPont-47054 are presented in Table 104 and 105. Fluazaindolizine and its metabolites were extracted from plant matrices using a mixture of methanol and water. The final determination of fluazaindolizine and its metabolites was performed by LC-MS/MS.

The LOQ was defined by the lowest fortification level successfully tested, which was 0.01 mg/kg in tomato (high acid/high water), wheat grain (high starch), oranges (high acid), corn stover (dry), soya bean seed (high oil/high protein) and dried peas (high protein). Good linearity was observed in the range of 0.2-10 ng/mL (nominal) for fluazaindolizine and its metabolites IN-REG72, IN-RYC33, IN-R2W56, IN-QEK31, IN-A5760, IN-F4106, IN-UJV12, IN-QZY47, IN-TQD54 (IN-UNS90) and IN-TMQ01 (IN-RSU03). These ranges corresponded to residue values of approximately 0.004-0.2 mg/kg for watery, oily, and acidic crops and 0.006-0.3 mg/kg for dry crops. At least five-point standard curves were prepared utilizing matrix-matched standards with r^2 values ≥ 0.9989 (pre-hydrolysis) and ≥ 0.9954 (post-hydolysis).

The stability of pre- and post-hydrolysis extracts from matrices fortified at 0.01and 0.1 mg/kg, as well as matrix-matched calibration standards was investigated for three matrices (grape, spinach, wheat straw) following 14–15 days storage at 5 °C. Mean recoveries of fluazaindolizine, IN-F4106, IN-QEK31, IN-QZY47, IN-R2W56, IN-REG72, IN-RSU03, and IN-RYC33 pre-hydrolysis were 96-106 percent with RSDs ≤8 percent. Mean post-hydrolysis recoveries of IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 were 96-113 percent with RSDs of 1–14 percent. The matrix-matched standards showed linearity with r^2 values of ≥0.999, and RSDs for the response factors of ≤18 percent.

Recovery data to validate the optional SPA cleanup step were provided for tomato, reflecting one sample fortified with each analyte at the LOQ and 10×LOQ, which indicated adequate recovery for each of the pre- and post-hydrolysis analytes.

Table 98 Recovery data (n=5) for the analytical method for the determination of fluazaindolizine residues in food of plant origin (Rebstock, 2021 DuPont-37832, Revision No. 1)

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	% RSD
		Fluazaindolizine: 466 → 157 m/z		IN-R2W56: 279 → 247 m/z	
Tomatoes	0.01	88	3	91	2
(pre-hydrolysis)	0.1	92	4	91	1
Wheat Grain	0.01	84	3	99	7
(pre-hydrolysis)	0.1	88	5	102	13
Oranges	0.01	83	6	89	5
(pre-hydrolysis)	0.1	88	1	94	2
Corn Stover	0.01	85	3	83	5
(pre-hydrolysis)	0.1	86	4	89	3
Soya bean Seed	0.01	83	3	87	3
(pre-hydrolysis)	0.1	82	2	92	1
Dried Peas	0.01	94	3	84	2
(pre-hydrolysis)	0.1	94	1	88	3
		IN-REG72: 452 → 123 m/z		IN-RYC33: 264 → 157 m/z	
Tomatoes	0.01	91	2	91	2
(pre-hydrolysis)	0.1	93	3	91	2
Wheat Grain	0.01	84	5	91	2
(pre-hydrolysis)	0.1	81	6	92	2
Oranges	0.01	87	3	89	3
(pre-hydrolysis)	0.1	91	1	92	2
Corn Stover	0.01	86	1	88	3
(pre-hydrolysis)	0.1	87	3	90	3
Soya bean Seed	0.01	78	3	90	1
(pre-hydrolysis)	0.1	78	3	90	2
Dried Peas	0.01	95	2	95	4
(pre-hydrolysis)	0.1	96	1	94	1
		IN-F4106: 220 → 78 m/z		IN-QEK31: 265 → 184 m/z	
Tomatoes	0.01	93	4	93	2
(pre-hydrolysis)	0.1	93	5	91	4
Tomatoes	0.01	92	8	86	3
(post-hydrolysis)	0.1	83	6	96	2
Wheat Grain	0.01	91	7	84	4
(pre-hydrolysis)	0.1	95	5	84	3
Wheat Grain	0.01	89	16	76	8
(post-hydrolysis)	0.1	82	4	79	5
Oranges	0.01	86	6	81	2
(pre-hydrolysis)	0.1	95	4	92	3
Oranges	0.01	89	6	82	2
(post-hydrolysis)	0.1	78	5	80	2
Corn Stover	0.01	100	5	80	5
(pre-hydrolysis)	0.1	86	6	85	3
Corn Stover	0.01	95	21	83	3
(post-hydrolysis)	0.1	84	5	79	2
Soya bean Seed	0.01	93	7	91	1
(pre-hydrolysis)	0.1	90	1	92	2
Soya bean Seed	0.01	91	27	81	3
(post-hydrolysis)	0.1	91	4	84	4
Dried Peas	0.01	85	10	96	4

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	% RSD
(pre-hydrolysis)	0.1	86	2	92	2
Dried Peas[c]	0.01	88	13	81	6
(post-hydrolysis)[A]	0.1	93	2	81	1
		IN-QZY47: 307 → 220 m/z		IN-TMQ01 (IN-RSU03)[C]: 308 → 220 m/z	
Tomatoes	0.0089	92	2	91	4
(pre-hydrolysis)	0.089	88	1	92	3
Tomatoes	0.0089	106	6	94	4
(post-hydrolysis)	0.089	100	3	86	3
Wheat Grain	0.0089	83	1	87	3
(pre-hydrolysis)	0.089	87	4	92	3
Wheat Grain	0.0089	95	8	94	4
(post-hydrolysis)	0.089	85	2	86	2
Oranges	0.0089	83	3	94	4
(pre-hydrolysis)	0.089	94	2	94	2
Oranges	0.0089	80	6	85	3
(post-hydrolysis)	0.089	79	2	80	3
Corn Stover	0.0089	72	7	82	5
(pre-hydrolysis)	0.089	72	4	91	3
Corn Stover	0.0089	81	5	97	6
(post-hydrolysis)	0.089	79	3	90	3
Soya bean Seed	0.0089	90	2	89	4
(pre-hydrolysis)	0.089	88	2	90	1
Soya bean Seed	0.0089	82	3	86	5
(post-hydrolysis)	0.089	86	3	87	5
Dried Peas	0.0089	86	3	96	2
(pre-hydrolysis)	0.089	90	1	98	1
Dried Peas	0.0089	89	3	91	5
(post-hydrolysis)	0.089	94	2	93	3
		IN-TQD54 (IN-UNS90)[D]: 294 m/z → 206 m/z		IN-UJV12: 293 → 206 m/z	
Tomatoes	0.0089	103	4	97	5
(post-hydrolysis)	0.089	87	3	87	2
Wheat Grain	0.0089	89	2	89	6
(post-hydrolysis)	0.089	84	3	85	2
Oranges	0.0089	84	6	84	3
(post-hydrolysis)	0.089	83	2	82	2
Corn Stover	0.0089	83	8	86	7
(post-hydrolysis)	0.089	80	5	77	4
Soya bean Seed	0.0089	90	5	82	12
(post-hydrolysis)	0.089	89	4	85	4
Dried Peas	0.0089	87	8	92	10
(post-hydrolysis)	0.089	94	3	94	1
		IN-A5760: 206 → 122 m/z			
Tomatoes	0.01	104	6		
(post-hydrolysis)	0.1	88	1		
Wheat Grain	0.01	91	16		
(post-hydrolysis)	0.1	83	3		
Oranges	0.01	83	3		
(post-hydrolysis)	0.1	81	2		
Corn Stover[g]	0.01	100	14		
(post-hydrolysis)[B]	0.1	84	5		
Soya bean Seed[d]	0.01	99	18		
(post-hydrolysis)	0.1	84	5		
Dried Peas	0.01	119	12		
(post-hydrolysis)	0.1	97	4		

Notes:

[A] Post-hydrolysis dried peas: 220 → 156 m/z used for quantitation of IN-F4106.

[B] Post-hydrolysis corn stover and post-hydrolysis soya bean seed: 206 → 142 m/z used to quantify IN-A5760.

[C] IN-TMQ01 is the *R* enantiomer contained in IN-RSU03 (racemic mixture).

[D] IN-TQD54 is the *R* enantiomer contained in IN-UNS90 (racemic mixture).

Confirmation of results obtained by this LC-MS/MS method was performed by quantifying a separate daughter ion signal for each analyte. The recovery data obtained using the confirmatory procedure are summarised in Table 99.

Table 99 Recovery data (n=5) for the analytical method for the determination of fluazaindolizine residues in food of plant origin (Rebstock, 2021 DuPont-37832, Revision No. 1)

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
		Fluazaindolizine: 466 → 142 m/z		IN-R2W56: 279 → 219 m/z	
Tomatoes	0.01	83	8.1	98	1.3
(pre hydrolysis)	0.1	93	2.9	101	1.5
Soya beans	0.01	87	2.8	109	1.4
(pre hydrolysis)	0.1	84	3.6	103	1.8
Grapes	0.01	105	6	106	2.3
(pre hydrolysis)	0.1	112	7.1	100	5.6
Wheat straw	0.01	99	10	91	7.3
(pre hydrolysis)	0.1	90	4.8	92	6.3
		IN-REG72: 452 → 123 m/z		IN-RYC33: 264 → 219 m/z	
Tomatoes	0.01	96	4.5	96	4.5
(pre hydrolysis)	0.1	102	5.4	102	5.4
Soya beans	0.01	72	2.1	113	1.3
(pre hydrolysis)	0.1	74	0.8	109	2.3
Grapes	0.01	103	5.3	97	8
(pre hydrolysis)[A]	0.1	112	9	95	5
Wheat straw	0.01	98	6	99	6.3
(pre hydrolysis)	0.1	99	5.5	90	4.8
		IN-F4106: 220 → 141 m/z		IN-QEK31: 265 → 219 m/z	
Tomatoes	0.01	88	2.8	78	6.7
(pre hydrolysis)	0.1	101	3.8	99	1.6
Tomatoes	0.01	83	7.5	82	5.7
(post hydrolysis)	0.1	87	4.8	90	6.8
Soya beans	0.01	107	3.3	105	3.4
(pre hydrolysis)	0.1	107	1.2	100	2.1
Soya beans	0.01	108	8.6	81	5
(post hydrolysis)	0.1	104	5.8	77	2.4
Grapes	0.01	106	0.8	106	5.7
(pre hydrolysis)	0.1	105	3.9	112	6.3
Grapes	0.01	105	13	95	14
(post hydrolysis)	0.1	107	2.2	93	1.3
Wheat straw	0.01	98	4.2	93	6.9
(pre hydrolysis)	0.1	94	5	92	10
Wheat straw	0.01	108	15	93	12
(post hydrolysis)	0.1	86	1.7	100	4.1
		IN-QZY47: 307 → 156 m/z		IN-TMQ01 (IN-RSU03)[E]: 308 → 156 m/z	
Tomatoes	0.01	94	4.5	89	4.3
(pre hydrolysis)	0.1	107	3.1	106	1.6
Tomatoes	0.01	104	6.2	82	5.7
(post hydrolysis)	0.1	107	4.4	90	6.8
Soya beans	0.01	105	2.9	104	2.2

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
(pre hydrolysis)	0.1	107	1.9	105	1
Soya beans	0.01	102	8.5	85	5.6
(post hydrolysis)	0.1	104	2.6	89	3
Grapes	0.01	108	5.6	101	7.2
(pre hydrolysis)	0.1	106	4.3	106	3
Grapes	0.01	106	3.2	100	11
(post hydrolysis)[B]	0.1	107	2	106	4.6
Wheat straw	0.01	90	12	80	7.9
(pre hydrolysis)	0.1	85	9.9	97	14
Wheat straw	0.01	103	5	98	4.1
(post hydrolysis)	0.1	111	3.1	101	3
		IN-TQD54 (IN-UNS90)[F]: 294 → 206 m/z		IN-UJV12: 293 → 142 m/z	
Tomatoes	0.01	84	18	93	4.3
(post hydrolysis)	0.1	93	4.5	93	1.8
Soya beans	0.01	84	12	96	11
(post hydrolysis)[D]	0.1	90	5.6	83	3.4
Grapes	0.01	96	6.8	90	9.1
(post hydrolysis)[B]	0.1	108	2.8	89	2.9
Wheat straw	0.01	94	5.2	94	9.6
(post hydrolysis)[C]	0.1	99	3.6	103	3.6
		IN-A5760: 206 → 122 m/z			
Tomatoes	0.01	89	6		
(post hydrolysis)	0.1	87	1.3		
Soya beans	0.01	94	5.2		
(post hydrolysis)	0.1	89	5.4		
Grapes	0.01	98	7.7		
(post hydrolysis)	0.1	97	1.5		
Wheat straw	0.01	80	5.8		
(post hydrolysis)	0.1	88	1.3		

Notes:

[A] Interference observed in first validation set (Control 1, LOQ 1-3, 10×LOQ 1), confirmatory recoveries for IN-RYC33 in grapes based partly on background subtracted data. Interference not present for second validation set.

[B] Post-hydrolysis grapes: 307 → 220 m/z used for confirmation of IN-QZY47, 308 → 220 m/z used for confirmation of IN-TMQ01 (IN-RSU03), 294 → 206 m/z used for confirmation of IN-TQD54 (IN-UNS90).

[C] Post-hydrolysis wheat straw: 294 → 206 m/z used for confirmation of IN-TQD54 (IN-UNS90), 293 → 78 m/z used for confirmation of IN-UJV12.

[D] Post-hydrolysis soya beans: Confirmatory recoveries for IN-UJV12 based on background subtracted data as there was an interference present in the control sample.

[E] IN-TMQ01 is the *R* enantiomer contained in IN-RSU03 (racemic mixture).

[F] IN-TQD54 is the *R* enantiomer contained in IN-UNS90 (racemic mixture).

Confirmation data was also generated during method validation performed at a contractor facility under DuPont-37832, Revision No. 1. These data are summarised in Table 100.

Table 100 Confirmation data for the analytical method for the determination of fluazaindolizine residues in food of plant origin (Rebstock, 2021 DuPont 37832, Revision No. 1) (n=5)

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
		Fluazaindolizine: 466 → 142 m/z		IN-R2W56: 279 → 219 m/z	
Tomato	0.01	90	4	91	2
(pre-hydrolysis)	0.1	92	2.5	91	1

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
Wheat Grain	0.01	91	2	96	8
(pre-hydrolysis)	0.1	89	3.3	102	11
Orange	0.01	87	3	87	5.9
(pre-hydrolysis)	0.1	88	1.1	93	1.7
Corn Stover	0.01	85	3.6	87	5
(pre-hydrolysis)	0.1	86	3	88	3.6
Soya bean Seed	0.01	83	3.7	88	3.5
(pre-hydrolysis)	0.1	82	2.3	88	0.8
Dried Pea	0.01	96	5	86	4.1
(pre-hydrolysis)	0.1	96	3	88	1.9
		IN-REG72: 452 → 143 m/z		IN-RYC33: 264 → 219 m/z	
Tomato	0.01	94	1	91	1.4
(pre-hydrolysis)	0.1	92	3.4	90	3.2
Wheat Grain	0.01	82	6.3	93	5.1
(pre-hydrolysis)	0.1	80	6.3	92	1.8
Orange	0.01	86	3	91	4.7
(pre-hydrolysis)	0.1	88	1.5	93	0.6
Corn Stover	0.01	85	4.8	88	4
(pre-hydrolysis)	0.1	86	5.1	91	3.7
Soya bean Seed	0.01	78	4	92	0.6
(pre-hydrolysis)	0.1	78	2.5	91	2
Dried Pea	0.01	92	6.9	93	1.8
(pre-hydrolysis)	0.1	95	1.7	95	1.7
		IN-F4106: 220 → 141 m/z		IN-QEK31: 265 → 157 m/z	
Tomato	0.01	89	4.5	77	3.9
(pre-hydrolysis)	0.1	91	3.4	82	3.3
Tomato	0.01	114	3.7	77	3.9
(post-hydrolysis)	0.1	86	4.6	82	3.3
Wheat Grain	0.01	101	4.1	84	2.6
(pre-hydrolysis)	0.1	90	5.3	85	1.5
Wheat Grain[A]	0.01	81	9.4	78	6.2
(post-hydrolysis)	0.1	84	3.6	79	5.9
Orange	0.01	97	7.8	85	2.5
(pre-hydrolysis)	0.1	87	5.4	92	2
Orange[A]	0.01	86	9.5	83	3
(post-hydrolysis)	0.1	77	3.5	81	1.4
Corn Stover	0.01	90	15	81	3.5
(pre-hydrolysis)	0.1	86	11	83	3.1
Corn Stover	0.01	95	5.9	84	5.6
(post-hydrolysis)	0.1	84	1.4	80	1.9
Soya bean Seed	0.01	95	13	91	2
(pre-hydrolysis)	0.1	91	2.9	92	1.9
Soya bean Seed	0.01	80	11	77	4.6
(post-hydrolysis)	0.1	89	6.6	84	4.7
Dried Pea	0.01	89	23	95	4.5
(pre-hydrolysis)	0.1	96	3	91	2.4
Dried Pea[A]	0.01	111	19	86	3.3
(post-hydrolysis)	0.1	96	2.8	91	0.6
		IN-QZY47: 307 → 156 m/z		IN-TMQ01 (IN-RSU03)[C]: 308 → 156 M/Z	
Tomato	0.0089	93	7.8	91	1.5
(pre-hydrolysis)	0.089	90	1.7	92	4.5
Tomato	0.0089	105	5.6	94	2.7
(post-hydrolysis)	0.089	100	3.7	86	4.5
Wheat Grain	0.0089	85	5	87	2.9
(pre-hydrolysis)	0.089	87	3.6	91	2.4

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
Wheat Grain	0.0089	94	9.1	89	2.1
(post-hydrolysis)	0.089	83	5.4	87	2.1
Orange	0.0089	83	4.8	91	5.6
(pre-hydrolysis)	0.089	90	2.3	92	3.4
Orange	0.0089	79	7.4	79	4.6
(post-hydrolysis)	0.089	81	2.1	82	2.4
Corn Stover	0.0089	73	10	85	9.3
(pre-hydrolysis)	0.089	70	3.1	89	4.8
Corn Stover	0.0089	78	8	93	9.1
(post-hydrolysis)	0.089	80	7	87	1.8
Soya bean Seed	0.0089	90	6.6	92	4.1
(pre-hydrolysis)	0.089	88	2.5	91	1.7
Soya bean Seed	0.0089	83	7.2	83	7.2
(post-hydrolysis)	0.089	88	3.5	88	3.5
Dried Pea	0.0089	81	9.4	98	3.9
(pre-hydrolysis)	0.089	89	1.8	96	2.7
Dried pea	0.0089	109	9.9	87	10
(post-hydrolysis)	0.089	94	2.9	95	3
		IN-TQD54 (IN-UNS90)[D]: 294 → 142 m/z		IN-UJV12: 293 → 142 m/z	
Tomatoes	0.0089	99	5.9	102	2
(post-hydrolysis)	0.089	84	3.4	85	2.1
Wheat Grain	0.0089	93	10	-	-
(post-hydrolysis)	0.089	86	4	-	-
Orange	0.0089	80	4.1	93	8
(post-hydrolysis)	0.089	85	2.8	83	7.6
Corn Stover	0.0089	86	14	-	-
(post-hydrolysis)	0.089	80	7.4	-	-
Soya bean Seed	0.0089	75	15	84	19
(post-hydrolysis)	0.089	85	4.6	89	4.3
Dried Pea[B]	0.0089	98	5.8	85	16
(post-hydrolysis)	0.089	93	6.5	94	1.4
		IN-A5760: 206 → 142 m/z			
Tomatoes	0.01	95	5.5		
(post-hydrolysis)	0.1	88	1.7		
Wheat Grain	0.01	95	16		
(post-hydrolysis)	0.1	89	1.9		
Orange	0.01	84	6.7		
(post-hydrolysis)	0.1	80	4.6		
Dried Pea	0.01	97	19		
(post-hydrolysis)	0.1	96	3.2		

Notes:

[A] Post-hydrolysis wheat grain, post-hydrolysis orange and post-hydrolysis dried pea: 220 m/z → 156 m/z used for confirmation of IN-F4106 residues.

[B] Post-hydrolysis dried peas: 293 m/z → 78 m/z used for confirmation of IN-UJV12 residues.

[C] IN-TMQ01 is the *R* enantiomer contained in IN-RSU03 (racemic mixture).

[D] IN-TQD54 is the *R* enantiomer contained in IN-UNS90 (racemic mixture).

This method is suitable for residue data collection for risk assessment purposes and may also be considered suitable for enforcement of the MRL of fluazaindolizine (parent). The instrumentation required to perform both the analysis and confirmatory methods is available in most well equipped analytical laboratories.

Method DuPont-47054 (Gesell, 2020 DuPont-47054, Revision No. 2)

Method DuPont-47054 is a modification of method DuPont-33681. The residue method for the determination of fluazaindolizine residues involves simple extraction, clean-up, and analytical determination by LC-MS/MS detection. Samples were also analysed after hydrolysis to determine if conjugated residue was present. The extraction procedure implemented in Part One (pre-hydrolysis) is identical for each of the methods, and it is the acidic hydrolysis step described in Part Two of the method that was the focus of the validation work.

The recoveries of IN-QEK31, IN-F4106, IN-A5760, IN-TQD54 (IN-UNS90), IN-TMQ01 (IN-RSU03), IN-QZY47 and IN-UJV12 from crop samples fortified at 0.01 (LOQ) and 0.10 mg/kg and carried through the subsequent hydrolysis and clean-up procedures support the satisfactory performance of this method.

The solvent mixture used for extracting fluazaindolizine and its metabolites from crop samples (70/30 methanol/water) are based on the same solvent mixture/sample ratio used in the primary crop metabolism studies conducted with radiolabelled fluazaindolizine (DuPont-34947, DuPont-34948, DuPont-34946, DuPont-43220 and DuPont-41849). The extraction efficiency data ranged from 72.2 to 93.9 percent and averaged 84.6 percent of the total radioactivity in the various commodities.

The fortification data reported in the method for determination of residues in limes, dried peas, tomatoes, and soya beans are summarised in Table 101. The average recovery were within the range 70 to 120 percent (75–118 percent), with RSD values ≤ 20 percent (2–11 percent).

Recovery and repeatability data for the determination of IN-QEK31, IN-F4106, IN-A5760, IN-TQD54 (IN-UNS90), IN-TMQ01 (IN-RSU03), IN-QZY47 and IN-UJV12 in crops using method DuPont-47054 are presented in Table 101. Fluazaindolizine and its metabolites were extracted from plant matrices using a mixture of methanol and water. The final determination of fluazaindolizine and its metabolites was performed by LC-MS/MS.

The LOQ was defined by the lowest fortification level successfully tested, which was 0.01 mg/kg in tomatoes, dried peas, soya beans and limes. The percentRSDs ranged from 2 to 11. Calibration curves over the range 0.1–50 ng/mL for IN-QEK31, IN-A5760 and IN-TMQ01 (IN-RSU03) and 0.2–50 ng/mL for IN-F4106, IN-UJV12, IN-QZY47 and IN-TQD54 (IN-UNS90). These ranges corresponded to residue values of 0.003-1.7 mg/kg and 0.003-0.7 mg/kg, respectively. At least five-point standard curves were prepared utilizing standards in solvent with r^2 for calibration curves all > 0.9995.

Method DuPont-47054 is suitable to determine residues of fluazaindolizine and metabolites in the matrices investigated. The specificity of the method is provided using a mass selective detector. The method was successfully validated for fluazaindolizine metabolites.

Table 101 Recovery data (n=5) for the analytical method for the determination of fluazaindolizine residues in food of plant origin (Gesell, 2020 DuPont-47054, Revision No. 2)

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
		IN-F4106: 220 → 141 m/z		IN-UJV12: 293 → 206 m/z	
Tomatoes	0.01	91	11	86	11
	0.1	81	5	80	6
Soya beans	0.01	90	11	84	8
	0.1	81	3	72	5
Limes	0.01	75	4	71	6
	0.1	79	4	77	3
Dried Peas	0.01	98	11	86	7
	0.1	89	5	86	10
		IN-QZY47: 307 → 220 m/z		IN-TQD54 (IN-UNS90)[A]: 294 → 206 m/z	

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
Tomatoes	0.01	112	9	105	11
	0.1	103	6	91	7
Soya beans	0.01	100	3	93	5
	0.1	94	4	87	7
Limes	0.01	98	5	88	4
	0.1	111	3	99	5
Dried Peas	0.01	101	6		
	0.1	106	6		
		IN-A5760: 206 → 142 m/z		IN-TMQ01 (IN-RSU03)[B]: 308 → 220 m/z	
Tomatoes	0.01	107	9	95	9
	0.1	107	4	92	1
Soya beans	0.01	112	9	97	3
	0.1	90	3	89	4
Limes	0.01	107	5	93	6
	0.1	109	2	92	5
Dried Peas	0.01	113	9	83	4
	0.1	118	5	74	5
		IN-QEK31: 265 → 184 m/z			
Tomatoes	0.01	93	7		
	0.1	87	5		
Soya beans	0.01	100	6		
	0.1	81	4		
Limes	0.01	91	3		
	0.1	94	4		
Dried Peas	0.01	81	4		
	0.1	78	2		

Notes:

[A] IN-TQD54 is the *R* enantiomer contained in IN-UNS90 (racemic mixture).

[B] IN-TMQ01 is the *R* enantiomer contained in IN-RSU03 (racemic mixture).

Reproducibility

A successful ILV demonstrates the reproducibility of this method.

Table 102 Independent laboratory validation data (n=5) for the analytical method for the determination of fluazaindolizine residues in food of plant origin (Rutt, 2017 DuPont-45659)

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
		IN-F4106: 220 → 141 m/z		IN-UJV12: 293 → 206 m/z	
Tomatoes	0.01	81	7	73	7
	0.1	84	4	73	3
Limes	0.01	84	8	75	6
	0.1	87	9	81	10
Dried Peas	0.01	96	8	85	8
	0.1	78	7	83	8
		IN-QZY47: 307 → 220 m/z		IN-TQD54 (IN-UNS90)[A]: 294 → 206 m/z	
Tomatoes	0.01	86	5	93	7
	0.1	86	3	99	5
Limes	0.01	85	10	91	8
	0.1	89	10	102	9
Dried Peas	0.01	83	4	79	3
	0.1	85	8	88	10

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
		IN-A5760: 206 → 142 m/z		IN-TMQ01 (IN-RSU03)[B]: 308 → 220 m/z	
Tomatoes	0.01	105	9	93	1
	0.1	98	3	96	3
Limes	0.01	79	12	79	12
	0.1	82	10	84	11
Dried Peas	0.01	85	14	96	15
	0.1	96	9	86	6
		IN-QEK31: 265 → 184 m/z			
Tomatoes	0.01	94	6		
	0.1	94	4		
Limes	0.01	75	16		
	0.1	76	11		
Dried peas	0.01	95	12		
	0.1	86	7		

Notes:

[A] IN-TQD54 is the *R* enantiomer contained in IN-UNS90 (racemic mixture).

[B] IN-TMQ01 is the *R* enantiomer contained in IN-RSU03 (racemic mixture).

Confirmatory method

Confirmation of results obtained by this LC-MS/MS method was performed by quantifying a separate daughter ion signal for each analyte. The recovery data obtained using the confirmatory procedure are summarised in Table 103.

Table 103 Confirmatory data (n=5)for the analytical method for the determination of fluazaindolizine residues in food of plant origin (Gesell, 2020 DuPont-47054, Revision No. 2)

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
		IN-F4106: 220 → 156 m/z		IN-UJV12: 293 → 142 m/z	
Tomatoes	0.01	96	11	89	13
	0.1	81	5	76	5
Soya beans	0.01	98	9	90	12
	0.1	83	4	78	5
Limes	0.01	78	8	76	8
	0.1	79	3	79	5
Dried Peas	0.01	88	10	93	12
	0.1	80	8	88	7
		IN-QZY47: 307 → 156 m/z		IN-TQD54 (IN-UNS90)[A]: 294 → 142 m/z	
Tomatoes	0.01	115	8	101	7
	0.1	101	5	92	6
Soya beans	0.01	109	5	101	12
	0.1	88	3	93	7
Limes	0.01	100	9	89	5
	0.1	106	7	102	4
Dried Peas	0.01	102	10	89	13
	0.1	108	5	85	5
		IN-A5760: 206 → 122 m/z		IN-TMQ01 (IN-RSU03)[B]: 308 → 156 m/z	
Tomatoes	0.01	101	6	94	5
	0.1	93	2	90	2
Soya beans	0.01	106	7	94	16
	0.1	84	2	90	7
Limes	0.01	107	9	86	11

Matrix	Fortification level (mg/kg)	Mean (%)	% RSD	Mean (%)	%RSD
	0.1	99	7	86	4
Dried Peas	0.01	104	9	73	8
	0.1	118	4	74	6
		IN-QEK31: 265 → 192 m/z			
Tomatoes	0.01	88	2		
	0.1	85	4		
Soya beans	0.01	96	4		
	0.1	81	4		
Limes	0.01	83	4		
	0.1	91	4		
Dried Peas	0.01	78	4		
	0.1	76	3		

Notes:

[A] IN-TQD54 is the *R* enantiomer contained in IN-UNS90 (racemic mixture).

[B] IN-TMQ01 is the *R* enantiomer contained in IN-RSU03 (racemic mixture).

The modification hydrolysis conditions and clean-up in the residue method for the determination of residues of IN-QEK31, IN-F4106, IN-A5760, IN-UNS90, IN-TMQ01, IN-QZY47, and IN-UJV12 in tomatoes, soya beans, limes, and dried peas was successfully validated over the range of 0.01–0.1 mg/kg, with an LOQ of 0.01 mg/kg.

Method DuPont-39990, AP.224685A and B (Brown 2020 DuPont-39990)

The recovery data reported in the methods for determining residues of fluazaindolizine and its metabolites in tomato, grape, wheat grain, dried bean, wheat straw and soya bean seed are summarised in Table 104. For some metabolites, analyte standards were provided in salt form. MW back calculations are required to correct these values since this discrepancy was not taken into consideration during weighing of the analytical standards. MW conversions would result in a 0.89 conversion factor; therefore, actual values fortified were approximately 0.0089 and 0.089 mg/kg.

Average recoveries for pre- and post-hydrolysis fortification samples were within 70–120 percent, with percentRSD values ≤ 20 percent, with a few, minor exceptions: In each case other transitions were available that met performance criteria. Pre-hydrolysis results for individual ion transitions outside acceptable ranges were wheat grain IN-REG72 two transitions individual mean recoveries 65 percent and 69 percent and one transition for others, IN-QZY47 individual mean recovery 69 percent; wheat straw fluazaindolizine RSD 22 percent; IN-F4106 individual mean recovery 67 percent, IN-QZY47 69 percent, IN-QEK31 69 percent; dried beans fluazaindolizine 68 percent. Post-hydrolysis tomato fluazaindolizine RSD 25 percent, IN-RSU03 RSD 21 percent, IN-QEK31 RSD 21 percent;wheat grain IN-F4106 RSD 21 percent, IN-UNS90 RSD 23 percent; wheat straw IN-QZY47 RSD 21 percent, soya bean seed IN-UNS90 individual mean recovery 69 percent, dried bean IN-UJV12 RSD 31 percent, IN-UNS90 RSD 24 percent.

The LOQ of the method for quantifying residues of fluazaindolizine and its metabolites is nominally 0.01 mg/kg. Good linearity was observed in the range of 0.15 to 10 ng/mL (nominal) for fluazaindolizine and its metabolites IN-REG72, IN-QEK31, IN-F4106, IN-QZY47, IN-TMQ01 (IN-RSU03), IN-RCY33, and IN-R2W56 (pre-hydrolysis). Similarly, good linearity was observed in the range of 0.15 to 10 ng/mL for IN-F4106, IN-TMQ01 (IN-RSU03), IN-QZY47, IN-UJV12, IN-A5760, IN-TQD54 (IN-UNS90), and IN-QEK31 (post-hydrolysis). These ranges correspond to residue values of approximately 0.003–0.2 mg/kg. The observed r^2 values were ≥0.99. At least five-point standard curves were prepared utilizing standards in solvent.

The majority of the relative standard deviations for all analytes and concentration levels, in each matrix were less than 20 percent, with a few minor exceedances. Therefore, it can be concluded that the repeatability of this method is adequate for the determination of residues of fluazaindolizine and its metabolites in tomatoes, wheat grain, grapes, wheat straw, soya bean seeds, and dried beans (pre-and post-hydrolysis) (Table 104.

Table 104 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in tomato – pre-hydrolysis (Brown, 2020 DuPont-39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
Fluazaindolizine	466.0 → 156.8	0.01	5	78	12
		0.1	5	84	3
	466.0 → 142.0	0.01	5	71	9
		0.1	5	84	4
IN-F4106	220.0 → 140.9	0.01	5	93	6
		0.1	5	92	3
	220.0 → 156.1	0.01	5	78	10
		0.1	5	91	2
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	76	7
		0.089	5	88	2
	308.0 → 156.1	0.0089	5	81	7
		0.089	5	89	2
	308.0 → 141.0	0.0089	5	75	10
		0.089	5	82	1
IN-REG72	451.9 → 122.8	0.01	5	84	8
		0.1	5	100	1
	451.9 → 143.1	0.01	5	84	12
		0.1	5	98	1
IN-QZY47	306.9 → 220.0	0.0089	5	74	6
		0.089	5	90	2
	306.9 → 156.0	0.0089	5	74	5
		0.089	5	85	3
	306.9 → 77.9	0.0089	5	78	11
		0.089	5	84	2
IN-QEK31	264.9 → 246.9	0.01	5	80	7
		0.1	5	93	2
	264.9 → 218.9	0.01	5	78	6
		0.1	5	94	2
	264.9 → 192.0	0.01	5	78	6
		0.1	5	94	1
	264.9 → 184.0	0.01	5	86	11
		0.1	5	93	2
IN-RYC33	263.9 → 246.8	0.01	5	84	7
		0.1	5	99	2
	263.9 → 218.9	0.01	5	81	5
		0.1	5	98	2
	263.9 → 192.0	0.01	5	83	5
		0.1	5	99	3
	263.9 → 184.1	0.01	5	84	4
		0.1	5	93	2
IN-R2W56	278.8 → 246.8	0.01	5	83	5
		0.1	5	90	4
	278.8 → 219.1	0.01	5	82	5
		0.1	5	90	4

Table 105 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in wheat grain – pre-hydrolysis (Brown, 2020 DuPont 39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
Fluazaindolizine	466.0 → 156.8	0.01	5		5
		0.1	5		8
	466.0 → 142.0	0.01	5	82	8
		0.1	5	74	6
IN-F4106	220.0 → 140.9	0.01	5	90	10
		0.1	5	83	4
	220.0 → 156.1	0.01	5	87	7
		0.1	5	78	5
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	81	6
		0.089	5	77	3
	308.0 → 156.1	0.0089	5	83	9
		0.089	5	77	3
	308.0 → 141.0	0.0089	5	84	3
		0.089	5	78	1
IN-REG72	451.9 → 122.8	0.01	5	71	4
		0.1	5	69	6
	451.9 → 143.1	0.01	5	71	6
		0.1	5	65	6
IN-QZY47	306.9 → 220.0	0.0089	5	80	7
		0.089	5	69	3
	306.9 → 156.0	0.0089	5	86	13
		0.089	5	74	5
	306.9 → 77.9	0.0089	5	75	11
		0.089	5	73	2
IN-QEK31	264.9 → 246.9	0.01	5	95	3
		0.1	5	101	2
	264.9 → 218.9	0.01	5	93	2
		0.1	5	102	1
	264.9 → 192.0	0.01	5	90	5
		0.1	5	100	2
	264.9 → 184.0	0.01	5	94	4
		0.1	5	100	1
IN-RYC33	263.9 → 246.8	0.01	5	93	4
		0.1	5	102	3
	263.9 → 218.9	0.01	5	89	4
		0.1	5	102	3
	263.9 → 192.0	0.01	5	89	2
		0.1	5	101	4
	263.9 → 184.1	0.01	5	93	3
		0.1	5	101	4
IN-R2W56	278.8 → 246.8	0.01	5	104	2
		0.1	5	110	4
	278.8 → 219.1	0.01	5	101	3
		0.1	5	110	3

Table 106 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in grape–pre-hydrolysis (Brown, 2020 DuPont-39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg) [AB]	N	Mean (%)	% RSD
Fluazaindolizine	466.0 → 156.8	0.01	5	105	4
		0.1	5	110	3
	466.0 → 142.0	0.01	5	100	4
		0.1	5	111	2
IN-F4106	220.0 → 140.9	0.01	5	103	5
		0.1	5	109	2
	220.0 → 156.1	0.01	5	105	5
		0.1	5	110	1
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	105	6
		0.089	5	110	7
	308.0 → 156.1	0.0089	5	110	2
		0.089	5	110	9
	308.0 → 141.0	0.0089	5	107	7
		0.089	5	110	10
IN-REG72	451.9 → 122.8	0.01	5	104	3
		0.1	5	107	1
	451.9 → 143.1	0.01	5	111	5
		0.1	5	108	1
IN-QZY47	306.9 → 220.0	0.0089	5	111	12
		0.089	5	113	10
	306.9 → 156.0	0.0089	5	110	9
		0.089	5	112	8
	306.9 → 77.9	0.0089	5	111	17
		0.089	5	114	9
IN-QEK31	264.9 → 246.9	0.01	5	97	4
		0.1	5	103	5
	264.9 → 218.9	0.01	5	96	1
		0.1	5	104	5
	264.9 → 192.0	0.01	5	95	2
		0.1	5	105	5
	264.9 → 184.0	0.01	5	94	4
		0.1	5	103	5
IN-RYC33	263.9 → 246.8	0.01	5	97	1
		0.1	5	102	2
	263.9 → 218.9	0.01	5	97	2
		0.1	5	103	2
	263.9 → 192.0	0.01	5	97	2
		0.1	5	103	2
	263.9 → 184.1	0.01	5	96	1
		0.1	5	103	2
IN-R2W56	278.8 → 246.8	0.01	5	95	1
		0.1	5	100	2
	278.8 → 219.1	0.01	5	96	1
		0.1	5	101	2

Table 107 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in wheat straw–pre-hydrolysis (Brown, 2020 DuPont 39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
Fluazaindolizine	466.0 → 156.8	0.01	5	98	18
		0.1	5	111	11
	466.0 → 142.0	0.01	5	99	22
		0.1	5	102	12
IN-F4106	220.0 → 140.9	0.01	5	71	13
		0.1	5	80	7
	220.0 → 156.1	0.01	5	67	6
		0.1	5	81	7
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	76	9
		0.089	5	80	4
	308.0 → 156.1	0.0089	5	78	11
		0.089	5	81	5
	308.0 → 141.0	0.0089	5	75	10
		0.089	5	81	5
IN-REG72	451.9 → 122.8	0.01	5	85	12
		0.1	5	99	11
	451.9 → 143.1	0.01	5	86	9
		0.1	5	97	11
IN-QZY47	306.9 → 220.0	0.0089	5	79	8
		0.089	5	83	5
	306.9 → 156.0	0.0089	5	81	7
		0.089	5	84	5
	306.9 → 77.9	0.0089	5	69	8
		0.089	5	78	8
IN-QEK31	264.9 → 246.9	0.01	5	76	4
		0.1	5	76	3
	264.9 → 218.9	0.01	5	69	6
		0.1	5	79	4
	264.9 → 192.0	0.01	5	76	7
		0.1	5	78	2
	264.9 → 184.0	0.01	5	72	3
		0.1	5	78	3
IN-RYC33	263.9 → 246.8	0.01	5	73	6
		0.1	5	77	6
	263.9 → 218.9	0.01	5	70	11
		0.1	5	77	6
	263.9 → 192.0	0.01	5	72	4
		0.1	5	77	7
	263.9 → 184.1	0.01	5	76	7
		0.1	5	76	8
IN-R2W56	278.8 → 246.8	0.01	5	77	6
		0.1	5	80	8
	278.8 → 219.1	0.01	5	75	7
		0.1	5	81	8

Table 108 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in soya bean – pre-hydrolysis (Brown, 2020 DuPont-39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
Fluazaindolizine	466.0 → 156.8	0.01	5	74	17
		0.1	5	103	3
	466.0 → 142.0	0.01	5	88	7
		0.1	5	103	5
IN-F4106	220.0 → 140.9	0.01	5	94	9
		0.1	5	97	4
	220.0 → 156.1	0.01	5	82	6
		0.1	5	96	4
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	94	3
		0.089	5	98	3
	308.0 → 156.1	0.0089	5	95	7
		0.089	5	97	4
	308.0 → 141.0	0.0089	5	97	5
		0.089	5	98	4
IN-REG72	451.9 → 122.8	0.01	5	84	10
		0.1	5	94	9
	451.9 → 143.1	0.01	5	72	9
		0.1	5	95	6
IN-QZY47	306.9 → 220.0	0.0089	5	97	11
		0.089	5	94	6
	306.9 → 156.0	0.0089	5	76	15
		0.089	5	90	7
	306.9 → 77.9	0.0089	5	82	18
		0.089	5	97	6
IN-QEK31	264.9 → 246.9	0.01	5	92	4
		0.1	5	95	3
	264.9 → 218.9	0.01	5	93	5
		0.1	5	97	3
	264.9 → 192.0	0.01	5	92	4
		0.1	5	96	3
	264.9 → 184.0	0.01	5	94	5
		0.1	5	96	4
IN-RYC33	263.9 → 246.8	0.01	5	93	4
		0.1	5	94	3
	263.9 → 218.9	0.01	5	94	7
		0.1	5	95	3
	263.9 → 192.0	0.01	5	92	5
		0.1	5	96	3
	263.9 → 184.1	0.01	5	90	9
		0.1	5	96	3
IN-R2W56	278.8 → 246.8	0.01	5	95	5
		0.1	5	98	4
	278.8 → 219.1	0.01	5	93	5
		0.1	5	97	4

Table 109 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in dried beans – pre-hydrolysis (Brown, 2020 DuPont-39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
Fluazaindolizine	466.0 → 156.8	0.01	5	86	5
		0.1	5	75	11
	466.0 → 142.0	0.01	5	68	5
		0.1	5	80	9
IN-F4106	220.0 → 140.9	0.01	5	83	11
		0.1	5	85	3
	220.0 → 156.1	0.01	5	83	23
		0.1	5	84	5
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	86	7
		0.089	5	87	4
	308.0 → 156.1	0.0089	5	89	9
		0.089	5	89	4
	308.0 → 141.0	0.0089	5	85	13
		0.089	5	88	3
IN-REG72	451.9 → 122.8	0.01	5	81	9
		0.1	5	79	4
	451.9 → 143.1	0.01	5	84	10
		0.1	5	80	1
IN-QZY47	306.9 → 220.0	0.0089	5	84	9
		0.089	5	85	2
	306.9 → 156.0	0.0089	5	86	12
		0.089	5	86	3
	306.9 → 77.9	0.0089	5	87	9
		0.089	5	83	2
IN-QEK31	264.9 → 246.9	0.01	5	83	7
		0.1	5	85	2
	264.9 → 218.9	0.01	5	85	9
		0.1	5	84	1
	264.9 → 192.0	0.01	5	82	9
		0.1	5	85	2
	264.9 → 184.0	0.01	5	81	7
		0.1	5	82	3
IN-RYC33	263.9 → 246.8	0.01	5	86	8
		0.1	5	82	2
	263.9 → 218.9	0.01	5	83	9
		0.1	5	81	2
	263.9 → 192.0	0.01	5	84	8
		0.1	5	82	2
	263.9 → 184.1	0.01	5	78	9
		0.1	5	82	4
IN-R2W56	278.8 → 246.8	0.01	5	92	9
		0.1	5	86	3
	278.8 → 219.1	0.01	5	89	10
		0.1	5	87	2

Table 110 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in tomato – post-hydrolysis (Brown, 2020 DuPont-39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
IN-F4106	220.0 → 140.9	0.01	5	95	25
		0.1	5	95	6
	220.0 → 156.1	0.01	5	100	16
		0.1	5	95	7
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	88	19
		0.089	5	88	8
	308.0 → 156.1	0.0089	5	89	14
		0.089	5	87	9
	308.0 → 141.0	0.0089	5	90	21
		0.089	5	86	9
IN-QZY47	306.9 → 220.0	0.0089	5	94	20
		0.089	5	95	8
	306.9 → 156.0	0.0089	5	90	18
		0.089	5	96	6
	306.9 → 77.9	0.0089	5	99	17
		0.089	5	96	9
IN-UJV12	292.9 → 205.9	0.0089	5	87	19
		0.089	5	81	8
	292.9 → 141.9	0.0089	5	90	20
		0.089	5	82	10
	292.9 → 77.8	0.0089	5	85	14
		0.089	5	84	8
IN-A5760	206.0 → 142.0	0.01	5	102	14
		0.1	5	93	6
	206.0 → 122.0	0.01	5	93	16
		0.1	5	93	7
IN-TQD54 (IN-UNS90)	293.9 → 205.8	0.0089	5	90	18
		0.089	5	84	9
	293.9 → 141.7	0.0089	5	-	-
		0.089	5	-	-
	293.9 → 78.0	0.0089	5	84	20
		0.089	5	83	9
IN-QEK31	264.9 → 246.9	0.01	5	-	-
		0.1	5	-	-
	264.9 → 218.9	0.01	5	-	-
		0.1	5	-	-
	264.9 → 192.0	0.01	5	-	-
		0.1	5	-	-
	264.9 → 184.0	0.01	5	87	21
		0.1	5	91	8

Table 111 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in wheat grain –post-hydrolysis (Brown, 2020 DuPont-39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
IN-F4106	220.0 → 140.9	0.01	5	86	21
		0.1	5	88	8
	220.0 → 156.1	0.01	5	97	20
		0.1	5	89	8
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	76	8

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
		0.089	5	82	5
	308.0 → 156.1	0.0089	5	85	8
		0.089	5	82	3
	308.0 → 141.0	0.0089	5	75	5
		0.089	5	81	5
IN-QZY47	306.9 → 220.0	0.0089	5	88	4
		0.089	5	92	3
	306.9 → 156.0	0.0089	5	92	6
		0.089	5	92	3
	306.9 → 77.9	0.0089	5	98	8
		0.089	5	88	3
IN-UJV12	292.9 → 205.9	0.0089	5	77	8
		0.089	5	88	6
	292.9 → 141.9	0.0089	5	80	19
		0.089	5	86	6
	292.9 → 77.8	0.0089	5	77	3
		0.089	5	84	8
IN-A5760	206.0 → 142.0	0.01	5	90	12
		0.1	5	91	8
	206.0 → 122.0	0.01	5	88	11
		0.1	5	92	7
IN-TQD54 (IN-UNS90)	293.9 → 205.8	0.0089	5	-	-
		0.089	5	-	-
	293.9 → 141.7	0.0089	5	90	15
		0.089	5	85	6
	293.9 → 78.0	0.0089	5	94	23
		0.089	5	90	6
IN-QEK31	264.9 → 246.9	0.01	5	88	5
		0.1	5	84	4
	264.9 → 218.9	0.01	5	-	-
		0.1	5	-	-
	264.9 → 192.0	0.01	5	-	-
		0.1	5	-	-
	264.9 → 184.0	0.01	5	82	13
		0.1	5	86	3

Table 112 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in grapes–post-hydrolysis (Brown, 2020 DuPont-39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
IN-F4106	220.0 → 140.9	0.01	5	81	13
		0.1	5	91	2
	220.0 → 156.1	0.01	5	80	18
		0.1	5	88	3
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	83	6
		0.089	5	86	7
	308.0 → 156.1	0.0089	5	78	6
		0.089	5	85	7
	308.0 → 141.0	0.0089	5	86	5
		0.089	5	86	7
IN-QZY47	306.9 → 220.0	0.0089	5	82	7
		0.089	5	97	7
	306.9 → 156.0	0.0089	5	83	11

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
		0.089	5	96	6
	306.9 → 77.9	0.0089	5	77	6
		0.089	5	103	7
IN-UJV12	292.9 → 205.9	0.0089	5	81	10
		0.089	5	94	6
	292.9 → 141.9	0.0089	5	89	6
		0.089	5	94	5
	292.9 → 77.8	0.0089	5	71	5
		0.089	5	94	4
IN-A5760	206.0 → 142.0	0.01	5	92	8
		0.1	5	87	5
	206.0 → 122.0	0.01	5	83	11
		0.1	5	90	7
IN-TQD54 (IN-UNS90)	293.9 → 205.8	0.0089	5	80	6
		0.089	5	88	8
	293.9 → 141.7	0.0089	5	90	14
		0.089	5	94	9
	293.9 → 78.0	0.0089	5	85	19
		0.089	5	89	7
IN-QEK31	264.9 → 246.9	0.01	5	76	7
		0.1	5	88	8
	264.9 → 218.9	0.01	5	79	13
		0.1	5	86	8
	264.9 → 192.0	0.01	5	84	8
		0.1	5	90	7
	264.9 → 184.0	0.01	5	79	10
		0.1	5	89	8

Table 113 Validation data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in wheat straw–post-hydrolysis (Brown, 2020 DuPont-39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
IN-F4106	220.0 → 140.9	0.01	5	98	19
		0.1	5	90	11
	220.0 → 156.1	0.01	5	92	17
		0.1	5	89	7
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	99	4
		0.089	5	95	6
	308.0 → 156.1	0.0089	5	95	4
		0.089	5	94	7
	308.0 → 141.0	0.0089	5	94	4
		0.089	5	93	6
IN-QZY47	306.9 → 220.0	0.0089	5	99	8
		0.089	5	95	6
	306.9 → 156.0	0.0089	5	109	9
		0.089	5	95	7
	306.9 → 77.9	0.0089	5	90	21
		0.089	5	88	9
IN-UJV12	292.9 → 205.9	0.0089	5	84	6
		0.089	5	88	5
	292.9 → 141.9	0.0089	5	70	10
		0.089	5	88	4
	292.9 → 77.8	0.0089	5	82	12

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
		0.089	5	88	9
IN-A5760	206.0 → 142.0	0.01	5	84	6
		0.1	5	87	7
	206.0 → 122.0	0.01	5	82	11
		0.1	5	86	7
IN-TQD54 (IN-UNS90)	293.9 → 205.8	0.0089	5	92	6
		0.089	5	91	6
	293.9 → 141.7	0.0089	5	83	16
		0.089	5	93	5
	293.9 → 78.0	0.0089	5	86	6
		0.089	5	84	10
IN-QEK31	264.9/246.9	0.01	5	111	5
		0.1	5	96	6
	264.9 → 218.9	0.01	5	100	6
		0.1	5	103	7
	264.9 → 192.0	0.01	5	103	7
		0.1	5	101	7
	264.9 → 184.0	0.01	5	103	4
		0.1	5	101	7

Table 114 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in soya bean –post-hydrolysis (Brown, 2020 DuPont-39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
IN-F4106	220.0 → 140.9	0.01	5	80	12
		0.1	5	76	2
	220.0 → 156.1	0.01	5	74	10
		0.1	5	75	4
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	82	5
		0.089	5	89	5
	308.0 → 156.1	0.0089	5	83	3
		0.089	5	90	6
	308.0 → 141.0	0.0089	5	93	6
		0.089	5	90	4
IN-QZY47	306.9 → 220.0	0.0089	5	91	9
		0.089	5	107	4
	306.9 → 156.0	0.0089	5	110	8
		0.089	5	105	4
	306.9 → 77.9	0.0089	5	-	-
		0.089	5	-	-
IN-UJV12	292.9 → 205.9	0.0089	5	116	5
		0.089	5	99	5
	292.9 → 141.9	0.0089	5	111	20
		0.089	5	103	4
	292.9 → 77.8	0.0089	5	105	19
		0.089	5	99	8
IN-A5760	206.0 → 142.0	0.01	5	80	10
		0.1	5	75	7
	206.0 → 122.0	0.01	5	93	5
		0.1	5	80	5
IN-TQD54 (IN-UNS90)	293.9 → 205.8	0.0089	5	69	15
		0.089	5	88	10
	293.9 → 141.7	0.0089	5	99	13

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
		0.089	5	88	7
	293.9 → 78.0	0.0089	5	94	17
		0.089	5	90	6
IN-QEK31	264.9 → 246.9	0.01	5	-	-
		0.1	5	-	-
	264.9 → 218.9	0.01	5	-	-
		0.1	5	-	-
	264.9 → 192.0	0.01	5	-	-
		0.1	5	-	-
	264.9 → 184.0	0.01	5	76	6
		0.1	5	75	6

Table 115 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in dried beans – post-hydrolysis (Brown, 2020 DuPont-39990, Revision No. 1)

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
IN-F4106	220.0 → 140.9	0.01	5	94	4
		0.1	5	91	7
	220.0 → 156.1	0.01	5	70	13
		0.1	5	93	10
IN-TMQ01 (IN-RSU03)	308.0 → 219.8	0.0089	5	85	5
		0.089	5	88	6
	308.0 → 156.1	0.0089	5	80	13
		0.089	5	86	8
	308.0 → 141.0	0.0089	5	87	12
		0.089	5	85	6
IN-QZY47	306.9 → 220.0	0.0089	5	90	6
		0.089	5	98	9
	306.9 → 156.0	0.0089	5	99	8
		0.089	5	101	10
	306.9 → 77.9	0.0089	5	110	14
		0.089	5	100	12
IN-UJV12	292.9 → 205.9	0.0089	5	96	6
		0.089	5	96	10
	292.9 → 141.9	0.0089	5	97	11
		0.089	5	93	9
	292.9 → 77.8	0.0089	5	80	31
		0.089	5	96	9
IN-A5760	206.0 → 142.0	0.01	5	91	4
		0.1	5	92	9
	206.0 → 122.0	0.01	5	88	8
		0.1	5	92	9
IN-TQD54 (IN-UNS90)	293.9 → 205.8	0.0089	5	-	-
		0.089	5	-	-
	293.9 → 141.7	0.0089	5	100	15
		0.089	5	92	10
	293.9 → 78.0	0.0089	5	74	24
		0.089	5	86	10
IN-QEK31	264.9 → 246.9	0.01	5	89	4
		0.1	5	88	9
	264.9 → 218.9	0.01	5	75	18
		0.1	5	86	10
	264.9 → 192.0	0.01	5	89	5

Analyte	Q1/Q3	Fortification level (mg/kg)	N	Mean (%)	% RSD
		0.1	5	95	9
	264.9 → 184.0	0.01	5	96	7
		0.1	5	91	10

Radiovalidation

MacDonald (2018 DuPont-48155) studied the hydrolysis step in the analytical method using selected samples from crop rotation and plant metabolism studies which utilised [Ph-^{14}C]- or ([IP-5,8a-^{14}C]-fluazaindolizine. For both sites of label, samples of 30DAA wheat hay, 30DAA radish roots. 30DAA mature spinach and soya bean hay were taken from the confined crop rotation study (DuPont-34945, Revision No. 1) and soya bean seeds were taken from the plant metabolism study (DuPont-34948).

Samples were, or had been, extracted with a methanol:water (70:30) mixture which removed the majority of the radioactive residues (77.1–95.8 percent TRR). The initial extraction solvents used in this study (methanol:water (7:3)) are identical to those used in the analytical method for the determination of fluazaindolizine and metabolites in crops using LC-MS/MS (DuPont-33861). The resulting extracts were combined to form a single extract, and a 10 mL aliquot was evaporated and hydrolysed with 4N HCl (*ca.* 100 °C, 1 hour). The samples were processed using SPE and LC conducted on any fractions which contained significant levels of radioactivity. Recoveries of the radioactive residues were measured throughout. The overall recoveries were calculated as a percentage of the extracted radioactive residues (percent TER).

With the exception of [IP-5,8a-^{14}C]fluazaindolizine soya bean seeds, subsamples of homogenised tissue were freshly extracted three times with methanol:water (7:3) at ambient temperature. Each extract was separated from solids by centrifugation. [IP-5,8a-^{14}C]fluazaindolizine soya bean seeds extracts were generated in the exact same way under DuPont-34948. For each sample, extracts 1-3 were combined to form a single extract.

Acid hydrolysis, using HCl, was conducted on combined extracts for each commodity. A 10 mL aliquot of was reduced to *ca.* 0.5 mL under a stream of nitrogen gas at 60 °C prior to reconstitution in 4M HCl and incubated at 100 °C for one hour. Hydrolysed samples were cleaned up by Oasis HLB SPE column previously conditioned with acetonitrile followed by 0.1M HCl. The hydrolysate was loaded onto the column which was rinsed with 1 percent acetic acid; twice with 5 mL of acetonitrile:50 mM ammonium acetate (90:10) and finally with acetonitrile. The acetonitrile:ammonium acetate eluates were reduced to dryness at 60 °C under a gentle stream of nitrogen and reconstituted in 1 mL of 50 mM ammonium acetate for analysis by LC-UV. Reference standards were analysed under the same conditions. Quantification of radioactivity in the column effluent was confirmed by fraction collection.

For each sample, the main components were hydrolysed to the expected products that were observed previously when crop sample extracts were hydrolysed under milder conditions (*ca.* 1N HCl at 80 °C for *ca.* 16 hrs). For example, fluazaindolizine was hydrolysed to IN-F4106/IN-QEK31, IN-REG72 was hydrolysed to IN-A5760/IN-QEK31, conjugates of IN-QZY47 were hydrolysed to IN-QZY47 and IN-UGA20 was hydrolysed to IN-QEK31.

Radiovalidation of residue extraction method

The radiochromatograms of the extracted ^{14}C using the metabolism based extraction method and that used in method DuPont-33861, Revision No. 3 were compared. The extraction solvents and proportions of solvent to sample in both methods are identical. Extractability of the majority of the ^{14}C was similar in

both the metabolism and method DuPont-33861 with both generating similar HPLC profiles, an example for hay from the [Ph-^{14}C]fluazaindolizine is shown in Table 116 with the extraction efficiency ≥ 67 percent for each analyte (Table 116).

Table 116 Comparison of the extraction efficiency of fluazaindolizine and various metabolites using the residue analytical method and the extraction method used in soya bean metabolism study ([Ph-^{14}C]fluazaindolizine hay)

Component	Metabolism Method		Method DuPont-33861		Extraction Efficiency (hydrolysis) [A] (% TRR)
	% TRR	mg/kg	% TRR	mg/kg	
Fluazaindolizine	6.1	0.040	4.8	0.032	80
IN-REG72	1.2	0.008	1.4	0.009	113
Malonyl conjugate of IN-QZY47 (IN-TUT81)	53.5	0.353	47.0	0.308	87
IN-QZY47	5.1	0.033	3.8	0.025	76
IN-RSU03 (IN-TMQ01)	0.5	0.003	-	-	- [B]
IN-F4106	2.7	0.018	1.9	0.012	67
Conjugate of IN-UJV12	2.4	0.016	1.7	0.011	69

Notes:
[A] 100×(% TRR method DuPont-33861)/(%TRR metabolism method) = % extraction efficiency of each analyte.
[B] IN-RSU03 levels were too low to determine extraction efficiency in this case.

Multiresidue methods

The applicability of the US Food and Drug Administration, Pesticide Analytical Manual (PAM), Volume I multiresidue methods for analysis of fluazaindolizine, IN-F4106, IN-QEK31, INQZY47 and IN-TMQ01 were evaluated by Ballard (2018 DuPont-42610). The study specifically evaluated the usefulness of the MRM described in the FDA PAM – Volume I, Appendix II, Third Edition, Jan. 1994: Multi-residue Protocols A, B, C, D, E, and F only for measuring residues of fluazaindolizine and its four metabolites. Protocols A, B, and C were tested to provide fluorescence detection and GC chromatography detection information. Protocol A was tested using instructions in PAM I, Section 401, module DL2 to determine if the compound was naturally fluorescent as required in the protocol. If the compound was fluorescent and provided reasonable sensitivity, this would have required additional work (Section 401 E1 + C1) to determine stability of chemical in methanol, short-term and long-term recovery of chemical through charcoal/silanised Celite clean up column and recovery through completed method with one substrate.

For protocol A, fluazaindolizine and IN-QEK31 were not fluorescent while IN-F4106, IN-QZY47 and IN-TMQ01 were weakly fluorescent. Protocol B was tested as per protocol guidelines for the carboxylic acid metabolites, IN-QEK31, IN-QZY47 and IN-TMQ01 (IN-RSU03). The methyl ester for the metabolite IN-QEK31 (IN-R2W56) was provided and run through GC module DG1. A peak was detected with suitable sensitivity to continue testing. All metabolites, IN-QEK31, IN-QZY47 and IN-TMQ01 (IN-RSU03), were methylated per Section 402 C1b. None of the compounds were successfully recovered through the Florisil® clean-up. Further testing through Protocol B was discontinued.

Section 302 GLC responses were determined by Protocol C guidelines for compounds fluazaindolizine, IN-QEK31, IN-F4106, IN-QZY47, and IN-TMQ01 (IN-RSU03). Compounds IN-F4106 and IN-QZY47 were deemed suitable for GC analysis under DG1 conditions and Protocol D was conducted according to the decision tree.

Extraction without clean-up was not attempted since the only module with adequate sensitivity, DG1, utilised electron capture detection. Compounds IN-F4106 and IN-QZY47 were tested through Section 302 C5 Florisil® clean up. Neither compound was adequately recovered so further work on Protocol D was discontinued.

Protocol E testing was not attempted because no compounds were successfully recovered through Protocol D (Section 302). Protocol F was tested as per protocol guidelines for IN-F4106 and IN-QZY47. The two components were tested through Section 304 C1 and C2 Florisil® Clean up. Neither compound was adequately recovered so further work on Protocol F was discontinued. Protocol G was not conducted because none of the compounds were substituted ureas.

In conclusion, the FDA MRMs are not suitable for detection and enforcement of MRL for fluazaindolizine, IN-QEK31, IN-F4106, IN-QZY47 or IN-TMQ01 (IN-RSU03) in non-fatty or fatty matrices.

Multi-Residue Method DFG-S19 was assessed for the detection, quantification, and confirmation of residues of fluazaindolizine and its metabolites IN-QEK31, IN-F4106, IN-A5760, IN-RYC33, IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 in tomato, soya bean, grapefruit, and wheat straw (Čermák and Kwiecien 2017, DuPont-42611). Samples were extracted with acetone using a homogenizer, and water is added beforehand in an amount that takes into account the water content of the matrix so that acetone/water ratio remains constant at 2/1. For wheat grain and soya bean seed, the water is heated to 40 °C and samples were allowed to soak for approximately 30 minutes. For grapefruit, a pH value is adjusted to approximately pH 7 by adding sodium hydrogen carbonate.

After addition of sodium chloride and ethyl acetate/cyclohexane (1/1) and repeated homogenization, the organic layer containing fluazaindolizine and its metabolites, is allowed to separate from the aqueous layer. The evaporated residue of an aliquot of the organic phase is cleaned up by gel permeation chromatography (GPC) on Bio Beads S-X3 (polystyrene gel) using a mixture of ethyl acetate and cyclohexane (1/1) as eluent and an automated gel permeation chromatograph. The residue-containing GPC fraction is concentrated, re-dissolved in the chromatography solvent and analyzed by LC-MS/MS.

The recovery data reported in the method for determining residues of fluazaindolizine and its metabolites in tomato, soya bean, grapefruit, and wheat straw are summarised in Table 117.

Good linearity was observed in the range of 0.20 to 20 ng/mL for fluazaindolizine and its metabolites IN-QEK31, IN-F4106, IN-A5760, IN-RYC33, IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90. These ranges correspond to residue values of approximately 0.002-0.2 mg/kg. The observed coefficients of determination r^2 were found to be ≥0.99. At least five-point standard curves were prepared utilizing standards in solvent.

The method was not validated due to unsatisfactory recovery for fluazaindolizine in wheat straw and grapefruit, for IN-QEK31, IN-TMQ01, and IN-UNS90 in all matrices except tomato and for IN-UJV12 and IN-QZY47 in all matrices. The determination of IN-4106, IN-A5760, and IN-RYC33 was successful in all matrices (except some small limitation in wheat straw). Repeatability is only adequate (RSD < 20 percent) for the determination of residues of IN-F4106, IN-A5760, and IN-RYC33 in tomato, soya bean, and grapefruit. In conclusion, the method was only validated for IN-F4106, IN-A5760, and IN-RYC33 in tomato, soya bean, and grapefruit at a LOQ of 0.01 mg/kg.

Table 117 Recovery data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in commodities of plant origin (Čermák, Kwiecien, 2017 DuPont-42611)

Matrix	Fortification level (mg/kg)	N	Mean (percent)	RSD	Mean (%)	% RSD
			Fluazaindolizine: 466 → 157 m/z		IN-QEK31: 265 → 247 m/z	
Tomato	0.01	5	113	2.1	116	1.1
	0.1	5	108	1.6	97	4.0
Soya bean	0.01	5	87	14	31	3.0
	0.1	5	78	8.1	19	5.8

Matrix	Fortification level (mg/kg)	N	Mean (percent)	RSD	Mean (%)	% RSD
Grapefruit	0.01	5	53	13		
	0.1	5	46	20		
Wheat Straw	0.01	5	0	0		
	0.1	5	18	20		
			IN-RYC33: 264 → 247 m/z		IN-F4106: 220 → 156 m/z	
Tomato	0.01	5	119	0.8	114	5.6
	0.1	5	114	2.7	109	3.7
Soya bean	0.01	5	92	6.4	100	8.5
	0.1	5	83	7.1	89	3.7
Grapefruit	0.01	5	87	3.2	108	3.1
	0.1	5	85	4.8	104	3.6
Wheat Straw	0.01	5	72	15	91	11
	0.1	5	68	5.4	84	15
			IN-A5760: 206 → 122 m/z		IN-TMQ01: 308 → 220 m/z	
Tomato	0.01	5	116	3.6	104	4.0
	0.1	5	110	1.9	101	2.0
Soya bean	0.01	5	97	4.8	0	0
	0.1	5	89	5.6	17	8.0
Grapefruit	0.01	5	89	2.3		
	0.1	5	84	5.2		
Wheat Straw	0.01	5	78	19		
	0.1	5	62	18		
			IN-UNS90: 294 → 206 m/z		IN-UJV12: 295 → 57 m/z	
Tomato	0.01	5	91	4.9		
	0.1	5	88	3.5		
Soya bean	0.1	5	6	7.0		

Confirmatory method

Confirmation of results obtained by this HPLC-MS/MS method was performed by quantifying a separate daughter ion signal for each analyte. The recovery data obtained using the confirmatory procedure are summarised in Table 118. For fluazaindolizine, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90, sufficient recovery was not obtained for most of the matrices under investigation. The method was considered validated for the determination of residues of IN-F4106, IN-A5760, and IN-RYC33 in tomato, soya bean, and grapefruit, but not wheat straw.

Table 118 Confirmation data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in commodities of plant origin (Čermák., Kwiecien, 2017 DuPont-42611)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			Fluazaindolizine: 468 → 247 m/z		IN-QEK31: 265 → 219 m/z	
Tomato	0.01	5	112	4.9	107	4.5
	0.1	5	102	1.4	99	4.1
Soya bean	0.01	5	106	4.6		
	0.1	5	101	7.9	17	6.5
Grapefruit	0.01	5	57	11		
	0.1	5	48	18		
Wheat Straw	0.1	5	20	3.1		
			IN-RYC33: 264 → 219 m/z		IN-F4106: 220 → 78 m/z	
Tomato	0.01	5	118	1.9	113	1.6
	0.1	5	113	2.3	109	2.7

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
Soya bean	0.01	5	92	6.6	101	8.1
	0.1	5	83	7.1	93	4.7
Grapefruit	0.01	5	86	3.2	102	4.4
	0.1	5	85	2.9	104	5.4
Wheat Straw	0.01	5	73	14	98	14
	0.1	5	68	4.5	89	15
			IN-A5760: 206 → 78 m/z		IN-TMQ01: 308 → 156 m/z	
Tomato	0.01	5	112	3.4	102	7.2
	0.1	5	112	1.3	102	2.6
Soya bean	0.01	5	97	5.0		
	0.1	5	89	6.0	17	8.0
Grapefruit	0.01	5	89	3.3		
	0.1	5	85	5.5		
Wheat Straw	0.01	5	78	18		
	0.1	5	61	16		
			IN-UNS90: 294 → 78 m/z			
Tomato	0.01	5	95	6.0		
	0.1	5	91	5.7		
Soya bean	0.1	5	6	2.5		
	0.1	5	0	0		

Substrates of animal origin - Description of methods for animal matrices

The study summary presented below encompasses the regulatory studies completed to create a suitable method for monitoring animal tissues for fluazaindolizine residue. An additional validation of this method was performed at Charles River Laboratories in the United Kingdom to support cattle feeding study under DuPont-42572, Revision No. 1 and is presented as an additional summary at the end of this section.

Table 119 Overview of the LC-MS/MS methods for determination of fluazaindolizine in animal commodities

Method	Charles River AP.225144.02 used in feeding study (modification of DuPont 39226) Du-Pont 42572	DuPont 39226 (see also modification Charles River AP.225144.02 used in feeding study)
Analytes	Fluazaindolizine, IN-AS5760, IN-F4106, IN-REG72, IN-QEK31, IN-R2W56 and IN-RYC33	
Extraction	Samples are homogenized and extracted with 0.01 M ammonium formate in 9:1 acetonitrile:water.	
Clean-up	The resulting extracts were cleaned-up using sequential dispersive SPE steps prior to LC-MS/MS analysis. A Strong Cation Exchange (SCX) step was followed by Strong Anion Exchange/C18/formic acid step to reduce matrix interferences. The cleaned-up extracts were concentrated under N_2 and diluted with water:formic acid (100:0.01 equivalent to 0.003 M formic acid)	
Analytical column	Phenomenex Kinetex Biphenyl column, 4.6×100 mm. 2.6 micron particle size	Agilent Zorbax Eclipse Plus® Phenyl-Hexyl column, 2.1×50 mm, 1.8 micron particle size
Mobile phase	Gradient mobile phase from water:methanol (75:25) with 0.01 % formic acid to methanol with 0.01 % formic acid	Gradient mobile phase water and 0.01 % formic acid in methanol
Ionization	ESI in the negative ion mode for fluazaindolizine, IN-AS5760, IN-F4106, IN-REG72 and the positive ion mode for IN-QEK31, IN-R2W56 and IN-RYC33	

Method	Charles River AP.225144.02 used in feeding study (modification of DuPont 39226) Du-Pont 42572	DuPont 39226 (see also modification Charles River AP.225144.02 used in feeding study)
Transitions	Fluazaindolizine: 466 → 157 (quantification) and 466 → 142 (confirmation) IN-AS5760: 206 → 122 (quantification) and 206 → 142 (confirmation) IN-F4106 : 220 → 156 (quantification) and 220 → 141 (confirmation) IN-QEK31: 265 → 219, (quantification) and 265 → 184 (confirmation) IN-R2W56: 279 → 247 (quantification) and 279 → 219 (confirmation) IN-REG72: 452 → 123 (quantification) and 452 → 244 (confirmation) IN-RYC33: 264 → 247 (quantification) and 264 → 192 (confirmation)	Fluazaindolizine: 466 → 157 (quantification) and 466 → 142 (confirmation) IN-AS5760: 206 → 142 (quantification) and 206 → 122 (confirmation) IN-F4106 : 220 → 156 (quantification) and 220 → 141 (confirmation) IN-QEK31: 265 → 184, (quantification) and 265 → 219 (confirmation) IN-R2W56: 279 → 247 (quantification) and 279 → 219 (confirmation) IN-REG72: 452 → 123 (quantification) and 452 → 244 (confirmation) IN-RYC33: 264 → 157 (quantification) and 264 → 184 (confirmation)
LOQ	0.01 mg/kg per analyte before adjusting for parent equivalent	
Linearity (r^2)	0.1-25 ng/mL r^2≥0.9932 for solvent standards and 0.9927 for fat matrix-matched standards	0.05-5 ng/mL r^2≥0.9997

Method DuPont-39226, Revision No. 1

Method DuPont-39226, Revision No. 1 (Klems, 2017) was developed for the detection, quantification, and confirmation of residues of fluazaindolizine and its metabolites IN-REG72, IN-QEK31, IN-F4106, IN-A5760 and IN-RYC33 in milk (cream, skim and whole), chicken eggs (yolks and whites), bovine muscle (ground beef), beef fat and beef liver.

The recovery data reported in the method for determining fluazaindolizine residues in animal tissues are summarised in Table 120. Recoveries outside the 70-120 percent range occurred for liver fortified at 0.01 mg/kg with fluazaindolizine (69 percent) and IN-QEK31 (65 percent); muscle fortified with IN-QEK31 at 0.01 mg/kg (68 percent); whole milk fortified with fluazaindolizine at the 0.01 mg/kg (123 percent) and 0.1 mg/kg (121 percent); whole milk fortified with IN-RYC33 at 0.01 mg/kg (64 percent); skim milk fortified with fluazaindolizine at 0.01 mg/kg (125 percent); and cream fortified with fluazaindolizine at 0.01 mg/kg (129 percent) and 0.1 mg/kg (125 percent). At the 0.010 and 0.1 mg/kg fortification levels, the mean recoveries at each level were within the range of 77–117 percent, with RSDs of 1–15 percent. Good linearity was observed in the range of 0.05 to 5.0 ng/mL for fluazaindolizine and its metabolites IN-REG72, IN-RYC33, IN-QEK31, IN-A5760 and IN-F4106. These ranges correspond to residue values of approximately 0.005-0.5 mg/kg. At least five-point standard curves were prepared utilizing standards in solvent with r2 ≥0.9997.

The LOQ of the method proposed for quantifying fluazaindolizine and metabolites is 0.01 mg/kg. Analysis of control samples consistently showed no detectable residues of fluazaindolizine or its metabolites. The response in the area of the fluazaindolizine and its metabolite peaks always corresponded to less than 30 percent of the LOQ. Relative standard deviations of less than 20 percent were consistently obtained for fortifications made at 0.01 mg/kg for each matrix and 0.1 mg/kg.

Table 120 Recovery data for the analytical method for the determination of fluazaindolizine residues in commodities of animal origin (Klems, 2017 DuPont-39226, Revision No. 1)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			Fluazaindolizine: 466 → 157 m/z		IN-REG72: 452 → 123 m/z	
Liver	0.01	5	88	13	96	6
	0.1	5	95	4	96	4
Beef Fat	0.01	5	106	8	100	2

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
	0.1	5	114	3	111	4
Beef Muscle	0.01	5	101	3	86	5
	0.1	5	101	2	93	3
Egg Yolk	0.01	5	95	9	86	9
	0.1	5	102	4	93	4
Egg White	0.01	5	99	8	98	4
	0.1	5	102	4	108	2
Whole Milk	0.01	5	113	2	110	2
	0.1	5	117	2	111	3
Skim Milk	0.01	5	105	4	96	1
	0.1	5	102	3	101	2
Heavy Cream	0.01	5	111	4	95	5
	0.1	5	109	2	107	1
			IN-RYC33: 264 → 157 m/z		IN-F4106: 220 → 156 m/z	
Liver	0.01	5	95	5	96	5
	0.1	5	92	2	94	2
Beef Fat	0.01	5	92	3	95	4
	0.1	5	90	2	100	3
Beef Muscle	0.01	5	88	4	99	2
	0.1	5	92	2	94	2
Egg Yolk	0.01	5	96	6	94	4
	0.1	5	97	3	95	2
Egg White	0.01	5	99	6	103	5
	0.1	5	101	1	104	2
Whole Milk	0.01	5	103	3	101	5
	0.1	5	110	4	104	2
Skim Milk	0.01	5	97	5	102	6
	0.1	5	99	3	95	2
Heavy Cream	0.01	5	102	4	106	6
	0.1	5	103	1	105	4
			IN-QEK31: 265 → 184 m/z		IN-A5760: 206 → 142 m/z	
Liver	0.01	5	88	6	111	7
	0.1	5	84	4	107	2
Beef Fat	0.01	5	88	7	100	6
	0.1	5	92	3	104	2
Beef Muscle	0.01	5	77	10	112	4
	0.1	5	82	4	113	2
Egg Yolk	0.01	5	77	6	93	3
	0.1	5	87	6	95	3
Egg White	0.01	5	90	5	103	5
	0.1	5	94	4	103	2
Whole Milk	0.01	5	105	7	114	4
	0.1	5	106	4	112	3
Skim Milk	0.01	5	103	13	100	2
	0.1	5	106	2	93	2
Heavy Cream	0.01	5	92	15	106	8
	0.1	5	101	1	114	3

The method's reproducibility was demonstrated by an independent laboratory validation (Xu, 2012, DuPont-44351) for the determination of fluazaindolizine and metabolites in liver, eggs and muscle. The recovery data is summarised in Table 121.

The ILV laboratory modified the solution used for preparation of analytical standards from 0.1 M aqueous formic acid: methanol (9:1) to 0.01 M formic acid in water:acetonitrile (80:20) containing 0.001

M ammonium formate and the solution for final dilution of extracts from 0.1M aqueous formic acid to 12.2 percent acetonitrile in water. In addition the amount of SCX sorbent was reduced for eggs and linear regression used for calculating analytical results.

Table 121 Independent laboratory validation data for the analytical method for the determination of fluazaindolizine residues in animal tissue (Xu, 2017 DuPont-44351)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			Fluazaindolizine: 466 → 157 m/z		IN-REG72: 452 → 123 m/z	
Egg (Chicken)	0.01	5	111	3.1	96	3
	0.1	5	102	1.8	102	2.3
Liver (Bovine)	0.01	5	94	21	85	4.4
	0.1	5	94	4.1	83	12
Muscle (Bovine)	0.01	5	117	4.6	109	2.9
	0.1	5	114	1.4	112	1.6
			IN-RYC33: 264 → 157 m/z		IN-F4106 220 m/z → 156 m/z	
Egg (Chicken)	0.01	5	85	6.8	91	5.5
	0.1	5	97	1.9	99	3.7
Liver (Bovine)	0.01	5	95	5.3	95	4.3
	0.1	5	100	1.4	99	3.1
Muscle (Bovine)	0.01	5	97	4.4	94	5.7
	0.1	5	100	1.4	102	2.3
			IN-QEK31 265 m/z → 184 m/z		IN-A5760 206 m/z → 142 m/z	
Egg (Chicken)	0.01	5	77	5.8	101	12
	0.1	5	76	1.4	101	5.6
Liver (Bovine)	0.01	5	81	9.4	114	3.9
	0.1	5	86	2	122	2.6
Muscle (Bovine)	0.01	5	92	2.4	108	9.6
	0.1	5	82	0.8	107	4.3

Extraction efficiency

The solvents and procedures for extracting fluazaindolizine and its metabolites from animal matrices in this method are the same as those used in the radio-validated extractions performed in DuPont-33572, Revision No. 1 and DuPont-33573, Revision No. 1 (Extraction efficiency of 9/1 acetonitrile/0.1 M aqueous ammonium formate) and ranged from 80.4 to 99.2 percent TRR, mean 91.4 percent TRR.

Confirmatory method

Confirmation of results obtained by LC-MS/MS method DuPont-39226 was performed by quantifying a separate daughter ion signal for each analyte. The recovery data obtained using the confirmatory procedure are summarised in Table 122.

Table 122 Confirmation data for the analytical method for the determination of fluazaindolizine residues in processed commodities origin (Klems, 2017 DuPont-39226, Revision No. 1)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			Fluazaindolizine: 466 → 142 m/z		IN-REG72: 452 → 244 m/z	
Liver	0.01	5	85	11	98	4
	0.1	5	85	9	94	3
Beef Fat	0.01	5	98	8	103	4
	0.1	5	102	7	105	1
Beef Muscle	0.01	5	96	8	82	13

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
	0.1	5	98	6	94	3
Egg Yolk	0.01	5	95	13	81	5
	0.1	5	96	9	88	6
Egg White	0.01	5	109	6	95	7
	0.1	5	107	5	101	2
Whole Milk	0.01	5	111	9	108	4
	0.1	5	111	7	117	1
Skim Milk	0.01	5	112	8	101	8
	0.1	5	102	3	103	2
Heavy Cream	0.01	5	116	9	105	8
	0.1	5	115	7	114	4
			IN-RYC33: 264 → 184 m/z		IN-F4106: 220 → 141 m/z	
Liver	0.01	5	97	10	100	3
	0.1	5	89	4	96	3
Beef Fat	0.01	5	90	8	96	8
	0.1	5	90	3	102	2
Beef Muscle	0.01	5	88	11	93	5
	0.1	5	83	4	96	1
Egg Yolk	0.01	5	99	9	98	5
	0.1	5	93	4	97	2
Egg White	0.01	5	100	2	99	4
	0.1	5	100	4	104	2
Whole Milk	0.01	5	89	19	106	4
	0.1	5	100	3	101	4
Skim Milk	0.01	5	96	13	98	6
	0.1	5	91	5	97	1
Heavy Cream	0.01	5	99	11	96	14
	0.1	5	101	4	96	3
			IN-QEK31: 265 → 219 m/z		IN-A5760: 206 → 122 m/z	
Liver	0.01	5	80	11	110	3
	0.1	5	77	2	107	2
Beef Fat	0.01	5	110	9	102	4
	0.1	5	96	2	105	1
Beef Muscle	0.01	5	83	10	117	4
	0.1	5	87	5	116	1
Egg Yolk	0.01	5	82	9	97	3
	0.1	5	78	6	99	3
Egg White	0.01	5	89	12	105	3
	0.1	5	90	4	102	1
Whole Milk	0.01	5	103	8	112	3
	0.1	5	101	3	114	2
Skim Milk	0.01	5	105	3	100	4
	0.1	5	101	3	101	2
Heavy Cream	0.01	5	97	12	111	5
	0.1	5	92	1	116	2

Method DuPont-42572, Revision No. 1

Method DuPont-42572 developed for the determination of fluazaindolizine and metabolites is a modification of method DuPont 39226 and it was used used in feeding studies. Recovery data is shown in Table 123

Table 123 Recovery data for the analytical method for the determination of fluazaindolizine residues in animal commodities (Harris, 2018 DuPont-42572, Revision No. 1)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			Fluazaindolizine: 466 → 157 m/z		IN-REG72: 452 → 123 m/z	
Bovine Muscle	0.01	5	117	5	95	5
	0.1	5	109	9	90	4
Bovine Liver	0.01	5	108	3	91	11
	0.1	5	101	4	82	4
Bovine Fat	0.01	5	100	9	104	8
	0.1	5	91	7	96	6
Bovine Whole Milk	0.01	5	105	6	94	16
	0.1	5	98	4	101	9
Bovine Skimmed Milk	0.01	5	87	12	96	10
	0.1	5	89	14	94	8
Bovine Cream	0.01	5	97	3	98	3
	0.1	5	95	3	94	4
Chicken Egg White	0.01	5	112	4	109	8
	0.1	5	98	8	95	6
Chicken Egg Yolk	0.01	5	102	7	93	7
	0.1	5	96	5	83	5
			IN-RYC33: 264 → 247 m/z		IN-F4106: 220 → 156 m/z	
Bovine Muscle	0.01	5	104	3	99	10
	0.1	5	102	1	97	3
Bovine Liver	0.01	5	104	1	94	3
	0.1	5	100	2	92	3
Bovine Fat	0.01	5	99	7	93	8
	0.1	5	94	8	94	4
Bovine Whole Milk	0.01	5	109	2	102	5
	0.1	5	107	1	104	2
Bovine Skimmed Milk	0.01	5	105	3	108	4
	0.1	5	103	1	100	3
Bovine Cream	0.01	5	105	3	99	4
	0.1	5	102	3	93	5
Chicken Egg White	0.01	5	103	5	103	14
	0.1	5	96	8	94	4
Chicken Egg Yolk	0.01	5	98	5	101	14
	0.1	5	88	3	95	2
			IN-QEK31: 265 → 219 m/z		IN-A5760: 206 → 122 m/z	
Bovine Muscle	0.01	5	86	5	99	4
	0.1	5	87	1	97	5
Bovine Liver	0.01	5	95	3	100[A]	5
	0.1	5	87	3	95	5
Bovine Fat	0.01	5	91	7	98	5
	0.1	5	88	4	95	5
Bovine Whole Milk	0.01	5	96	4	101	5
	0.1	5	98	2	106	3
Bovine Skimmed Milk	0.01	5	93	5	97	5
	0.1	5	93	1	98	2
Bovine Cream	0.01	5	94	2	93	4

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
	0.1	5	91	4	92	4
Chicken Egg White	0.01	5	91	10	102	3
	0.1	5	94	16	93	4
Chicken Egg Yolk	0.01	5	78	6	99	4
	0.1	5	74	4	90	3
			IN-R2W56: 279 → 247 m/z			
Bovine Muscle	0.01	5	99	4		
	0.1	5	97	2		
Bovine Liver	0.01	5	101	2		
	0.1	5	93	3		
Bovine Fat	0.01	5	93	8		
	0.1	5	87	13		
Bovine Whole Milk	0.01	5	106	2		
	0.1	5	104	2		
Bovine Skimmed Milk	0.01	5	99	2		
	0.1	5	97	1		
Bovine Cream	0.01	5	101	1		
	0.1	5	96	3		
Chicken Egg White	0.01	5	109	5		
	0.1	5	100	8		
Chicken Egg Yolk	0.01	5	106	5		
	0.1	5	93	3		

Notes:

[A] Value of 918 percent considered an outlier and not included in the calculation of mean and % RSD.

The method's reproducibility was demonstrated by an independent laboratory validation for fluazaindolizine, IN-REG72, IN-QEK31, IN-F4106, IN-RYC33 and IN-A5760. The recovery data obtained during the ILV is summarised in Table 124. IN-R2W56 was not included in the ILV as the residue of this molecule was deemed to be insignificant.

Table 124 Independent laboratory recovery data for the analytical method for the determination of fluazaindolizine residues in animal tissue (Xu, 2017 DuPont-44351)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			Fluazaindolizine: 466 → 157 m/z		IN-REG72: 452 → 123 m/z	
Egg (Chicken)	0.01	5	111	3.1	96	3
	0.1	5	102	1.8	102	2.3
Liver (Bovine)	0.01	5	94	21	85	4.4
	0.1	5	94	4.1	83	12
Muscle (Bovine)	0.01	5	117	4.6	109	2.9
	0.1	5	114	1.4	112	1.6
			IN-RYC33: 264 → 157 m/z		IN-F4106: 220 → 156 m/z	
Egg (Chicken)	0.01	5	85	6.8	91	5.5
	0.1	5	97	1.9	99	3.7
Liver (Bovine)	0.01	5	95	5.3	95	4.3
	0.1	5	100	1.4	99	3.1
Muscle (Bovine)	0.01	5	97	4.4	94	5.7
	0.1	5	100	1.4	102	2.3
			IN-QEK31: 265 → 184 m/z		IN-A5760: 206 → 142 m/z	
Egg (Chicken)	0.01	5	77	5.8	101	12

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
	0.1	5	76	1.4	101	5.6
Liver (Bovine)	0.01	5	81	9.4	114	3.9
	0.1	5	86	2	122	2.6
Muscle (Bovine)	0.01	5	92	2.4	108	9.6
	0.1	5	82	0.8	107	4.3

Confirmatory method

Confirmation of results obtained by this LC-MS/MS method was performed by quantifying a separate daughter ion signal for each analyte. The recovery data obtained using the confirmatory procedure are summarised in Table 125. Individual mean recoveries for samples fortified at 0.01 mg/kg or 0.1 mg/kg were 81–113 percent with RSDs 1–16 percent.

Table 125 Confirmation data for the analytical method for the determination of fluazaindolizine residues in animal tissues (Harris, 2018 DuPont-42572, Revision No. 1)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			Fluazaindolizine: 466 → 142 m/z		IN-REG72: 452 → 244 m/z	
Bovine Muscle	0.01	5	113	6	98	5
	0.1	5	108	9	91	4
Bovine Liver	0.01	5	97	3	93	12
	0.1	5	100	4	81	2
Bovine Fat	0.01	5	100	8	102	6
	0.1	5	90	6	96	6
Bovine Whole Milk	0.01	5	102	1	94	15
	0.1	5	98	5	99	8
Bovine Skimmed Milk	0.01	5	87	13	94	7
	0.1	5	89	13	94	8
Bovine Cream	0.01	5	94	4	99	4
	0.1	5	95	3	94	5
Chicken Egg White	0.01	5	112	5	110	12
	0.1	5	96	7	97	6
Chicken Egg Yolk	0.01	5	102	4	93	6
	0.1	5	93	4	82	4
			IN-RYC33: 264 → 192 m/z		IN-F4106: 220 → 141 m/z	
Bovine Muscle	0.01	5	102	2	100	6
	0.1	5	103	2	99	3
Bovine Liver	0.01	5	104	2	95	8
	0.1	5	98	2	95	3
Bovine Fat	0.01	5	97	7	98	7
	0.1	5	91	9	96	3
Bovine Whole Milk	0.01	5	108	3	101	6
	0.1	5	108	1	108	3
Bovine Skimmed Milk	0.01	5	102	3	101	3
	0.1	5	103	2	102	2
Bovine Cream	0.01	5	107	2	97	4
	0.1	5	102	4	94	4
Chicken Egg White	0.01	5	106	6	102	6
	0.1	5	97	8	96	2
Chicken Egg Yolk	0.01	5	98	3	102	9
	0.1	5	87	2	95	3
			IN-QEK31: 265 → 184 m/z		IN-A5760: 206 → 142 m/z	

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
Bovine Muscle	0.01	5	86	6	97	4
	0.1	5	87	2	100	6
Bovine Liver	0.01	5	95	3	102[A]	6
	0.1	5	89	4	94	3
Bovine Fat	0.01	5	92	7	97	9
	0.1	5	88	4	94	5
Bovine Whole Milk	0.01	5	95	1	102	11
	0.1	5	97	1	105	2
Bovine Skimmed Milk	0.01	5	91	2	105	5
	0.1	5	92	2	99	2
Bovine Cream	0.01	5	96	3	91	4
	0.1	5	92	4	91	4
Chicken Egg White	0.01	5	91	8	107	7
	0.1	5	95	16	93	3
Chicken Egg Yolk	0.01	5	77	5	97	4
	0.1	5	73	5	92	1
			IN-R2W56: 279 → 219 m/z			
Bovine Muscle	0.01	5	98	4		
	0.1	5	95	1		
Bovine Liver	0.01	5	101	1		
	0.1	5	92	3		
Bovine Fat	0.01	5	93	7		
	0.1	5	87	13		
Bovine Whole Milk	0.01	5	103	2		
	0.1	5	101	2		
Bovine Skimmed Milk	0.01	5	96	2		
	0.1	5	94	2		
Bovine Cream	0.01	5	99	3		
	0.1	5	94	3		
Chicken Egg White	0.01	5	110	5		
	0.1	5	100	8		
Chicken Egg Yolk	0.01	5	106	4		
	0.1	5	94	3		

Notes:

[A] Value of 891 percent considered an outlier and not included in the calculation of mean and percent RSD.

The analytical method validation described in DuPont-44365 (Čermák and Kwiecien, 2017) was performed for the detection, quantification, and confirmation of residues of fluazaindolizine and its metabolites IN-REG72, IN-QEK31, IN-F4106, IN-A5760, and IN-RYC33 in milk, egg, bovine meat, fat, and liver, using Multi-Residue Method DFG-S19. The method LOQ was 0.01 mg/kg and the LOD was 30 percent of the LOQ, or 0.003 mg/kg.

Samples were extracted with acetone using a homogenizer. Water is added beforehand in an amount that takes into account the natural water content of the specimen so that during extraction, the acetone/water ratio remains constant at 2/1. For milk, a liquid-liquid extraction was performed using sodium chloride and dichloromethane. Eggs and tissues samples were previouly homogenized with a mixture of ethyl acetate/cyclohexane (1/1) and sodium chloride and fat tissue samples were dissolved in a mixture of ethyl acetate and cyclohexane (1/1). Following the separation of phases, an aliquot of the organic phase was evaporated and then cleaned-up by GPC on Bio Beads S-X3 (polystyrene gel) using a mixture of ethyl acetate/cyclohexane (1/1) as the eluent. The GPC fractions were concentrated, re-dissolved in the HPLC solvent and analysed by LC-MS/MS. Validation data is shown in Table 126.

Table 126 Validation data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in commodities of animal origin (Čermák, Kwiecien, 2017 DuPont-44365)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			Fluazaindolizine: 466 → 157 m/z		IN-REG72: 452 → 123 m/z	
Egg	0.01	5	0	0	0	0
	0.1	5	14	11.8	0	0
Milk	0.01	5	73	5.8	62	21
	0.1	5	76	5.8	58	16
Bovine Meat	0.01	5	50	24	0	0
	0.1	5	31	31	40	24
Bovine Fat	0.01	5	0	0	0	0
	0.1	5	0	0	0	0
			IN-QEK31: 265 → 247 m/z		IN-F4106: 220 → 156 m/z	
Egg	0.01	5	0	0	70	5.9
	0.1	5	0	0	72	4.2
Milk	0.01	5	0	0	84	5.1
	0.1	5	8	37	80	4.4
Bovine Meat	0.01	5	34	20	89	12
	0.1	5	26	24	95	2.1
Bovine Fat	0.01	5	0	0	90	2.9
	0.1	5	0	0	84	1.6
			IN-A5760: 206 → 122 m/z		IN-RYC33: 264 → 247 m/z	
Egg	0.01	5	80	4.5	89	3.0
	0.1	5	81	6.0	92	4.3
Milk	0.01	5	87	1.2	85	2.6
	0.1	5	81	3.6	84	3.8
Bovine Meat	0.01	5	80	4.5	85	19
	0.1	5	81	6.0	97	4.8
Bovine Fat	0.01	5	96	3.4	94	1.5
	0.1	5	92	1.8	91	2.2

Confirmatory method

Confirmation of results obtained by this LC-MS/MS method was performed by quantifying a separate daughter ion signal for each analyte (Table 127).

Table 127 Confirmatory data for the analytical method for the determination of residues of fluazaindolizine and its metabolites in commodities of animal origin (Čermák, Kwiecien, 2017 DuPont-44365)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			Fluazaindolizine: 468 → 247 m/z		IN-REG72: 454 → 247 m/z	
Egg	0.01	5	0	0	0	0
	0.1	5	13	9.8	0	0
Milk	0.01	5	77	5.3	65	22
	0.1	5	68	6.6	59	16
Bovine Meat	0.01	5	48	25	0	0
	0.1	5	32	31	41	27
Bovine Fat	0.01	5	0	0	0	0
	0.1	5	0	0	0	0
			IN-QEK31: 265 → 219 m/z		IN-F4106: 220 → 78 m/z	
Egg	0.01	5	0	0	71	5.3
	0.1	5	0	0	72	3.9

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
Milk	0.01	5	0	0	85	3.7
	0.1	5	8	35	79	4.6
Bovine Meat	0.01	5	35	16	89	11
	0.1	5	25	25	93	2.1
Bovine Fat	0.01	5	0	0	91	3.9
	0.1	5	0	0	84	1.8
			IN-A5760: 206 → 78 m/z		IN-RYC33: 264 → 219 m/z	
Egg	0.01	5	79	3.7	87	3.0
	0.1	5	83	5.6	89	4.3
Milk	0.01	5	86	3.8	83	3.8
	0.1	5	83	3.7	82	5.7
Bovine Meat	0.01	5	91	10	84	18
	0.1	5	96	4.1	95	5.6
Bovine Fat	0.01	5	98	2.1	96	2.1
	0.1	5	92	2.9	90	1.9

An ILV of the multiresidue method DFG S19 (DuPont 49359) for the determination of metabolites of fluazaindolizine in animal tissues by LC-MS/MS was reported by Schernikau and Colorado (2017). The results are shown in Table 128.

Table 128 Validation data for the method ILV for the determination of residues of the metabolites of fluazaindolizine in commodities of animal origin (Schernikau and Colorado, 2017 DuPont-49359)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			IN-F4106: 220 → 156 m/z		IN-A5760: 206 → 122 m/z	
Milk	0.01	5	105	2.1	110	3.1
	0.1	5	103	4.2	110	6.3
Bovine Meat	0.01	5	119	7.9	119	4.2
	0.1	5	99	13	99	15
Bovine Fat	0.01	5	106	2.1	108	2.0
	0.1	5	105	1.4	109	2.7
			IN-RYC33: 264 → 247 m/z			
Milk	0.01	5	104	5.4		
	0.1	5	92	4.0		
Bovine Meat	0.01	5	92	2.2		
	0.1	5	81	11		
Bovine Fat	0.01	5	119	2.3		
	0.1	5	106	2.0		

Confirmation of results obtained by this LC-MS/MS method was performed by quantifying a separate daughter ion signal for each analyte (Table 129)

Table 129 Confirmatory data for the method ILV for the determination of residues of the metabolites of fluazaindolizine in commodities of animal origin (Schernikau and Colorado, 2017 DuPont-49359)

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
			IN-F4106: 220 → 78 m/z		IN-A5760: 206 → 78 m/z	
Milk	0.01	5	107	3.0	109	5.4
	0.1	5	107	5.7	108	5.2
Bovine Meat	0.01	5	117	8.2	112	4.6
	0.1	5	98	12	96	13

Matrix	Fortification level (mg/kg)	N	Mean (%)	% RSD	Mean (%)	% RSD
Bovine Fat	0.01	5	106	3.8	106	0.9
	0.1	5	106	0.9	110	1.9
			IN-RYC33: 264 → 219 m/z			
Milk	0.01	5	101	4.3		
	0.1	5	95	4.0		
Bovine Meat	0.01	5	92	3.1		
	0.1	5	81	12		
Bovine Fat	0.01	5	118	3.1		
	0.1	5	118	2.6		

STABILITY OF PESTICIDES RESIDUES IN STORED ANALYTICAL SAMPLES

Rebstock (2021 DuPont-39883, Revision No. 1) studied the freezer storage stability of crops fortified with fluazaindolizine and metabolites. Separate representative homogenised control crop sample replicates were fortified with 0.2 mg/kg each of fluazaindolizine and metabolites (IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-R2W56, IN-REG72, IN-RYC33, IN-TMQ01, IN-UJV12, and IN-UNS90). Samples were stored at approximately -20 °C. Samples were analysed using procedures based on DuPont-33861, Revision No. 3.

Analysis of crop samples for fluazaindolizine-related residues involves extraction followed by quantification of fluazaindolizine and seven metabolites (IN-F4106, IN-QEK31, IN-QZY47, IN-R2W56, IN-REG72, IN-TMQ01 (IN-RSU03), and IN-RYC33). An aliquot of the extract then undergoes a hydrolysis procedure and SPE clean-up prior to a second analysis for seven metabolites (IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-TMQ01 (IN-RSU03), IN-UJV12 and IN-UNS90). The post-hydrolysis analysis was not included in this study as there was no freezer storage of samples after hydrolysis. The time intervals between fortification of samples for freezer storage and analysis of 0-day time point samples ranged from 0 to 3 days depending on the analyte and matrix combinations, with the exception of IN-RYC33 in dried pea seeds at day zero that was re-analysed 16 days after fortification.

Residues of fluazaindolizine and metabolites (IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-R2W56, IN-REG72, IN-RYC33, IN-TMQ01, IN-UJV12, and IN-UNS90) are stable in representative crop matrices for at least 23 months (pea hay), 24 months (wheat grain, oranges, field corn stover, and dried pea seeds), or 33 months (tomato and soya bean seed) when stored at -20 °C. The results are shown in Table 130.

Table 130 Summary of fluazaindolizine and metabolite residues in crop samples fortified at 0.20 mg/kg and stored at approximately -20 °C

Storage interval (months)	Residue (mg/kg)		Procedural recovery (%)		Residue (mg/kg)		Procedural recovery (%)	
Tomato								
	Fluazaindolizine				IN-AS5760			
0	0.18	0.19	-	-	0.18	0.19	-	-
3	0.17	0.15	91	91	0.19	0.18	94	90
6	0.16	0.16	95	96	0.19	0.19	94	93
12	0.16	0.16	99	98	0.19	0.19	94	95
18	0.16	0.17	103	104	0.20	0.21	100	104
24	0.16	0.16	101	99	0.20	0.20	102	101
33+	0.15	0.16	96	98	0.19	0.21	96	106
	IN-F4106				IN-QEK31			
0	0.18	0.18	-	-	0.18	0.18	-	-
3	0.18	0.18	91	90	0.21	0.22	104	108

Storage interval (months)	Residue (mg/kg)		Procedural recovery (%)		Residue (mg/kg)		Procedural recovery (%)	
6	0.19	0.18	96	92	0.20	0.20	102	99
12	0.19	0.19	95	96	0.19	0.19	95	95
18	0.19	0.20	96	98	0.20	0.21	102	103
24	0.20	0.20	98	99	0.20	0.21	98	103
33+	0.20	0.20	98	99	0.20	0.20	99	101
	IN-QZY47				IN-R2W56			
0	0.18	0.18	-	-	0.18	0.19	-	-
3	0.21	0.21	104	108	0.19	0.18	93	89
6	0.19	0.19	102	99	0.19	0.19	96	97
12	0.19	0.19	95	95	0.20	0.19	99	96
18	0.20	0.21	102	103	0.21	0.21	105	106
24	0.19	0.19	98	103	0.21	0.21	105	104
33+	0.19	0.21	99	101	0.20	0.20	101	100
	IN-REG72				IN-RYC33			
0	0.18	0.18	-	-	0.19	0.19	-	-
3	0.19	0.18	93	90	0.18	0.18	92	91
6	0.20	0.21	105	101	0.19	0.19	97	97
12	0.20	0.19	98	96	0.18	0.19	89	93
18	0.20	0.20	100	102	0.20	0.20	100	102
24	0.21	0.21	103	104	0.20	0.20	100	101
33+	0.20	0.19	98	94	0.20	0.20	98	98
	IN-TMQ01				IN-UJV12			
0	0.18	0.18	-	-	0.19	0.19	-	-
3	0.18	0.18	91	90	0.20	0.21	102	105
6	0.19	0.19	94	95	0.19	0.19	96	95
12	0.18	0.19	92	94	0.18	0.18	92	92
18	0.20	0.21	101	104	0.20	0.21	98	104
24	0.19	0.19	96	93	0.20	0.20	101	100
33+	0.18	0.19	88	95	0.20	0.20	100	101
	IN-UNS90							
0	0.18	0.19	-	-				
3	0.21	0.20	105	99				
6	0.19	0.18	96	92				
12	0.19	0.19	95	95				
18	0.20	0.21	102	104				
24	0.20	0.19	99	97				
33+	0.19	0.20	93	101				
Wheat grain								
	Fluazaindolizine				IN-AS5760			
0	0.17	0.16	-	-	0.19	0.19	-	-
3	0.16	0.16	78	80	0.19	0.19	94	96
6	0.15	0.16	76	80	0.19	0.19	96	94
12	0.16	0.16	82	79	0.18	0.18	90	89
18	0.16	0.15	81	74	0.20	0.19	99	97
24	0.15	0.15	75	76	0.18	0.18	90	90
	IN-F4106				IN-QEK31			
0	0.18	0.19	-	-	0.17	0.17	-	-
3	0.19	0.19	97	97	0.19	0.19	94	97
6	0.19	0.19	96	94	0.19	0.18	96	92
12	0.17	0.17	85	84	0.17	0.17	86	83
18	0.19	0.18	94	91	0.19	0.19	97	95
24	0.19	0.18	93	90	0.18	0.18	90	92
	IN-QZY47				IN-R2W56			
0	0.17	0.17	-	-	0.18	0.19	-	-

Storage interval (months)	Residue (mg/kg)		Procedural recovery (%)		Residue (mg/kg)		Procedural recovery (%)	
3	0.18	0.18	90	92	0.19	0.19	95	96
6	0.18	0.18	91	92	0.19	0.19	97	94
12	0.17	0.16	84	82	0.18	0.18	91	89
14	0.19	0.19	93	94				
18	0.19	0.19	95	93	0.21	0.20	106	98
24	0.18	0.18	89	90	0.18	0.18	89	92
	IN-REG72				IN-RYC33			
0	0.15	0.15	-	-	0.16	0.17	-	-
3	0.17	0.17	86	87	0.19	0.19	94	95
6	0.16	0.16	81	82	0.19	0.18	95	89
12	0.15	0.15	75	75	0.18	0.17	88	85
14	0.17	0.16	84	81				
18	0.16	0.15	79	74	0.20	0.19	98	96
24	0.15	0.16	76	82	0.18	0.18	91	91
	IN-TMQ01				IN-UJV12			
0	0.18	0.18	-	-	0.16	0.16	-	-
3	0.18	0.18	90	88	0.18	0.18	91	90
6	0.18	0.18	88	91	0.18	0.18	89	90
12	0.17	0.17	85	84	0.17	0.17	86	83
14					0.18	0.19	90	94
18	0.17	0.17	86	85	0.20	0.18	98	92
24	0.17	0.16	85	81	0.18	0.18	92	88
	IN-UNS90							
0	0.16	0.16	-	-				
3	0.18	0.18	90	90				
6	0.18	0.18	92	91				
12	0.17	0.17	84	83				
18	0.18	0.18	90	89				
24	0.18	0.17	89	86				
Orange								
	Fluazaindolizine				IN-AS5760			
0	0.19	0.20	-	-	0.19	0.19	-	-
3	0.18	0.18	88	89	0.17	0.18	87	88
6	0.20	0.20	101	100	0.20	0.20	99	98
12	0.19	0.20	95	98	0.19	0.19	95	95
18	0.20	0.20	101	102	0.19	0.19	97	97
24	0.21	0.21	104	104	0.21	0.20	104	102
	IN-F4106				IN-QEK31			
0	0.19	0.19	-	-	0.21	0.21	-	-
3	0.17	0.18	87	90	0.19	0.19	93	94
6	0.20	0.20	99	99	0.20	0.20	99	100
12	0.19	0.18	96	92	0.19	0.19	93	95
18	0.20	0.20	98	100	0.20	0.19	102	97
24	0.21	0.20	103	98	0.20	0.20	102	99
	IN-QZY47				IN-R2W56			
0	0.20	0.21	-	-	0.20	0.20	-	-
3	0.19	0.19	93	93	0.18	0.18	91	91
6	0.20	0.20	100	100	0.19	0.19	96	97
12	0.18	0.19	92	93	0.19	0.19	94	94
18	0.19	0.19	95	96	0.20	0.20	100	99
24	0.20	0.19	100	94	0.19	0.19	97	94
	IN-REG72				IN-RYC33			
0	0.20	0.20	-	-	0.20	0.20	-	-
3	0.18	0.18	88	89	0.18	0.18	91	92

Storage interval (months)	Residue (mg/kg)		Procedural recovery (%)		Residue (mg/kg)		Procedural recovery (%)	
6	0.20	0.20	102	102	0.20	0.20	98	98
12	0.20	0.19	98	97	0.19	0.19	93	93
18	0.20	0.20	99	101	0.20	0.20	99	99
24	0.20	0.21	102	104	0.19	0.19	95	96
	IN-TMQ01				IN-UJV12			
0	0.20	0.19	-	-	0.21	0.21	-	-
3	0.17	0.17	85	86	0.18	0.19	91	94
6	0.19	0.20	97	98	0.19	0.20	96	99
12	0.18	0.19	92	94	0.19	0.19	96	94
18	0.19	0.20	97	101	0.20	0.20	98	98
24	0.20	0.19	98	95	0.19	0.19	97	95
	IN-UNS90							
0	0.21	0.20	-	-				
3	0.19	0.19	93	93				
6	0.20	0.20	98	100				
12	0.19	0.19	94	95				
18	0.19	0.20	97	100				
24	0.20	0.19	100	93				
Field corn stover								
	Fluazaindolizine				IN-AS5760			
0	0.15	0.15	-	-	0.16	0.15	-	-
3	0.17	0.17	86	85	0.19	0.18	94	89
6	0.18	0.18	92	90	0.18	0.18	90	92
12	0.16	0.19	82	93	0.18	0.18	88	90
18	0.16	0.16	79	78	0.18	0.18	88	91
24	0.16	0.15	79	77	0.18	0.17	89	87
	IN-F4106				IN-QEK31			
0	0.15	0.16	-	-	0.16	0.15	-	-
3	0.17	0.19	86	94	0.18	0.17	89	86
6	0.17	0.20	86	101	0.18	0.18	91	89
12	0.18	0.18	92	88	0.17	0.18	86	89
18	0.17	0.17	87	85	0.18	0.18	90	90
24	0.18	0.17	91	83	0.18	0.18	90	91
	IN-QZY47				IN-R2W56			
0	0.14	0.14	-	-	0.15	0.14	-	-
3	0.16	0.16	81	81	0.18	0.17	88	86
6	0.15	0.15	76	74	0.16	0.17	82	87
12	0.15	0.16	75	81	0.17	0.18	87	89
18	0.16	0.16	81	81	0.16	0.18	82	88
24	0.16	0.16	80	79	0.18	0.18	88	89
	IN-REG72				IN-RYC33			
0	0.15	0.15	-	-	0.15	0.15	-	-
3	0.18	0.18	89	90	0.18	0.18	90	92
6	0.18	0.18	89	89	0.18	0.18	88	90
12	0.17	0.18	85	89	0.17	0.18	84	88
18	0.17	0.17	87	86	0.17	0.18	86	90
24	0.17	0.17	85	84	0.18	0.17	90	86
	IN-TMQ01				IN-UJV12			
0	0.17	0.16	-	-	0.15	0.15	-	-
3	0.20	0.19	98	94	0.17	0.16	83	80
6	0.18	0.17	90	87	0.16	0.15	78	75
12	0.17	0.18	87	90	0.15	0.16	77	82
18	0.17	0.17	83	84	0.16	0.16	82	82
24	0.17	0.16	82	82	0.16	0.17	81	83

Storage interval (months)	Residue (mg/kg)		Procedural recovery (%)		Residue (mg/kg)		Procedural recovery (%)	
	IN-UNS90							
0	0.17	0.16	-	-				
3	0.18	0.17	88	86				
6	0.18	0.17	91	84				
12	0.17	0.19	86	93				
18	0.17	0.18	87	90				
24	0.18	0.18	90	88				
Pea hay								
	Fluazaindolizine				IN-AS5760			
0	0.18	0.18	-	-	0.19	0.19	-	-
1week	0.20	0.20	98	100	0.19	0.20	97	101
1	0.19	0.19	95	93	0.19	0.18	95	91
3	0.19	0.18	94	91	0.19	0.19	96	95
6	0.19	0.18	95	92	0.19	0.18	94	92
12	0.19	0.19	96	94	0.19	0.20	94	98
18	0.19	0.19	94	95	0.20	0.20	101	101
22+	0.17	0.17	83	87	0.18	0.17	90	87
	IN-F4106				IN-QEK31			
0	0.18	0.19	-	-	0.19	0.19	-	-
1week	0.20	0.21	100	105	0.19	0.19	97	97
1	0.19	0.19	95	94	0.20	0.19	99	97
3	0.19	0.18	94	91	0.19	0.18	97	91
6	0.18	0.17	92	87	0.19	0.18	93	92
12	0.18	0.19	92	97	0.19	0.19	95	93
18	0.18	0.20	90	99	0.19	0.19	95	96
22+	0.18	0.18	89	89	0.19	0.19	95	94
	IN-QZY47				IN-R2W56			
0	0.19	0.17	-	-	0.18	0.19	-	-
1week	0.18	0.20	92	101	0.19	0.19	94	95
1	0.18	0.20	88	100	0.20	0.20	98	98
3	0.21	0.19	104	93	0.20	0.19	99	97
6	0.19	0.18	93	92	0.19	0.18	93	92
12	0.18	0.19	89	93	0.18	0.19	90	93
15	0.18	0.18	91	89				
18	0.18	0.19	90	95	0.19	0.19	94	95
22+	0.17	0.17	84	87	0.21	0.20	104	100
	IN-REG72				IN-RYC33			
0	0.18	0.17	-	-	0.19	0.19	-	-
1week	0.19	0.20	96	98	0.19	0.19	93	96
1	0.19	0.18	96	92	0.20	0.20	98	98
3	0.19	0.18	97	92	0.20	0.19	98	96
6	0.19	0.18	95	92	0.19	0.19	95	93
12	0.18	0.18	91	91	0.19	0.19	93	93
18	0.18	0.18	92	91	0.20	0.19	98	96
22+	0.18	0.18	89	89	0.20	0.19	100	97
	IN-TMQ01				IN-UJV12			
0	0.18	0.17	-	-	0.18 0.1	0.18 0.19	-	-
1week	0.18	0.19	88	93	0.18	0.18	90	91
1	0.18	0.18	89	92	0.18	0.18	91	92
3	0.20	0.19	99	93	0.22	0.20	109	99
6	0.17	0.17	85	87	0.19	0.18	94	91
12	0.19	0.19	93	94	0.18	0.19	90	95
					0.18	0.18	91	89
18	0.19	0.19	95	97	0.18	0.18	92	92

Storage interval (months)	Residue (mg/kg)		Procedural recovery (%)		Residue (mg/kg)		Procedural recovery (%)	
22+	0.16	0.17	78	86	0.18	0.17	92	84
	IN-UNS90							
0	0.19	0.19	-	-				
1week	0.18	0.19	90	95				
1	0.18	0.18	92	92				
3	0.22	0.19	111	95				
6	0.19	0.18	95	89				
12	0.19	0.19	95	95				
18	0.18	0.19	92	97				
22+	0.19	0.18	94	89				
Soya bean seeds								
	Fluazaindolizine				IN-AS5760			
0	0.16	0.16	-	-	0.16	0.16	-	-
3	0.18	0.16	90	82	0.20	0.19	102	94
4+								
6	0.16	0.16	82	82	0.19	0.19	94	93
12	0.18	0.17	91	85	0.17	0.17	86	87
18	0.18	0.18	89	90	0.20	0.20	99	98
24	0.17	0.17	87	86	0.19	0.18	93	92
33+	0.15	0.15	75	76	0.16	0.17	82	84
	IN-F4106				IN-QEK31			
0	0.18	0.17	-	-	0.18	0.19	-	-
3					0.19	0.17	97	83
4+	0.18	0.21	92	106				
6	0.19	0.19	96	95	0.21	0.20	103	99
12	0.18	0.18	90	90	0.19	0.19	93	94
18	0.19	0.19	93	95	0.19	0.19	94	96
24	0.19	0.18	93	92	0.19	0.19	94	97
33+	0.16	0.16	79	79	0.19	0.19	94	94
	IN-QZY47				IN-R2W56			
0	0.17	0.17	-	-	0.18	0.18	-	-
3					0.19	0.17	78	86
4+	0.19	0.18	93	91				
6	0.18	0.19	91	96	0.18	0.18	91	89
12	0.18	0.18	89	88	0.18	0.18	96	99
18	0.18	0.19	92	95	0.19	0.19	99	104
24	0.18	0.16	88	82	0.19	0.19	71	75
33+	0.15	0.16	76	80	0.18	0.18	116	119
	IN-REG72				IN-RYC33			
0	0.15	0.15	-	-	0.18	0.17	-	-
3	0.16	0.15	82	76				
4+					0.19	0.20	97	102
6	0.16	0.16	82	81	0.20	0.21	104	98
12	0.16	0.15	80	77	0.18	0.18	91	89
18	0.17	0.17	84	87	0.19	0.19	94	97
24	0.17	0.17	83	87	0.19	0.19	94	95
33+	0.15	0.15	74	73	0.19	0.18	93	90
	IN-TMQ01				IN-UJV12			
0	0.19	0.18	-	-	0.17	0.17	-	-
3	0.20	0.18	102	92				
4+					0.17	0.17	87	85
6	0.19	0.19	96	97	0.18	0.18	90	89
12	0.19	0.19	94	95	0.17	0.16	83	81
18	0.20	0.21	102	103	0.18	0.18	91	90

Storage interval (months)	Residue (mg/kg)		Procedural recovery (%)		Residue (mg/kg)		Procedural recovery (%)	
24	0.18	0.18	90	90	0.16	0.16	81	79
33+	0.16	0.16	81	79	0.15	0.15	75	74
	IN-UNS90							
0	0.19	0.18	-	-				
3	0.20	0.17	99	86				
6	0.20	0.20	100	102				
12	0.19	0.18	93	92				
18	0.20	0.19	101	95				
24	0.17	0.17	87	85				
33+	0.16	0.16	82	79				
Dried pea seeds								
	Fluazaindolizine				IN-AS5760			
0	0.18	0.18	-	-	0.18	0.17	-	-
3	0.18	0.20	92	100	0.18	0.21	90	106
6	0.19	0.19	96	96	0.18	0.18	92	91
12	0.19	0.18	93	92	0.16	0.17	79	85
18	0.19	0.20	95	99	0.19	0.19	97	95
24	0.18	0.18	92	92	0.17	0.17	87	86
	IN-F4106				IN-QEK31			
0	0.18	0.16	-	-	0.17	0.16	-	-
3	0.19	0.19	96	97	0.19	0.19	94	96
6	0.19	0.19	93	93	0.19	0.19	97	96
12	0.17	0.17	83	83	0.18	0.18	89	88
18	0.19	0.20	95	102	0.19	0.20	96	99
24	0.19	0.19	96	95	0.21	0.21	105	102
	IN-QZY47				IN-R2W56			
0	0.16	0.16	-	-	0.19	0.18	-	-
3	0.18	0.18	81	93	0.17	0.15	87	75
6	0.18	0.18	89	90	0.19	0.18	93	90
12	0.17	0.16	78	82	0.19	0.17	93	85
18	0.18	0.20	91	91	0.19	0.20	95	101
24	0.18	0.17	72	76	0.19	0.19	96	94
	IN-REG72				IN-RYC33			
0	0.19	0.18	-	-	0.13	0.16	-	-
3	0.18	0.21	92	103	0.17	0.16	84	78
6	0.18	0.18	89	91	0.19	0.19	93	97
12	0.18	0.18	90	89	0.17	0.17	87	85
18	0.18	0.19	88	95	0.19	0.20	95	98
24	0.21	0.19	104	93	0.20	0.20	102	99
	IN-TMQ01				IN-UJV12			
0	0.19	0.19	-	-	0.16	0.16	-	-
3	0.18	0.18	92	90	0.18	0.19	88	93
6	0.18	0.19	92	93	0.18	0.18	89	88
12	0.17	0.17	84	83	0.17	0.17	84	84
18	0.18	0.19	88	93	0.19	0.21	94	104
24	0.19	0.18	93	89	0.17	0.17	87	87
	IN-UNS90							
0	0.17	0.17	-	-				
3	0.18	0.19	89	96				
6	0.19	0.19	96	96				
12	0.17	0.16	87	81				
18	0.18	0.20	92	102				
24	0.19	0.18	95	90				

Maximum storage intervals for supervised residue trials are listed in Table 131

Table 131 Maximum periods of frozen storage before extraction for residue analysis

	Maximum periods of frozen storage before analysis	
	Days	Months
Cucurbit vegetables	470[A]	15.4
Fruiting vegetables	683	22.5
Carrots	152	5.0
Potatoes	481	15.8
Tomato processing	616	20.2
Potato processing	391	12.8
Wheat processing	197	6.5
Field corn processing	217	7.1
Soya bean processing	127	4.2
Strawberry processing	313	10.3
Row crop/ crop rotation North American region	619[B]	20.3[B]
Row crop/ crop rotation Europe	907[C]	31[C,D]
Vegetable and fruit crop rotation North America region	616	20.2
Vegetable and fruit crop rotation Europe	647	22
Limited field crop rotation North America region	881	28.9
Limited field crop rotation Europe	–	24
Demonstrated stability	Days	Months
High water (tomatoes)		33
High acid (oranges)		24
Very dry (field corn stover, pea hay)		24/23
High starch (wheat grain)		24
High oil (soya bean seeds)		33
High protein (pea seeds, dry)		24

Notes:

[A] 443 days or about 14.6 months for analysis of two melon peel samples.

[B] 18 exceptions for re-analysis of samples. Maximum storage intervals for reanalysis of samples were 631 days/20.8 months for soya beans, 1001 days/32.9 months for peas and 812 days/26.6 months for corn. No residue values were changed as a result of reanalysis of samples.

[C] Maximum of 25 months for pea forage, dried peas, oilseed rape straw, maize forage, maize immature ears, maize grain, maize stover, wheat hay and wheat grain. A maximum 29 months for pea vines, pea hay, oilseed rape seed, oilseed rape forage and wheat straw and a maximum of 31 months for wheat forage.

[D] The majority of samples in the study were analysed after less than 24 months of frozen storage.

Dunlop *et al*. (2019 DuPont-42563, Revision No 1) studied the freezer storage stability of fluazaindolizine and metabolites in bovine whole milk, fat, muscle, liver and kidney. Separate representative homogenised control milk and tissue samples were fortified with 0.1 mg/kg each of fluazaindolizine and metabolites (IN-REG72, IN-F4106, IN-A5760, IN-QEK31, IN-RYC33, IN-R2W56). Samples were stored at about -20 °C. Samples were analysed for residues of fluazaindolizine, IN-AS5760, IN-F4106, IN-QEK31, IN-R2W56, IN-REG72 and IN-RYC33 using Charles River Analytical Procedure AP.225144.02, a modified version of method DuPont-39226 (LOQ 0.01 mg/kg for each analyte). The results of the stability testing are given by commodity and analysis interval in Table 132 below.

Fluazaindolizine and metabolites (IN-A5760, IN-F4106, IN-QEK31, IN-R2W56, IN-REG72, IN-RYC33) are stable on frozen storage in milk for at least 6.8 months, in muscle for at least 6.7 months, in fat for at least 8.5 months and for analytes other than IN-R2W56 in kidney for at least 8.3 months. IN-R2W56 was stable in kidney for 7 days but not in a sample stored for 250 days. Fluazaindolizine, IN-

QEK31 and IN-R2W56 were stable in liver for at least 0.77 months (23 days), IN-RYC33 for 0.47 months (14 days) and IN-REG72, IN-F4106 and IN-A5760 for 0.23 months (7 days) (Table 133).

Table 132 Summary of fluazaindolizine and metabolite residues in animal commodities fortified at 0.1 mg/kg and stored at approximately -20 °C

Storage interval (days)	Residue (mg/kg)		Procedural recovery (percent)		Residue (mg/kg)		Procedural recovery (percent)	
Milk								
	Fluazaindolizine				IN-QEK31			
0	0.079	0.099	-	-	0.101	0.109	-	-
7	0.084	0.095	80	84	0.088	0.096	91	88
14	0.103	0.085	104	109	0.106	0.102	104	103
206/265	0.102	0.127	103	101	0.096	0.105	104	104
	IN-REG72				IN-F4106			
0	0.086	0.102	-	-	0.099	0.105	-	-
7	0.088	0.096	84	88	0.092	0.097	96	95
14	0.106	0.096	110	111	0.109	0.103	104	103
206	0.092	0.122	94	103	0.100	0.124	99	102
	IN-A5760				IN-RYC33			
0	0.102	0.110	-	-	0.104	0.112		
7	0.092	0.094	94	92	0.090	0.095	95	92
14	0.111	0.103	106	107	0.111	0.017	114	112
206/265	0.101	0.121	107	104	0.103	0.111	109	108
	IN-R2W56							
0	0.105	0.114	-	-				
7	0.091	0.095	93	93				
14	0.110	0.107	108	107				
265	0.097	0.105	103	103				
Muscle								
	Fluazaindolizine				IN-QEK31			
0	0.096	0.105	-	-	0.090	0.097	-	-
7	0.083	0.106	113	105	0.099	0.101	103	95
14	0.100	0.093	108	104	0.073	0.074	76	76
200/281	0.111	0.110	106	112	0.107	0.118	100	80
	IN-REG72				IN-F4106			
0	0.091	0.097	-	-	0.102	0.108	-	-
7	0.082	0.097	106	100	0.093	0.104	106	101
14	0.091	0.083	99	94	0.096	0.092	102	99
200	0.092	0.094	94	99	0.097	0.099	96	109
	IN-A5760				IN-RYC33			
0	0.095	0.102	-	-	0.096	0.104		
7	0.097	0.105	110	100	0.101	0.105	117	108
14	0.098	0.094	101	101	0.097	0.105	109	101
200	0.093	0.093	103	82	0.116	0.111	95	89
	IN-R2W56							
0	0.097	0.105	-	-				
7	0.097	0.102	109	101				
14	0.094	0.100	103	100				
200	0.123	0.117	99	90				
Fat								
	Fluazaindolizine				IN-QEK31			
0	0.101	0.104	-	-	0.097	0.098	-	-
7	0.108	0.105	103	107	0.111	0.114	104	114
14					0.086	0.083	101	94

Storage interval (days)	Residue (mg/kg)		Procedural recovery (percent)		Residue (mg/kg)		Procedural recovery (percent)	
255	0.112	0.103	117	91	0.085	0.082	98	102
	IN-REG72				IN-F4106			
0	0.092	0.096	-	-	0.086	0.086	-	-
7	0.116	0.112	108	112	0.111	0.106	101	108
14	0.104	0.097	119	117	0.100	0.089	112	110
255	0.088	0.081	98	106	0.095	0.095	99	102
	IN-A5760				IN-RYC33			
0	0.096	0.094	-	-	0.101	0.102	-	-
7	0.111	0.109	99	109	0.119	0.116	109	116
14	0.095	0.092	113	104	0.090	0.088	111	103
255	0.090	0.090	97	103	0.093	0.090	96	102
	IN-R2W56							
0	0.097	0.097	-	-				
7	0.117	0.112	105	114				
14	0.092	0.090	111	104				
255	0.094	0.090	99	105				
Kidney								
	Fluazaindolizine				IN-QEK31			
0	0.095	0.115	-	-	0.093	0.106	-	-
7					0.081	0.068	68	79
250	0.107	0.119	135	114	0.091	0.095	86	79
	IN-REG72				IN-F4106			
0	0.095	0.106	-	-	0.094	0.109	-	-
14	0.079	0.089	87	105	0.095	0.104	94	122
250	0.092	0.102	107	93	0.088	0.100	109	94
	IN-A5760				IN-RYC33			
0	0.072	0.082	-	-	0.104	0.119	-	-
7					0.097	0.103	102	101
14	0.091	0.108	96	119				
250	0.101	0.110	120	102	0.065	0.070	92	82
	IN-R2W56							
0	0.098	0.114	-	-				
7	0.078	0.100	100	84				
250	0.026	0.028	91	78				
Liver								
	Fluazaindolizine				IN-QEK31			
0	0.093	0.108	-	-	0.081	0.086	-	-
7					0.152	0.169	98	107
14					0.113	0.067	67	113
0					0.087	0.088	87	88
23	0.099	0.116	128	118	0.077	0.085	92	82
	IN-REG72				IN-F4106			
0	0.108	0.113	-	-	0.099	0.111	-	-
7	0.075	0.086	83	89	0.094	0.102	97	107
		IN-A5760				IN-RYC33		
0	0.095	0.093			0.092	0.093	-	-
7	0.091	0.099	99	105	0.101	0.019	117	125
14					0.097	0.109	109	99
	IN-R2W56							
0	0.094	0.093	-	-				
7	0.034	0.032	113	121				
14	0.031	0.104	101	33				
0	0.094	0.101	-	-				

Storage interval (days)	Residue (mg/kg)		Procedural recovery (percent)		Residue (mg/kg)	Procedural recovery (percent)
23	0.080	0.090	104	93		

Table 133 Frozen stability of fluazaindolizine and metabolites in bovine matrices

Matrix	Maximum freezer storage interval (days)	Demonstrated stability on frozen storage (days)
Whole Milk	125	206
Skimmed Milk	107	206
Cream	112	206
Muscle	83	200
Liver	9	7 (IN-REG72, IN-F4106, IN-A5760) 14 (IN-RYC33) 23 (fluazaindolizine, IN-QEK31, IN-R2W56)
Kidney	7[A]	7 (IN-R2W56) 250 (all other analytes)
Fat	93	255

Notes:

[A] 134 days for the repeat analysis from one animal in the 2 ppm dose group.

Note: The initial stability analyses for IN-R2W56 in liver showed too much variability in the recovery values. Therefore, the liver stability assessment was restarted for this analyte.

USE PATTERN

Fluazaindolizine is a nematicide that controls or supresses parasitic root-knot nematodes in cucurbit vegetables, non-cucurbit fruiting vegetables and also carrots and tuberous and corm vegetables. The use patterns relevant for this evaluation are shown in Table 134.

Table 134 Fluazaindolizine use patterns

Crop	Country	Application rate (kg ai/ha)	Spray volume (L/ha)	No. (interval, days)	PHI (days)	Comments
Tuberous and corm vegetables (crop subgroup 1C)	Canada	1.12-2.24, max 2.24/year	>140, incorporate 10-15 cm soil	2 (14)	40	Pre-plant of broadcast followed by soil incorporation.
		1.12-2.24, max 2.24/year (10.1-20.25 g ai/100 m based on 90 cm row spacing)		2 (14)	40	In-furrow
		0.56-1.12, max 2.24/year		2 (14)	40	Supplemental in-season chemigation following pre-plant or at-plant application
Carrot	Canada	1.12-2.24, max 2.24/year	>140, incorporate 10-	2 (14)	65	Pre-plant or broadcast followed by soil incorporation.

Crop	Country	Application rate (kg ai/ha)	Spray volume (L/ha)	No. (interval, days)	PHI (days)	Comments
		0.56-1.12, max 2.24/year	15 cm soil	2 (14)	65	Chemigation (post-plant). Supplemental in-season chemigation following pre-plant or at-plant application
Cucurbit vegetables (crop group 9)	Canada	1.12-2.24, max 2.24/year	>140, incorporate 10-15 cm soil	4 (14)	1	Pre-plant or broadcast followed by soil incorporation.
		1.12-2.24, max 2.24/year		4 (14)	1	Chemigation (pre-plant or at-plant)
		0.56-1.12, max 2.24/year		4 (14)	1	Chemigation (post-plant)
Fruiting vegetables (crop group 8-09)	Canada	1.12-2.24, max 2.24/year	>140, incorporate 10-15 cm soil	3 (14)	1	Pre-plant or broadcast followed by soil incorporation.
		1.12-2.24, max 2.24/year		3 (14)	1	Chemigation (pre-plant or at-plant)
		0.56-1.12, max 2.24/year		3 (14)	1	Chemigation (post-plant)
Cucurbits (field and protected)	Australia	2, max 2/year		2	0	Drip/trickle irrigation at establishment up to 3 days before to 1 day after planting, soil applied and incorporated by irrigation of mechanical incoporation up to 3 days before planting.
		1+1, max 2/year		2	0	Pre-plant (up to 3 days before to 1 day after planting) & post-plant drip irrigation (14-28 days after transplanting)
		1 or 2, max 2/year			0	Post-plant drip irrigation)
Fruiting vegetables (field and protected)	Australia	2, max 2/year		2	0	Drip/trickle irrigation at establishment up to 3 days before to 1 day after planting, soil applied and incorporated by irrigation of mechanical incoporation up to 3 days before planting.
		1+1, max 2/year		2	0	Pre-plant (up to 3 days before to 1 day after planting) & post-plant drip irrigation (14-28 days after transplanting)
		1 or 2, max 2/year			0	Post-plant drip irrigation
Root & tuber vegetables	Australia	2, max 2/year			Not required	Pre-plant incorporated or in-furrow soil treatment, up to 3 days before planting
Sweet potato	Australia	2, max 2/year			Not required	At establishment drip/trickle irrigation, apply 3 days before to 3 days after planting
		2, max 2/year			Not required	Soil applied and incorporated by irrigation of mechanical incorporation, apply up to 3 days before transplanting

Crop	Country	Application rate (kg ai/ha)	Spray volume (L/ha)	No. (interval, days)	PHI (days)	Comments
		1+1, max 2/year		2	Not required	Pre-plant (up to 3 days before to 1 day after planting) & post-plant drip irrigation (14-21 days after transplanting)
		1 or 2, max 2/year			Not required	Post-plant drip irrigation, do not apply later than 21 days after transplanting
Eggplant (protected)	Mexico	0.5-1.0, max 2 applications/season with max 4 applications/year		2	1	Perform 1 application at least one day before transplantation through drip irrigation
Chili (protected)	Mexico			2	1	
Pepper (protected)	Mexico			2	1	
Tomato (protected)	Mexico			2	1	
Pumpkin (protected)	Mexico			2	1	
Zucchini (protected)	Mexico			2	1	
Melon (protected)	Mexico			2	1	
Cucumber (protected)	Mexico			2	1	
Watermelon (protected)	Mexico			2	1	
Eggplant (field)	Mexico	0.75-1.0, max 2 applications/season with max 4 applications/year		2	1	
Chili (field)	Mexico			2	1	
Tomato (field)	Mexico			2	1	
Pepper (field)	Mexico			2	1	
Pumpkin (field)	Mexico	0.5-0.75, max 2 applications/season with max 4 applications/year		2	1	
Zucchini (field)	Mexico			2	1	
Melon (field)	Mexico			2	1	
Cucumber (field)	Mexico			2	1	
Watermelon (field)	Mexico			2	1	
Potato (field)	Mexico	0.75-1.0, max 2 applications/season with max 4 applications/year	350-450	2	1	Application to the bottom of the furrow at the time of planting. Make 1 application at the time of sowing directed to the bottom of the furrow.

Notes:

Tuberous and corm vegetables (Canada Crop subgroup 1C): Arrowroot, chayote root, Chinese artichoke, Jerusalem artichoke, edible canna, chufa, dasheen, ginger, potato, sweet potato, and true yam.

Cucurbit vegetables (Canada Crop group 9): (Chayote, Chinese waxgourd, citron melon, cucumber, gherkin, edible gourd [hyotan, cucuzza, hechima and Chinese okra], Momordica spp. [balsam apple, balsam pear, bitter melon and Chinese cucumber], muskmelon [true cantaloupe, cantaloupe, casaba, crenshaw melon, golden pershaw melon, honeydew melon, honey balls, mango melon, Persian melon, pineapple melon, Santa Claus melon and snake melon], pumpkin, summer squash [crookneck squash, scallop squash, straightneck squash, vegetable marrow and zucchini], winter squash [butternut squash, calabaza, hubbard squash, acorn squash and spaghetti squash], and watermelon).

Fruiting vegetables (Canada Crop group 8-09): African eggplant, currant tomato, eggplant, garden huckleberry, goji berry, ground cherry, martynia, okra, pea eggplant, pepino, bell pepper, non-bell pepper, scarlet eggplant, sunberry, tomatillo and tomato.

Australia: ALL CROPS: Fluazaindolizine rates should only be applied to the portion of the field/greenhouse that requires protection from nematode infestation. For example, if the inter-row accounts for 30 percent of the area the use rate over the full hectare will be 2 or 4 litres per ha × 70 percent.

The product label from Canada also includes information on plant-back intervals. All crops on the label may be replanted at any time following the last application of Salibro Nematicide. All other crops listed below can be planted 14 days following the last application of Salibro Nematicide.

Rotational Crops	Planting Time from Last Application
Carrots, CSG1C, CG8-09, CG9[A]	Immediately
Root vegetables, except sugar beets (CSG1B, except carrot roots)	14 days
Leaves of root and tuber vegetables (crop group 2)	
Bulb vegetables (crop group 3-07)	
Leafy vegetables (crop group 4-13)	
Brassica head and stem vegetable (crop group 5-13)	
Legume vegetables, succulent or dried (crop group 6)	
Foliage of legume vegetables (crop group 7)	
Low growing berries (crop subgroup 13-07G)	
Cereal grain (crop group 15)	
Forage, fodder, and straw of cereal grains (crop group 16)	
Grass forage, fodder, and hay (crop group 17)	
Oilseeds revised (crop group 20)	
Stalk, stem, and leaf petioles (crop group 22)	
All other crops	365 days

Notes:

[A] Tuberous and corm vegetables (Canada crop subgroup 1C); Cucurbit vegetables (Canada crop group 9); Fruiting vegetables (Canada crop group 8-09)

Mexico labels state that the following crops can be replanted immediately after the last application: carrots, cucurbits, solanacea, trees that will not bear fruit 12 months after application (citrus, stone fruit trees and tree nuts), vines and potatoes. The following crops can be replanted 14 days after the last application: brassicaceae, bulbs, cereals, leafy vegetables, legumes, strawberry, pastures established in paddocks, oilseeds, roots and tubers, asparagus and celery

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials for fluazaindolizine on the following crops:

Crop	Table No.
Cucumber	135
Melon	136, 137
Squash	138
Tomato	139
Pepper	140
Carrot	141
Potato	142

Trials were generally well documented, with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Control samples are indicated in the summary tables with a "c". Unless stated otherwise, residue data are recorded unadjusted for recovery.

Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Trial designs used non-replicated plots. Field reports provided data on the sprayers used, plot size, field sample size and sampling date.

Residue concentrations are expressed in terms of the individual compounds and not as fluazaindolizine equivalents. In some studies, a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

To convert residues in the tables to fluazaindolizine equivalents, correction for differences in molecular weight need to be made. The required correction factors are:

Analyte	Fluazaindolizine MW/metabolite MW	Correction factor
IN-A5760	468.2/207.6	2.26
IN-F4106	468.2/221.7	2.11
IN-QEK31	468.2/264.6	1.77
IN-QZY47	468.2/308.7	1.52
IN-TMQ01	468.2/309.7	1.51
IN-UJV12	468.2/294.7	1.59
IN-UNS90	468.2/295.7	1.58

The sum of IN-A5760, IN-F4106, IN-QZY47 and IN-TMQ01, all expressed in terms of fluazaindolzine is required for use in dietary risk assessment. This is calculated as 2.26×IN-A5760 + 2.11×IN-F4106 + 1.52×IN-QZY47 + 1.51×IN-TMQ01 and reported in the Tables below.

Curcubits

Shepard (2020 DuPont-40063) conducted residue trials on cucurbit vegetables at a number of sites (13 cucumber, 13 melon, 12 summer squash) in the Canada and the United States in 2014/2015. The results are shown in Tables 135 to 138.

At each location, separate plots were treated with fluazaindolizine SC formulation was applied as soil applications (drip/drench or directed spray) at planting and then 15±2 days and 1 day before expected maturity and at the other plot as four applications at 1±2 day intervals starting 43±3 days before expected maturity. No adjuvants were used in any of the trials.

The maximum interval of frozen storage before analysis was 470 days for cucurbits with extracts quantified within 33 days of extraction. Fruit was analysed for residues of fluazaindolizine and compounds hydrolysed with acid to IN-A5760, IN-F4016, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12 and IN-UNS90 (IN-TQD54) using the analytical method DuPont-33861, rev. 3, with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg for each analyte. Acceptable concurrent recovery data were obtained for all matrices.

Table 135 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post hydrolysis[C]) in cucumber from trials conducted in Canada and the United States

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	N-A5760	IN-F4106	IN-QEK31	IN-QZY 47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
Branchton, ON, Canada 2014 Talladega	3 (57 12) 11/6 7/8 19/8 dripline	1.14 0.56 0.56	1	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	0.012	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.011	<0.01	<0.01	<0.01	0.0755
			7	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	0.01[A]	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada 2014 Talladega	4 (14 22 12) 2/7 16/7 7/8 19/8 dripline	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada 2014 Darlington	3 (53 13) 21/6 13/8 26/8 dripline	1.12 0.56 0.56	1	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	0.01[A]	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	0.01[A]	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	0.010	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	0.012	ND	ND	ND	
				ND	ND	ND	ND	0.01[A]	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	0.0755
			36	ND	ND	ND	ND	0.010	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON,	4 (15 15 13)	0.56 0.56	1	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY 47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
Canada 2014 Darlington	14/7 29/7 13/8 26/8 dripline	0.56 0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada 2014 Intimidator	3 (48 12) 19/6 6/8 18/8 dripline	1.12 0.56 0.56	1	ND	ND	<0.01	0.020	0.070	<0.01	0.014	ND	
				ND	ND	<0.01	0.021	0.079	<0.01	0.014	ND	
			Mean	<0.01	<0.01	<0.01	0.0205	0.0745	<0.01	0.014	<0.01	0.1720
			7	ND	ND	<0.01	0.013	0.051	<0.01	0.011	ND	
				ND	ND	ND	0.011	0.039	<0.01	0.010	ND	
			Mean	<0.01	<0.01	<0.01	0.012	0.045	<0.01	0.0105	<0.01	0.1272
			16	ND	ND	<0.01	0.043	0.099	0.01[A]	0.017	ND	
				ND	ND	<0.01	0.018	0.047	<0.01	0.01[A]	ND	
			Mean	<0.01	<0.01	<0.01	0.0305	0.073	<0.01	0.0135	<0.01	0.1698
			22	ND	ND	<0.01	0.028	0.073	<0.01	0.018	ND	
				ND	ND	<0.01	0.035	0.075	<0.01	0.015	ND	
			Mean	<0.01	<0.01	<0.01	0.0315	0.074	<0.01	0.0165	<0.01	0.1713
			30	ND	ND	<0.01	0.027	0.075	<0.01	0.013	ND	
				ND	ND	ND	0.017	0.053	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.022	0.064	<0.01	<0.0115	<0.01	0.1561
			37	ND	ND	<0.01	0.020	0.067	<0.01	0.016	ND	
				ND	ND	<0.01	0.017	0.055	<0.01	0.011	ND	
			Mean	<0.01	<0.01	<0.01	0.0185	0.061	<0.01	0.0135	<0.01	0.1515
Branchton, ON, Canada 2014 Intimidator	4 (12 16 12) 9/7 21/7 6/8 18/8 dripline	0.56 0.56 0.56 0.56	1	ND	ND	ND	0.01[A]	0.025	ND	<0.01	ND	
				ND	ND	<0.01	<0.01	0.021	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.01	0.023	<0.01	<0.01	<0.01	0.0938
			7	ND	ND	<0.01	0.01[A]	0.034	<0.01	<0.01	ND	
				ND	ND	ND	<0.01	0.024	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.029	<0.01	<0.01	<0.01	0.1029
			16	ND	ND	<0.01	0.015	0.033	<0.01	<0.01	ND	
				ND	ND	<0.01	0.018	0.048	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.0165	0.0405	<0.01	<0.01	<0.01	0.1204
			22	ND	ND	<0.01	0.013	0.031	<0.01	<0.01	ND	
				ND	ND	<0.01	0.012	0.026	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.0125	0.0285	<0.01	<0.01	<0.01	0.1021
			30	ND	ND	<0.01	0.011	0.039	<0.01	<0.01	ND	
				ND	ND	<0.01	0.010	0.038	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.0105	0.0385	<0.01	<0.01	<0.01	0.1173
			37	ND	ND	ND	0.010	0.027	<0.01	<0.01	ND	
				ND	ND	ND	<0.01	0.028	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0275	<0.01	<0.01	<0.01	0.1006
St. Marc-sur-Richelieu, QC, Canada 2014 Magic	3 (45 13) 9/6 24/7 6/8 drench	1.12 0.56 0.56	1	<0.01	ND	0.024	0.016	0.021	ND	<0.01	ND	
				ND	ND	0.019	0.011	0.020	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.0215	0.0135	0.0205	<0.01	<0.01	<0.01	0.1142
			6	ND	ND	0.018	0.015	0.026	ND	<0.01	ND	
				ND	ND	0.018	0.018	0.027	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.018	0.0165	0.0265	<0.01	<0.01	<0.01	0.1160
			14	ND	ND	0.01[A]	0.017	0.019	ND	<0.01	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY 47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
				ND	ND	0.016	0.021	0.027	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.013	0.019	0.023	<0.01	<0.01	<0.01	0.1001
			21	ND	ND	0.015	0.018	0.021	ND	<0.01	ND	
				ND	ND	0.020	0.031	0.037	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.0175	0.0245	0.029	<0.01	<0.01	<0.01	0.1187
			28	ND	ND	0.012	0.021	0.033	ND	<0.01	ND	
				ND	ND	0.011	0.014	0.027	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.0115	0.0175	0.030	<0.01	<0.01	<0.01	0.1076
			34	ND	ND	0.016	0.025	0.041	ND	<0.01	ND	
				ND	ND	0.013	0.022	0.035	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.0145	0.0235	0.038	<0.01	<0.01	<0.01	0.1261
St. Marc-sur-Richelieu, QC, Canada 2014 Magic	4 (13 15 13) 26/6 9/7 24/7 6/8 drench	0.56 0.56 0.56 0.56	1	<0.01	ND	0.020	0.013	0.020	ND	<0.01	ND	
				ND	ND	0.017	0.014	0.026	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.0185	0.0135	0.023	<0.01	<0.01	<0.01	0.1117
			6	ND	ND	0.022	0.020	0.033	ND	<0.01	ND	
				ND	ND	0.018	0.018	0.031	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.020	0.019	0.032	<0.01	<0.01	<0.01	0.1285
			14	ND	ND	0.015	0.015	0.025	ND	<0.01	ND	
				ND	ND	0.012	0.012	0.024	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.0135	0.0135	0.0245	<0.01	<0.01	<0.01	0.1034
			21	ND	ND	0.013	0.015	0.023	ND	<0.01	ND	
				ND	ND	0.010	0.016	0.023	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.0115	0.0155	0.023	<0.01	<0.01	<0.01	0.0969
			28	ND	ND	0.011	0.016	0.027	ND	<0.01	ND	
				ND	ND	0.017	0.026	0.040	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.014	0.021	0.0335	<0.01	<0.01	<0.01	0.1182
			34	ND	ND	0.014	0.016	0.025	ND	<0.01	ND	
				ND	ND	0.012	0.017	0.033	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.013	0.0165	0.029	<0.01	<0.01	<0.01	0.1092
St. Marc-sur-Richelieu, QC, Canada 2014 Intimidator	3 (45 13) 9/6 24/7 6/8 drench	1.12 0.56 0.56	1	ND	ND	<0.01	<0.01	0.015	ND	<0.01	ND	
				<0.01	ND	<0.01	<0.01	0.014	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0145	<0.01	<0.01	<0.01	0.0808
			6	ND	ND	ND	<0.01	0.017	ND	<0.01	ND	
				ND	ND	<0.01	<0.01	0.019	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.018	<0.01	<0.01	<0.01	0.0862
			143	ND	ND	ND	<0.01	0.018	ND	<0.01	ND	
				ND	ND	ND	<0.01	0.016	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.017	<0.01	<0.01	<0.01	0.0846
			21	ND	ND	ND	<0.01	0.015	ND	<0.01	ND	
				ND	ND	ND	<0.01	0.023	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.019	<0.01	<0.01	<0.01	0.0877
			28	ND	ND	ND	<0.01	0.018	ND	<0.01	ND	
				ND	ND	ND	<0.01	0.021	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0195	<0.01	<0.01	<0.01	0.0884
			34	ND	ND	ND	<0.01	0.020	ND	<0.01	ND	
				ND	ND	ND	<0.01	0.024	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.022	<0.01	<0.01	<0.01	0.0922
St. Marc-sur-Richelieu, QC, Canada, 2014 Intimidator	4 (13 15 13) 26/6 9/7 24/7 6/8 drench	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	0.01 [A]	0.028	ND	<0.01	ND	
				<0.01	ND	<0.01	<0.01	0.025	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0265	<0.01	<0.01	<0.01	0.0991
			6	ND	ND	<0.01	<0.01	0.033	ND	<0.01	ND	
				ND	ND	<0.01	0.010	0.036	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0345	<0.01	<0.01	<0.01	0.1112
			14	ND	ND	ND	<0.01	0.028	ND	<0.01	ND	
				ND	ND	ND	<0.01	0.020	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.024	<0.01	<0.01	<0.01	0.0953
			21	ND	ND	<0.01	0.010	0.035	<0.01	<0.01	ND	
				ND	ND	ND	<0.01	0.028	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0315	<0.01	<0.01	<0.01	0.1067

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY 47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			28	ND	ND	ND	<0.01	0.031	<0.01	<0.01	ND	
				ND	ND	ND	0.011	0.037	<0.01	0.011	ND	
			Mean	<0.01	<0.01	<0.01	0.0105	0.034	<0.01	<0.0105	<0.01	0.1105
			34	ND	ND	ND	0.012	0.038	<0.01	0.01[A]	ND	
				ND	ND	ND	0.010	0.034	<0.01	0.011	ND	
			Mean	<0.01	<0.01	<0.01	0.011	0.036	<0.01	0.0105	<0.01	0.1135
Chula, GA, United States, 2014 Thunder	3 (27, 12) Drip irr	1.12 0.56 0.56	1	0.042	ND	0.037	0.046	0.065	<0.01	<0.01	ND	
				0.041	ND	0.029	0.045	0.061	<0.01	<0.01	ND	
			Mean	0.0415	<0.01	0.033	0.0455	0.063	<0.01	<0.01	<0.01	0.2031
			7	0.043	ND	0.040	0.061	0.12	0.011	0.011	ND	
				0.049	ND	0.043	0.064	0.11	0.01[A]	0.011	ND	
			Mean	0.046	<0.01	0.0415	0.0625	0.115	0.0105	0.011	<0.01	0.3008
			15	0.021	ND	0.030	0.049	0.11	0.018	0.013	ND	
				0.025	ND	0.040	0.057	0.13	0.022	0.014	ND	
			Mean	0.023	<0.01	0.035	0.053	0.12	0.020	0.013	<0.01	0.3091
			22	<0.01	ND	0.011	0.021	0.068	<0.01	0.014	ND	
				<0.01	ND	0.012	0.028	0.072	0.010	0.016	ND	
			Mean	<0.01	<0.01	0.0115	0.0245	0.070	<0.01	0.015	<0.01	0.1684
			29	<0.01	ND	0.011	0.028	0.095	0.016	0.013	ND	
				<0.01	ND	<0.01	0.026	0.083	0.013	0.010	ND	
			Mean	<0.01	<0.01	<0.0105	0.027	0.089	0.0145	0.0115	<0.01	0.2019
			36	<0.01	ND	<0.01	0.044	0.10	0.019	0.014	ND	
				ND	ND	<0.01	0.040	0.10	0.015	0.014	ND	
			Mean	<0.01	<0.01	<0.01	0.042	0.10	0.017	0.014	<0.01	0.2214
Chula, GA, United States, 2014 Thunder	4 (10, 16, 12) drip irr	0.56 0.56 0.56 0.56	1	0.055	ND	0.045	0.054	0.073	<0.01	<0.01	ND	
				0.052	ND	0.040	0.055	0.077	<0.01	<0.01	ND	
			Mean	0.0535	<0.01	0.0425	0.0545	0.075	<0.01	<0.01	<0.01	0.2414
			7	0.045	<0.01	0.057	0.060	0.11	0.011	0.011	ND	
				0.046	<0.01	0.060	0.070	0.11	0.012	<0.01	ND	
			Mean	0.0455	<0.01	0.0585	0.065	0.11	0.0115	<0.0105	<0.01	0.3306
			15	0.030	ND	0.042	0.070	0.14	0.022	0.017	ND	
				0.026	ND	0.043	0.063	0.15	0.024	0.016	ND	
			Mean	0.028	<0.01	0.0425	0.0665	0.145	0.023	0.0165	<0.01	0.3674
			22	<0.01	ND	0.014	0.028	0.080	0.01[A]	0.015	ND	
				<0.01	ND	0.014	0.031	0.085	0.011	0.014	ND	
			Mean	<0.01	<0.01	0.014	0.0295	0.0825	0.0105	0.0145	<0.01	0.1934
			29	<0.01	ND	0.013	0.037	0.10	0.015	0.011	ND	
				<0.01	ND	0.014	0.034	0.096	0.014	0.012	ND	
			Mean	<0.01	<0.01	0.0135	0.0355	0.098	0.0145	0.0115	<0.01	0.2219
			36	0.01[A]	ND	0.014	0.043	0.12	0.025	0.016	ND	
				<0.01	ND	0.014	0.046	0.11	0.019	0.015	ND	
			Mean	<0.01	<0.01	0.014	0.0445	0.115	0.022	0.0155	<0.01	0.2602
Athens, GA, United States, 2014 Poinsett 76	3 (64, 14) drip tape	1.12 0.56 0.56	1	ND	ND	<0.01	0.011	0.030	0.014	0.014	ND	
				ND	ND	<0.01	0.011	0.027	0.013	0.015	<0.01	
			Mean	<0.01	<0.01	<0.01	0.011	0.0285	0.0135	0.0145	<0.01	0.1074
			7	ND	ND	<0.01	<0.01	0.036	0.011	0.015	ND	
				ND	ND	<0.01	<0.01	0.027	0.011	0.016	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0315	0.011	0.0155	<0.01	0.1082
			15	ND	ND	<0.01	<0.01	0.036	<0.01	0.013	ND	
				ND	ND	<0.01	<0.01	0.026	<0.01	0.012	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.031	<0.01	0.0125	<0.01	0.1059
	4 (14, 14, 14) drip tape	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	<0.01	0.026	<0.01	<0.01	ND	
				<0.01	ND	<0.01	0.010	0.031	0.011	0.010	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0285	<0.0105	<0.01	<0.01	0.1029
			7	ND	ND	<0.01	<0.01	0.031	<0.01	0.010	ND	
				ND	ND	<0.01	0.011	0.037	<0.01	0.015	ND	
			Mean	<0.01	<0.01	<0.01	<0.0105	0.034	<0.01	0.0125	<0.01	0.1105
			15	ND	ND	<0.01	<0.01	0.023	<0.01	<0.01	ND	
				ND	ND	<0.01	<0.01	0.028	<0.01	<0.01	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY 47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	<0.01	<0.01	<0.01	<0.01	0.0255	<0.01	<0.01	<0.01	0.0976
Hobe Sound, FL, United States, 2014 Impact	3 (64, 14) Drench Drip Inj Drip Inj	1.12 0.56 0.56	1	0.012	ND	0.011	<0.01	0.012	ND	ND	ND	
				0.012	ND	0.011	<0.01	0.013	ND	ND	ND	
			Mean	0.012	<0.01	0.011	<0.01	0.0125	<0.01	<0.01	<0.01	0.0799
			7	<0.01	ND	0.01[A]	<0.01	0.015	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	0.014	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0145	<0.01	<0.01	<0.01	0.0808
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	<0.01	ND	0.013	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.01	<0.01	0.0687
			22	ND	ND	<0.01	ND	0.012	ND	ND	ND	
				ND	ND	<0.01	ND	0.012	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.012	<0.01	<0.01	<0.01	0.0770
			29	ND	ND	<0.01	ND	0.014	ND	ND	ND	
				ND	ND	<0.01	ND	0.012	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	<0.01	0.0786
			36	ND	ND	<0.01	ND	0.015	ND	<0.01	ND	
				ND	ND	<0.01	ND	0.011	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	<0.01	0.0786
Hobe Sound, FL, United States, 2014 Impact	4 (14 14 14) All drip inj	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				0.01[A]	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	<0.01	ND	0.01[A]	<0.01	<0.01	ND	ND	ND	
				<0.01	ND	0.011	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	0.0105	<0.01	<0.01	<0.01	<0.01	<0.01	0.0751
			15	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Lime Springs, IA, United States, 2014 Homemade Pickles	3 (56 14) all drench	1.12 0.56 0.56	1	0.011	ND	ND	ND	<0.01	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.0105	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	<0.01	ND	ND	ND	ND	ND	ND	ND	
				<0.01	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	0.013	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.01	<0.01	0.0763
			35	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Lime Springs, IA, United States,	4 (14 15 14) drench	0.56 0.56 0.56 0.56	1	<0.01	ND	ND	ND	ND	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	<0.01	ND	ND	ND	ND	ND	ND	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY 47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
2014 Homemade Pickles				0.01[A]	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	<0.01	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	0.01[A]	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Stewardson, IL, United States, 2014 Talladega	3 (46 13) drench	1.11 0.56 0.56	1	0.010	ND	<0.01	0.01[A]	0.020	ND	<0.01	ND	
				0.011	ND	<0.01	<0.01	0.022	ND	<0.01	ND	
			Mean	0.0105	<0.01	<0.01	<0.01	0.021	<0.01	<0.01	<0.01	0.0907
			8	ND	ND	<0.01	<0.01	0.029	ND	<0.01	ND	
				<0.01	ND	<0.01	<0.01	0.050	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0395	<0.01	<0.01	<0.01	0.1188
			15	<0.01	ND	<0.01	<0.01	0.038	ND	<0.01	ND	
				ND	ND	<0.01	<0.01	0.042	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.040	<0.01	<0.01	<0.01	0.1196
			22	ND	ND	<0.01	<0.01	0.040	<0.01	<0.01	ND	
				ND	ND	<0.01	<0.01	0.038	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.039	<0.01	<0.01	<0.01	0.1181
			28	ND	ND	<0.01	<0.01	0.027	ND	<0.01	ND	
				ND	ND	<0.01	<0.01	0.034	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0305	<0.01	<0.01	<0.01	0.1052
			36	ND	ND	<0.01	<0.01	0.033	ND	<0.01	ND	
				ND	ND	<0.01	<0.01	0.027	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.030	<0.01	<0.01	<0.01	0.1044
Stewardson, IL, United States, 2014 Talladega	4 (14 14 13) drench	0.56 0.55 0.56 0.56	1	0.018	ND	0.014	0.014	0.022	ND	<0.01	ND	
				0.013	ND	0.012	0.012	0.020	ND	<0.01	ND	
			Mean	0.0155	<0.01	0.013	0.013	0.021	<0.01	<0.01	<0.01	0.0971
			8	<0.01	ND	<0.01	0.01[A]	0.042	<0.01	<0.01	ND	
				<0.01	ND	<0.01	<0.01	0.038	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.040	<0.01	<0.01	<0.01	0.1196
			15	<0.01	ND	<0.01	0.015	0.055	<0.01	0.011	ND	
				<0.01	ND	<0.01	0.012	0.054	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.0135	0.0545	<0.01	<0.0105	<0.01	0.1416
			22	ND	ND	<0.01	<0.01	0.031	ND	<0.01	ND	
				ND	ND	<0.01	0.012	0.042	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.011	0.0365	<0.01	<0.01	<0.01	0.1143
			28	ND	ND	<0.01	<0.01	0.036	ND	<0.01	ND	
				ND	ND	<0.01	<0.01	0.034	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.035	<0.01	<0.01	<0.01	0.1120
			36	ND	ND	<0.01	<0.01	0.042	ND	<0.01	ND	
				ND	ND	<0.01	<0.01	0.030	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.036	<0.01	<0.01	<0.01	0.1135
York, NE, United States, 2014 Burpless II	3 (51 15) 16/6 6/8 21/8 drench	1.12 0.56 0.56	1	0.033	ND	0.022	0.022	ND	ND	ND	ND	
				0.10	ND	0.052	0.063	ND	ND	ND	ND	
			Mean	0.0665	<0.01	0.037	0.0425	<0.01	<0.01	<0.01	<0.01	0.1310
			7	ND	ND	ND	ND	<0.01	ND	ND	ND	
				0.013	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.0115	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY 47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			22	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	<0.01	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
York, NE, United States, 2014 Burpless II 9/7 23/7 6/8 21/8	4 (14 14 15) drench	0.56 0.56 0.56 0.56	1	0.028	ND	0.020	0.019	<0.01	ND	ND	ND	
				0.044	ND	0.020	0.024	ND	ND	ND	ND	
			Mean	0.036	<0.01	0.020	0.0215	<0.01	<0.01	<0.01	<0.01	0.0951
			7	0.037	ND	0.022	0.022	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	0.0235	<0.01	<0.016	<0.016	<0.01	<0.01	<0.01	<0.01	0.0867
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
York, NE, United States, 2014 Burpless II 16/6 6/8 21/8	3 (51 15) drench	2.24 1.12 1.12	1	0.12	ND	0.060	0.067	ND	ND	ND	ND	
				0.031	ND	0.016	0.018	ND	ND	ND	ND	
			Mean	0.0755	<0.01	0.038	0.0425	<0.01	<0.01	<0.01	<0.01	0.1331
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				0.022	ND	0.014	0.014	<0.01	ND	ND	ND	
			Mean	<0.016	<0.01	<0.012	<0.012	<0.01	<0.01	<0.01	<0.01	0.0782
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	<0.01	ND	<0.01	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	0.015	ND	0.011	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.0125	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	<0.01	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
York, NE, United States, 2014 Burpless II 9/7 23/7 6/8 21/8	4 (14 14 15) drench	1.12 1.12 1.12 1.12	1	0.015	ND	0.010	<0.01	<0.01	ND	ND	ND	
				0.092	ND	0.053	0.052	<0.01	ND	ND	ND	
			Mean	0.0535	<0.01	0.0315	0.031	<0.01	<0.01	<0.01	<0.01	0.1194
			7	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				0.040	ND	0.024	0.025	<0.01	ND	ND	ND	
			Mean	<0.025	<0.01	<0.017	<0.0175	<0.01	<0.01	<0.01	<0.01	0.0888
			15	<0.01	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	
				ND	ND	<0.01	ND	<0.01	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	<0.01	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY 47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Uvalde, TX, United States, 2014 Explorer	3 (68 14) Drip irr	1.13 0.55 0.55	1	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			23	ND	ND	<0.01	ND	<0.01	<0.01	<0.01	ND	
				ND	ND	<0.01	ND	0.013	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.01	<0.01	0.0763
			30	ND	ND	0.022	<0.01	0.056	0.017	0.010	ND	
				<0.01	ND	0.015	<0.01	0.050	0.013	0.011	ND	
			Mean	<0.01	<0.01	0.0185	<0.01	0.053	0.015	0.0105	<0.01	0.1648
Uvalde, TX, United States, 2014 Explorer	4 (24 4 14)	0.55 0.55 0.55 0.55	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	0.01	ND	0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			23	ND	ND	<0.01	ND	0.013	<0.01	<0.01	ND	
				ND	ND	0.013	<0.01	0.022	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.0115	<0.01	0.0175	<0.01	<0.01	<0.01	0.0886
			30	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	0.015	<0.01	0.016	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.0125	<0.01	<0.013	<0.01	<0.01	<0.01	0.0838

Notes:

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] SUM = 2.26×IN-A5760 + 2.11×IN-F4106 + 1.52×IN-QZY47 + 1.51×IN-TMQ01.

[C] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 136 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post hydrolysis[B]) in melon pulp and peel from trials conducted in Canada and the United States

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
Branchton, ON, Canada, 2014 Primo Trial 16 02 **pulp**	3 (79 14) 11/6 29/8 2/9 dripline	1.11 0.56 0.56	2	ND	ND	ND	ND	0.010	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			6	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	0.01[A]	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			14	ND	ND	ND	ND	0.013	<0.01	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.01	<0.01	0.0763

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			21	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			28	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
Branchton, ON, Canada, 2014 Primo Trial 16 03 **pulp**	4 (13 15 14) 1/8 14/8 29/8 12/9 dripline	0.56 0.56 0.56 0.56	2	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			6	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			14	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			21	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			28	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
Branchton, ON, Canada, 2014 Primo Trial 16 02 **peel**	3 (79 14) dripline	1.11 0.56 0.56	2	ND	ND	ND	ND	0.015	<0.01	ND	ND	
				ND	ND	ND	ND	0.011	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	<0.01	
			6	ND	ND	ND	ND	0.013	<0.01	ND	ND	
				ND	ND	ND	ND	0.017	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.015	<0.01	<0.01	<0.01	
			14	<0.01	ND	<0.01	<0.01	0.029	0.01 [A]	ND	ND	
				<0.01	ND	<0.01	ND	0.014	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0215	<0.01	<0.01	<0.01	
			21	ND	ND	ND	ND	<0.01	<0.01	ND	ND	
				ND	ND	ND	ND	0.010	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			28	ND	ND	ND	ND	0.012	<0.01	ND	ND	
				ND	ND	ND	ND	0.015	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0135	<0.01	<0.01	<0.01	
Branchton, ON, Canada, 2014 Primo Trial 16 03 **peel**	4 (13 15 14) dripline	0.56 0.56 0.56 0.56	2	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	0.011	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	<0.01	
			6	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			14	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			21	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			28	ND	ND	ND	ND	0.010	ND	ND	ND	
				ND	ND	<0.01	ND	0.012	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	
Branchton, ON, Canada, 2014 Avatar Trial 17 02 **pulp**	3 (71 14) 18/6/ 28/8 11/9 dripline	1.12 0.56 0.56	1	ND	ND	ND	ND	0.013	<0.01	ND	ND	
				ND	ND	ND	ND	0.013	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	<0.01	0.0786
			7	ND	ND	ND	ND	0.011	<0.01	ND	ND	
				ND	ND	ND	ND	0.010	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0105	<0.01	<0.01	<0.01	0.0748
			15	ND	ND	ND	ND	0.013	<0.01	ND	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0115	<0.01	<0.01	<0.01	0.0763
			21	ND	ND	ND	ND	0.011	<0.01	ND	ND	
				ND	ND	ND	ND	0.011	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	0.0755
			29	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	0.012	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.011	<0.01	<0.01	<0.01	0.0755
Branchton, ON, Canada, 2014 Avatar Trial 17 03 **pulp**	4 (13 15 14) 31/7 13/8 28/8 11/9 dripline	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			7	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			21	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			29	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
Branchton, ON, Canada, 2014 Avatar Trial 17 02 **peel**	3 (71 14) dripline	1.12 0.56 0.56	1	<0.01	ND	<0.01	<0.01	0.026	<0.01	ND	ND	
				ND	ND	<0.01	ND	0.022	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.024	<0.01	<0.01	<0.01	
			7	ND	ND	ND	ND	0.016	<0.01	ND	ND	
				<0.01	ND	ND	<0.01	0.019	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0175	<0.01	<0.01	<0.01	
			15	ND	ND	ND	ND	0.018	<0.01	ND	ND	
				<0.01	ND	ND	ND	0.013	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0155	<0.01	<0.01	<0.01	
			21	ND	ND	ND	ND	0.017	<0.01	ND	ND	
				ND	ND	ND	ND	0.020	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0185	<0.01	<0.01	<0.01	
			29	ND	ND	ND	ND	0.013	<0.01	ND	ND	
				ND	ND	ND	ND	0.018	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0155	<0.01	<0.01	<0.01	
Branchton, ON, Canada, 2014 Avatar Trial 17 03 **peel**	4 (13 15 14) dripline	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	0.010	<0.01	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01[A]	<0.01	<0.01	<0.01	
			7	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	0.010	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	0.011	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	<0.01	
			21	ND	ND	ND	ND	0.01[A]	<0.01	ND	ND	
				ND	ND	ND	ND	0.011	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0105	<0.01	<0.01	<0.01	
			29	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	0.01[A]	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
Branchton, ON, Canada, 2014 Sugar Cube Trial 18 02 **pulp**	3 (80 14) 20/6 8/9 22/9	1.12 0.56 0.56	1	ND	ND	ND	ND	0.027	0.011	ND	ND	
				ND	ND	ND	ND	0.030	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0285	<0.0105	<u><0.01</u>	<u><0.01</u>	0.1029
			7	ND	ND	ND	<0.01	0.031	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.043	0.012	ND	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
	dripline		Mean	<0.01	<0.01	<0.01	<0.01	0.037	<0.011	<0.01	<0.01	0.1166
			16	ND	ND	ND	<0.01	0.040	0.012	ND	ND	
				ND	ND	ND	ND	0.033	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0365	<0.011	<0.01	<0.01	0.1158
			23	ND	ND	ND	<0.01	0.044	0.013	ND	ND	
				ND	ND	ND	<0.01	0.052	0.012	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.048	0.0125	<0.01	<0.01	0.1355
Branchton, ON, Canada, 2014 Sugar Cube Trial 18 03 **pulp**	4 (14 14 14) 11/8 25/8 8/9 22/9 dripline	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	0.019	<0.01	ND	ND	
				ND	ND	ND	ND	0.014	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0165	<0.01	<0.01	<0.01	0.0839
			7	ND	ND	ND	ND	0.027	<0.01	ND	ND	
				ND	ND	ND	ND	0.014	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0205	<0.01	<0.01	<0.01	0.0900
			16	ND	ND	ND	ND	0.018	<0.01	ND	ND	
				ND	ND	ND	ND	0.015	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.017	<0.01	<0.01	<0.01	0.0839
			23	ND	ND	ND	ND	0.018	<0.01	ND	ND	
				ND	ND	ND	ND	0.021	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0195	<0.01	<0.01	<0.01	0.0884
Branchton, ON, Canada, 2014 Sugar Cube Trial 18 02 **peel**	3 (80 14) dripline	1.12 0.56 0.56	1	0.014	ND	<0.01	0.013	0.052	0.025	ND	ND	
				ND	ND	<0.01	<0.01	0.052	0.019	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.0115	0.052	0.022	<0.01	<0.01	
			7	<0.01	ND	0.010	0.011	0.057	0.022	ND	ND	
				0.017	ND	0.018	0.020	0.090	0.032	<0.01	ND	
			Mean	<0.0135	<0.01	0.014	0.0155	0.0735	0.027	<0.01	<0.01	
			16	<0.01	<0.01	<0.01	0.010	0.078	0.029	<0.01	ND	
				<0.01	ND	<0.01	<0.01	0.060	0.022	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.069	0.0255	<0.01	<0.01	
			23	ND	<0.01	<0.01	<0.01	0.075	0.031	<0.01	<0.01	
				ND	<0.01	<0.01	<0.01	0.10	0.025	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0875	0.028	<0.01	<0.01	
Branchton, ON, Canada, 2014 Sugar Cube Trial 18 03 **peel**	4 (14 14 14) dripline	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	<0.01	0.032	0.011	ND	ND	
				<0.01	ND	<0.01	<0.01	0.027	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0295	<0.0105	<0.01	<0.01	
			7	<0.01	<0.01	0.01[A]	<0.01	0.047	0.013	ND	ND	
				0.014	ND	0.01[A]	0.011	0.029	<0.01	ND	ND	
			Mean	<0.012	<0.01	0.01[A]	<0.0105	0.038	<0.0115	<0.01	<0.01	
			16	<0.01	<0.01	<0.01	<0.01	0.047	0.013	ND	ND	
				<0.01	ND	<0.01	<0.01	0.031	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.039	<0.0115	<0.01	<0.01	
			23	<0.01	<0.01	<0.01	<0.01	0.036	0.011	ND	ND	
				<0.01	<0.01	<0.01	<0.01	0.043	0.012	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0395	0.0115	<0.01	<0.01	
St. Marc-sur-Richelieu, QC, Canada, 2014 Goddess Trial 19 02 **pulp**	3 (63 15) 10/6 12/8 27/8 Drench	1.12 0.56 0.56	1	ND	ND	ND	ND	0.025	<0.01	ND	ND	
				ND	ND	ND	ND	0.024	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0245	<0.01	<0.01	<0.01	0.0960
			6	ND	ND	ND	ND	0.018	<0.01	ND	ND	
				ND	ND	ND	ND	0.018	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.018	<0.01	<0.01	<0.01	0.0862
			14	ND	ND	ND	ND	0.017	<0.01	ND	ND	
				ND	ND	ND	ND	0.015	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.016	<0.01	<0.01	<0.01	0.0831
			21	ND	ND	ND	ND	0.011	ND	ND	ND	
				ND	ND	ND	ND	0.011	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	0.0755
St. Marc-sur-Richelieu, QC, Canada, 2014	4 (14 13 15) 16/7	0.56 0.56 0.56	1	ND	ND	ND	ND	0.01[A]	ND	ND	ND	
				ND	ND	ND	ND	0.01[A]	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.01[A]	<0.01	<0.01	<0.01	0.074

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
Goddess Trial 19 03 **pulp**	30/7 12/827 /8 drench	0.56	6	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			14	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			21	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
St. Marc-sur-Richelieu, QC, Canada, 2014 Goddess ss Trial 19 02 **peel**	3 (63 15) Drench	1.12 0.56 0.56	1	<0.01	ND	<0.01	<0.01	0.049	0.032	ND	<0.01	
				<0.01	ND	<0.01	<0.01	0.054	0.030	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0525	0.031	<0.01	<0.01	
			6	ND	ND	<0.01	<0.01	0.044	0.021	ND	ND	
				<0.01	ND	0.014	<0.01	0.043	0.021	ND	<0.01	
			Mean	<0.01	<0.01	0.012	<0.01	0.0435	0.021	<0.01	<0.01	
			14	ND	ND	<0.01	<0.01	0.041	0.019	ND	ND	
				ND	ND	<0.01	<0.01	0.032	0.015	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0365	0.017	<0.01	<0.01	
			21	ND	ND	ND	<0.01	0.025	0.012	ND	ND	
				ND	ND	ND	<0.01	0.025	0.010	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.025	0.011	<0.01	<0.01	
St. Marc-sur-Richelieu, QC, Canada, 2014 Goddess ss Trial 19 03 **peel**	4 (14 13 15) drench	0.56 0.56 0.56 0.56	1	ND	ND	<0.01	ND	0.017	<0.01	ND	ND	
				0.01[A]	ND	0.012	<0.01	0.025	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.011	<0.01	0.021	<0.01	<0.01	<0.01	
			6	ND	ND	<0.01	ND	0.020	<0.01	ND	ND	
				ND	ND	<0.01	ND	0.020	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.020	<0.01	<0.01	<0.01	
			14	ND	ND	<0.01	ND	0.017	<0.01	ND	ND	
				ND	ND	<0.01	<0.01	0.020	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0185	<0.01	<0.01	<0.01	
			21	ND	ND	ND	<0.01	0.020	<0.01	ND	ND	
				ND	ND	ND	ND	0.013	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.016	<0.01	<0.01	<0.01	
St. Marc-sur-Richelieu, QC, Canada, 2014 Magnifisweet Trial 20 02 **pulp**	3 (63 15) 10/6 12/8 27/8 drench	1.12 0.56 0.56	1	ND	ND	ND	ND	0.017	<0.01	ND	ND	
				<0.01	<0.01	<0.01	<0.01	0.016	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.017	<0.01	<0.01	<0.01	0.0839
			6	ND	ND	ND	ND	0.013	<0.01	ND	ND	
				ND	ND	ND	ND	0.010	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.012	<0.01	<0.01	<0.01	0.0763
			14	ND	ND	ND	ND	0.012	<0.01	ND	ND	
				ND	ND	ND	ND	0.013	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	<0.01	0.0778
			21	ND	ND	ND	ND	0.01[A]	ND	ND	ND	
				ND	ND	ND	ND	0.01[A]	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.01[A]	<0.01	<0.01	<0.01	0.074
			28	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			34	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
St. Marc-sur-Richelieu, QC, Canada, 2014 Magnifisweet Trial 20 03 **pulp**	4 (14 13 15) 16/7 30/7 12/827 /8 drench	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	0.016	<0.01	ND	ND	
				ND	ND	ND	ND	0.014	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.015	<0.01	<0.01	<0.01	0.0816
			6	ND	<0.01	ND	ND	0.012	<0.01	ND	ND	
				ND	ND	ND	ND	0.013	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.012	<0.01	<0.01	<0.01	0.0778
			14	ND	ND	ND	ND	0.013	<0.01	ND	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
				ND	ND	ND	ND	0.011	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.012	<0.01	<0.01	<0.01	0.0770
			21	ND	ND	ND	ND	0.012	ND	ND	ND	
				ND	ND	ND	ND	0.011	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.012	<0.01	<0.01	<0.01	0.0763
			28	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	0.015	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0125	<0.01	<0.01	<0.01	0.0778
			34	ND	ND	ND	ND	<0.01	<0.01	ND	ND	
				ND	ND	ND	ND	0.012	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	0.0755
St. Marc-sur-Richelieu, QC, Canada, 2014 Magnifisweet Trial 20 02 **peel**	3 (63 15) drench	1.12 0.56 0.56	1	0.043	ND	0.035	0.035	0.022	0.012	ND	ND	
				0.22	ND	0.16	0.14	0.023	0.013	ND	ND	
			Mean	0.13	<0.01	0.096	0.088	0.022	0.013	<0.01	<0.01	
			6	<0.01	ND	<0.01	<0.01	0.017	0.010	ND	ND	
				ND	ND	<0.01	<0.01	0.018	0.012	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0175	0.011	<0.01	<0.01	
			14	0.016	ND	0.016	0.016	0.019	0.010	ND	ND	
				ND	ND	<0.01	<0.01	0.020	0.011	ND	ND	
			Mean	<0.013	<0.01	<0.013	<0.013	0.0195	0.0105	<0.01	<0.01	
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			34	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
St. Marc-sur-Richelieu, QC, Canada, 2014 Magnifisweet Trial 20 03 **peel**	4 (14 13 15) drench	0.56 0.56 0.56 0.56	1	0.018	ND	0.015	0.014	0.020	0.01[A]	ND	ND	
				<0.01	ND	0.011	<0.01	0.019	<0.01	ND	ND	
			Mean	<0.014	<0.01	0.013	<0.012	0.0195	<0.01	<0.01	<0.01	
			6	ND	ND	<0.01	<0.01	0.015	<0.01	ND	ND	
				ND	ND	<0.01	<0.01	0.015	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.015	<0.01	<0.01	<0.01	
			14	ND	ND	<0.01	<0.01	0.021	0.01[A]	ND	ND	
				ND	ND	<0.01	<0.01	0.015	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.018	<0.01	<0.01	<0.01	
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			34	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
Chula, GA, United States, 2014 Athena Trial 14 02 **pulp**	3 (50 14) drip inj	1.12 0.56 0.56	1	<0.01	ND	0.017	0.017	0.064	0.017	<0.01	ND	
				<0.01	ND	0.018	0.018	0.056	0.014	ND	ND	
			Mean	<0.01	<0.01	0.0175	0.0175	0.060	0.0155	<0.01	<0.01	0.1741
			7	<0.01	ND	0.022	0.017	0.067	0.015	ND	ND	
				<0.01	ND	0.019	0.019	0.062	0.014	<0.01	ND	
			Mean	<0.01	<0.01	0.0205	0.018	0.0645	0.0145	<0.01	<0.01	0.1858
			14	<0.01	ND	0.027	0.025	0.074	0.017	<0.01	ND	
				0.010	ND	0.026	0.030	0.084	0.021	<0.01	ND	
			Mean	<0.01[A]	<0.01	0.0265	0.0275	0.079	0.019	<0.01	<0.01	0.2273
			22	ND	ND	0.017	0.025	0.10	0.018	<0.01	ND	
				<0.01	ND	0.019	0.033	0.10	0.019	<0.01	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	<0.01	<0.01	0.018	0.029	0.10	0.0185	<0.01	<0.01	0.2405
			28	ND	ND	0.017	0.041	0.11	0.024	<0.01	ND	
				ND	<0.01	<0.01	0.041	0.11	0.024	<0.01	ND	
			Mean	<0.01	<0.01	0.0135	0.041	0.11	0.024	<0.01	<0.01	0.2545
			36	ND	ND	0.012	0.038	0.11	0.028	<0.01	ND	
				ND	ND	0.016	0.033	0.098	0.020	<0.01	ND	
			Mean	<0.01	<0.01	0.014	0.0355	0.1045	0.024	<0.01	<0.01	0.2465
Chula, GA, United States, 2014 Athena Trail 14 03 03 melon **pulp**	4 (14 14 14) drip inj	0.56 0.56 0.56 0.56	1	<0.01	ND	0.028	0.025	0.12	0.029	<0.01	ND	
				<0.01	ND	0.022	0.022	0.093	0.024	<0.01	ND	
			Mean	<0.01	<0.01	0.025	0.0235	0.1065	0.0265	<0.01	<0.01	0.2772
			7	<0.01	ND	0.027	0.031	0.12	0.027	<0.01	ND	
				<0.01	ND	0.030	0.030	0.094	0.021	<0.01	ND	
			Mean	<0.01	<0.01	0.0285	0.0305	0.107	0.024	<0.01	<0.01	0.2816
			14	0.01[A]	<0.01	0.038	0.035	0.11	0.028	<0.01	ND	
				0.01	<0.01	0.043	0.041	0.13	0.035	<0.01	ND	
			Mean	0.01	<0.01	0.0405	0.038	0.12	0.0315	<0.01	<0.01	0.3380
			22	<0.01	<0.01	0.025	0.043	0.13	0.026	<0.01	ND	
				<0.01	<0.01	0.023	0.036	0.13	0.027	<0.01	ND	
			Mean	<0.01	<0.01	0.024	0.0395	0.13	0.0265	<0.01	<0.01	0.3109
			28	<0.01	ND	0.021	0.044	0.15	0.035	<0.01	<0.01	
				<0.01	ND	0.024	0.034	0.12	0.023	<0.01	ND	
			Mean	<0.01	<0.01	0.0225	0.039	0.135	0.029	<0.01	<0.01	0.3191
			36	ND	ND	0.014	0.041	0.12	0.026	<0.01	ND	
				---	---	---	---	---	---	---	---	
			Mean	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	0.2738
Chula, GA, United States, 2014 Athena 02 melon **peel**	3 (50 14) drip inj	1.12 0.56 0.56	1	<0.01	<0.01	0.036	0.035	0.096	0.034	ND	ND	
				<0.01	<0.01	0.037	0.031	0.084	0.028	ND	ND	
			Mean	<0.01	<0.01	0.0365	0.033	0.090	0.031	<0.01	<0.01	
			7	<0.01	<0.01	0.039	0.033	0.12	0.029	ND	ND	
				<0.01	0.010	0.041	0.037	0.11	0.032	ND	ND	
			Mean	<0.01	<0.01	0.040	0.035	0.115	0.0315	<0.01	<0.01	
			14	<0.01	ND	0.034	0.037	0.13	0.027	<0.01	ND	
				<0.01	ND	0.042	0.043	0.14	0.030	<0.01	ND	
			Mean	<0.01	<0.01	0.038	0.040	0.135	0.0285	<0.01	<0.01	
			22	<0.01	<0.01	0.031	0.048	0.19	0.041	0.01[A]	ND	
				<0.01	<0.01	0.041	0.049	0.16	0.034	<0.01	ND	
			Mean	<0.01	<0.01	0.036	0.0485	0.175	0.0375	<0.01	<0.01	
			28	<0.01	<0.01	0.019	0.042	0.16	0.037	<0.01	ND	
				<0.01	<0.01	0.016	0.040	0.16	0.038	<0.01	ND	
			Mean	<0.01	<0.01	0.0175	0.041	0.16	0.0375	<0.01	<0.01	
			36	ND	<0.01	0.024	0.040	0.17	0.041	<0.01	ND	
				ND	<0.01	0.027	0.038	0.16	0.035	<0.01	ND	
			Mean	<0.01	<0.01	0.0255	0.039	0.165	0.038	<0.01	<0.01	
Chula, GA, United States, 2014 Athena 03 **peel**	4 (14 14 14) drip inj	0.56 0.56 0.56 0.56	1	<0.01	0.011	0.060	0.059	0.21	0.064	<0.01	ND	
				<0.01	<0.01	0.051	0.046	0.14	0.047	ND	ND	
			Mean	<0.01	<0.0105	0.0555	0.0525	0.175	0.0555	<0.01	<0.01	
			7	<0.01	0.011	0.053	0.046	0.23	0.057	<0.01	ND	
				<0.01	0.01[A]	0.051	0.041	0.17	0.041	<0.01	ND	
			Mean	<0.01	<0.0105	0.052	0.0435	0.20	0.049	<0.01	<0.01	
			14	<0.01	<0.01	0.056	0.045	0.21	0.041	<0.01	ND	
				0.011	0.012	0.066	0.063	0.23	0.061	0.011	<0.01	
			Mean	<0.0105	<0.011	0.061	0.054	0.22	0.051	<0.0105	<0.01	
			22	<0.01	<0.01	0.045	0.059	0.19	0.054	<0.01	ND	
				<0.01	<0.01	0.041	0.053	0.20	0.049	<0.01	ND	
			Mean	<0.01	<0.01	0.043	0.056	0.195	0.0515	<0.01	<0.01	
			28	<0.01	0.010	0.045	0.061	0.25	0.070	<0.01	<0.01	
				<0.01	<0.01	0.046	0.057	0.19	0.047	<0.01	ND	
			Mean	<0.01	<0.01	0.0455	0.059	0.22	0.0585	<0.01	<0.01	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			36	ND	<0.01	0.024	0.045	0.21	0.049	<0.01	ND	
Lime Springs, IA, United States, 2014 Minnesota Midget Trial 15 02 **pulp**	3 (65 20) Drench	1.12 0.56 0.56	1	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			6	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			13	<0.01	ND	<0.01	ND	<0.01	ND	ND	ND	
				0.010	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			20	<0.01	ND	ND	ND	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
Lime Springs, IA, United States, 2014 Minnesota Midget Trial 15 03 **pulp**	4 (14 14 20) drench	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				<0.01	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			13	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			20	ND	ND	ND	ND	ND	ND	ND	ND	
				<0.01	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
Lime Springs, IA, United States, 2014 Minnesota Midget Trial 14 05 **pulp**	4 (14 14 20) drench	1.12 1.12 1.12 1.12	1	ND	ND	ND	ND	ND	ND	ND	ND	
				<0.01	ND	<0.01	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			6	<0.01	ND	<0.01	ND	<0.01	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			13	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			20	ND	ND	ND	ND	<0.01	ND	ND	ND	
				<0.01	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
Lime Springs, IA, United States, 2014 Minnesota Midget Trial 15 02 **peel**	3 (65 20) Drench	1.12 0.56 0.56	1	0.019	ND	0.016	0.013	<0.01	ND	ND	ND	
				0.020	ND	0.014	0.014	<0.01	ND	ND	ND	
			Mean	0.0195	<0.01	0.015	0.0135	<0.01	<0.01	<0.01	<0.01	
			6	<0.01	ND	0.01[A]	<0.01	0.010	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			13	<0.01	ND	0.010	<0.01	0.011	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	<0.01	
			20	ND	ND	<0.01	ND	0.01[A]	ND	ND	ND	
				<0.01	ND	0.013	<0.01	0.017	ND	ND	ND	
			Mean	<0.01	<0.01	0.0115	<0.01	0.0135	<0.01	<0.01	<0.01	
Lime Springs, IA, United States, 2014 Minnesota Midget Trial 15 03 **peel**	4 (14 14 20) drench	0.56 0.56 0.56 0.56	1	0.046	ND	0.029	0.030	<0.01	ND	ND	ND	
				0.069	ND	0.040	0.043	<0.01	ND	ND	ND	
			Mean	0.0575	<0.01	0.0345	0.0365	<0.01	<0.01	<0.01	<0.01	
			6	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				0.012	ND	0.012	<0.01	<0.01	ND	ND	ND	
			Mean	<0.011	<0.01	<0.011	<0.01	<0.01	<0.01	<0.01	<0.01	
			13	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			20	<0.01	ND	<0.01	ND	<0.01	ND	ND	ND	
				0.023	<0.01	0.024	0.015	<0.01	ND	ND	ND	
			Mean	<0.0165	<0.01	0.017	<0.0125	<0.01	<0.01	<0.01	<0.01	
Lime Springs, IA, United States, 2014 Minnesota Midget Trial 15 05 **peel**	4 (14 14 20) drench	1.12 1.12 1.12 1.12	1	0.058	ND	0.032	0.034	<0.01	ND	ND	ND	
				0.039	ND	0.022	0.025	<0.01	ND	ND	ND	
			Mean	0.0485	<0.01	0.027	0.0295	<0.01	<0.01	<0.01	<0.01	
			6	0.025	ND	0.022	0.018	<0.01	ND	ND	ND	
				0.012	ND	0.011	<0.01	<0.01	ND	ND	ND	
			Mean	0.0185	<0.01	0.0165	<0.014	<0.01	<0.01	<0.01	<0.01	
			13	0.048	<0.01	0.044	0.035	0.014	ND	ND	ND	
				0.031	ND	0.030	0.023	<0.01	ND	ND	ND	
			Mean	0.0395	<0.01	0.037	0.029	<0.012	<0.01	<0.01	<0.01	
			20	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
Carlyle, IL, United States, 2014 Athena Trial 21 02 **pulp**	3 (67 14) drip	1.12 0.56 0.56	1	ND	ND	ND	<0.01	0.021	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.018	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0195	<0.01	<0.01	<0.01	0.0884
			6	ND	ND	ND	ND	0.012	ND	ND	ND	
				ND	ND	ND	<0.01	0.014	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	<0.01	0.0786
			14	ND	ND	ND	<0.01	0.016	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.015	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0155	<0.01	<0.01	<0.01	0.0824
			21	ND	ND	ND	<0.01	0.019	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.022	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0205	<0.01	<0.01	<0.01	0.0900
			28	ND	ND	ND	<0.01	0.019	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.017	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.018	<0.01	<0.01	<0.01	0.0862
Carlyle, IL, United States, 2014 Athena Trial 21 03 **pulp**	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	0.011	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	<0.01	0.0748
			6	ND	ND	ND	<0.01	0.011	<0.01	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	<0.01	0.0748
			14	ND	ND	ND	<0.01	0.011	ND	ND	ND	
				ND	ND	ND	ND	0.012	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0115	<0.01	<0.01	<0.01	0.0763
			21	ND	ND	ND	<0.01	0.015	<0.01	ND	ND	
				ND	ND	ND	ND	0.012	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0135	<0.01	<0.01	<0.01	0.0793
			28	ND	ND	ND	<0.01	0.013	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.015	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.014	<0.01	<0.01	<0.01	0.0801
Carlyle, IL, United States, 2014 Athena Trial 21 02 **peel**	3 (67 14) drip	1.12 0.56 0.56	1	0.014	ND	<0.01	0.013	0.028	<0.01	ND	ND	
				0.014	ND	<0.01	0.012	0.030	<0.01	ND	ND	
			Mean	0.014	<0.01	<0.01	0.0125	0.029	<0.01	<0.01	<0.01	
			6	ND	ND	ND	<0.01	0.020	<0.01	ND	ND	
				<0.01	ND	ND	<0.01	0.024	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.022	<0.01	<0.01	<0.01	
			14	ND	ND	<0.01	<0.01	0.029	<0.01	ND	ND	
				ND	ND	<0.01	<0.01	0.028	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0285	<0.01	<0.01	<0.01	
			21	ND	ND	ND	<0.01	0.028	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.036	0.011	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.032	<0.0105	<0.01	<0.01	
			28	ND	ND	ND	<0.01	0.035	0.010	ND	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
				<0.01	ND	<0.01	<0.01	0.035	0.01[A]	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.035	0.01[A]	<0.01	<0.01	
Carlyle, IL, United States, 2014 Athena Trial 21 03 **peel**	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	<0.01	0.020	<0.01	ND	ND	
				<0.01	ND	<0.01	<0.01	0.015	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0175	<0.01	<0.01	<0.01	
			6	ND	ND	<0.01	<0.01	0.022	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.016	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.019	<0.01	<0.01	<0.01	
			14	ND	ND	<0.01	<0.01	0.019	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.020	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0195	<0.01	<0.01	<0.01	
			21	ND	ND	<0.01	<0.01	0.026	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.022	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.024	<0.01	<0.01	<0.01	
			28	ND	ND	ND	<0.01	0.027	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.028	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0275	<0.01	<0.01	<0.01	
Uvalde, TX, United States, 2014 Primo Trial 22 02 **pulp**	3 (91 14) drip irr	1.13 0.55 0.55	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	0.013	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.01	<0.01	0.0763
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
Uvalde, TX, United States, 2014 Primo Trial 22 03 **pulp**	4 (14 13 14) drip irr	0.55 0.55 0.55 0.55	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
Uvalde, TX, United States, 2014 Primo Trial 22 02 **peel**	3 (91 14) drip irr	1.13 0.55 0.55	1	ND	ND	ND	ND	<0.01	ND	ND	ND	
				<0.01	ND	0.013	<0.01	0.032	0.014	ND	ND	
			Mean	<0.01	<0.01	<0.0115	<0.01	<0.021	<0.012	<0.01	<0.01	
			7	ND	ND	<0.01	ND	<0.01	<0.01	ND	ND	
				ND	ND	<0.01	ND	<0.01	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			14	ND	ND	<0.01	ND	<0.01	<0.01	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
Uvalde, TX, United States, 2014 Primo Trial 22 03 **peel**	4 (14 13 14) drip irr	0.55 0.55 0.55 0.55	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			14	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
Yuma, AZ, United States, 2014 Fiji Trial 23 02	3 (76 14) drip	1.12 0.56 0.56	1	ND	ND	ND	ND	<0.01	<0.01	ND	ND	
				ND	ND	ND	ND	0.031	0.015	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0205	<0.0125	<0.01	<0.01	0.0937
			7	ND	ND	ND	ND	0.021	0.011	<0.01	ND	
				ND	ND	ND	ND	0.030	0.014	<0.01	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
pulp			Mean	<0.01	<0.01	<0.01	<0.01	0.0255	0.0125	<0.01	<0.01	0.1013
			15	ND	ND	ND	ND	0.026	0.017	<0.01	ND	
				ND	ND	ND	ND	0.030	0.022	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.028	0.0195	<0.01	<0.01	0.1157
			23	ND	ND	ND	ND	0.018	0.015	<0.01	ND	
				ND	ND	<0.01	ND	0.040	0.038	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.029	0.0265	<0.01	<0.01	0.1278
Yuma, AZ, United States, 2014 Fiji Trial 23 03 **pulp**	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	0.035	0.013	<0.01	ND	
				ND	ND	ND	ND	0.033	0.017	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.034	0.015	<0.01	<0.01	0.1180
			7	ND	ND	ND	ND	<0.01	<0.01	ND	ND	
				ND	ND	ND	ND	0.033	0.016	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0215	<0.013	<0.01	<0.01	0.0960
			15	ND	ND	0.011	<0.01	0.080	0.044	<0.01	<0.01	
				ND	ND	<0.01	ND	0.039	0.020	<0.01	ND	
			Mean	<0.01	<0.01	<0.0105	<0.01	0.0595	0.032	<0.01	<0.01	0.1835
			23	ND	ND	<0.01	ND	0.019	0.01[A]	<0.01	ND	
				ND	ND	0.011	<0.01	0.082	0.042	<0.01	ND	
			Mean	<0.01	<0.01	<0.0105	<0.01	0.0505	0.026	<0.01	<0.01	0.1608
Yuma, AZ, United States, 2014 Fiji Trial 23 02 **peel**	3 (76 14) drip	1.12 0.56 0.56	1	ND	ND	ND	ND	0.018	0.011	ND	ND	
				ND	ND	<0.01	ND	0.052	0.053	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.035	0.032	<0.01	<0.01	
			7	0.010	ND	0.010	<0.01	0.028	0.028	ND	ND	
				<0.01	<0.01	0.015	<0.01	0.046	0.042	ND	ND	
			Mean	<0.01	<0.01	0.0125	<0.01	0.037	0.035	<0.01	<0.01	
			15	<0.01	ND	0.018	<0.01	0.033	0.045	ND	<0.01	
				<0.01	<0.01	0.015	<0.01	0.044	0.053	ND	<0.01	
			Mean	<0.01	<0.01	0.0165	<0.01	0.0385	0.049	<0.01	<0.01	
			23	ND	ND	0.010	ND	0.024	0.037	ND	<0.01	
				ND	ND	0.018	<0.01	0.048	0.080	<0.01	<0.01	
			Mean	<0.01	<0.01	0.014	<0.01	0.036	0.0585	<0.01	<0.01	
Yuma, AZ, United States, 2014 Fiji Trial 23 03 **peel**	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	ND	0.013	<0.01	0.049	0.035	ND	ND	
				<0.01	<0.01	0.022	<0.01	0.045	0.050	ND	ND	
			Mean	<0.01	<0.01	0.0175	<0.01	0.047	0.0425	<0.01	<0.01	
			7	<0.01	ND	0.011	<0.01	<0.01	<0.01	ND	ND	
				<0.01	<0.01	0.027	<0.01	0.046	0.047	ND	<0.01	
			Mean	<0.01	<0.01	0.019	<0.01	<0.028	<0.0285	<0.01	<0.01	
			15	0.012	<0.01	0.058	0.018	0.089	0.11	ND	<0.01	
				ND	<0.01	0.027	<0.01	0.050	0.050	ND	<0.01	
			Mean	<0.011	<0.01	0.0425	<0.014	0.0695	0.08	<0.01	<0.01	
			23	<0.01	<0.01	0.019	<0.01	0.030	0.026	ND	ND	
				ND	<0.01	0.034	0.010	0.098	0.097	<0.01	<0.01	
			Mean	<0.01	<0.01	0.0265	<0.01	0.064	0.0615	<0.01	<0.01	
Porterville, CA, United States, 2014 Hales Best Jumbo Trial 24 02 **pulp**	3 (45 14) drip	1.12 0.56 0.56	1	ND	ND	0.01[A]	<0.01	0.031	0.013	ND	ND	
				ND	ND	0.010	<0.01	0.031	<0.01	ND	ND	
			Mean	<0.01	<0.01	0.01[A]	<0.01	0.031	0.0115	<0.01	<0.01	0.1082
			6	ND	ND	0.012	<0.01	0.043	0.018	ND	ND	
				ND	ND	0.013	<0.01	0.037	0.012	ND	ND	
			Mean	<0.01	<0.01	0.0125	<0.01	0.040	0.015	<0.01	<0.01	0.1324
			14	ND	ND	0.016	<0.01	0.047	0.017	<0.01	ND	
				ND	ND	0.016	<0.01	0.050	0.018	ND	ND	
			Mean	<0.01	<0.01	0.016	<0.01	0.0485	0.0175	<0.01	<0.01	0.1565
			21	ND	ND	0.013	<0.01	0.062	0.020	ND	ND	
				ND	ND	0.016	<0.01	0.050	0.017	<0.01	ND	
			Mean	<0.01	<0.01	0.0145	<0.01	0.056	0.0185	<0.01	<0.01	0.1662
			28	ND	ND	0.024	<0.01	0.072	0.022	<0.01	ND	
				ND	ND	0.016	<0.01	0.058	0.020	<0.01	ND	
			Mean	<0.01	<0.01	0.020	<0.01	0.065	0.021	<0.01	<0.01	0.1953

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			35	ND	ND	0.018	<0.01	0.052	0.021	<0.01	ND	
				ND	ND	0.017	<0.01	0.062	0.027	<0.01	ND	
			Mean	<0.01	<0.01	0.0175	<0.01	0.057	0.024	<0.01	<0.01	0.1824
Porterville, CA, United States, 2014 Hales Best Jumbo Trial 24 03 **pulp**	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	ND	<0.01	<0.01	0.046	0.016	ND	ND	
				ND	ND	<0.01	<0.01	0.045	0.014	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0455	0.015	<0.01	<0.01	0.1355
			6	ND	ND	0.013	<0.01	0.047	0.011	ND	ND	
				ND	ND	0.013	<0.01	0.058	0.016	<0.01	ND	
			Mean	<0.01	<0.01	0.013	<0.01	0.0525	0.0135	<0.01	<0.01	0.1502
			14	ND	ND	0.016	<0.01	0.058	0.019	<0.01	ND	
				ND	ND	0.016	<0.01	0.056	0.021	ND	ND	
			Mean	<0.01	<0.01	0.016	<0.01	0.057	0.020	<0.01	<0.01	0.1732
			21	ND	ND	0.018	<0.01	0.047	0.018	ND	ND	
				ND	ND	ND	<0.01	0.058	0.021	<0.01	ND	
			Mean	<0.01	<0.01	<0.014	<0.01	0.0525	0.020	<0.01	<0.01	0.1614
			28	ND	ND	0.028	<0.01	0.077	0.030	<0.01	ND	
				ND	ND	0.028	<0.01	0.049	0.019	<0.01	ND	
			Mean	<0.01	<0.01	0.028	<0.01	0.063	0.0245	<0.01	<0.01	0.2144
			35	ND	ND	0.022	<0.01	0.077	0.027	<0.01	<0.01	
				ND	ND	0.020	<0.01	0.060	0.027	<0.01	<0.01	
			Mean	<0.01	<0.01	0.021	<0.01	0.0685	0.027	<0.01	<0.01	0.2118
Porterville, CA, United States, 2014 Hales Best Jumbo Trial 24 02 **peel**	3 (45 14) drip	1.12 0.56 0.56	1	<0.01	<0.01	0.022	0.014	0.058	0.028	<0.01	ND	
				0.013	<0.01	0.020	0.017	0.047	0.017	<0.01	ND	
			Mean	0.0115	<0.01	0.021	0.0155	0.0525	0.0225	<0.01	<0.01	
			6	0.01[A]	<0.01	0.029	0.020	0.087	0.050	<0.01	ND	
				0.016	<0.01	0.036	0.023	0.067	0.034	<0.01	ND	
			Mean	0.013	<0.01	0.0325	0.0215	0.077	0.042	<0.01	<0.01	
			14	<0.01	<0.01	0.031	0.016	0.068	0.039	<0.01	ND	
				<0.01	<0.01	0.032	0.019	0.073	0.053	<0.01	ND	
			Mean	<0.01	<0.01	0.0315	0.0175	0.0705	0.046	<0.01	<0.01	
			21	<0.01	<0.01	0.039	0.018	0.12	0.071	<0.01	ND	
				<0.01	<0.01	0.037	0.016	0.086	0.062	<0.01	ND	
			Mean	<0.01	<0.01	0.038	0.017	0.103	0.0665	<0.01	<0.01	
			28	<0.01	0.010	0.053	0.023	0.16	0.097	<0.01	<0.01	
				0.012	<0.01	0.046	0.024	0.095	0.064	<0.01	<0.01	
			Mean	<0.011	<0.01	0.0495	0.0235	0.1275	0.0805	<0.01	<0.01	
			35	<0.01	<0.01	0.042	0.017	0.11	0.080	<0.01	<0.01	
				0.011	<0.01	0.053	0.027	0.12	0.11	<0.01	<0.01	
			Mean	<0.0105	<0.01	0.0475	0.022	0.115	0.095	<0.01	<0.01	
Porterville, CA, United States, 2014 Hales Best Jumbo Trial 24 03 **peel**	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	0.01[A]	<0.01	0.023	0.015	0.082	0.028	<0.01	ND	
				<0.01	<0.01	0.025	0.016	0.10	0.036	<0.01	<0.01	
			Mean	<0.01	<0.01	0.024	0.0155	0.091	0.032	<0.01	<0.01	
			6	0.012	<0.01	0.041	0.019	0.11	0.040	<0.01	<0.01	
				<0.01	<0.01	0.038	0.019	0.13	0.058	<0.01	<0.01	
			Mean	<0.011	<0.01	0.0395	0.019	0.12	0.049	<0.01	<0.01	
			14	<0.01	<0.01	0.033	0.014	0.11	0.048	<0.01	<0.01	
				<0.01	<0.01	0.042	0.016	0.099	0.053	<0.01	<0.01	
			Mean	<0.01	<0.01	0.0375	0.015	0.0995	0.0505	<0.01	<0.01	
			21	<0.01	<0.01	0.038	0.011	0.12	0.051	<0.01	ND	
				<0.01	<0.01	0.045	0.014	0.14	0.071	<0.01	<0.01	
			Mean	<0.01	<0.01	0.0415	0.0125	0.13	0.061	<0.01	<0.01	
			28	0.022	<0.01	0.058	0.029	0.16	0.052	<0.01	<0.01	
				<0.01	<0.01	0.060	0.018	0.17	0.097	<0.01	<0.01	
			Mean	<0.016	<0.01	0.059	0.0235	0.165	0.0745	<0.01	<0.01	
			35	<0.01	0.011	0.059	0.020	0.19	0.10	0.010	<0.01	
				<0.01	<0.01	0.060	0.019	0.16	0.093	0.011	<0.01	
			Mean	<0.01	<0.0105	0.0595	0.0195	0.175	0.0965	0.0105	<0.01	
Yuba City, CA,	3 (59	1.12	1	ND	ND	ND	ND	0.025	<0.01	ND	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
United States,	14)	0.56		ND	ND	ND	<0.01	0.022	<0.01	ND	ND	
2014 Hales	drip irr	0.56	Mean	<0.01	<0.01	<0.01	<0.01	0.0235	<0.01	<0.01	<0.01	0.0945
Best Jumbo			7	ND	ND	ND	<0.01	0.026	<0.01	ND	ND	
Trial 25				ND	ND	ND	<0.01	0.030	0.011	<0.01	ND	
02			Mean	<0.01	<0.01	<0.01	<0.01	0.028	<0.0105	<u><0.01</u>	<u><0.01</u>	0.1021
pulp			14	ND	ND	ND	<0.01	0.024	<0.01	ND	ND	
				ND	ND	ND	<0.01	0.029	0.01[A]	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0265	<0.01	<0.01	<0.01	0.0991
			21	ND	ND	ND	<0.01	0.032	0.01[A]	ND	ND	
				ND	ND	ND	<0.01	0.027	0.011	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0295	0.0105	<0.01	<0.01	0.1044
			28	ND	ND	ND	<0.01	0.045	0.011	<0.01	ND	
				ND	ND	ND	<0.01	0.051	0.012	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.048	0.0115	<0.01	<0.01	<u>0.1340</u>
			35	ND	ND	ND	<0.01	0.034	0.01[A]	<0.01	ND	
				ND	ND	ND	<0.01	0.027	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0305	<0.01	<0.01	<0.01	0.1052
Yuba City, CA,	4 (14	0.56	1	ND	ND	ND	ND	<0.01	<0.01	ND	ND	
United States,	14 14)	0.56		ND	ND	ND	ND	<0.01	ND	ND	ND	
2014 Hales	drip irr	0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
Best Jumbo		0.56	7	ND	ND	ND	ND	<0.01	ND	ND	ND	
Trial 25				ND	ND	ND	ND	<0.01	ND	ND	ND	
03			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
pulp			14	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			21	ND	ND	ND	ND	0.012	<0.01	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.074
			28	ND	ND	ND	ND	0.015	<0.01	ND	ND	
				ND	ND	ND	ND	0.014	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0145	<0.01	<0.01	<0.01	0.0755
			35	ND	ND	ND	ND	0.014	<0.01	ND	ND	
				ND	ND	ND	ND	0.011	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0125	<0.01	<0.01	<0.01	0.0808
Yuba City, CA,	3 (59	1.12	1	ND	ND	<0.01	<0.01	0.041	0.022	ND	ND	
United States,	14)	0.56		ND	ND	<0.01	<0.01	0.035	0.019	ND	ND	
2014 Hales	drip irr	0.56	Mean	<0.01	<0.01	<0.01	<0.01	0.038	0.0215	<0.01	<0.01	
Best Jumbo			7	ND	ND	<0.01	<0.01	0.038	0.025	<0.01	ND	
Trial 25				ND	ND	<0.01	<0.01	0.038	0.024	ND	ND	
02			Mean	<0.01	<0.01	<0.01	<0.01	0.038	0.0245	<0.01	<0.01	
peel			14	ND	ND	<0.01	<0.01	0.036	0.021	<0.01	ND	
				ND	ND	<0.01	<0.01	0.041	0.021	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0385	0.021	<0.01	<0.01	
			21	ND	ND	<0.01	<0.01	0.047	0.024	<0.01	ND	
				ND	ND	<0.01	<0.01	0.035	0.025	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.041	0.0245	<0.01	<0.01	
			28	ND	ND	ND	<0.01	0.042	0.028	ND	ND	
				ND	ND	ND	<0.01	0.053	0.026	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0475	0.027	<0.01	<0.01	
			35	ND	ND	ND	<0.01	0.033	0.015	ND	ND	
				ND	ND	ND	<0.01	0.026	0.016	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0295	0.0155	<0.01	<0.01	
Yuba City, CA,	4 (14	0.56	1	ND	ND	<0.01	ND	0.010	<0.01	ND	ND	
United States,	14 14)	0.56		ND	ND	<0.01	ND	0.012	<0.01	ND	ND	
2014 Hales	drip irr	0.56	Mean	<0.01	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	
Best Jumbo		0.56	7	ND	ND	<0.01	ND	0.010	<0.01	ND	ND	
Trial 25				ND	ND	<0.01	ND	0.013	<0.01	ND	ND	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
03 **peel**			Mean	<0.01	<0.01	<0.01	<0.01	0.0115	<0.01	<0.01	<0.01	
			14	ND	ND	<0.01	ND	0.012	<0.01	ND	ND	
				ND	ND	<0.01	ND	0.011	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0115	<0.01	<0.01	<0.01	
			21	ND	ND	ND	ND	0.016	0.011	ND	ND	
				ND	ND	ND	ND	<0.01	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.013	<0.01	<0.01	<0.01	
			28	ND	ND	ND	ND	0.014	<0.01	ND	ND	
				ND	ND	ND	ND	0.013	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0135	<0.01	<0.01	<0.01	
			35	ND	ND	ND	ND	0.021	<0.01	ND	ND	
				ND	ND	<0.01	ND	0.017	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.019	<0.01	<0.01	<0.01	
Sanger, CA, United States, 2014 Summer Dew Trial 26 02 **pulp**	3 (62 14) soil-directed 2×drip	1.13 0.56 0.56	1	ND	<0.01	0.048	0.044	0.077	0.017	<0.01	<0.01	
				ND	ND	0.028	0.054	0.12	0.028	<0.01	0.01 [A]	
			Mean	<0.01	<0.01	0.038	0.049	0.0985	0.0225	<0.01	<0.01	0.2865
			6	ND	ND	0.021	0.048	0.092	0.027	<0.01	<0.01	
				ND	<0.01	0.062	0.094	0.20	0.046	0.015	0.011	
			Mean	<0.01	<0.01	0.0415	0.071	0.146	0.0365	<0.0125	<0.0105	0.3872
			14	ND	ND	0.018	0.045	0.072	0.021	<0.01	<0.01	
				ND	ND	0.026	0.045	0.092	0.026	<0.01	<0.01	
			Mean	<0.01	<0.01	0.022	0.045	0.082	0.0235	<0.01	<0.01	0.2291
			21	ND	ND	0.017	0.035	0.047	0.017	<0.01	<0.01	
				ND	ND	0.023	0.047	0.082	0.032	0.011	<0.01	
			Mean	<0.01	<0.01	0.020	0.041	0.0645	0.0245	<0.0105	<0.01	0.1998
			28	ND	ND	0.027	0.039	0.041	0.016	<0.01	<0.01	
				ND	ND	0.013	0.039	0.044	0.014	<0.01	<0.01	
			Mean	<0.01	<0.01	0.020	0.039	0.0425	0.015	<0.01	<0.01	0.1520
			35	ND	ND	0.022	0.042	0.085	0.038	0.011	<0.01	
				ND	<0.01	0.032	0.077	0.23	0.061	0.014	0.011	
			Mean	<0.01	<0.01	0.027	0.0595	0.1575	0.0495	0.0125	<0.0105	0.3937
Sanger, CA, United States, 2014 Summer Dew Trial 26 03 **pulp**	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	<0.01	0.060	0.061	0.091	0.023	<0.01	<0.01	
				ND	ND	0.026	0.064	0.15	0.034	0.010	<0.01	
			Mean	<0.01	<0.01	0.043	0.0625	0.1205	0.0285	<0.01	<0.01	0.3395
			6	ND	<0.01	0.040	0.053	0.077	0.017	<0.01	<0.01	
				ND	<0.01	0.033	0.043	0.071	0.015	<0.01	<0.01	
			Mean	<0.01	<0.01	0.0365	0.048	0.074	0.016	<0.01	<0.01	0.2363
			14	ND	ND	0.051	0.044	0.061	0.018	<0.01	<0.01	
				ND	<0.01	0.058	0.059	0.082	0.027	<0.01	<0.01	
			Mean	<0.01	<0.01	0.0545	0.0515	0.0715	0.0225	<0.01	<0.01	0.2802
			21	ND	<0.01	0.047	0.060	0.074	0.023	<0.01	<0.01	
				ND	<0.01	0.062	0.069	0.088	0.031	<0.01	<0.01	
			Mean	<0.01	<0.01	0.0545	0.0645	0.081	0.027	<0.01	<0.01	0.3105
			28	ND	<0.01	0.042	0.045	0.053	0.020	<0.01	<0.01	
				ND	<0.01	0.051	0.048	0.064	0.022	0.01 [A]	<0.01	
			Mean	<0.01	<0.01	0.0465	0.0465	0.0585	0.021	<0.01	<0.01	0.2413
			35	ND	<0.01	0.050	0.063	0.085	0.025	0.011	<0.01	
				ND	0.01 [A]	0.051	0.075	0.078	0.026	0.011	<0.01	
			Mean	<0.01	<0.01	0.0505	0.069	0.0815	0.0255	0.011	<0.01	0.2915
Sanger, CA, United States, 2014 Summer Dew Trial 26 02 **peel**	3 (62 14) soil-directed 2×drip	1.13 0.56 0.56	1	<0.01	0.015	0.083	0.029	0.13	0.036	<0.01	<0.01	
				ND	0.013	0.046	0.033	0.18	0.063	<0.01	<0.01	
			Mean	<0.01	0.014	0.0645	0.031	0.155	0.0495	<0.01	<0.01	
			6	ND	0.012	0.034	0.024	0.13	0.043	<0.01	<0.01	
				ND	0.028	0.080	0.054	0.34	0.085	0.015	0.011	
			Mean	<0.01	0.020	0.057	0.039	0.235	0.064	<0.0125	<0.0105	
			14	ND	0.017	0.042	0.035	0.12	0.044	<0.01	<0.01	
				ND	0.017	0.043	0.031	0.24	0.064	0.011	<0.01	
			Mean	<0.01	0.017	0.0425	0.033	0.18	0.054	<0.0105	<0.01	

Location, year variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			21	ND	0.010	0.031	0.035	0.19	0.045	<0.01	<0.01	
				ND	0.023	0.053	0.045	0.18	0.077	0.013	0.010	
			Mean	<0.01	0.0165	0.042	0.040	0.185	0.061	<0.0115	<0.01	
			28	ND	0.019	0.052	0.040	0.15	0.044	<0.01	<0.01	
				ND	0.012	0.026	0.036	0.16	0.045	<0.01	<0.01	
			Mean	<0.01	0.0155	0.039	0.038	0.155	0.0445	<0.01	<0.01	
			35	ND	0.018	0.043	0.051	0.39	0.13	0.019	0.017	
				ND	0.027	0.056	0.13	0.64	0.17	0.022	0.013	
			Mean	<0.01	0.0225	0.0495	0.0905	0.515	0.15	0.0205	0.015	
Sanger, CA, United States, 2014 Summer Dew Trial 26 03 **peel**	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	<0.01	0.023	0.11	0.052	0.15	0.052	<0.01	<0.01	
				<0.01	0.014	0.051	0.052	0.30	0.066	0.01 [A]	<0.01	
			Mean	<0.01	0.0185	0.0805	0.052	0.225	0.059	<0.01	<0.01	
			6	ND	0.015	0.071	0.034	0.13	0.029	<0.01	<0.01	
				ND	0.020	0.081	0.046	0.15	0.040	<0.01	<0.01	
			Mean	<0.01	0.0175	0.076	0.040	0.14	0.0345	<0.01	<0.01	
			14	ND	0.023	0.088	0.035	0.11	0.032	<0.01	<0.01	
				ND	0.032	0.10	0.053	0.17	0.048	0.010	<0.01	
			Mean	<0.01	0.0275	0.094	0.044	0.14	0.040	<0.01	<0.01	
			21	ND	0.034	0.11	0.075	0.20	0.060	0.013	<0.01	
				ND	0.047	0.14	0.078	0.19	0.085	0.015	<0.01	
			Mean	<0.01	0.0405	0.125	0.0765	0.195	0.0725	0.014	<0.01	
			28	ND	0.023	0.081	0.038	0.14	0.055	0.010	<0.01	
				ND	0.034	0.10	0.051	0.17	0.059	0.014	<0.01	
			Mean	<0.01	0.0285	0.0905	0.0445	0.155	0.057	0.012	<0.01	
			35	ND	0.050	0.12	0.059	0.23	0.084	0.020	0.012	
				ND	0.050	0.12	0.074	0.22	0.089	0.019	0.011	
			Mean	<0.01	0.050	0.12	0.0665	0.2225	0.0865	0.0195	0.0115	

Notes:

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] SUM = 2.26×IN-A5760 + 2.11×IN-F4106 + 1.52×IN-QZY47 + 1.51×IN-TMQ01.

[C] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 137 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post hydrolysis) in whole melon, calculated from pulp and peel residues (ND = 0, >ND but <LOQ = LOQ)

Location, year, variety	Rate (kg ai/ha)	Fraction pulp	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Branchton, ON, Canada, 2014 Primo	1.11 0.56 0.56	0.54 0.57	2	0.01	0.01	0.01	0.01	0.012	0.01	0.01	0.01
				0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
		0.56 0.60	6	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
				0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.012	0.01	0.01	0.01
		0.59 0.58	14	0.01	0.01	0.01	0.01	0.020	0.01	0.01	0.01
				0.01	0.01	0.01	0.01	0.012	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.016	0.01	0.01	0.01
		0.58 0.57	21	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
				0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.65 0.62	28	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
				0.01	0.01	0.01	0.01	0.012	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
Branchton, ON, Canada,	0.56 0.56	0.59 0.56	2	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
				0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01

Location, year, variety	Rate (kg ai/ha)	Fraction pulp	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
2014 Primo	0.56		Mean	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
	0.56	0.59	6	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.55		0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.57	14	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.46		0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.55	21	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.59		0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.59	28	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.61		0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
Branchton, ON, Canada, 2014 Avatar	1.12	0.62	1	0.01	0.01	0.01	0.01	0.018	0.01	0.01	0.01
	0.56	0.59		0.01	0.01	0.01	0.01	0.017	0.01	0.01	0.01
	0.56		Mean	0.01	0.01	0.01	0.01	0.017	0.01	0.01	0.01
		0.65	7	0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
		0.61		0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
		0.65	15	0.01	0.01	0.01	0.01	0.015	0.01	0.01	0.01
		0.66		0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
		0.57	21	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
		0.57		0.01	0.01	0.01	0.01	0.015	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
		0.55	29	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
		0.55		0.01	0.01	0.01	0.01	0.015	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
Branchton, ON, Canada, 2014 Avatar	0.56	0.69	1	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
	0.56	0.70		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
	0.56		Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
	0.56	0.67	7	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.68		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.59	15	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.58		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.60	21	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.58		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.58	29	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.59		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Branchton, ON, Canada, 2014 Sugar Cube	1.12	0.53	1	0.012	0.01	0.01	0.011	0.039	0.018	0.01	0.01
	0.56	0.53		0.01	0.01	0.01	0.01	0.040	0.014	0.01	0.01
	0.56		Mean	0.011	0.01	0.01	0.01	0.040	0.016	0.01	0.01
		0.51	7	0.01	0.01	0.01	0.01	0.044	0.016	0.01	0.01
		0.55		0.013	0.01	0.014	0.014	0.064	0.021	0.01	0.01
			Mean	0.012	0.01	0.012	0.012	0.054	0.018	0.01	0.01
		0.53	16	0.01	0.01	0.01	0.01	0.058	0.020	0.01	0.01
		0.45		0.01	0.01	0.01	0.01	0.048	0.017	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.053	0.018	0.01	0.01
		0.59	23	0.01	0.01	0.01	0.01	0.057	0.020	0.01	0.01
		0.60		0.01	0.01	0.01	0.01	0.071	0.017	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.064	0.019	0.01	0.01
Branchton, ON, Canada, 2014 Sugar Cube	0.56	0.55	1	0.01	0.01	0.01	0.01	0.025	0.01	0.01	0.01
	0.56	0.54		0.01	0.01	0.01	0.01	0.020	0.01	0.01	0.01
	0.56		Mean	0.01	0.01	0.01	0.01	0.022	0.01	0.01	0.01
	0.56	0.54	7	0.01	0.01	0.01	0.01	0.036	0.011	0.01	0.01
		0.56		0.012	0.01	0.01	0.01	0.021	0.01	0.01	0.01

Location, year, variety	Rate (kg ai/ha)	Fraction pulp	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Mean	0.011	0.01	0.01	0.01	0.028	0.011	0.01	0.01
		0.55	16	0.01	0.01	0.01	0.01	0.031	0.011	0.01	0.01
		0.48		0.01	0.01	0.01	0.01	0.023	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.027	0.011	0.01	0.01
		0.61	23	0.01	0.01	0.01	0.01	0.025	0.01	0.01	0.01
		0.61		0.01	0.01	0.01	0.01	0.030	0.011	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.027	0.011	0.01	0.01
St. Marc-sur-Richelieu, QC, Canada, 2014 Goddess	1.12	0.66	1	0.01	0.01	0.01	0.01	0.033	0.018	0.01	0.01
	0.56	0.63		0.01	0.01	0.01	0.01	0.035	0.017	0.01	0.01
	0.56		Mean	0.01	0.01	0.01	0.01	0.034	0.017	0.01	0.01
		0.65	6	0.01	0.01	0.01	0.01	0.027	0.014	0.01	0.01
		0.67		0.01	0.01	0.011	0.01	0.026	0.014	0.01	0.01
			Mean	0.01	0.01	0.011	0.01	0.027	0.014	0.01	0.01
		0.66	14	0.01	0.01	0.01	0.01	0.025	0.013	0.01	0.01
		0.60		0.01	0.01	0.01	0.01	0.022	0.012	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.023	0.012	0.01	0.01
		0.52	21	0.01	0.01	0.01	0.01	0.018	0.011	0.01	0.01
		0.53		0.01	0.01	0.01	0.01	0.018	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.018	0.01	0.01	0.01
St. Marc-sur-Richelieu, QC, Canada, 2014 Goddess	0.56	0.66	1	0.01	0.01	0.01	0.01	0.012	0.01	0.01	0.01
	0.56	0.68		0.01	0.01	0.011	0.01	0.015	0.01	0.01	0.01
	0.56		Mean	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
	0.56	0.63	6	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
		0.65		0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
		0.56	14	0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
		0.58		0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
		0.51	21	0.01	0.01	0.01	0.01	0.015	0.01	0.01	0.01
		0.54		0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
St. Marc-sur-Richelieu, QC, Canada, 2014 Magnifisweet	1.12	0.59	1	0.024	0.01	0.020	0.020	0.019	0.011	0.01	0.01
	0.56	0.62		0.089	0.01	0.067	0.059	0.019	0.011	0.01	0.01
	0.56		Mean	0.056	0.01	0.043	0.040	0.019	0.011	0.01	0.01
		0.57	6	0.01	0.01	0.01	0.01	0.015	0.010	0.01	0.01
		0.58		0.01	0.01	0.01	0.01	0.013	0.011	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.014	0.010	0.01	0.01
		0.63	14	0.012	0.01	0.012	0.012	0.015	0.01	0.01	0.01
		0.64		0.011	0.01	0.01	0.01	0.016	0.01	0.01	0.01
			Mean	0.01	0.01	0.011	0.011	0.015	0.01	0.01	0.01
		0.60	21	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.56		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.59	28	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.56		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.55	34	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.57		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
St. Marc-sur-Richelieu, QC, Canada, 2014 Magnifisweet	0.56	0.66	1	0.013	0.01	0.012	0.011	0.017	0.01	0.01	0.01
	0.56	0.63		0.01	0.01	0.01	0.01	0.016	0.01	0.01	0.01
	0.56		Mean	0.011	0.01	0.011	0.011	0.017	0.01	0.01	0.01
	0.56	0.58	6	0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
		0.60		0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
		0.63	14	0.01	0.01	0.01	0.01	0.016	0.01	0.01	0.01
		0.62		0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
		0.58	21	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
		0.53		0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01

Location, year, variety	Rate (kg ai/ha)	Fraction pulp	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Mean	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
		0.60	28	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.58		0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
		0.56	34	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.54		0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
Chula, GA, United States, 2014 Athena	1.12	0.34	1	0.01	0.01	0.030	0.029	0.085	0.028	0.01	0.007
	0.56	0.33		0.01	0.01	0.031	0.027	0.075	0.023	0.007	0.007
	0.56		Mean	0.01	0.01	0.030	0.028	0.080	0.026	0.008	0.007
		0.31	7	0.01	0.01	0.034	0.028	0.104	0.025	0.007	0.007
		0.30		0.01	0.01	0.034	0.032	0.095	0.027	0.01	0.007
			Mean	0.01	0.01	0.034	0.030	0.099	0.026	0.008	0.007
		0.35	14	0.01	0.01	0.032	0.033	0.110	0.023	0.01	0.006
		0.37		0.01	0.01	0.036	0.038	0.119	0.027	0.01	0.006
			Mean	0.01	0.01	0.034	0.035	0.115	0.025	0.01	0.006
		0.31	22	0.01	0.01	0.027	0.041	0.162	0.034	0.01	0.007
		0.31		0.01	0.01	0.034	0.044	0.141	0.029	0.01	0.007
			Mean	0.01	0.01	0.030	0.042	0.152	0.032	0.01	0.007
		0.37	28	0.01	0.01	0.018	0.042	0.142	0.032	0.01	0.006
		0.35		0.01	0.01	0.014	0.040	0.143	0.033	0.01	0.007
			Mean	0.01	0.01	0.016	0.041	0.142	0.033	0.01	0.006
		0.36	36	0.01	0.01	0.020	0.039	0.148	0.036	0.01	0.006
		0.37		0.01	0.01	0.023	0.036	0.137	0.029	0.01	0.006
			Mean	0.01	0.01	0.021	0.038	0.143	0.033	0.01	0.006
Chula, GA, United States, 2014 Athena	0.56	0.39	1	0.01	0.011	0.047	0.046	0.175	0.050	0.01	0.006
	0.56	0.41		0.01	0.01	0.039	0.036	0.121	0.038	0.01	0.006
	0.56		Mean	0.01	0.01	0.043	0.041	0.148	0.044	0.01	0.006
	0.56	0.36	7	0.01	0.011	0.044	0.041	0.191	0.046	0.01	0.006
		0.34		0.01	0.01	0.044	0.037	0.144	0.034	0.01	0.007
			Mean	0.01	0.01	0.044	0.039	0.167	0.040	0.01	0.007
		0.35	14	0.01	0.01	0.050	0.042	0.175	0.036	0.01	0.007
		0.37		0.01	0.011	0.057	0.055	0.193	0.051	0.011	0.006
			Mean	0.01	0.011	0.054	0.048	0.184	0.044	0.01	0.006
		0.32	22	0.01	0.01	0.039	0.054	0.171	0.045	0.01	0.007
		0.32		0.01	0.01	0.035	0.048	0.178	0.042	0.01	0.007
			Mean	0.01	0.01	0.037	0.051	0.174	0.043	0.01	0.007
		0.40	28	0.01	0.01	0.035	0.054	0.210	0.056	0.01	0.010
		0.39		0.01	0.01	0.037	0.048	0.163	0.038	0.01	0.006
			Mean	0.01	0.01	0.036	0.051	0.187	0.047	0.01	0.008
		0.39	36	0.01	0.01	0.020	0.043	0.175	0.040	0.01	0.006
			Mean	0.01	0.01	0.020	0.043	0.175	0.040	0.01	0.006
Lime Springs, IA, United States, 2014 Minnesota Midget	1.12	0.30	1	0.016	0.01	0.014	0.012	0.010	0.01	0.01	0.01
	0.56	0.29		0.017	0.01	0.013	0.013	0.010	0.01	0.01	0.01
	0.56		Mean	0.017	0.01	0.014	0.012	0.010	0.01	0.01	0.01
		0.38	6	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.41		0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.39	13	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
		0.43		0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.46	20	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.39		0.01	0.01	0.012	0.01	0.014	0.01	0.01	0.01
			Mean	0.01	0.01	0.011	0.01	0.012	0.01	0.01	0.01
Lime Springs, IA, United States, 2014 Minnesota Midget	0.56	0.35	1	0.033	0.01	0.022	0.023	0.01	0.01	0.01	0.01
	0.56	0.34		0.049	0.01	0.030	0.032	0.01	0.01	0.01	0.01
	0.56		Mean	0.041	0.01	0.026	0.027	0.01	0.01	0.01	0.01
	0.56	0.41	6	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.39		0.011	0.01	0.011	0.01	0.01	0.01	0.01	0.01
			Mean	0.011	0.01	0.011	0.01	0.01	0.01	0.01	0.01

Location, year, variety	Rate (kg ai/ha)	Fraction pulp	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
		0.36	13	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.36		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.38	20	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.40		0.018	0.01	0.018	0.013	0.01	0.01	0.01	0.01
			Mean	0.014	0.01	0.014	0.012	0.01	0.01	0.01	0.01
Lime Springs, IA, United States, 2014 Minnesota Midget	1.12	0.40	1	0.039	0.01	0.023	0.024	0.01	0.01	0.01	0.01
	1.12	0.37		0.028	0.01	0.018	0.019	0.01	0.01	0.01	0.01
	1.12		Mean	0.034	0.01	0.020	0.022	0.01	0.01	0.01	0.01
	1.12	0.42	6	0.019	0.01	0.017	0.015	0.010	0.01	0.01	0.01
		0.40		0.011	0.01	0.011	0.01	0.01	0.01	0.01	0.01
			Mean	0.015	0.01	0.014	0.012	0.01	0.01	0.01	0.01
		0.41	13	0.033	0.01	0.030	0.025	0.012	0.01	0.01	0.01
		0.37		0.023	0.01	0.023	0.018	0.010	0.01	0.01	0.01
			Mean	0.028	0.01	0.026	0.022	0.011	0.01	0.01	0.01
		0.33	20	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.36		0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
Carlyle, IL, United States, 2014 Athena Trial 21 02 pulp	1.12	0.36	1	0.013	0.01	0.01	0.012	0.025	0.01	0.01	0.01
	0.56	0.39		0.012	0.01	0.01	0.011	0.025	0.01	0.01	0.01
	0.56		Mean	0.012	0.01	0.01	0.012	0.025	0.01	0.01	0.01
		0.45	6	0.01	0.01	0.01	0.01	0.016	0.01	0.01	0.01
		0.41		0.01	0.01	0.01	0.010	0.020	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.018	0.01	0.01	0.01
		0.54	14	0.01	0.01	0.01	0.010	0.022	0.01	0.01	0.01
		0.53		0.01	0.01	0.01	0.010	0.021	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.010	0.022	0.01	0.01	0.01
		0.54	21	0.01	0.01	0.01	0.010	0.023	0.01	0.01	0.01
		0.52		0.01	0.01	0.01	0.010	0.029	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.010	0.026	0.01	0.01	0.01
		0.51	28	0.01	0.01	0.01	0.010	0.027	0.01	0.01	0.01
		0.49		0.01	0.01	0.01	0.010	0.026	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.010	0.026	0.01	0.01	0.01
Carlyle, IL, United States, 2014 Athena	0.56	0.38	1	0.01	0.01	0.01	0.01	0.017	0.01	0.01	0.01
	0.56	0.42		0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
	0.56		Mean	0.01	0.01	0.01	0.01	0.015	0.01	0.01	0.01
	0.56	0.41	6	0.01	0.01	0.01	0.01	0.017	0.01	0.01	0.01
		0.43		0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.015	0.01	0.01	0.01
		0.53	14	0.01	0.01	0.01	0.01	0.015	0.01	0.01	0.01
		0.54		0.01	0.01	0.01	0.01	0.016	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.015	0.01	0.01	0.01
		0.57	21	0.01	0.01	0.01	0.01	0.020	0.01	0.01	0.01
		0.53		0.01	0.01	0.01	0.01	0.017	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.018	0.01	0.01	0.01
		0.51	28	0.01	0.01	0.01	0.01	0.020	0.01	0.01	0.01
		0.52		0.01	0.01	0.01	0.01	0.021	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.021	0.01	0.01	0.01
Uvalde, TX, United States, 2014 Primo	1.13	0.60	1	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
	0.55	0.65		0.01	0.01	0.011	0.01	0.02	0.011	0.01	0.01
	0.55		Mean	0.01	0.01	0.01	0.01	0.015	0.011	0.01	0.01
		0.63	7	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.58		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.60	14	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.57		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Uvalde, TX, United States, 2014 Primo	0.55	0.60	1	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
	0.55	0.58		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
	0.55		Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01

Location, year, variety	Rate (kg ai/ha)	Fraction pulp	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
	0.55	0.54	7	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.62		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.57	14	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.55		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Yuma, AZ, United States, 2014 Fiji	1.12	0.48	1	0.01	0.01	0.01	0.01	0.014	0.011	0.01	0.01
	0.56	0.46		0.01	0.01	0.01	0.01	0.042	0.036	0.01	0.01
	0.56		Mean	0.01	0.01	0.01	0.01	0.028	0.023	0.01	0.01
		0.55	7	0.01	0.01	0.01	0.01	0.024	0.019	0.01	0.01
		0.58		0.01	0.01	0.012	0.01	0.037	0.026	0.01	0.01
			Mean	0.01	0.01	0.011	0.01	0.030	0.022	0.01	0.01
		0.66	15	0.01	0.01	0.013	0.01	0.028	0.026	0.01	0.01
		0.67		0.01	0.01	0.012	0.01	0.035	0.032	0.01	0.01
			Mean	0.01	0.01	0.012	0.01	0.031	0.029	0.01	0.01
		0.63	23	0.01	0.01	0.01	0.01	0.020	0.023	0.01	0.01
		0.60		0.01	0.01	0.013	0.01	0.043	0.055	0.01	0.01
			Mean	0.01	0.01	0.012	0.01	0.032	0.039	0.01	0.01
Yuma, AZ, United States, 2014 Fiji	0.56	0.51	1	0.01	0.01	0.011	0.01	0.042	0.024	0.01	0.01
	0.56	0.48		0.01	0.01	0.016	0.01	0.039	0.034	0.01	0.01
	0.56		Mean	0.01	0.01	0.014	0.01	0.041	0.029	0.01	0.01
	0.56	0.60	7	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
		0.57		0.01	0.01	0.017	0.01	0.039	0.029	0.01	0.01
			Mean	0.01	0.01	0.014	0.01	0.024	0.020	0.01	0.01
		0.69	15	0.01	0.01	0.025	0.012	0.083	0.064	0.01	0.01
		0.76		0.01	0.01	0.014	0.01	0.042	0.027	0.01	0.01
			Mean	0.01	0.01	0.020	0.011	0.062	0.046	0.01	0.01
		0.59	23	0.01	0.01	0.014	0.01	0.023	0.017	0.01	0.01
		0.62		0.01	0.01	0.020	0.01	0.088	0.063	0.01	0.01
			Mean	0.01	0.01	0.017	0.01	0.056	0.040	0.01	0.01
Porterville, CA, United States, 2014 Hales Best Jumbo	1.12	0.77	1	0.01	0.01	0.013	0.011	0.037	0.016	0.01	0.01
	0.56	0.78		0.01	0.01	0.012	0.012	0.035	0.011	0.01	0.01
	0.56		Mean	0.01	0.01	0.012	0.011	0.036	0.014	0.01	0.01
		0.85	6	0.01	0.01	0.015	0.011	0.050	0.023	0.01	0.01
		0.83		0.01	0.01	0.017	0.012	0.042	0.016	0.01	0.01
			Mean	0.01	0.01	0.016	0.012	0.046	0.019	0.01	0.01
		0.82	14	0.01	0.01	0.019	0.011	0.051	0.021	0.01	0.01
		0.83		0.01	0.01	0.019	0.012	0.054	0.024	0.01	0.01
			Mean	0.01	0.01	0.019	0.011	0.052	0.022	0.01	0.01
		0.83	21	0.01	0.01	0.018	0.011	0.072	0.029	0.01	0.01
		0.83		0.01	0.01	0.020	0.011	0.056	0.025	0.01	0.01
			Mean	0.01	0.01	0.019	0.011	0.064	0.027	0.01	0.01
		0.84	28	0.01	0.01	0.029	0.012	0.086	0.034	0.01	0.01
		0.82		0.01	0.01	0.022	0.013	0.065	0.028	0.01	0.01
			Mean	0.01	0.01	0.025	0.012	0.076	0.031	0.01	0.01
		0.77	35	0.01	0.01	0.023	0.012	0.065	0.034	0.01	0.01
		0.83		0.01	0.01	0.023	0.013	0.072	0.041	0.01	0.01
			Mean	0.01	0.01	0.023	0.012	0.068	0.038	0.01	0.01
Porterville, CA, United States, 2014 Hales Best Jumbo	0.56	0.80	1	0.01	0.01	0.013	0.011	0.053	0.018	0.01	0.01
	0.56	0.80		0.01	0.01	0.013	0.011	0.056	0.018	0.01	0.01
	0.56		Mean	0.01	0.01	0.013	0.011	0.055	0.018	0.01	0.01
	0.56	0.86	6	0.01	0.01	0.017	0.011	0.056	0.015	0.01	0.01
		0.87		0.01	0.01	0.016	0.011	0.068	0.022	0.01	0.01
			Mean	0.01	0.01	0.017	0.011	0.062	0.018	0.01	0.01
		0.82	14	0.01	0.01	0.019	0.011	0.068	0.024	0.01	0.01
		0.83		0.01	0.01	0.020	0.011	0.063	0.027	0.01	0.01
			Mean	0.01	0.01	0.020	0.011	0.065	0.025	0.01	0.01
		0.89	21	0.01	0.01	0.020	0.010	0.055	0.022	0.01	0.009
		0.85		0.01	0.01	0.015	0.011	0.071	0.029	0.01	0.01
			Mean	0.01	0.01	0.018	0.010	0.063	0.025	0.01	0.009

Location, year, variety	Rate (kg ai/ha)	Fraction pulp	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
		0.82	28	0.012	0.01	0.033	0.013	0.092	0.034	0.01	0.01
		0.82		0.01	0.01	0.034	0.011	0.071	0.033	0.01	0.01
			Mean	0.011	0.01	0.034	0.012	0.081	0.033	0.01	0.01
		0.87	35	0.01	0.01	0.027	0.011	0.092	0.036	0.01	0.01
		0.81		0.01	0.01	0.028	0.012	0.079	0.040	0.01	0.01
			Mean	0.01	0.01	0.027	0.012	0.085	0.038	0.01	0.01
Yuba City, CA, United States, 2014 Hales Best Jumbo	1.12	0.45	1	0.01	0.01	0.01	0.01	0.034	0.017	0.01	0.01
	0.56	0.39		0.01	0.01	0.01	0.01	0.030	0.015	0.01	0.01
	0.56		Mean	0.01	0.01	0.01	0.01	0.032	0.016	0.01	0.01
		0.43	7	0.01	0.01	0.01	0.01	0.033	0.019	0.01	0.01
		0.45		0.01	0.01	0.01	0.01	0.034	0.018	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.034	0.018	0.01	0.01
		0.47	14	0.01	0.01	0.01	0.01	0.030	0.016	0.01	0.01
		0.43		0.01	0.01	0.01	0.01	0.036	0.016	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.033	0.016	0.01	0.01
		0.43	21	0.01	0.01	0.01	0.01	0.041	0.018	0.01	0.01
		0.45		0.01	0.01	0.01	0.01	0.031	0.019	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.036	0.018	0.01	0.01
		0.46	28	0.01	0.01	0.01	0.01	0.043	0.020	0.01	0.01
		0.43		0.01	0.01	0.01	0.01	0.052	0.020	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.048	0.020	0.01	0.01
		0.36	35	0.01	0.01	0.01	0.01	0.033	0.013	0.01	0.01
		0.39		0.01	0.01	0.01	0.01	0.026	0.014	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.030	0.013	0.01	0.01
Yuba City, CA, United States, 2014 Hales Best Jumbo	0.56	0.43	1	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
	0.56	0.29		0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
	0.56		Mean	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
	0.56	0.49	7	0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
		0.49		0.01	0.01	0.01	0.01	0.012	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
		0.50	14	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
		0.46		0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.011	0.01	0.01	0.01
		0.42	21	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
		0.44		0.01	0.01	0.01	0.01	0.010	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.012	0.01	0.01	0.01
		0.35	28	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
		0.37		0.01	0.01	0.01	0.01	0.013	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.014	0.01	0.01	0.01
		0.38	35	0.01	0.01	0.01	0.01	0.018	0.01	0.01	0.01
		0.36		0.01	0.01	0.01	0.01	0.015	0.01	0.01	0.01
			Mean	0.01	0.01	0.01	0.01	0.017	0.01	0.01	0.01
Sanger, CA, United States, 2014 Summer Dew	1.13	0.72	1	0.01	0.011	0.058	0.040	0.092	0.022	0.010	0.010
	0.56	0.75		0.01	0.011	0.032	0.049	0.135	0.037	0.010	0.010
	0.56		Mean	0.01	0.011	0.045	0.044	0.113	0.030	0.010	0.010
		0.71	6	0.01	0.011	0.025	0.041	0.103	0.032	0.010	0.010
		0.73		0.01	0.015	0.067	0.083	0.238	0.056	0.015	0.011
			Mean	0.01	0.013	0.046	0.062	0.170	0.044	0.013	0.011
		0.74	14	0.01	0.012	0.024	0.042	0.084	0.027	0.010	0.010
		0.75		0.01	0.012	0.030	0.042	0.129	0.036	0.010	0.010
			Mean	0.01	0.012	0.027	0.042	0.107	0.031	0.010	0.010
		0.76	21	0.01	0.01	0.020	0.035	0.081	0.024	0.010	0.010
		0.74		0.01	0.013	0.031	0.046	0.108	0.044	0.012	0.010
			Mean	0.01	0.012	0.026	0.041	0.094	0.034	0.011	0.010
		0.65	28	0.01	0.013	0.036	0.039	0.079	0.026	0.010	0.010
		0.60		0.01	0.011	0.018	0.038	0.090	0.026	0.010	0.010
			Mean	0.01	0.012	0.027	0.039	0.085	0.026	0.010	0.010
		0.62	35	0.01	0.013	0.030	0.045	0.200	0.073	0.014	0.013
		0.66		0.01	0.016	0.040	0.095	0.371	0.098	0.017	0.012
			Mean	0.01	0.014	0.035	0.070	0.285	0.086	0.015	0.012

Location, year, variety	Rate (kg ai/ha)	Fraction pulp	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Sanger, CA, United States, 2014 Summer Dew	0.56	0.71	1	0.01	0.014	0.075	0.058	0.108	0.031	0.010	0.010
	0.56	0.69		0.01	0.011	0.034	0.060	0.196	0.044	0.010	0.010
	0.56		Mean	0.01	0.013	0.054	0.059	0.152	0.038	0.010	0.010
	0.56	0.70	6	0.01	0.012	0.049	0.047	0.093	0.021	0.010	0.010
		0.71		0.01	0.013	0.047	0.044	0.094	0.022	0.010	0.010
			Mean	0.01	0.012	0.048	0.046	0.094	0.021	0.010	0.010
		0.74	14	0.01	0.013	0.060	0.042	0.074	0.022	0.010	0.010
		0.75		0.01	0.016	0.069	0.057	0.104	0.032	0.010	0.010
			Mean	0.01	0.014	0.065	0.050	0.089	0.027	0.010	0.010
		0.71	21	0.01	0.017	0.065	0.064	0.110	0.034	0.011	0.010
		0.71		0.01	0.021	0.084	0.072	0.117	0.046	0.011	0.010
			Mean	0.01	0.019	0.075	0.068	0.114	0.040	0.011	0.010
		0.67	28	0.01	0.014	0.055	0.043	0.082	0.032	0.010	0.010
		0.67		0.01	0.018	0.067	0.049	0.099	0.034	0.011	0.010
			Mean	0.01	0.016	0.061	0.046	0.091	0.033	0.011	0.010
		0.69	35	0.01	0.022	0.072	0.062	0.130	0.043	0.014	0.011
		0.66		0.01	0.023	0.074	0.075	0.126	0.047	0.014	0.010
			Mean	0.01	0.023	0.073	0.068	0.128	0.045	0.014	0.010

Table 138 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in squash from supervised trials conducted in Canada and the United States

Location Year variety	N (interval) days	Rate	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
Germansville, PA, United States, 2014 Superpik	3 (36 15) drench	1.12	0	ND	ND	ND	0.012	0.028	ND	<0.01	ND	
		0.56		ND	ND	ND	0.013	0.030	ND	<0.01	ND	
		0.56	Mean	<0.01	<0.01	<0.01	0.0125	0.029	<0.01	<0.01	<0.01	0.1029
			7	ND	ND	<0.01	0.013	0.030	ND	<0.01	ND	
				ND	ND	<0.01	0.013	0.030	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.013	0.030	<0.01	<0.01	<0.01	0.1044
			14	ND	ND	ND	<0.01	0.018	ND	<0.01	ND	
				ND	ND	<0.01	<0.01	0.024	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.021	<0.01	<0.01	<0.01	0.0907
			21	ND	ND	ND	<0.01	0.026	ND	<0.01	ND	
				ND	ND	<0.01	<0.01	0.028	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.027	<0.01	<0.01	<0.01	0.0998
			28	ND	ND	ND	0.013	0.039	ND	<0.01	ND	
				ND	ND	ND	0.012	0.039	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.012	0.039	<0.01	<0.01	<0.01	0.1181
			35	ND	ND	ND	<0.01	0.033	ND	0.011	ND	
				ND	ND	ND	<0.01	0.028	ND	0.011	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0305	<0.01	0.011	<0.01	0.1052
Germansville, PA, United States, 2014 Superpik	4 (12 14 15) drench	0.56	0	ND	ND	<0.01	0.016	0.032	ND	<0.01	ND	
		0.56		<0.01	ND	<0.01	0.017	0.034	ND	<0.01	ND	
		0.56	Mean	<0.01	<0.01	<0.01	0.0165	0.033	<0.01	<0.01	<0.01	0.1090
		0.56	7	ND	ND	<0.01	0.019	0.039	ND	<0.01	ND	
				ND	ND	<0.01	0.017	0.034	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.018	0.0365	<0.01	<0.01	<0.01	0.1143
			14	ND	ND	<0.01	0.012	0.031	ND	<0.01	ND	
				ND	ND	ND	0.013	0.030	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.0125	0.0305	<0.01	<0.01	<0.01	0.1052
			21	ND	ND	ND	0.011	0.032	ND	<0.01	ND	
				ND	ND	ND	<0.01	0.030	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.0105	0.031	<0.01	<0.01	<0.01	0.1059
			28	ND	ND	<0.01	0.015	0.042	ND	<0.01	ND	
				ND	ND	<0.01	0.016	0.053	ND	0.012	ND	
			Mean	<0.01	<0.01	<0.01	0.0155	0.0475	<0.01	<0.011	<0.01	0.1310
			35	ND	ND	ND	0.012	0.038	ND	0.012	ND	

Location Year variety	N (interval) days	Rate	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
				ND	ND	ND	0.013	0.039	ND	0.015	ND	
			Mean	<0.01	<0.01	<0.01	0.0125	0.0385	<0.01	0.0135	<0.01	0.1173
Chula, GA, United States, 2014 Yellow Crookneck	3 (27 12) drip inj	1.12 0.56 0.56	1	0.10	ND	0.076	0.10	0.047	ND	<0.01	ND	
				0.078	ND	0.053	0.096	0.046	ND	<0.01	ND	
			Mean	0.089	<0.01	0.0645	0.098	0.0465	<0.01	<0.01	<0.01	0.2445
			7	0.043	ND	0.043	0.097	0.10	ND	<0.01	ND	
				0.038	ND	0.036	0.080	0.088	ND	<0.01	ND	
			Mean	0.0405	<0.01	0.0395	0.0885	0.094	<0.01	<0.01	<0.01	0.2639
			15	0.020	ND	0.021	0.060	0.087	<0.01	0.010	ND	
				0.023	<0.01	0.024	0.073	0.086	<0.01	<0.01	ND	
			Mean	0.0215	<0.01	0.0225	0.0665	0.0865	<0.01	<0.01 [A]	<0.01	0.2167
			22	<0.01	<0.01	0.017	0.041	0.047	<0.01	0.011	ND	
				<0.01	ND	<0.01	0.034	0.063	<0.01	0.011	ND	
			Mean	<0.01	<0.01	0.0135	0.0375	0.055	<0.01	0.011	<0.01	0.1498
			29	<0.01	ND	<0.01	0.027	0.067	ND	0.017	ND	
				<0.01	<0.01	<0.01	0.024	0.057	ND	0.018	<0.01	
			Mean	<0.01	<0.01	<0.01	0.0255	0.062	<0.01	0.0175	<0.01	0.1530
			36	<0.01	<0.01	<0.01	0.030	0.060	ND	0.021	<0.01	
				<0.01	ND	<0.01	0.031	0.078	ND	0.020	<0.01	
			Mean	<0.01	<0.01	<0.01	0.0305	0.069	<0.01	0.0205	<0.01	0.1637
Branchton, ON, Canada, 2014 Senator Trial 31 12/6 18/7 31/7	3 (36 13) dripline	1.12 0.56 0.56	1	ND	ND	<0.01	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Senator 20/6 4/7 18/7 31/7	4 (14 14 14) dripline	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Golden Dawn Trial 32 24/6 7/8 19/8	3 (44 12) dripline	1.12 0.56 0.56	1	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	<0.01	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Golden Dawn	4 (14 21 12) 3/7 17/7 7/8 19/8 dripline	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740

Location Year variety	N (interval) days	Rate	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			15	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Spineless Beauty Trial 33	3 (35 13) 19/6 24/7 6/8 dripline	1.12 0.56 0.56	1	ND	ND	0.013	0.018	<0.01	<0.01	ND	ND	
				ND	ND	0.013	0.022	<0.01	<0.01	ND	ND	
			Mean	<0.01	<0.01	0.013	0.020	<0.01	<0.01	<0.01	<0.01	0.0803
			7	ND	ND	0.015	0.019	<0.01	<0.01	<0.01	ND	
				<0.01	<0.01	0.021	0.034	<0.01	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	0.018	0.0265	<0.01	<0.01	<0.01	<0.01	0.0909
			16	ND	<0.01	0.012	0.024	<0.01	<0.01	<0.01	<0.01	
				ND	<0.01	0.010	0.026	<0.01	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	0.011	0.025	<0.01	<0.01	<0.01	<0.01	0.0761
			23	ND	ND	<0.01	0.017	<0.01	ND	<0.01	ND	
				ND	ND	0.01 [A]	0.019	0.011	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.018	<0.0105	<0.01	<0.01	<0.01	0.0748
Branchton, ON, Canada, 2014 Spineless Beauty	4 (14 14 13) 26/6 10/7 24/7 6/8 dripline	0.56 0.56 0.56 0.56	1	<0.01	ND	0.014	0.016	<0.01	ND	ND	ND	
				<0.01	ND	0.013	0.013	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	0.0135	0.0145	<0.01	<0.01	<0.01	<0.01	0.0814
			7	<0.01	ND	0.019	0.024	<0.01	<0.01	<0.01	ND	
				ND	ND	0.014	0.017	<0.01	ND	<0.01	ND	
			Mean	<0.01	<0.01	0.0165	0.0205	<0.01	<0.01	<0.01	<0.01	0.0877
			16	ND	<0.01	<0.01	0.021	<0.01	<0.01	<0.01	<0.01	
				ND	<0.01	<0.01	0.019	<0.01	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.020	<0.01	<0.01	<0.01	<0.01	0.0740
			23	ND	ND	<0.01	0.013	<0.01	ND	<0.01	ND	
				ND	<0.01	<0.01	0.017	<0.01	<0.01	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.015	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Zucchini vert foncé Trial 34	3 (38 14) 8/6 16/7 30/7 drench	1.12 0.56 0.56	1	<0.01	ND	<0.01	0.012	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	0.010	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	<0.01	0.012	<0.01	ND	ND	ND	
				ND	ND	<0.01	0.017	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0145	<0.01	<0.01	<0.01	<0.01	0.0740
			13	ND	ND	<0.01	0.012	<0.01	ND	ND	ND	
				ND	ND	<0.01	0.016	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.014	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	<0.01	0.012	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	<0.01	0.010	<0.01	ND	ND	ND	
				ND	ND	<0.01	0.011	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0105	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	<0.01	0.013	0.010	ND	<0.01	ND	
				ND	ND	<0.01	0.013	<0.01	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	0.013	<0.01 [A]	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Zucchini vert foncé 19/6 2/7 16/7	4 (13 14 14) drench	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	0.011	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	<0.01	0.010	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			13	ND	ND	<0.01	0.011	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	

Location Year variety	N (interval) days	Rate	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
30/7			Mean	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	0.012	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	<0.01	0.011	<0.01	ND	ND	ND	
				ND	ND	<0.01	0.012	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Golden Glory Trial 35	3 (38 14) 8/6 16/7 30/7 drench	1.12 0.56 0.56	1	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			13	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Golden Glory	4 (13 14 14) 19/6 2/7 16/7 30/7 drench	0.56 0.56 0.56 0.56	1	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	0.012	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			13	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	0.01[A]	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	0.012	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.011	<0.01	<0.01	<0.01	0.0755
			35	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	0.01[A]	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Chula, GA, United States, 2014 Yellow Crookneck	4 (10 16 12) drip inj	0.56 0.56 0.56 0.56	1	0.065	ND	0.052	0.081	0.042	ND	<0.01	ND	
				0.072	ND	0.058	0.087	0.049	ND	<0.01	ND	
			Mean	0.0685	<0.01	0.055	0.084	0.0455	<0.01	<0.01	<0.01	0.2229
			7	0.036	ND	0.038	0.084	0.099	ND	<0.01	ND	
				0.038	<0.01	0.043	0.082	0.098	<0.01	<0.01	ND	
			Mean	0.037	ND	0.0405	0.083	0.0985	<0.01	<0.01	<0.01	0.2729
			15	0.014	ND	0.017	0.055	0.077	ND	0.010	ND	
				0.018	<0.01	0.021	0.070	0.088	<0.01	0.011	<0.01	
			Mean	0.016	<0.01	0.019	0.0625	0.0825	<0.01	0.0105	<0.01	0.2032
			22	<0.01	ND	<0.01	0.027	0.038	ND	0.010	ND	
				<0.01	ND	<0.01	0.034	0.044	ND	0.010	ND	
			Mean	<0.01	<0.01	<0.01	0.0305	0.041	<0.01	0.010	<0.01	0.1211
			29	<0.01	<0.01	0.01[A]	0.026	0.070	ND	0.020	ND	

Location Year variety	N (interval) days	Rate	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
				<0.01	<0.01	0.011	0.037	0.045	ND	0.018	<0.01	
			Mean	<0.01	<0.01	0.0105	0.0315	0.0575	<0.01	0.019	<0.01	0.1473
			36	<0.01	ND	<0.01	0.034	0.045	ND	0.021	<0.01	0.1272
Hobe Sound, FL, United States, 2014 Enterprise	3 (34 14) drench 2×drip	1.12 0.56 0.56	1	0.012	ND	0.010	0.012	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	0.011	<0.01	<0.01	<0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			7	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	0.01 [A]	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Hobe Sound, FL, United States, 2014 Enterprise	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	<0.01	ND	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Richland, IA, United States, 2014 Yellow Crookneck OG	3 (47 14) dripline	1.12 0.56 0.56	1	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			34	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Richland, IA, United States, 2014 Yellow Crookneck OG	4 (15 13 14) dripline	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	

Location Year variety	N (interval) days	Rate	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			34	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
York, NE, United States, 2014 Black Magic	3 (51 15) drench	1.12 0.56 0.56	1	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				0.046	ND	0.028	0.027	ND	ND	ND	ND	
			Mean	0.028	<0.01	0.019	0.0185	<0.01	<0.01	<0.01	<0.01	0.0930
			7	ND	ND	<0.01	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	<0.01	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
York, NE, United States, 2014 Black Magic	4 (14 14 15) drench	0.56 0.56 0.56 0.56	1	0.018	ND	0.012	0.011	ND	ND	ND	ND	
				0.073	ND	0.040	0.042	ND	ND	ND	ND	
			Mean	0.0455	<0.01	0.026	0.0265	<0.01	<0.01	<0.01	<0.01	0.1078
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	<0.01	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Porterville, CA, United States, 2014 Caserta	3 (36 13) drip	1.12 0.56 0.56	1	<0.01	ND	<0.01	0.01[A]	ND	ND	ND	ND	
				<0.01	ND	0.01[A]	0.015	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0125	<0.01	<0.01	<0.01	<0.01	0.0740
			6	<0.01	ND	<0.01	0.01[A]	ND	ND	<0.01	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	<0.01	ND	<0.01	0.01[A]	ND	ND	<0.01	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	0.01[A]	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	<0.01	0.01[A]	<0.01	ND	ND	ND	

Location Year variety	N (interval) days	Rate	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Porterville, CA, United States, 2014 Caserta	4 (14 14 13)	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Madera, CA, United States, 2014 Black Beauty [D] Trial 38	3 46 14) soil directed	1.13 0.56 0.56	1	ND	ND	<0.01	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Madera, CA, United States, 2014 Black Beauty [D] Trial 38	4 (14 14 14) soil directed	0.57 0.56 0.56 0.56	1	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740

Notes:

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] SUM = 2.26×IN-A5760 + 2.11×IN-F4106 + 1.52×IN-QZY47 + 1.51×IN-TMQ01.

[C] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

[D] The total per hectare rate was concentrated in the application band for all sites, with the exception of Trial 38.

Fruiting Vegetables

Shepard (2020 DuPont-40062) conducted residue trials on fruiting vegetables at a number of sites (22 tomato, 21 pepper) in Canada and the United States in 2014/2015.

At each location, separate plots were treated with fluazaindolizine SC formulation was applied as soil applications (drip/drench or directed spray) at planting at 1.12 kg ai/ha and then 15±2 days and 1 day before expected maturity at 0.56 kg ai/ha and at the other plot as four applications at 14±2 day intervals starting 43±3 days before expected maturity and at 0.56 kg ai/ha. No adjuvants were used in any of the trials.

The maximum interval of frozen storage before analysis was 683 days for tomatoes and peppers prior to extraction. Fruit was analysed for residues of fluazaindolizine and its metabolites IN-A5760, IN-F4016, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12 and IN-UNS90 (IN-TQD54) using the analytical method DuPont-33861, rev. 3, with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg for each analyte. Acceptable concurrent recovery data were obtained for all matrices.

Table 139 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in tomato from supervised trials conducted in Canada and the United States

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
Branchton, ON, Canada, 2014 TSH25 (Large) Trial 07	3 (74 14) 12/6 25/8 8/9 Drip	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			30	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			37	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 TSH25 (Large)	4 (19 12 14) 25/7 13/8 25/8 8/9 drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			30	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			37	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 TSH28 (Large) Trial 08	3 (69 14) 18/6 26/8 9/9 drip	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			78	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 TSH28 (Large)	4 (15 13 14) 29/7 13/8 26/8 9/9 drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			8	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Crista (Large) Trial 09	3 (58 15) 14/6 11/8 26/8 drip	1.12 0.56 0.56	1	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			8	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	<0.018	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	0.011	ND	<0.01	ND	<0.01	ND	<0.01	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
			Mean	<0.01	<0.0145	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0842
			36	ND	<0.01	ND	0.012	ND	<0.01	ND	0.012	
				ND	0.011	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.0105	<0.01	<0.011	<0.01	<0.01	<0.01	<0.011	0.0751
Branchton, ON, Canada, 2014 Crista (Large)	4 (14 18 15) 10/7 24/7 11/8 26/8 drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			8	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	ND	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Smarty (Grape) Trial 10	3 (56 14) 12/6 7/8 21/8 drip	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	<0.01	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	<0.01	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Smarty (Grape)	4 (14 22 14) 2/78 16/7 7/8 21/8 drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014	3 (55 14) 14/6	1.12 0.56	1	ND	ND	ND	<0.01	ND	0.014	ND	<0.01	
				ND	ND	ND	<0.01	ND	0.013	ND	<0.01	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
Cupid (Grape) Trial 11	8/8 22/8 drip	0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0135	<0.01	<0.01	0.0793
			7	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	ND	0.01[A]	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	0.01[A]	ND	0.01[A]	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Cupid (Grape)	4 (14 23 14) 2/7 16/7 8/8 22/8drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	ND	0.011	ND	<0.01	
				ND	ND	ND	<0.01	ND	0.01[A]	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0105	<0.01	<0.01	0.0748
			28	ND	ND	ND	<0.01	ND	0.01[A]	ND	<0.01	
				ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	0.013	ND	0.012	ND	<0.01	
				ND	ND	ND	0.016	ND	0.012	ND	0.01[A]	
			Mean	<0.01	<0.01	<0.01	0.0145	<0.01	0.012	<0.01	<0.01	0.0770
St. Marc-sur-Richelieu, QC, Canada, 2014 Mountain Spring (Large) Trial 12	3 (59 15) 7/6/1 5/8 20/8 drench	1.12 0.56 0.56	1	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				NDB	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			13	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Mountain Spring	4 (13 14 15) 9/7 22/7 5/8 20/8 drench	0.56 0.56 0.56 0.56	1	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			13	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	ND	ND	ND	ND	ND	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	<0.01	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Sweet 100 (Cherry) Trial 13	3 (59 15) 7/6 5/8 20/8 drench	1.12 0.56 0.56	1	<0.01	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				<0.01	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	<0.01	<0.01	<0.01	<0.01	ND	<0.01	ND	ND	
				<0.01	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			13	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	NDB	NDB	NDB	NDB	NDB	NDB	NDB	NDB	
				NDB	NDB	NDB	<0.01[B]	NDB	<0.01[B]	NDB	NDB	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	NDB	NDB	NDB	NDB	NDB	NDB	NDB	NDB	
				NDB	NDB	NDB	<0.01[B]	NDB	NDB	NDB	NDB	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	NDB	NDB	NDB	NDB	NDB	NDB	NDB	NDB	
				NDB	NDB	NDB	NDB	NDB	NDB	NDB	NDB	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Sweet 100 (Cherry)	4 (13 14 15) 9/7 22/7 5/8 20/8 drench	0.56 0.56 0.56 0.56	1	<0.01	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				<0.01	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	<0.01	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			13	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	NDB	NDB	NDB	<0.01[B]	NDB	<0.01[B]	NDB	NDB	
				NDB	NDB	NDB	<0.01[B]	NDB	<0.01[B]	NDB	NDB	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	NDB	<0.01[B]	NDB	<0.01[B]	NDB	<0.01[B]	NDB	NDB	
				NDB	<0.01[B]	NDB	<0.01[B]	NDB	<0.01[B]	NDB	NDB	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	NDB	NDB	NDB	NDB	NDB	<0.01[B]	NDB	NDB	
				NDB	<0.01[B]	NDB	<0.01[B]	NDB	<0.01[B]	NDB	NDB	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Germansville, PA, United States, 2014 Scarlet Red (Large)	3 (73 14) drench	1.12 0.56 0.56	1	ND	0.014	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	0.011	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	0.0125	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0797
			7	ND	0.011	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	0.012	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	0.0115	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0774
			14	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	0.010	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			20	ND	0.011	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			27	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			34	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Germansville, PA, United States, 2014 Scarlet Red (Large)	4 (14 14 14) drench	0.56 0.56 0.56 0.56	1	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.013	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.01	0.0763
			14	ND	0.011	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
			Mean	<0.01	<0.0105	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0751
			20	ND	0.011	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.0105	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0751
			27	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	0.010	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01[A]	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			34	ND	<0.01	ND	ND	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Athens, GA, United States, 2014 Tobago (Large)	3 (70 14) drip	1.12 0.56 0.56	1	ND	<0.01	ND	0.011	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	0.01[A]	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.0105	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
				ND	<0.01	ND	0.010	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Athens, GA, United States, 2014 Tobago (Large)	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	<0.01	ND	0.013	ND	ND	ND	ND	
				ND	<0.01	ND	0.010	ND	ND	ND	ND	
			Mean	<u><0.01</u>	<0.01	<0.01	0.0115	<u><0.01</u>	<u><0.01</u>	<u><0.01</u>	<u><0.01</u>	0.0740
			7	ND	<0.01	ND	0.012	ND	ND	ND	<0.01	
				ND	<0.01	ND	0.013	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.0125	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	0.013	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	0.013	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	0.011	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	<0.01	ND	0.015	ND	ND	ND	<0.01	
				ND	0.011	ND	0.017	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.0105	<0.01	0.016	<0.01	<0.01	<0.01	<0.01	<u>0.0751</u>
			36	ND	<0.01	ND	0.018	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	0.017	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<u>0.0175</u>	<0.01	<0.01	<0.01	<0.01	0.0740
Tifton, GA, United States, 2014 Red	3 (56 11) drip	1.12 0.56	1	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
Candy F1- (Grape)		0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			20	ND	ND	ND	ND	ND	ND	ND	<0.01	
				ND	ND	ND	ND	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			32	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Tifton, GA, United States, 2014 Red Candy F1- (Grape)	4 (18 14 11) drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			20	ND	ND	ND	ND	ND	ND	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	0.011	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	0.0748
			32	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Groveland, FL, United States, 2015 Early Girl (Large)	3 (50 14) drip	1.12 0.56 0.56	1	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Groveland, FL, United States, 2015 Early Girl (Large)	4 (11 19 14) drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	0.011	ND	<0.01	ND	ND	
				ND	<0.01	ND	0.011	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	0.011	ND	0.012	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.0105	<0.01	<0.011	<0.01	<0.01	<0.01	<0.01	0.0751
			36	ND	<0.01	ND	0.01[A]	ND	<0.01	ND	ND	
				ND	<0.01	ND	0.013	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0115	<0.01	<0.01	<0.01	<0.01	0.0740
Hobe Sound, FL, United States, 2014 BHN 762 (Cherry)	3 (53 14) drip	1.12 0.56 0.56	1	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	0.012	ND	ND	ND	ND	
				ND	<0.01	ND	0.016	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.014	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	<0.01	ND	0.013	ND	ND	ND	ND	
				ND	<0.01	ND	0.016	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0145	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	<0.01	ND	0.012	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.011	<0.01	<0.01	<0.01	<0.01	0.0740
Hobe Sound, FL, United States, 2014 BHN 762 (Cherry)	4 (13 14 14) drip	0.56 0.56 0.56 0.56	1	ND	<0.01	ND	0.018	ND	ND	ND	ND	
				ND	<0.01	ND	0.015	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0165	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	0.016	ND	ND	ND	ND	
				ND	<0.01	ND	0.014	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.015	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	0.021	ND	ND	ND	ND	
				ND	<0.01	ND	0.021	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.021	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	<0.01	ND	0.022	ND	ND	ND	ND	
				ND	<0.01	ND	0.021	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0215	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	<0.01	ND	0.024	ND	ND	ND	ND	
				ND	<0.01	ND	0.017	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0205	<0.01	<0.01	<0.01	<0.01	0.0740
Fitchburg, WI, United States, 2014 Mountain Fresh (Large)	3 (63 16) drench	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Fitchburg, WI, United States, 2014 Mountain Fresh (Large)	4 (14 14 16) drench	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
York, NE, United States, 2014 Beefmaster (Large) Trial 14	3 (57 13) 10/6 6/8 19/8 drench	1.12 0.56 0.56	1	0.023 [C]	NDC	0.013 [C]	0.014 [C]	NDC	NDC	NDC	NDC	
				0.11 [C]	NDC	0.051 [C]	0.062 [C]	NDC	NDC	NDC	NDC	
			Mean	0.0665	<0.01	0.032	0.038	<0.01	<0.01	<0.01	<0.01	0.1204
			7	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	<0.01	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
York, NE, United States, 2014 Beefmaster (Large)	4 (14 14 13) 9/7 23/7 6/8 19/8 drench	0.56 0.56 0.56 0.56	1	0.010	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
York, NE, United States, 2014	3 (48 13) 10/6	2.24 1.12	1	0.023	ND	0.015	0.014	ND	ND	ND	ND	
				0.022	ND	0.013	0.015	ND	ND	ND	ND	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
Beefmaster (Large) Trial 15	6/8 19/8 drench	1.12	Mean	0.0225	<0.01	0.014	0.0145	<0.01	<0.01	<0.01	<0.01	0.0824
			7	ND <0.01	ND <0.01	ND ND	ND <0.01	ND ND	ND ND	ND ND	ND <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND ND	ND <0.01	ND ND	ND <0.01	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	<0.01 ND	<0.01 <0.01	ND ND	<0.01 ND	ND ND	ND ND	ND ND	ND <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND ND	<0.01 ND	ND ND	<0.01 ND	ND ND	ND ND	ND ND	<0.01 ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	<0.01 ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
York, NE, United States, 2014 Beefmaster (Large)	4 (14 14 13) 9/7 23/7 6/8 19/8 drench	1.12 1.12 1.12 1.12	1	0.032 0.024	<0.01 ND	0.021 0.012	0.027 0.015	ND ND	ND ND	ND ND	ND ND	
			Mean	0.028	<0.01	0.0165	0.021	<0.01	<0.01	<0.01	<0.01	0.0877
			7	<0.01 ND	<0.01 <0.01	<0.01 ND	<0.01 <0.01	ND ND	ND ND	ND ND	<0.01 ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND <0.01	<0.01 <0.01	ND <0.01	<0.01 <0.01	ND ND	ND ND	ND ND	<0.01 <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND ND	<0.01 <0.01	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	<0.01 ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND ND	<0.01 ND	ND ND	ND ND	ND ND	ND ND	ND ND	<0.01 ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND ND	ND <0.01	ND ND	ND <0.01	ND ND	ND ND	ND ND	ND <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Lime Springs, IA, United States, 2014 Sweet 100 (Cherry)	3 (48 13) drench	1.12 0.56 0.56	1	<0.01 [B] <0.01 [B]	NDB NDB	NDB NDB	<0.01 [B] <0.01 [B]	NDB NDB	NDB NDB	NDB NDB	NDB NDB	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	<0.01 [B] NDE	NDB NDB	NDB NDB	<0.01 [B] <0.01 [B]	NDB NDB	NDB NDB	NDB NDB	NDB NDB	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Lime Springs, IA, United States, 2014 Sweet 100 (Cherry) 3 instead of 4 sprays	3 (14 16) drench	0.56 0.56 0.56	1	NDB NDB	NDB NDB	NDB NDB	<0.01 [B] <0.01 [B]	NDB NDB	NDB NDB	NDB NDB	NDB NDB	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	NDB NDB	NDB NDB	NDB NDB	<0.01 [B] <0.01 [B]	NDB NDB	NDB NDB	NDB NDB	NDB NDB	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	<0.01	ND	ND	ND	<0.01	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	<0.01	
				ND	ND	ND	ND	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Yuma, AZ, United States, 2015 Mountain Fresh (Large)	3 (74 13) drip	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Yuma, AZ, United States, 2015 Mountain Fresh (Large)	4 (15 14 13) drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	<0.01	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Yuba City, CA, United States, 2014 Sweet Million –(Cherry)	3 (48 14) drip	1.12 0.56 0.56	0	ND	ND	ND	<0.01	<0.01	0.013	ND	ND	
				ND	<0.01	ND	<0.01	<0.01	0.021	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.017	<0.01	<0.01	0.0846
			6	ND	<0.01	ND	<0.01	<0.01	0.014	ND	<0.01	
				ND	<0.01	ND	<0.01	<0.01	0.019	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0165	<0.01	<0.01	0.0838
			14	ND	<0.01	ND	<0.01	ND	0.010	ND	ND	
				ND	<0.01	ND	<0.01	ND	0.010	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.010	<0.01	<0.01	0.0740
			21	ND	<0.01	ND	<0.01	<0.01	0.018	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	0.010	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.014	<0.01	<0.01	0.0800
			28	ND	<0.01	ND	<0.01	<0.01	0.019	ND	<0.01	
				ND	<0.01	ND	0.010	<0.01	0.020	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0195	<0.01	<0.01	0.0883
			35	ND	0.01 [A]	ND	<0.01	ND	0.016	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	0.012	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.014	<0.01	<0.01	0.0800
Yuba City, CA, United States,	4 (14 13 14) drip	0.56 0.56	0	<0.01	<0.01	ND	<0.01	<0.01	0.016	ND	ND	
				ND	<0.01	ND	<0.01	<0.01	0.017	ND	ND	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
2014 Sweet Million –(Cherry)		0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0165	<0.01	<0.01	0.0838
		0.56	6	ND	<0.01	ND	<0.01	<0.01	0.012	ND	ND	
				ND	<0.01	ND	<0.01	0.015	0.031	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0125	0.0215	<0.01	<0.01	0.0952
			14	ND	<0.01	<0.01	<0.01	<0.01	0.029	ND	<0.01	
				ND	<0.01	<0.01	<0.01	<0.01	0.021	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.025	<0.01	<0.01	0.0967
			21	ND	<0.01	<0.01	<0.01	<0.01	0.016	ND	<0.01	
				ND	<0.01	<0.01	<0.01	<0.01	0.024	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.020	<0.01	<0.01	0.0891
			28	ND	<0.01	ND	<0.01	ND	0.01[A]	ND	ND	
				ND	<0.01	ND	<0.01	<0.01	0.021	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0155	<0.01	<0.01	0.0823
			35	ND	<0.01	ND	<0.01	<0.01	0.019	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	0.01[A]	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0145	<0.01	<0.01	0.0808
Sanger, CA, United States, 2014 Q-21 (Large) Trial 18	3 (83 14) soil directed 2×drip	1.11	1	0.011	0.14	0.030	0.17	0.020	0.064	ND	0.029	
		0.56		<0.01	0.15	0.029	0.19	0.023	0.063	<0.01	0.031	
		0.56	Mean	<0.0105	0.145	0.0295	0.18	0.0215	0.0635	<0.01	0.030	0.5185
			6	0.013	0.17	0.044	0.23	0.027	0.062	ND	0.046	
				0.012	0.18	0.042	0.23	0.020	0.055	<0.01	0.036	
			Mean	0.0125	0.175	0.043	0.23	0.0235	0.0585	<0.01	0.041	0.6103
			14	ND	0.19	0.036	0.26	0.019	0.089	<0.01	0.043	
				ND	0.26	0.055	0.36	0.037	0.11	<0.01	0.056	
			Mean	<0.01	0.225	0.0455	0.31	0.028	0.0995	<0.01	0.0495	0.7973
			21	ND	0.18	0.029	0.24	0.012	0.077	<0.01	0.040	
				ND	0.22	0.039	0.30	0.016	0.077	<0.01	0.054	
			Mean	<0.01	0.20	0.034	0.27	0.014	0.077	<0.01	0.047	0.6613
			28	ND	0.23	0.034	0.31	0.018	0.11	<0.01	0.051	
				ND	0.20	0.031	0.30	0.014	0.096	<0.01	0.044	
			Mean	<0.01	0.215	0.0325	0.305	0.016	0.103	<0.01	0.0475	0.7343
			35	ND	0.20	0.027	0.30	0.012	0.11	<0.01	0.045	
				ND	0.25	0.030	0.37	0.016	0.10	<0.01	0.053	
			Mean	<0.01	0.225	0.0285	0.335	0.014	0.105	<0.01	0.049	0.7485
Sanger, CA, United States, 2014 Q-21 (Large) Trial 18	4 (13 16 14) drip	0.56	1	0.014	0.24	0.059	0.61	0.050	0.13	<0.01	0.060	
		0.56		0.024	0.24	0.070	0.42	0.066	0.13	<0.01	0.062	
		0.56	Mean	0.019	0.24	0.0645	0.515	0.058	0.13	<0.01	0.061	0.9630
		0.56	6	0.028	0.21	0.069	0.45	0.027	0.077	<0.01	0.038	
				0.022	0.19	0.058	0.34	0.020	0.060	ND	0.036	
			Mean	0.025	0.20	0.0635	0.395	0.0235	0.0685	<0.01	0.037	0.7251
			14	ND	0.20	0.046	0.32	0.019	0.078	<0.01	0.037	
				<0.01	0.26	0.056	0.43	0.018	0.079	<0.01	0.050	
			Mean	<0.01	0.23	0.051	0.375	0.0185	0.0785	<0.01	0.0435	0.7741
			21	ND	0.22	0.033	0.36	0.015	0.081	<0.01	0.054	
				ND	0.17	0.024	0.27	0.012	0.075	ND	0.038	
			Mean	<0.01	0.195	0.0285	0.315	0.0135	0.078	<0.01	0.046	0.6391
			28	ND	0.22	0.028	0.36	0.018	0.10	<0.01	0.043	
				ND	0.22	0.028	0.37	0.014	0.097	<0.01	0.045	
			Mean	<0.01	0.22	0.028	0.365	0.016	0.0985	<0.01	0.044	0.7293
			35	ND	0.20	0.025	0.35	0.011	0.12	<0.01	0.050	
				ND	0.36	0.054	0.55	0.029	0.16	<0.01	0.072	
			Mean	<0.01	0.28	0.0395	0.45	0.020	0.14	<0.01	0.061	0.9579
Porterville, CA, United States, 2014 Gardner Delight (Cherry)	3 (59 14) drench 2×drip	1.12	1	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
		0.56		ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
		0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	<0.01	ND	ND	ND	<0.01	ND	ND	
				ND	<0.01	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	<0.01	ND	ND	ND	<0.01	ND	ND	
				ND	<0.01	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Porterville, CA, United States, 2014 Gardner Delight (Cherry)	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	<0.01	ND	0.013	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	ND	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	<0.01	ND	ND	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Paso Robles, CA, United States, 2014 Cherry 100 (Cherry)	3 (83 13) drip	1.12 0.56 0.57	1	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Paso Robles, CA, United States, 2014 Cherry 100 (Cherry)	4 (15 12 13) drip	0.55 0.57 0.56 0.57	1	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	<0.01	<0.01	<0.01	ND	ND	ND	ND	
				ND	0.01[A]	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01[A]	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	<0.01	<0.01	ND	ND	ND	ND	ND	
				ND	0.01[A]	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	0.012	ND	<0.01	ND	ND	ND	ND	
				ND	0.012	ND	<0.01	ND	ND	ND	ND	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
			Mean	<0.01	0.012	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0785
			35	ND	0.012	ND	<0.01	ND	ND	ND	ND	
				ND	0.014	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	0.013	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0808
Fresno, CA, United States, 2014 Dri219 (Large)	3 (78 14) drip	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	<0.01	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	<0.01	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Fresno, CA, United States, 2014 Dri219 (Large)	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	<0.01	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	<0.01	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Madera, CA, United States, 2014 Quality 27 (Large)	3 (60 14) soil directed	1.12 0.56 0.56	1	NDD	NDD	NDD	NDD	NDD	NDD	NDD	NDD	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Madera, CA, United States,	4 (14 14 14) soil	0.56 0.56	1	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	

Location, year Variety, trial	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [F]
2014 Quality 27 (Large)	directed	0.56 0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND ND	<0.01 <0.01	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND ND	<0.01 <0.01	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740

Notes:

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] Average of duplicate analyses.

[C] Average of triplicate analyses.

[D] Treated sample inadvertently used as a control.

[E] Residues in untreated control samples were ND for all analytes with the exception of one tomato control sample with post hydrolysis residues of 0.010 and 0.018 mg/kg in IN-A5760 and IN QEK31, respectively.

[F] SUM = 2.26×IN-A5760 + 2.11×IN-F4106 + 1.52×IN-QZY47 + 1.51×IN-TMQ01.

[G] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 140 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post- hydrolysis) in peppers from supervised trials conducted in Canada and the United States

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [C]
Branchton, ON, Canada, 2014 Socrates (Bell) Trial 27 13/6 8/8 21/8	3 (56 13) drip	1.12 0.56 0.56	1	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Socrates (Bell)	4 (14 14 13) 11/7 25/7 8/8 21/8	0.56 0.56 0.56 0.56	1	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [C]
	drip		Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Aristotle (Bell) Trial 28	3 (57 14) 18/6 14/8 28 drip	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			8	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	<0.01	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Aristotle (Bell)	4 (14 13 14) 18/7 1/8 14/8 28/8 drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			8	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Tomcat (Bell) Trial 29	3 (51 12) 16/6 6/8 18/8 drip	1.12 0.56 0.56	1	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	<0.01	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			16	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			23	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	<0.01	<0.01	ND	0.01 [A]	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [c]
			30	ND	<0.01	ND	<0.01	ND	0.011	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	0.0748
			37	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	0.012	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.011	<0.01	<0.01	0.0755
Branchton, ON, Canada, 2014 Tomcat (Bell)	4 (19 14 12) 4/7 23/7 6/8 18/8 drip	0.56 0.56 0.56 0.56	1	ND	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			16	ND	ND	<0.01	<0.01	ND	<0.01	ND	ND	
				ND	ND	<0.01	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			23	ND	ND	<0.01	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			30	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			37	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Long Red Cyanne (non-Bell) Trial 30	3 (56 15) 12/6 7/8 22/8 drip	1.12 0.56 0.56	1	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	<0.01	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2014 Long Red Cyanne (non-Bell)	4 (14 21 15) 3/7 17/7 7/8 22/8 drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON,	3 (51 13)	1.12	1	ND	ND	ND	ND	ND	ND	ND	ND	

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [c]
Canada, 2014	18/6	0.56		ND	ND	ND	ND	ND	ND	ND	ND	
Crimson Hot	8/8	0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
(non-Bell) Trial	21/8		7	ND	ND	ND	ND	ND	ND	ND	ND	
31	drip			ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON,	4 (14 14 13)	0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
Canada, 2014	25/6	0.56		ND	ND	ND	ND	ND	ND	ND	ND	
Crimson Hot	8/8	0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
(non-Bell)	21/8	0.56	7	ND	ND	ND	ND	ND	ND	ND	ND	
11/7	drip			ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON,	3 (52 12)	1.12	1	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	ND	
Canada, 2014	16/6	0.56		ND	<0.01	<0.01	<0.01	ND	<0.01	ND	ND	
Hungarian Wax	7/8	0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
(non-Bell) Trial	19/8		6	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	ND	
32	drip			ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	<0.01	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON,	4 (12 8 12)	0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
Canada, 2014	18/7	0.56		ND	ND	ND	ND	ND	ND	ND	ND	
Hungarian Wax	30/7	0.56	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
(non-Bell)	7/8	0.56	6	ND	ND	ND	ND	ND	ND	ND	ND	
	19/8			ND	ND	ND	ND	ND	ND	ND	ND	
	drip		Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [C]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Socrates (Bell) Trial 33	3 (56 14) 2/6 28/6 11/8 drench	1.12 0.56 0.56	1	0.020	ND	0.017	0.014	ND	ND	ND	ND	
				0.031	ND	0.019	0.020	ND	ND	ND	ND	
			Mean	0.0255	<0.01	0.018	0.017	<0.01	<0.01	<0.01	<0.01	0.0909
			7	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	<0.01	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Socrates (Bell)	4 (14 14 14) 30/6 14/7 28/7 11/8 drench	0.56 0.56 0.56 0.56	1	0.012	ND	0.01[A]	0.010	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	0.011	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Aristotle X3R (Bell) Trial 34	3 (56 14) 17/6 12/8 26/8 drench	1.12 0.56 0.56	1	ND	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [C]
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Aristotle X3R (Bell)	4 (14 13 14) 16/7 30/7 12/8 26/8 d rench	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	<0.01	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Long Rouge (non-Bell) Trial 35	3 (50 14) 17/6 6/8 20/8 drench	1.12 0.56 0.56	1	0.011	ND	<0.01	<0.01	ND	ND	ND	ND	
				0.012	ND	0.010	<0.01	ND	ND	ND	ND	
			Mean	0.0115	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Long Rouge (non-Bell)	4 (15 13 14) 9/7 24/7 6/8 20/8 drench	0.56 0.56 0.56 0.56	1	0.032	ND	0.019	0.019	ND	ND	ND	ND	
				0.021	ND	0.015	0.015	ND	ND	ND	ND	
			Mean	0.0265	<0.01	0.017	0.017	<0.01	<0.01	<0.01	<0.01	0.0888
			6	ND	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	0.010	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Jaune Hongrois	3 (56 14) 17/6 12/8 26/8	1.12 0.56 0.56	1	<0.01	<0.01	<0.01	<0.01	ND	ND	ND	ND	
				0.011	ND	0.01 [A]	<0.01	ND	ND	ND	ND	
			Mean	<0.0105	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	<0.01	ND	ND	ND	ND	ND	ND	

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [C]
(non-Bell) Trial 36	drench			ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	<0.01	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	<0.01	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	<0.01	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2014 Jaune Hongrois (non-Bell)	4 (14 13 14) 16/7 30/7 12/8 26/8 drench	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	<0.01	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	<0.01	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Athens, GA, United States, 2014 Aristotle (Bell)	3 (63 14) drip	1.12 0.56 0.56	1	ND	0.011	0.015	<0.01	ND	0.034	ND	0.021	
				ND	0.010	0.014	<0.01	ND	0.034	ND	0.020	
			Mean	<0.01	0.0105	0.0145	<0.01	<0.01	0.034	<0.01	0.0205	0.1209
			7	ND	0.01[A]	0.012	<0.01	ND	0.030	ND	0.021	
				ND	<0.01	0.016	<0.01	ND	0.031	ND	0.018	
			Mean	<0.01	<0.01	0.014	<0.01	<0.01	0.0305	<0.01	0.0195	0.1134
			15	ND	<0.01	0.014	<0.01	ND	0.022	ND	0.013	
				ND	<0.01	0.011	<0.01	ND	0.020	ND	0.012	
			Mean	<0.01	<0.01	0.0125	<0.01	<0.01	0.021	<0.01	0.0125	0.0959
			22	ND	<0.01	0.01[A]	<0.01	ND	0.022	ND	0.015	
				ND	<0.01	<0.01	<0.01	ND	0.016	ND	0.014	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.019	<0.01	0.0145	0.0876
			29	ND	<0.01	<0.01	<0.01	ND	0.017	ND	0.012	
				ND	<0.01	<0.01	<0.01	ND	0.022	ND	0.016	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0195	<0.01	0.014	0.0883
			36	ND	<0.01	<0.01	0.012	ND	0.022	ND	0.017	
				ND	<0.01	<0.01	0.011	ND	0.019	ND	0.016	
			Mean	<0.01	<0.01	<0.01	0.0115	<0.01	0.0205	<0.01	0.0165	0.0899
Athens, GA, United States, 2014 Aristotle (Bell)	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	<0.01	<0.01	<0.01	ND	0.013	ND	<0.01	
				ND	<0.01	0.010	0.01[A]	ND	0.016	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0145	<0.01	<0.01	0.0808
			7	ND	<0.01	<0.01	<0.01	ND	0.015	ND	0.01[A]	
				ND	<0.01	0.011	0.012	ND	0.020	ND	0.013	
			Mean	<0.01	<0.01	<0.0105	<0.011	<0.01	0.0175	<0.01	0.0115	0.0864
			15	ND	<0.01	0.011	0.010	ND	0.021	ND	0.012	
				ND	<0.01	<0.01	<0.01	ND	0.017	ND	<0.01	
			Mean	<0.01	<0.01	<0.0105	<0.01[A]	<0.01	0.019	<0.01	<0.011	0.0886
			22	ND	<0.01	<0.01	<0.01	ND	0.015	ND	0.010	
				ND	<0.01	<0.01	<0.01	ND	0.016	ND	0.010	

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [C]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0155	<0.01	0.010	0.0823
			29	ND	<0.01	<0.01	<0.01	ND	0.021	ND	0.014	
				ND	<0.01	<0.01	0.011	ND	0.022	ND	0.014	
			Mean	<0.01	<0.01	<0.01	<0.0105	<0.01	0.0215	<0.01	0.014	0.0914
			36	ND	<0.01	<0.01	0.010	ND	0.018	ND	0.013	
				ND	ND	<0.01	<0.01	ND	0.013	ND	0.01[A]	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0155	<0.01	0.0115	0.0823
Tifton, GA, United States, 2014 Jalapeno (non-Bell)	3 (56 11) drip	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			20	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			32	ND	<0.01	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Tifton, GA, United States, 2014 Jalapeno (non-Bell)	4 (18 14 11) drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			20	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			32	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Center Hill, FL, United States, 2014 Tomcat (Bell)	3 (82 14) drip	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [c]
Center Hill, FL, United States, 2014 Tomcat (Bell)	4 (9 19 14) drip	0.56 0.56 0.56 0.56	1	ND ND	ND ND	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	<0.01 <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND ND	ND ND	ND ND	<0.01 ND	ND ND	<0.01 <0.01	ND ND	<0.01 ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND ND	ND ND	ND ND	<0.01 ND	ND ND	<0.01 ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND ND	ND ND	ND ND	<0.01 ND	ND ND	<0.01 ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND ND	ND ND	ND ND	<0.01 ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			36	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Richland, IA, United States, 2014 California Wonder (Bell)	3 (90 14) drip	1.12 0.56 0.56	1	<0.01 ND	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	ND ND	<0.01 <0.01	ND ND	ND <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			13	ND <0.01	ND ND	<0.01 <0.01	<0.01 <0.01	ND ND	<0.01 <0.01	ND ND	<0.01 <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND ND	<0.01 <0.01	ND ND	<0.01 ND	ND ND	<0.01 <0.01	ND ND	<0.01 <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND ND	ND <0.01	ND ND	ND <0.01	ND ND	<0.01 <0.01	ND ND	ND <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND ND	ND ND	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Richland, IA, United States, 2014 California Wonder (Bell)	4 (13 15 14) drip	0.56 0.56 0.56 0.56	1	ND ND	ND ND	ND <0.01	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND <0.01	ND ND	<0.01 <0.01	<0.01 ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			13	ND ND	ND ND	<0.01 <0.01	<0.01 <0.01	ND ND	<0.01 ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND ND	ND ND	ND ND	ND <0.01	ND ND	ND <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND ND	ND ND	ND ND	ND <0.01	ND ND	ND <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Lime Springs, IA, United States, 2014 Vangard (Bell) 02	3 (68 14) drench	1.12 0.56 0.56	1	<0.01 ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [c]
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Lime Springs, IA, United States, 2014 Vangard (Bell) 03	4 (28 13 14) drench	0.56 0.56 0.56 0.56	1	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Lime Springs, IA, United States, 2014 Vangard (Bell) 04	3 (68 14) drench	2.24 1.12 1.12	1	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Lime Springs, IA, United States, 2014 Vangard (Bell) 05	4 (28 13 14) drench	1.12 1.12 1.12 1.12	1	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	0.013	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.0115	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [C]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				0.010	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Uvalde, TX, United States, 2014 Capistrand (Bell)	3 (105 14) drip	0.55 0.55 0.55	1	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND[B]	ND[B]	ND[B]	ND[B]	ND[B]	ND[B]	ND[B]	ND[B]	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			30	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			37	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Uvalde, TX, United States, 2014 Capsitrand (Bell)	4 (13 14 14) drip	0.55 0.55 0.55 0.55	1	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	<0.01	0.012	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	<0.01	0.014	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.012	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	<0.01	ND	0.011	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			30	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			37	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Claude, TX, United States, 2014 Big Jim Numex (non-Bell) Trial 39	3 (92 14) drench	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Claude, TX, United States, 2014 Big Jim	4 (14 14 14) drench	0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [c]
Numex (non-Bell) Trial 39		0.56	6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Fresno, CA, United States, 2014 Classic (Bell) Trial 4-0	3 (57 14) drip	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	<0.01	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	ND	ND	<0.01	ND	<0.01	
				ND	ND	ND	ND	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Fresno, CA, United States, 2014 Classic (Bell)	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	<0.01	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	<0.01	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	<0.01	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Yuma, AZ, United States, 2015 Massivo (non-Bell) Trial 41	3 (67 14) drip	1.12 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [c]
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Yuma, AZ, United States, 2015 Massivo (non-Bell)	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			7	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			15	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			22	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			29	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Madera, CA, United States, 2014 Maccabi (Bell)	3 (53 14) soil directed	1.13 0.56 0.56	1	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	<0.01	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Madera, CA, United States, 2014 Maccabi (Bell)	4 (14 14 14) soil directed	0.56 0.56 0.56 0.56	1	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			6	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			14	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			21	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			28	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			35	ND	<0.01	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Sanger, CA, United States, 2014 Ancho (non-Bell) Trial 43	3 (67 14) soil directed 2×drip	1.12 0.56 0.56	1	ND	0.048	0.015	0.10	0.024	0.097	ND	0.040	
				<0.01	0.048	0.016	0.096	0.022	0.082	ND	0.035	
			Mean	<0.01	0.048	0.015	0.098	0.023	0.0895	<0.01	0.0375	0.3102
			6	ND	0.029	0.014	0.067	0.011	0.056	ND	0.021	
				<0.01	0.035	0.018	0.10	0.020	0.074	ND	0.029	

Location, year, variety	N (interval)days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [C]
			Mean	<0.01	0.032	0.016	0.08355	0.0155	0.065	<0.01	0.025	0.2278
			14	ND	0.025	0.011	0.070	0.013	0.045	ND	0.018	
				ND	0.030	0.011	0.075	0.017	0.057	ND	0.028	
			Mean	<0.01	0.0275	0.011	0.0725	0.015	0.051	<0.01	0.023	0.1852
			21	ND	0.013	ND	0.054	<0.01	0.031	ND	0.012	
				ND	0.016	<0.01	0.044	<0.01	0.031	ND	0.013	
			Mean	<0.01	0.0145	0.005	0.049	0.01	0.031	<0.01	0.0125	0.1159
			28	ND	0.032	0.016	0.073	0.014	0.019	ND	0.014	
				ND	0.036	0.022	0.090	0.020	0.024	<0.01	0.020	
			Mean	<0.01	0.034	0.019	0.0815	0.017	0.0215	<0.01	0.017	0.1752
			35	ND	0.018	<0.01	0.059	<0.01	0.023	ND	0.015	
				ND	0.018	<0.01	0.056	<0.01	0.026	ND	0.012	
			Mean	<0.01	0.018	<0.01	0.0575	<0.01	0.0245	<0.01	0.0145	0.1140
Sanger, CA, United States, 2014 Ancho (non-Bell)	4 (14 14 14) drip	0.56 0.56 0.56 0.56	1	<0.01	0.027	0.016	0.065	<0.01	0.017	ND	<0.01	
				<0.01	0.034	0.025	0.081	0.015	0.027	ND	0.016	
			Mean	<0.01	0.0305	0.0205	0.073	<0.0125	0.022	<0.01	<0.013	0.1644
			6	<0.01	0.026	0.018	0.069	<0.01	0.014	ND	<0.01	
				<0.01	0.034	0.026	0.089	0.013	0.023	ND	0.010	
			Mean	<0.01	0.030	0.022	0.079	<0.0115	0.0185	<0.01	<0.01	0.1596
			14	<0.01	0.037	0.028	0.10	0.022	0.025	ND	0.018	
				ND	0.037	0.015	0.075	0.016	0.020	ND	0.011	
			Mean	<0.01	0.037	0.0215	0.0875	0.019	0.0225	<0.01	0.0145	0.1918
			21	ND	0.037	0.013	0.089	0.015	0.022	ND	0.011	
				ND	0.027	0.012	0.080	0.011	0.017	ND	<0.01	
			Mean	<0.01	0.032	0.0125	0.0845	0.013	0.0195	<0.01	<0.0105	0.1479
			28	ND	0.027	<0.01	0.066	0.023	0.049	ND	0.030	
				ND	0.030	0.011	0.067	0.017	0.053	<0.01	0.034	
			Mean	<0.01	0.0285	0.0105	0.0665	0.020	0.051	<0.01	0.032	0.1940
			35	ND	0.039	0.014	0.12	0.020	0.023	ND	0.014	
				ND	0.023	0.011	0.070	0.011	0.013	ND	<0.01	
			Mean	<0.01	0.031	0.0125	0.095	0.0155	0.018	<0.01	<0.012	0.1472

Notes:

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] Treated sample inadvertently used as a control.

[C] SUM = 2.26×IN-A5760 + 2.11×IN-F4106 + 1.52×IN-QZY47 + 1.51×IN-TMQ01.

[D] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Carrots

Shepard (2020 DuPont-43192 rev 1) conducted residue trials on carrots at 11 sites in Canada and the United States in 2014/2015. At each location, separate plots were treated with fluazaindolizine SC formulation was applied as a single soil application (in-furrow spray) at planting at 2.24 kg ai/ha and at the other plot as a soil application (in-furrow spray) at planting followed by a soil directed spray 14±1 days later, both applications at 1.12 kg ai/ha. Adjuvants were not used at most of trial sites, with the exception of Uvalde, Texas, second of two applications and Jerome, Idaho, second of two applications which both included non-ionic surfactant at 0.25 percent. The first sample collected was of immature carrot roots with the remaining of mature carrots.

The maximum interval of frozen storage before analysis was 152 days before extraction. Carrots were analysed for residues of fluazaindolizine and its metabolites IN-A5760, IN-F4016, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12 and IN-TQD54 using the analytical method DuPont-33861, rev. 3, with an

LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg for each analyte. Acceptable concurrent recovery data were obtained for all matrices.

At the Richland IA, Woodland CA and Porterville CA trial sites, the per hectare application was concentrated in the band or furrow at planting and for the Woodland CA also for the later applications. The practice gave rise to higher residues compared to the other trials.

Table 141 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in carrot from trials conducted in Canada and the United States

Location, year, variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
Woodville, NS, Canada, 2015 Bolero	1 banded seed at planting	2.24	83	ND	ND	ND	ND	0.016	0.042	ND	<0.01	
				ND	ND	ND	ND	0.019	0.049	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0175	0.0455	<0.01	<0.01	0.1390
			88	ND	ND	ND	<0.01	0.017	0.053	ND	<0.01	
				ND	ND	ND	<0.01	0.017	0.054	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.017	0.0535	<0.01	<0.01	0.1503
			94	ND	ND	ND	<0.01	0.012	0.036	ND	ND	
				ND	ND	ND	ND	0.012	0.033	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.012	0.0345	<0.01	<0.01	0.1140
			99	ND	ND	ND	ND	0.011	0.041	ND	<0.01	
				ND	ND	ND	ND	0.012	0.040	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0115	0.0405	<0.01	<0.01	0.1223
			104	ND	ND	ND	ND	0.012	0.035	ND	ND	
				ND	ND	ND	ND	0.011	0.031	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0115	0.033	<0.01	<0.01	0.1110
			109	ND	ND	ND	ND	0.014	0.039	ND	<0.01	
				ND	ND	ND	ND	0.013	0.041	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0135	0.040	<0.01	<0.01	0.1246
			115	ND	ND	ND	ND	0.013	0.041	ND	ND	
				ND	ND	ND	ND	0.011	0.037	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.012	0.039	<0.01	<0.01	0.1208
Woodville, NS, Canada, 2015 Bolero	2 (14) banded seed at planting, cotyledon	1.12 1.11	69	ND ND	ND ND	ND ND	<0.01 <0.01	0.020 0.021	0.045 0.041	ND ND	<0.01 <0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0205	0.043	<0.01	<0.01	0.1398
			74	ND	ND	ND	<0.01	0.016	0.037	ND	<0.01	
				ND	ND	ND	<0.01	0.017	0.037	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0165	0.037	<0.01	<0.01	0.1247
			80	ND	ND	ND	<0.01	0.014	0.036	ND	<0.01	
				ND	ND	ND	<0.01	0.013	0.032	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0135	0.034	<0.01	<0.01	0.1156
			85	ND	ND	ND	ND	0.013	0.031	ND	<0.01	
				ND	ND	ND	<0.01	0.016	0.037	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0145	0.034	<0.01	<0.01	0.1171
			90	ND	ND	ND	ND	0.011	0.030	ND	<0.01	
				ND	ND	ND	ND	0.011	0.028	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.011	0.029	<0.01	<0.01	0.1042
			95	ND	ND	ND	ND	0.014	0.036	ND	<0.01	
				ND	ND	ND	<0.01	0.012	0.037	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.013	0.0365	<0.01	<0.01	0.1186
			101	ND	ND	ND	<0.01	0.011	0.038	ND	ND	
				ND	ND	ND	ND	0.013	0.037	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.012	0.0375	<0.01	<0.01	0.1186
Branchton, ON, Canada, 2015 Goldfinger Trial 03	1 broadcast pre-germination	2.19	59	<0.01 <0.01	ND ND	ND <0.01	0.011 0.017	0.042 0.049	0.063 0.064	ND ND	<0.01 <0.01	
			Mean	<0.01	<0.01	<0.01	0.014	0.0455	0.0635	<0.01	<0.01	0.2087
			84	ND	ND	ND	<0.01	0.011	0.042	ND	<0.01	
				ND	ND	ND	<0.01	0.012	0.036	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0115	0.039	<0.01	<0.01	0.1201
			90	ND	ND	ND	<0.01	0.011	0.032	ND	<0.01	

Location, year, variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
				ND	ND	ND	<0.01	0.011	0.029	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.011	0.0305	<0.01	<0.01	0.1065
			96	ND	ND	ND	<0.01	<0.01	0.036	ND	<0.01	
				ND	ND	ND	<0.01	<0.01	0.025	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0305	<0.01	<0.01	0.1050
			101	ND	ND	ND	<0.01	0.010	0.046	ND	<0.01	
				ND	ND	ND	<0.01	0.010	0.041	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.010	0.0435	<0.01	<0.01	0.1246
			105	ND	ND	ND	<0.01	<0.01	0.033	ND	<0.01	
				ND	ND	ND	<0.01	0.011	0.039	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0105	0.036	<0.01	<0.01	0.1140
			109	ND	ND	ND	<0.01	<0.01	0.036	ND	<0.01	
				ND	ND	ND	<0.01	0.01[A]	0.047	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0415	<0.01	<0.01	0.1216
Branchton, ON, Canada, 2015 Goldfinger	Broadcast pre-germination and pre-emergence	1.07 1.17	45	<0.01	ND	ND	0.015	0.044	0.073	ND	<0.01	
				<0.01	ND	<0.01	0.017	0.053	0.072	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.016	0.0485	0.0725	<0.01	<0.01	0.2269
			70	ND	ND	ND	<0.01	0.013	0.044	ND	<0.01	
				ND	ND	ND	<0.01	0.015	0.046	ND	<0.01	
			Mean	≤0.01	≤0.01	≤0.01	<0.01	0.014	0.045	≤0.01	≤0.01	0.1329
			76	ND	ND	ND	<0.01	0.010	0.033	ND	<0.01	
				ND	ND	ND	<0.01	0.01[A]	0.039	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.010	0.036	<0.01	<0.01	0.1133
			82	ND	ND	ND	0.014	0.011	0.041	ND	<0.01	
				ND	ND	ND	0.011	0.010	0.041	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.0125	0.0105	0.041	<0.01	<0.01	0.1216
			87	ND	ND	ND	0.012	0.01[A]	0.039	ND	<0.01	
				ND	ND	ND	<0.01	0.012	0.047	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.011	0.011	0.043	<0.01	<0.01	0.1254
			91	ND	ND	ND	<0.01	<0.01	0.032	ND	<0.01	
				ND	ND	ND	<0.01	0.011	0.045	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0105	0.0385	<0.01	<0.01	0.1178
			95	ND	ND	ND	0.010	<0.01	0.041	ND	<0.01	
				ND	ND	ND	0.01[A]	0.011	0.047	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.01[A]	<0.0105	0.044	<0.01	<0.01	0.1261
St-Marc-sur-Richelieu, QC, Canada, 2015 Naval	1 banded BBCH 00	2.23	79	ND	ND	ND	ND	<0.01	<0.01	ND	ND	
				ND	ND	ND	ND	<0.01	0.011	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	0.0748
St-Marc-sur-Richelieu, QC, Canada, 2015 Naval	1 banded BBCH 00	2.23	65	0.010	ND	<0.01	0.012	0.034	0.028	ND	ND	
				<0.01	ND	<0.01	0.011	0.028	0.020	ND	ND	
			Mean	≤0.01	≤0.01	≤0.01	0.0115	0.031	0.024	≤0.01	≤0.01	0.1271
			79	ND	ND	ND	ND	<0.01	0.010	ND	ND	
				ND	ND	ND	ND	<0.01	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			84	ND	ND	ND	ND	<0.01	<0.01	ND	ND	
				ND	ND	ND	ND	<0.01	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			89	<0.01	ND	ND	<0.01	<0.01	0.010	ND	ND	
				<0.01	ND	ND	<0.01	<0.01	0.012	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	0.0755
			94	ND	ND	ND	<0.01	<0.01	0.012	ND	ND	
				ND	ND	ND	<0.01	<0.01	0.014	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	0.0785
			99	ND	ND	ND	<0.01	<0.01	0.01[A]	ND	ND	
				ND	ND	ND	<0.01	<0.01	0.011	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0105	<0.01	<0.01	0.0748
			105	ND	ND	ND	ND	<0.01	<0.01	ND	ND	
				ND	ND	ND	ND	<0.01	0.011	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	0.0748

Location, year, variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
St-Marc-sur-Richelieu, QC, Canada, 2015 Naval	2 (13) banded BBCH00, BBCH 05	1.17 1.13	66	ND ND	ND ND	ND ND	ND ND	<0.01 <0.01	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St-Marc-sur-Richelieu, QC, Canada, 2015 Naval	2 (13) banded BBCH00, BBCH 05	1.17 1.13	52	<0.01 <0.01	ND ND	<0.01 <0.01	<0.01 <0.01	0.019 0.022	0.014 0.016	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0205	0.015	<0.01	<0.01	0.0975
			66	ND ND	ND ND	ND ND	ND ND	<0.01 <0.01	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			71	ND ND	ND ND	ND ND	ND ND	<0.01 <0.01	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			76	ND ND	ND ND	ND ND	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			81	ND ND	ND ND	ND ND	<0.01 ND	<0.01 <0.01	0.011 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	0.0748
			86	ND ND	ND ND	ND ND	<0.01 <0.01	<0.01 <0.01	0.011 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	0.0748
			92	ND ND	ND ND	ND ND	ND ND	<0.01 <0.01	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Oviedo, FL, United States, 2015 Nelson F1	1 banded at planting	2.22	56	<0.01 <0.01	ND ND	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			90	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			94	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			99	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	<0.01 ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			104	<0.01 <0.01	ND ND	ND ND	<0.01 ND	ND ND	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			109	<0.01 <0.01	ND ND	ND ND	ND <0.01	ND ND	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			114	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Oviedo, FL, United States, 2015 Nelson F1	2 (14) soil directed over row, BBCH 03-12	1.10 1.12	42	<0.01 <0.01	ND ND	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			76	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			80	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			85	<0.01 ND	ND ND	ND ND	<0.01 ND	ND ND	ND ND	ND ND	ND ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			90	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	ND ND	

Location, year, variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			95	ND	ND	ND	ND	ND	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			100	<0.01	ND	ND	ND	ND	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Willard, OH, United States, 2015 Yosemite 480	1 broadcast seed at planting	2.24	70	0.034	ND	0.021	0.025	<0.01	0.01[A]	ND	ND	
				0.046	ND	0.024	0.031	<0.01	0.014	ND	ND	
			Mean	0.040	<0.01	0.0225	0.028	<0.01	0.012	<0.01	<0.01	0.1034
			119	0.010	ND	<0.01	<0.01	<0.01	<0.01	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			124	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			129	0.010	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			134	0.017	ND	0.014	0.013	ND	<0.01	ND	ND	
				<0.01	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.0135	<0.01	<0.012	<0.0115	<0.01	<0.01	<0.01	<0.01	0.0782
			139	0.01[A]	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			144	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Willard, OH, United States, 2015 Yosemite 480	2 (13) broadcast seed and planting and seedling	1.11 1.13	57	0.014	ND	ND	ND	ND	ND	ND	ND	
				0.013	ND	<0.01	<0.01	<0.01	<0.01	ND	ND	
			Mean	0.0135	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			106	0.011	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.0105	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			111	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			116	0.010	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			121	0.011	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.0105	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			126	0.011	ND	<0.01	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.0105	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			131	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Richland, IA, United States, 2015 Sugarsnax 54	1 in-furrow preplant	2.26	62	0.24	0.011	0.12	0.25	0.47	0.57	<0.01	0.042	
				0.29	0.015	0.15	0.29	0.53	0.62	<0.01	0.042	
			Mean	0.265	0.013	0.135	0.27	0.50	0.595	<0.01	0.042	1.9727
			79	0.037	ND	0.019	0.051	0.15	0.32	ND	0.021	
				0.032	ND	0.018	0.044	0.12	0.23	ND	0.016	
			Mean	0.0345	<0.01	0.0185	0.0475	0.135	0.275	<0.01	0.0185	0.6821
			83	0.020	ND	0.011	0.032	0.098	0.23	ND	0.014	
				0.026	ND	0.014	0.039	0.11	0.23	ND	0.015	
			Mean	0.023	<0.01	0.0125	0.0355	0.104	0.23	<0.01	0.0145	0.5544
			88	0.036	<0.01	0.022	0.048	0.12	0.22	ND	0.015	
				0.033	<0.01	0.021	0.047	0.11	0.24	ND	0.016	

Location, year, variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	0.0345	<0.01	0.0215	0.0475	0.115	0.23	<0.01	0.0155	0.5901
			93	0.027	ND	0.019	0.033	0.11	0.20	ND	0.014	
				0.027	ND	0.017	0.031	0.081	0.17	ND	0.014	
			Mean	0.027	<0.01	0.018	0.032	0.0955	0.185	<0.01	0.014	0.4851
			98	<0.01	ND	ND	0.014	0.063	0.21	ND	0.014	
				<0.01	ND	<0.01	0.018	0.061	0.18	ND	0.012	
			Mean	<0.01	<0.01	<0.01	0.016	0.062	0.195	<0.01	0.013	0.4324
			103	0.010	ND	<0.01	0.020	0.080	0.22	ND	0.014	
				0.015	ND	0.011	0.023	0.072	0.20	ND	0.015	
			Mean	0.0125	<0.01	<0.0105	0.0215	0.076	0.21	<0.01	0.0145	0.4774
Richland, IA, United States, 2015 Sugarsnax 54	2 (14) In-furrow preplant, broadcast BBCH11	1.12 1.14	48	0.099	<0.01	0.051	0.094	0.13	0.17	ND	0.012	
				0.087	ND	0.041	0.078	0.12	0.15	ND	0.012	
			Mean	0.093	<0.01	0.046	0.086	0.125	0.16	<0.01	0.012	0.5513
			65	0.017	ND	<0.01	0.019	0.049	0.088	ND	<0.01	
				0.016	ND	0.01[A]	0.019	0.048	0.072	ND	<0.01	
			Mean	0.0165	<0.01	<0.01	0.019	0.0485	0.080	<0.01	<0.01	0.2382
			69	0.012	ND	<0.01	0.015	0.039	0.070	ND	<0.01	
				0.012	ND	<0.01	0.015	0.048	0.094	ND	<0.01	
			Mean	0.012	<0.01	<0.01	0.015	0.0435	0.082	<0.01	<0.01	0.2336
			74	0.030	ND	0.016	0.029	0.053	0.12	ND	<0.01	
				0.024	ND	0.013	0.024	0.051	0.11	ND	<0.01	
			Mean	0.027	<0.01	0.0145	0.0265	0.052	0.115	<0.01	<0.01	0.3059
			79	0.020	ND	0.01[A]	0.021	0.057	0.13	ND	<0.01	
				0.023	ND	0.011	0.024	0.056	0.14	ND	0.010	
			Mean	0.0215	<0.01	0.0105	0.0225	0.0565	0.135	<0.01	<0.01[A]	0.3345
			84	<0.01	ND	ND	<0.01	0.030	0.10	ND	<0.01	
				<0.01	ND	ND	<0.01	0.021	0.077	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0255	0.0885	<0.01	<0.01	0.2161
			89	<0.01	ND	ND	<0.01	0.032	0.097	ND	<0.01	
				<0.01	ND	ND	<0.01	0.036	0.11	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.034	0.1035	<0.01	<0.01	0.2517
Uvalde, TX, United States, 2015 Belgrado F1	1 in-furrow	2.25	123	0.025	ND	0.019	0.029	0.032	0.025	ND	ND	
				0.015	ND	0.011	0.017	0.025	0.022	ND	ND	
			Mean	0.020	<0.01	0.015	0.023	0.0285	0.0235	<0.01	<0.01	0.1331
			133	ND	ND	ND	<0.01	<0.01	0.014	ND	ND	
				<0.01	ND	ND	<0.01	<0.01	0.016	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.015	<0.01	<0.01	0.0816
			138	<0.01	ND	ND	<0.01	<0.01	0.014	ND	ND	
				ND	ND	ND	<0.01	<0.01	0.012	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	0.0785
			142	ND	ND	ND	<0.01	<0.01	0.013	ND	ND	
				ND	ND	ND	<0.01	<0.01	0.013	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	0.0785
			147	ND	ND	ND	ND	<0.01	0.013	ND	ND	
				ND	ND	ND	ND	<0.01	0.015	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.014	<0.01	<0.01	0.0800
			151	ND	ND	ND	ND	<0.01	0.011	ND	ND	
				<0.01	ND	ND	<0.01	<0.01	0.016	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0135	<0.01	<0.01	0.0793
			155	<0.01	ND	ND	ND	<0.01	0.015	ND	ND	
				ND	ND	ND	ND	<0.01	0.017	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.016	<0.01	<0.01	0.0831
Uvalde, TX, United States, 2015 Belgrado F1	2 (14) in-furrow, broadcast cotyledons	1.14 1.12	109	0.016	ND	0.016	0.021	0.029	0.022	ND	ND	
				0.020	ND	0.020	0.030	0.038	0.027	ND	ND	
			Mean	0.018	<0.01	0.018	0.0255	0.0335	0.0245	<0.01	<0.01	0.1485
			119	<0.01	ND	ND	<0.01	0.013	0.018	ND	ND	
				ND	ND	ND	<0.01	<0.01	0.017	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0115	0.0175	<0.01	<0.01	0.0876
			124	ND	ND	ND	<0.01	<0.01	0.015	ND	ND	
				ND	ND	ND	<0.01	<0.01	0.015	ND	ND	

Location, year, variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.015	<0.01	<0.01	0.0816
			128	ND	ND	ND	<0.01	<0.01	0.014	ND	ND	
				ND	ND	ND	<0.01	<0.01	0.012	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	0.0785
			133	<0.01	ND	ND	<0.01	<0.01	0.017	ND	ND	
				ND	ND	ND	ND	<0.01	0.016	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0165	<0.01	<0.01	0.0838
			137	<0.01	ND	ND	ND	<0.01	0.015	ND	ND	
				ND	ND	ND	ND	<0.01	0.016	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0155	<0.01	<0.01	0.0823
			141	ND	ND	ND	<0.01	<0.01	0.018	ND	ND	
				<0.01	ND	ND	ND	<0.01	0.017	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0175	<0.01	<0.01	0.0853
Woodland, CA, United States, 2015 Red Cored Chantenay	1 banded in-furrow	2.25	112	0.094	ND	0.057	0.086	0.091	0.068	ND	<0.01	
				0.061	ND	0.040	0.061	0.10	0.084	ND	<0.01	
			Mean	0.0775	<0.01	0.0485	0.0735	0.0955	0.076	<0.01	<0.01	0.3849
			145	0.014	ND	<0.01	0.020	0.032	0.032	ND	ND	
				0.017	ND	0.01A	0.025	0.048	0.045	ND	<0.01	
			Mean	0.0155	<0.01	<0.01	0.0225	0.040	0.0385	<0.01	<0.01	0.1626
			149	<0.01	ND	<0.01	<0.01	0.023	0.013	ND	ND	
				0.011	ND	<0.01	0.018	0.034	0.031	ND	ND	
			Mean	<0.0105	<0.01	<0.01	0.014	0.0285	0.022	<0.01	<0.01	0.1202
			154	<0.01	ND	<0.01	0.010	0.019	0.015	ND	ND	
				<0.01	ND	<0.01	0.012	0.025	0.027	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.011	0.022	0.021	<0.01	<0.01	0.1089
			159	<0.01	ND	<0.01	0.013	0.032	0.024	ND	ND	
				<0.01	ND	ND	0.010	0.023	0.021	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0115	0.0275	0.0225	<0.01	<0.01	0.1195
			164	<0.01	ND	<0.01	0.012	0.026	0.025	ND	ND	
				0.014	ND	<0.01	0.025	0.064	0.050	ND	<0.01	
			Mean	<0.012	<0.01	<0.01	0.0185	0.045	0.0375	<0.01	<0.01	0.1687
			168	<0.01	ND	<0.01	0.01[A]	0.017	0.016	ND	ND	
				0.011	ND	<0.01	0.017	0.042	0.023	ND	ND	
			Mean	<0.0105	<0.01	<0.01	0.0135	0.0295	0.0195	<0.01	<0.01	0.1180
Woodland, CA, United States, 2015 Red Cored Chantenay	2 (14) banded in-furrow, BBCH09	1.13 1.12	98	0.14	ND	0.088	0.11	0.13	0.079	ND	<0.01	
				0.059	ND	0.038	0.058	0.089	0.074	ND	<0.01	
			Mean	0.0995	<0.01	0.063	0.084	0.1095	0.0765	<0.01	<0.01	0.4375
			131	0.013	ND	<0.01	0.015	0.029	0.025	ND	ND	
				0.012	ND	<0.01	0.018	0.035	0.027	ND	ND	
			Mean	0.0125	<0.01	<0.01	0.0165	0.032	0.026	<0.01	<0.01	0.1316
			135	0.011	ND	<0.01	0.014	0.027	0.026	ND	ND	
				<0.01	ND	<0.01	0.016	0.025	0.027	ND	ND	
			Mean	<0.0105	<0.01	<0.01	0.015	0.026	0.0265	<0.01	<0.01	0.1232
			140	<0.01	ND	<0.01	0.012	0.024	0.022	ND	ND	
				<0.01	ND	<0.01	0.014	0.027	0.019	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.013	0.0255	0.0205	<0.01	<0.01	0.1134
			145	<0.01	ND	<0.01	0.013	0.024	0.026	ND	ND	
				<0.01	ND	<0.01	0.014	0.031	0.025	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0135	0.0275	0.0255	<0.01	<0.01	0.1240
			150	<0.01	ND	<0.01	0.011	0.026	0.020	ND	ND	
				0.01[A]	ND	<0.01	0.015	0.028	0.028	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.013	0.027	0.024	<0.01	<0.01	0.1210
			154	<0.01	ND	<0.01	0.011	0.029	0.023	ND	ND	
				<0.01	ND	<0.01	0.011	0.028	0.019	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.011	0.0285	0.021	<0.01	<0.01	0.1187
Fresno, CA, United States, 2015 Laguna F1 (prepared for consumption)	1 banded soil directed at plant	2.22	85	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Fresno, CA,	1 banded soil	2.22	52	<0.01	ND	ND	<0.01	<0.01	<0.01	ND	ND	

Location, year, variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
United States, 2015 Laguna F1	directed at plant			ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			85	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			90	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			95	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			100	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			105	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			109	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Fresno, CA, United States, 2015 Laguna F1	2 (14) banded soil directed at plant, soil directed BBH12	1.10 1.10	71	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Fresno, CA, United States, 2015 Laguna F1	2 (14) banded soil directed at plant, soil directed BBH12	1.10 1.10	38	ND	ND	ND	<0.01	<0.01	<0.01	ND	ND	
				<0.01	ND	ND	<0.01	<0.01	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			71	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			76	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			81	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			86	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			91	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			95	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Porterville, CA, United States, 2015 Danvers	1 banded	2.29	55	<0.01	<0.01	0.011	0.015	0.17	0.61	ND	0.031	
				0.012	ND	0.016	0.021	0.25	0.72	ND	0.040	
			Mean	0.011	<0.01	0.0135	0.018	0.21	0.665	<0.01	0.0355	1.374
			122	<0.01	ND	ND	0.013	0.078	0.35	ND	0.041	
				<0.01	ND	ND	0.014	0.10	0.43	ND	0.057	
			Mean	<0.01	<0.01	<0.01	0.0135	0.089	0.39	<0.01	0.049	0.7679
			126	<0.01	ND	ND	0.017	0.058	0.30	ND	0.041	
				<0.01	ND	<0.01	0.018	0.084	0.41	ND	0.042	
			Mean	<0.01	<0.01	<0.01	0.0175	0.071	0.355	<0.01	0.0415	0.6877
			131	<0.01	ND	ND	0.016	0.068	0.25	ND	0.022	
				<0.01	ND	ND	0.017	0.060	0.092	ND	0.024	
			Mean	<0.01	<0.01	<0.01	0.0165	0.064	0.171	<0.01	0.023	0.3992
			136	<0.01	ND	ND	0.012	0.058	0.27	ND	0.027	
				<0.01	ND	ND	0.010	0.074	0.30	ND	0.031	

Location, year, variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	<0.01	<0.01	<0.01	0.011	0.066	0.285	<0.01	0.029	0.5744
			140	ND	ND	ND	<0.01	0.049	0.24	ND	0.031	
				ND	ND	ND	<0.01	0.057	0.054	ND	0.026	
			Mean	<0.01	<0.01	<0.01	<0.01	0.053	0.147	<0.01	0.0285	0.3462
			145	<0.01	ND	ND	0.013	0.037	0.15	ND	0.029	
				ND	ND	ND	0.011	0.039	0.20	ND	0.030	
			Mean	<0.01	<0.01	<0.01	0.012	0.038	0.175	<0.01	0.0295	0.3657
Porterville, CA, United States, 2015 Danvers	2 (14) banded, broadcast BBCH09	1.12	41	0.012	ND	0.024	0.028	0.44	1.5	ND	0.077	
		1.11	108	ND	ND	ND	<0.01	0.046	0.17	ND	0.020	
				<0.01	ND	<0.01	0.015	0.065	0.24	ND	0.022	
			Mean	<0.01	<0.01	<0.01	0.0125	0.0555	0.205	<0.01	0.021	0.4376
			112	ND	ND	ND	<0.01	0.054	0.16	ND	0.024	
				ND	ND	ND	<0.01	0.045	0.12	ND	0.023	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0495	0.14	<0.01	0.0235	0.3303
			117	ND	ND	ND	<0.01	0.049	0.15	ND	0.012	
				ND	ND	ND	<0.01	0.053	0.26	ND	0.018	
			Mean	<0.01	<0.01	<0.01	<0.01	0.051	0.205	<0.01	0.015	0.4308
			122	ND	ND	ND	<0.01	0.036	0.17	ND	0.016	
				ND	ND	ND	<0.01	0.036	0.12	ND	0.013	
			Mean	<0.01	<0.01	<0.01	<0.01	0.036	0.145	<0.01	0.0145	0.3174
			126	ND	ND	ND	0.010	0.047	0.17	ND	0.013	
				ND	ND	ND	<0.01	0.034	0.15	ND	0.010	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0405	0.16	<0.01	0.0115	0.3469
			131	ND	ND	ND	<0.01	0.051	0.12	ND	0.020	
				ND	ND	ND	<0.01	0.042	0.12	ND	0.016	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0465	0.12	<0.01	0.018	0.2956
Jerome, ID, United States, 2015 Danvers 126	1 banded in-furrow at plant	2.30	103	ND	ND	ND	<0.01	0.021	0.045	ND	<0.01	
				ND	ND	ND	<0.01	0.030	0.056	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0255	0.0505	<0.01	<0.01	0.1587
Jerome, ID, United States, 2015 Danvers 126	1 banded in-furrow at plant	2.30	69	0.025	ND	0.014	0.037	0.12	0.24	ND	0.025	
				0.022	ND	0.011	0.030	0.11	0.24	ND	0.025	
			Mean	0.0235	<0.01	0.0125	0.0335	0.115	0.24	<0.01	0.025	0.5862
			103	<0.01	ND	<0.01	0.012	0.042	0.094	ND	<0.01	
				<0.01	ND	<0.01	0.012	0.045	0.073	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.012	0.0435	0.0835	<0.01	<0.01	0.2359
			107	<0.01	ND	ND	0.012	0.039	0.068	ND	<0.01	
				<0.01	ND	<0.01	0.015	0.056	0.099	ND	0.012	
			Mean	<0.01	<0.01	<0.01	0.0135	0.0475	0.0835	<0.01	<0.011	0.2420
			113	<0.01	ND	<0.01	0.016	0.058	0.14	ND	<0.01	
				<0.01	ND	<0.01	0.014	0.041	0.072	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.015	0.0495	0.106	<0.01	<0.01	0.2790
			117	<0.01	ND	<0.01	0.010	0.031	0.073	ND	<0.01	
				<0.01	ND	ND	0.012	0.027	0.080	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.011	0.029	0.0765	<0.01	<0.01	0.2033
			121	<0.01	ND	<0.01	0.015	0.029	0.081	ND	0.013	
				<0.01	ND	<0.01	0.010	0.028	0.050	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.0125	0.0285	0.0655	<0.01	<0.0115	0.1859
			126	<0.01	ND	ND	0.013	0.037	0.081	ND	<0.01	
				<0.01	ND	ND	0.011	0.024	0.074	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.012	0.0305	0.0775	<0.01	<0.01	0.2071
Jerome, ID, United States, 2015 Danvers 126	2 (14) banded in-furrow at plant, banded in-furrow cotyledon	1.13	89	ND	ND	ND	<0.01	0.020	0.055	ND	<0.01	
		1.13		ND	ND	ND	<0.01	0.029	0.076	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.0245	0.0655	<0.01	<0.01	0.1798
Jerome, ID, United States, 2015 Danvers 126	2 (14) banded in-furrow at plant, banded in-furrow	1.13	55	0.017	ND	<0.01	0.033	0.14	0.27	ND	0.023	
		1.13		0.022	ND	0.011	0.036	0.11	0.31	ND	0.030	
			Mean	0.0195	<0.01	<0.0105	0.0345	0.125	0.29	<0.01	0.0265	0.6727
			89	<0.01	ND	<0.01	0.011	0.036	0.10	ND	<0.01	

Location, year, variety	N (interval) days	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
	cotyledon			<0.01	ND	<0.01	<0.01	0.030	0.076	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	0.033	0.088	<0.01	<0.01	0.2267
			93	<0.01	ND	ND	0.013	0.027	0.11	ND	<0.01	
				<0.01	ND	ND	0.011	0.029	0.089	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.012	0.028	0.0995	<0.01	<0.01	0.2365
			99	<0.01	ND	ND	0.013	0.040	0.065	ND	<0.01	
				<0.01	ND	ND	0.011	0.027	0.061	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.012	0.0335	0.063	<0.01	<0.01	0.1898
			103	<0.01	ND	ND	0.013	0.036	0.075	ND	<0.01	
				<0.01	ND	ND	0.017	0.034	0.088	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.015	0.035	0.0815	<0.01	<0.01	0.2200
			107	<0.01	ND	ND	<0.01	0.019	0.056	ND	<0.01	
				<0.01	ND	<0.01	0.014	0.029	0.077	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.012	0.024	0.0665	<0.01	<0.01	0.1806
			112	<0.01	ND	ND	0.012	0.025	0.051	ND	<0.01	
				<0.01	ND	<0.01	0.015	0.043	0.066	ND	0.01[A]	
			Mean	<0.01	<0.01	<0.01	0.0135	0.034	0.0585	<0.01	<0.01	0.1837

Notes:

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] SUM = 2.26×IN-A5760 + 2.11×IN-F4106 + 1.52×IN-QZY47 + 1.51×IN-TMQ01.

[C] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-QD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Potatoes

Shepard (2020 DuPont-43193 rev 1) conducted residue trials on potatoes at 22 sites in the Canada and the United States in 2014/2015 (Table 142).

At each location, separate plots were treated with fluazaindolizine SC formulation applied as a soil application (in-furrow spray) at planting at 2.24 kg ai/ha and at the other plot as a soil application (in-furrow spray) at planting at 2.24 kg ai/ha followed by a soil directed spray 14±1 days later at 1.12 kg ai/ha. Adjuvants were not used at most of trial sites, with the exception of Lyons, New York, second of two applications included non-ionic surfactant at 0.25 percent. The per-hectare application rates were concentrated in the furrow for the applications at plant at 18 of the 22 test sites and were concentrated in a band over the row for the second application at 11 of the 22 test sites.

The maximum interval of frozen storage before analysis was 481 days before extraction. Potato tubers were analysed for residues of fluazaindolizine and its metabolites IN-A5760, IN-F4016, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12 and IN-TQD54 using the analytical method DuPont-33861, rev. 3, with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg for each analyte. Acceptable concurrent recovery data were obtained for all matrices.

Table 142 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in potato from trials conducted in Canada and the United States

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
Nictaux East, NS, Canada, 2015 Superior	1 banded in-furrow 11 days after plant	2.26	53	0.026	<0.01	0.019	0.030	<0.01	0.014	ND	<0.01	
				0.030	<0.01	0.026	0.032	0.011	0.016	ND	<0.01	
			Mean	0.028	<0.01	0.023	0.031	<0.0105	0.015	<0.01	<0.01	0.1097
			58	0.018	<0.01	0.018	0.026	0.011	0.022	ND	<0.01	
				0.027	<0.01	0.021	0.038	0.013	0.026	ND	<0.01	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	0.0225	<0.01	0.0195	0.032	0.012	0.024	<0.01	<0.01	0.1182
			63	0.016	<0.01	0.015	0.026	0.010	0.025	ND	<0.01	
				0.013	<0.01	0.013	0.024	<0.01	0.018	ND	<0.01	
			Mean	0.0145	<0.01	0.014	0.025	<0.01	0.0215	<0.01	<0.01	0.0998
			68	0.024	<0.01	0.020	0.028	0.011	0.021	ND	<0.01	
				0.011	<0.01	0.016	0.024	0.010	0.019	ND	<0.01	
			Mean	0.0175	<0.01	0.018	0.026	0.0105	0.020	<0.01	<0.01	0.1067
			73	0.014	<0.01	0.017	0.023	0.012	0.024	ND	<0.01	
				0.015	<0.01	0.013	0.021	0.012	0.022	ND	<0.01	
			Mean	0.0145	<0.01	0.015	0.022	0.012	0.023	<0.01	<0.01	0.1072
			78	0.014	<0.01	0.015	0.023	0.01[A]	0.018	ND	<0.01	
				0.016	<0.01	0.018	0.026	0.011	0.020	<0.01	0.01[A]	
			Mean	0.015	<0.01	0.0165	0.0245	0.0105	0.019	<0.01	<0.01	0.1021
Nictaux East, NS, Canada, 2015 Superior	2 (14) banded in-furrow 11 days after plant, broadcast some emergence	2.24 1.14	39	0.043	<0.01	0.034	0.049	0.017	0.022	ND	<0.01	
				0.039	<0.01	0.031	0.044	0.013	0.017	ND	<0.01	
			Mean	0.041	<0.01	0.0325	0.0465	0.015	0.0195	<0.01	<0.01	0.1434
			44	0.025	<0.01	0.022	0.039	0.016	0.024	ND	0.010	
				0.032	<0.01	0.027	0.045	0.016	0.023	ND	0.010	
			Mean	0.0285	<0.01	0.0245	0.042	0.016	0.0235	<0.01	0.010	0.1341
			49	0.019	<0.01	0.021	0.031	0.012	0.022	ND	<0.01	
				0.020	<0.01	0.022	0.031	0.013	0.022	ND	0.01[A]	
			Mean	0.0195	<0.01	0.0215	0.031	0.0125	0.022	<0.01	<0.01	0.1202
			54	0.012	<0.01	0.018	0.029	0.01[A]	0.017	ND	<0.01	
				0.025	<0.01	0.027	0.039	0.01[A]	0.018	ND	<0.01	
			Mean	0.0185	<0.01	0.0225	0.034	0.01[A]	0.0175	<0.01	<0.01	0.1117
			59	0.013	<0.01	0.014	0.021	<0.01	0.015	ND	<0.01	
				0.015	<0.01	0.019	0.025	0.01[A]	0.016	ND	<0.01	
			Mean	0.014	<0.01	0.0165	0.023	<0.01	0.0155	<0.01	<0.01	0.0960
			64	0.011	<0.01	0.014	0.024	0.01[A]	0.021	ND	<0.01	
				0.014	<0.01	0.017	0.030	<0.01	0.016	ND	<0.01	
			Mean	0.0125	<0.01	0.0155	0.027	<0.01	0.0185	<0.01	<0.01	0.0984
Woodville, NS, Canada, 2015 Russet Burbank	1 banded in-furrow at plant	2.22	85	<0.01	<0.01	0.015	0.017	0.028	0.021	<0.01	<0.01	
				0.016	<0.01	0.028	0.031	0.036	0.037	<0.01	0.010	
			Mean	0.013	<0.01	0.0215	0.024	0.032	0.029	<0.01	<0.01	0.1604
			90	0.015	ND	0.022	0.032	0.045	0.049	ND	0.011	
				0.013	<0.01	0.021	0.033	0.046	0.046	0.012	0.01[A]	
			Mean	0.014	<0.01	0.0215	0.0325	0.0455	0.0475	<0.011	0.0105	0.2089
			96	<0.01	<0.01	0.022	0.023	0.025	0.026	<0.01	<0.01	
				<0.01	<0.01	0.023	0.025	0.033	0.032	<0.01	0.01[A]	
			Mean	<0.01	<0.01	0.0225	0.024	0.029	0.029	<0.01	<0.01	0.1579
			101	ND	<0.01	0.015	0.015	0.028	0.023	<0.01	<0.01	
				<0.01	<0.01	0.018	0.023	0.034	0.030	<0.01	<0.01	
			Mean	<0.01	<0.01	0.0165	0.019	0.031	0.0265	<0.01	<0.01	0.1446
			106	0.01[A]	<0.01	0.021	0.027	0.028	0.031	<0.01	0.01[A]	
				0.014	<0.01	0.026	0.038	0.031	0.036	<0.01	0.011	
			Mean	0.012	<0.01	0.0235	0.0325	0.0295	0.0335	<0.01	0.0105	0.1676
			111	<0.01	<0.01	0.011	0.015	0.022	0.019	<0.01	<0.01	
				<0.01	<0.01	0.020	0.027	0.032	0.033	<0.01	0.01[A]	
			Mean	<0.01	<0.01	0.0155	0.021	0.027	0.026	<0.01	<0.01	0.1356
Woodville, NS, Canada, 2015 Russet Burbank	2 (14) banded in-furrow at plant, broadcast	1.12 1.11	71	0.019	<0.01	0.025	0.033	0.025	0.022	<0.01	<0.01	
				0.023	<0.01	0.028	0.040	0.034	0.029	<0.01	<0.01	
			Mean	0.021	<0.01	0.0265	0.0365	0.0295	0.0255	<0.01	<0.01	0.1619
			76	ND	<0.01	<0.01	0.01[A]	0.016	0.012	<0.01	<0.01	
				<0.01	<0.01	0.012	0.018	0.023	0.022	<0.01	<0.01	
			Mean	<0.01	<0.01	<0.011	0.014	0.0195	0.017	<0.01	<0.01	0.1011
			82	<0.01	<0.01	0.015	0.015	0.012	<0.01	ND	<0.01	
				<0.01	<0.01	0.013	0.015	0.015	0.011	<0.01	<0.01	
			Mean	<0.01	<0.01	0.014	0.015	0.0135	<0.0105	<0.01	<0.01	0.0885
			87	<0.01	<0.01	0.017	0.022	0.019	0.016	<0.01	<0.01	
				0.01[A]	<0.01	0.016	0.026	0.019	0.015	<0.01	<0.01	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	<0.01	<0.01	0.0165	0.024	0.019	0.0155	<0.01	<0.01	0.1097
			92	<0.01	<0.01	0.011	0.013	0.013	0.01[A]	<0.01	<0.01	
				<0.01	<0.01	0.015	0.016	0.018	0.014	<0.01	<0.01	
			Mean	<0.01	<0.01	0.013	0.0145	0.0155	0.012	<0.01	<0.01	0.0917
			97	<0.01	<0.01	0.010	0.013	0.012	<0.01	ND	<0.01	
				<0.01	<0.01	0.010	0.013	0.011	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	0.010	0.013	0.0115	<0.01	<0.01	<0.01	0.0763
New Glasgow PEI, Canada, 2015 Yukon Gold	1 banded in-furrow BBCH00	2.28	70	0.061	<0.01	0.037	0.056	0.044	0.050	<0.01	0.028	
				0.021	ND	0.012	0.025	0.029	0.017	<0.01	0.019	
			Mean	0.041	<0.01	0.0245	0.0405	0.0365	0.0335	<0.01	0.0235	0.1804
			76	0.046	ND	0.027	0.048	0.038	0.034	<0.01	0.028	
				0.020	ND	<0.01	0.026	0.028	0.022	<0.01	0.022	
			Mean	0.033	<0.01	<0.0185	0.037	0.033	0.028	<0.01	0.025	0.1541
			80	0.030	ND	0.020	0.039	0.045	0.041	<0.01	0.032	
				0.017	ND	<0.01	0.029	0.034	0.026	<0.01	0.025	
			Mean	0.0235	<0.01	<0.015	0.034	0.0395	0.0335	<0.01	0.0285	0.1649
			85	<0.01	ND	<0.01	0.011	0.022	0.016	ND	0.017	
				<0.01	ND	<0.01	0.013	0.020	0.014	ND	0.015	
			Mean	<0.01	<0.01	<0.01	0.012	0.021	0.015	<0.01	0.016	0.0983
			89	<0.01	ND	<0.01	0.018	0.031	0.031	<0.01	0.027	
				0.013	ND	0.012	0.022	0.033	0.029	<0.01	0.027	
			Mean	<0.0115	<0.01	<0.011	0.020	0.032	0.030	<0.01	0.027	0.1398
			94	0.015	<0.01	0.015	0.022	0.034	0.021	<0.01	0.021	
				<0.01	ND	<0.01	0.013	0.034	0.020	<0.01	0.019	
			Mean	<0.0125	<0.01	<0.0125	0.0175	0.034	0.0205	<0.01	0.020	0.1316
New Glasgow PEI, Canada, 2015 Yukon Gold	2 (13) banded in-furrow BBCH00, banded over row BBCH00	1.18 1.11	57	0.016	<0.01	0.012	0.027	0.046	0.044	<0.01	0.032	
				0.021	<0.01	0.016	0.029	0.039	0.034	<0.01	0.029	
			Mean	0.0185	<0.01	0.014	0.028	0.0425	0.039	<0.01	0.0305	0.1756
			63	0.010	ND	<0.01	0.022	0.038	0.033	<0.01	0.029	
				0.012	ND	<0.01	0.023	0.040	0.033	<0.01	0.029	
			Mean	0.011	<0.01	<0.01	0.0225	0.039	0.033	<0.01	0.029	0.1528
			67	0.01[A]	ND	<0.01	0.024	0.038	0.030	<0.01	0.031	
				<0.01	ND	<0.01	0.020	0.033	0.022	<0.01	0.026	
			Mean	<0.01	<0.01	<0.01	0.022	0.0355	0.026	<0.01	0.0285	0.1369
			72	<0.01	<0.01	<0.01	0.019	0.031	0.027	<0.01	0.027	
				<0.01	ND	<0.01	0.014	0.028	0.022	<0.01	0.025	
			Mean	<0.01	<0.01	<0.01	0.0165	0.0295	0.0245	<0.01	0.026	0.1255
			76	0.014	<0.01	0.014	0.024	0.039	0.036	<0.01	0.031	
				<0.01	ND	<0.01	0.018	0.032	0.025	<0.01	0.027	
			Mean	<0.012	<0.01	<0.012	0.021	0.0355	0.0305	<0.01	0.029	0.1479
			81	<0.01	ND	<0.01	0.019	0.034	0.029	<0.01	0.026	
				<0.01	ND	<0.01	0.020	0.030	0.026	<0.01	0.023	
			Mean	<0.01	<0.01	<0.01	0.0195	0.032	0.0275	<0.01	0.0245	0.1339
New Glasgow PEI, Canada, 2015 Russet Burbank	1 in-furrow at plant	2.20	103	0.058	<0.01	0.040	0.050	0.029	0.031	<0.01	0.010	
				0.024	<0.01	0.022	0.026	0.024	0.027	<0.01	0.01[A]	
			Mean	0.041	<0.01	0.031	0.038	0.0265	0.029	<0.01	0.010	0.1721
			108	0.039	<0.01	0.030	0.041	0.039	0.040	<0.01	0.015	
				0.048	<0.01	0.036	0.046	0.033	0.030	<0.01	0.012	
			Mean	0.0435	<0.01	0.033	0.0435	0.036	0.035	<0.01	0.0135	0.1998
			112	0.079	<0.01	0.049	0.066	0.048	0.053	0.012	0.017	
				0.022	<0.01	0.020	0.026	0.034	0.039	<0.01	0.013	
			Mean	0.0505	<0.01	0.0345	0.046	0.041	0.046	<0.011	0.015	0.2272
			116	0.026	<0.01	0.021	0.027	0.020	0.022	<0.01	<0.01	
				0.043	<0.01	0.030	0.042	0.030	0.038	<0.01	0.011	
			Mean	0.0345	<0.01	0.0255	0.0345	0.025	0.030	<0.01	<0.0105[A]	0.1597
			122	0.066	<0.01	0.043	0.057	0.036	0.039	<0.01	0.014	
				0.037	<0.01	0.026	0.038	0.033	0.034	<0.01	0.012	
			Mean	0.0515	<0.01	0.0345	0.0475	0.0345	0.0365	<0.01	0.013	0.2030
			126	0.036	<0.01	0.020	0.035	0.021	0.019	<0.01	0.010	
				0.039	<0.01	0.024	0.037	0.029	0.029	<0.01	0.012	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	0.0375	<0.01	0.022	0.036	0.025	0.024	<0.01	0.011	0.1433
New Glasgow PEI, Canada, 2015 Russet Burbank	2 (13) in-furrow at plant, banded over row	1.10 1.13	90	0.020	<0.01	0.026	0.029	0.029	0.030	<0.01	0.014	
				0.031	<0.01	0.034	0.036	0.032	0.037	<0.01	0.015	
			Mean	0.0255	<0.01	0.030	0.0325	0.0305	0.0335	<0.01	0.0145	0.1828
			95	0.035	<0.01	0.031	0.039	0.031	0.030	<0.01	0.015	
				0.030	0.01[A]	0.028	0.037	0.035	0.037	<0.01	0.015	
			Mean	0.0325	<0.01	0.0295	0.038	0.033	0.0335	<0.01	0.015	0.1856
			99	0.024	<0.01	0.026	0.032	0.035	0.039	<0.01	0.015	
				0.026	0.010	0.030	0.036	0.037	0.043	0.010	0.017	
			Mean	0.025	<0.01[A]	0.028	0.034	0.036	0.041	<0.01[A]	0.016	0.1983
			103	0.028	<0.01	0.025	0.032	0.038	0.046	<0.01	0.015	
				0.027	<0.01	0.024	0.032	0.035	0.038	<0.01	0.017	
			Mean	0.0275	<0.01	0.0245	0.032	0.0365	0.042	<0.01	0.016	0.1932
			109	0.023	<0.01	0.020	0.031	0.033	0.039	<0.01	0.016	
				0.037	0.01[A]	0.032	0.042	0.042	0.047	0.01[A]	0.017	
			Mean	0.030	<0.01	0.026	0.0365	0.0375	0.043	0.01	0.0165	0.1994
			113	0.032	0.011	0.030	0.051	0.037	0.048	0.014	0.023	
				0.030	<0.01	0.024	0.044	0.028	0.032	0.01[A]	0.017	
			Mean	0.031	<0.0105	0.027	0.0475	0.0325	0.040	0.012	0.020	0.1905
Branchton, ON, Canada, 2015 Chieftain	1 banded in-furrow/no crop	2.15	88	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			92	ND	ND	ND	<0.01	<0.01	ND	ND	<0.01	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			97	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			102	ND	ND	<0.01	<0.01	<0.01	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			106	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			111	ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
				ND	ND	<0.01	<0.01	<0.01	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Branchton, ON, Canada, 2015 Chieftain	2 (15) banded in-furrow no crop, banded over row/pre-emergence	1.09 1.10	73	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				<0.01	ND	ND	0.01[A]	<0.01	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			77	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			82	ND	ND	ND	0.010	<0.01	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			87	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			91	ND	ND	ND	0.01[A]	<0.01	ND	ND	<0.01	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			96	ND	ND	ND	<0.01	<0.01	ND	ND	<0.01	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2015 Chieftain	1 soil banded BBCH00	2.23	79	<0.01	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
				<0.01	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-	1 soil banded	2.23	79	<0.01	ND	<0.01	0.012	<0.01	<0.01	ND	<0.01	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
Richelieu, QC, Canada, 2015 Chieftain	BBCH00			<0.01	ND	<0.01	0.01[A]	<0.01	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			84	0.012	ND	<0.01	0.014	<0.01	<0.01	ND	<0.01	
				<0.01	ND	<0.01	0.01[A]	<0.01	<0.01	ND	ND	
			Mean	<0.011	<0.01	<0.01	0.012	<0.01	<0.01	<0.01	<0.01	0.0740
			89	0.014	ND	0.011	0.017	<0.01	<0.01	ND	<0.01	
				0.01[A]	ND	<0.01	0.014	<0.01	<0.01	ND	<0.01	
			Mean	0.012	<0.01	<0.0105	0.0155	<0.01	<0.01	<0.01	<0.01	0.0751
			94	<0.01	ND	<0.01	0.011	<0.01	<0.01	<0.01	<0.01	
				0.012	<0.01	<0.01	0.017	<0.01	<0.01	<0.01	<0.01	
			Mean	<0.011	<0.01	<0.01	0.014	<0.01	<0.01	<0.01	<0.01	0.0740
			99	<0.01	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
				<0.01	ND	<0.01	0.01[A]	<0.01	<0.01	<0.01	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			105	ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
				<0.01	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2015 Chieftain	2 (13) soil banded BBCH00, soil banded BBCH01	1.16 1.12	66	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
St. Marc-sur-Richelieu, QC, Canada, 2015 Chieftain	2 (13) soil banded BBCH00, soil banded BBCH01	1.16 1.12	66	<0.01	ND	ND	0.01[A]	<0.01	ND	ND	ND	
				<0.01	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			71	ND	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			76	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			81	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			86	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			92	ND	ND	ND	<0.01	ND	<0.01	ND	<0.01	
				<0.01	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Saskatoon, SK, Canada, 2015 Russet Burbank	1 in-furrow (directed)	2.25	96	0.048	ND	0.026	0.035	<0.01	<0.01	ND	ND	
				0.045	ND	0.024	0.033	<0.01	<0.01	ND	ND	
			Mean	0.0465	<0.01	0.025	0.034	<0.01	<0.01	<0.01	<0.01	0.1057
			102	0.021	ND	0.012	0.017	ND	ND	ND	ND	
				0.034	ND	0.019	0.025	ND	ND	ND	ND	
			Mean	0.0275	<0.01	0.0155	0.021	<0.01	<0.01	<0.01	<0.01	0.0856
			106	0.020	ND	0.012	0.016	ND	ND	ND	ND	
				0.019	ND	0.011	0.015	ND	ND	ND	ND	
			Mean	0.0195	<0.01	0.0115	0.0155	<0.01	<0.01	<0.01	<0.01	0.0772
			111	0.065	ND	0.035	0.044	ND	<0.01	ND	ND	
				0.024	ND	0.013	0.018	ND	ND	ND	ND	
			Mean	0.0445	<0.01	0.024	0.031	<0.01	<0.01	<0.01	<0.01	0.1035
			116	0.030	ND	0.015	0.024	ND	ND	ND	ND	
				0.023	ND	0.012	0.018	ND	ND	ND	ND	
			Mean	0.0265	<0.01	0.0135	0.021	<0.01	<0.01	<0.01	<0.01	0.0814
			120	0.027	ND	0.014	0.020	ND	ND	ND	ND	
				0.046	ND	0.024	0.032	ND	ND	ND	ND	
			Mean	0.0365	<0.01	0.019	0.026	<0.01	<0.01	<0.01	<0.01	0.0930
Saskatoon, SK, Canada, 2015 Russet	2 (14) in-furrow directed, in-furrow directed	1.11 1.13	82	0.026	ND	0.013	0.019	ND	ND	ND	ND	
				0.025	ND	0.015	0.017	ND	ND	ND	ND	
			Mean	0.0255	<0.01	0.014	0.018	<0.01	<0.01	<0.01	<0.01	0.0824

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
Burbank			88	0.019	ND	0.011	0.015	ND	ND	ND	ND	
				0.020	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	0.0195	<0.01	0.0105	0.0125	<0.01	<0.01	<0.01	<0.01	0.0751
			92	0.017	ND	<0.01	0.013	ND	ND	ND	ND	
				0.022	ND	0.011	0.016	ND	ND	ND	ND	
			Mean	0.0195	<0.01	0.0105	0.0145	<0.01	<0.01	<0.01	<0.01	0.0751
			97	0.030	ND	0.015	0.020	ND	ND	ND	ND	
				0.029	ND	0.016	0.019	ND	ND	ND	ND	
			Mean	0.0295	<0.01	0.0155	0.0195	<0.01	<0.01	<0.01	<0.01	0.0856
			102	0.024	ND	0.012	0.016	ND	ND	ND	ND	
				0.022	ND	0.011	0.015	ND	ND	ND	ND	
			Mean	0.023	<0.01	0.0115	0.0155	<0.01	<0.01	<0.01	<0.01	0.0772
			106	0.023	ND	0.012	0.016	ND	ND	ND	ND	
				0.024	ND	0.015	0.016	ND	ND	ND	ND	
			Mean	0.0235	<0.01	0.0135	0.016	<0.01	<0.01	<0.01	<0.01	0.0814
Glenboro, MB, Canada, 2015 Norland	1 in-furrow	2.18	84	0.011	ND	0.01[A]	0.020	<0.01	0.017	ND	<0.01	
				0.016	<0.01	0.015	0.027	<0.01	0.022	ND	<0.01	
			Mean	0.0135	<0.01	0.0125	0.0235	<0.01	0.0195	<0.01	<0.01	0.0936
			89	0.010	ND	0.012	0.021	<0.01	0.012	ND	<0.01	
				0.050	ND	0.036	0.053	<0.01	0.019	ND	<0.01	
			Mean	0.030	<0.01	0.024	0.037	<0.01	0.0155	<0.01	<0.01	0.1118
			93	0.019	<0.01	0.019	0.036	<0.01	0.028	ND	0.011	
				<0.01	ND	<0.01	0.021	<0.01	0.017	ND	<0.01	
			Mean	<0.0145	<0.01	<0.0145	0.0285	<0.01	0.0225	<0.01	<0.0105	0.1024
			98	0.01[A]	ND	0.01[A]	0.019	<0.01	0.014	ND	<0.01	
				0.024	ND	0.024	0.038	<0.01	0.028	ND	0.01[A]	
			Mean	0.017	<0.01	0.017	0.0285	<0.01	0.021	<0.01	<0.01	0.1054
			102	0.015	ND	0.015	0.031	<0.01	0.018	ND	<0.01	
				<0.01	ND	<0.01	0.020	<0.01	0.014	ND	<0.01	
			Mean	<0.0125	<0.01	<0.0125	0.0255	<0.01	0.016	<0.01	<0.01	0.0883
			106	0.01[A]	ND	0.013	0.026	<0.01	0.021	ND	<0.01	
				<0.01	ND	0.011	0.024	<0.01	0.017	ND	<0.01	
			Mean	<0.01	<0.01	0.012	0.025	<0.01	0.019	<0.01	<0.01	0.0918
Glenboro, MB, Canada, 2015 Norland	2 (14) in-furrow, broadcast soil	1.10 1.12	70	0.01[A]	ND	0.011	0.021	<0.01	0.013	ND	<0.01	
				0.014	ND	0.015	0.025	<0.01	0.015	ND	<0.01	
			Mean	0.012	<0.01	0.013	0.023	<0.01	0.014	<0.01	<0.01	0.0864
			75	0.015	<0.01	0.015	0.030	<0.01	0.023	ND	<0.01	
				0.012	ND	0.017	0.026	<0.01	0.011	ND	<0.01	
			Mean	0.0135	<0.01	0.016	0.028	<0.01	0.017	<0.01	<0.01	0.0972
			79	<0.01	ND	<0.01	0.017	<0.01	0.015	ND	<0.01	
				<0.01	ND	<0.01	0.014	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.0155	<0.01	0.0125	<0.01	<0.01	0.0778
			84	<0.01	ND	<0.01	0.011	ND	<0.01	ND	<0.01	
				0.010	ND	0.011	0.022	<0.01	0.013	ND	<0.01	
			Mean	<0.01	<0.01	<0.0105	0.0165	<0.01	0.0115	<0.01	<0.01	0.0773
			88	0.021	<0.01	0.021	0.045	<0.01	0.035	ND	0.012	
				0.015	ND	0.014	0.024	<0.01	0.013	ND	<0.01	
			Mean	0.018	<0.01	0.0175	0.0345	<0.01	0.024	<0.01	0.011	0.1110
			92	0.015	<0.01	0.017	0.037	<0.01	0.028	ND	0.010	
				0.068	<0.01	0.049	0.080	0.012	0.034	ND	0.013	
			Mean	0.0415	<0.01	0.033	0.0585	<0.011	0.031	<0.01	0.0115	0.1558
Geneva, MN, United States, 2015 Cascade	1 in-furrow at plant	2.27	94	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
North Rose, NY, United States, 2015 Red Norland	1 banded at plant	2.28	85	0.013	<0.01	0.013	0.030	0.015	0.047	<0.01	0.013	
				<0.01	<0.01	<0.01	0.018	0.011	0.043	<0.01	0.01[A]	
			Mean	<0.0115	<0.01	<0.0115	0.024	0.013	0.045	<0.01	0.0115	0.1346
			90	<0.01	ND	<0.01	0.013	<0.01	0.035	ND	0.01[A]	
				<0.01	<0.01	<0.01	0.022	0.014	0.049	ND	0.012	
			Mean	<0.01	<0.01	<0.01	0.0175	<0.012	0.042	<0.01	0.011	0.1254

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			95	ND	ND	ND	0.014	<0.01	0.032	ND	<0.01	
				ND	ND	ND	0.01[A]	<0.01	0.027	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.012	<0.01	0.0295	<0.01	<0.01	0.1034
			100	0.016	ND	0.013	0.027	<0.01	0.031	ND	0.011	
				0.011	<0.01	0.013	0.029	0.015	0.065	<0.01	0.021	
			Mean	0.0135	<0.01	0.013	0.028	<0.0125	0.048	<0.01	0.016	0.1415
			105	<0.01	ND	<0.01	0.018	<0.01	0.037	<0.01	0.011	
				0.018	<0.01	0.014	0.039	0.014	0.067	<0.01	0.020	
			Mean	<0.014	<0.01	<0.012	0.0285	<0.012	0.052	<0.01	0.0155	0.1447
			110	<0.01	ND	<0.01	0.019	<0.01	0.038	<0.01	0.011	
				0.01A	<0.01	<0.01	0.027	<0.01	0.052	<0.01	0.016	
			Mean	<0.01	<0.01	<0.01	0.023	<0.01	0.045	<0.01	0.0135	0.1269
North Rose, NY, United States, 2015 Red Norland	2 (14) banded at plant, banded/cracking through soil	1.14 1.15	71	ND	<0.01	<0.01	0.029	0.016	0.062	<0.01	0.014	
				<0.01	<0.01	0.01[A]	0.038	0.020	0.098	<0.01	0.022	
			Mean	<0.01	<0.01	<0.01	0.0335	0.018	0.080	<0.01	0.018	0.1919
			76	<0.01	<0.01	<0.01	0.041	0.026	0.13	<0.01	0.029	
				<0.01	<0.01	<0.01	0.034	0.021	0.099	<0.01	0.021	
			Mean	<0.01	<0.01	<0.01	0.0375	0.0235	0.1145	<0.01	0.025	0.2523
			81	<0.01	<0.01	0.013	0.050	0.028	0.13	<0.01	0.031	
				0.014	<0.01	0.019	0.060	0.027	0.12	<0.01	0.030	
			Mean	<0.0115	<0.01	0.016	0.055	0.0275	0.125	<0.01	0.0305	0.2869
			86	<0.01	<0.01	0.017	0.062	0.029	0.15	<0.01	0.037	
				<0.01	<0.01	0.012	0.054	0.025	0.15	<0.01	0.035	
			Mean	<0.01	<0.01	0.0145	0.058	0.027	0.15	<0.01	0.036	0.3207
			91	0.013	<0.01	0.028	0.087	0.051	0.22	0.013	0.053	
				<0.01	<0.01	0.014	0.057	0.025	0.16	<0.01	0.035	
			Mean	<0.0115	<0.01	0.021	0.072	0.038	0.19	0.0115	0.044	0.4116
			96	<0.01	<0.01	0.020	0.070	0.044	0.19	0.010	0.041	
				<0.01	<0.01	<0.01	0.039	0.016	0.10	<0.01	0.023	
			Mean	<0.01	<0.01	0.015	0.0545	0.030	0.145	<0.01	0.032	0.3188
Alton, NY, United States, 2015 Reba	1 banded in-furrow at plant	2.31	86	ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			92	ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			96	ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			101	ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			106	ND	ND	<0.01	<0.01	<0.01	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	<0.01	0.010	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			112	ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
				ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Alton, NY, United States, 2015 Reba	2 (15) banded in-furrow at plant, broadcast BBCH00	1.11 1.12	71	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			77	ND	ND	ND	ND	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			81	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			86	<0.01	ND	<0.01	0.013	ND	ND	ND	ND	
				ND	ND	ND	<0.01	ND	ND	ND	ND	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.01	<0.01	<0.01	0.0740
			91	ND	ND	<0.01	<0.01	ND	ND	ND	ND	
				ND	ND	<0.01	<0.01	<0.01	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			97	ND	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Germansville, PA, United States, 2015 Dark Red Norland	1 banded in-furrow at plant	2.25	76	0.016	<0.01	0.012	0.035	0.021	0.094	<0.01	0.017	
				0.017	<0.01	0.016	0.034	0.023	0.11	<0.01	0.020	
			Mean	0.0165	<0.01	0.014	0.0345	0.022	0.102	<0.01	0.0185	0.2396
			80	0.01[A]	<0.01	<0.01	0.024	0.014	0.081	<0.01	0.014	
				0.010	<0.01	<0.01	0.028	0.016	0.088	<0.01	0.017	
			Mean	0.01[A]	<0.01	<0.01	0.026	0.015	0.0845	<0.01	0.0155	0.1941
			85	<0.01	<0.01	<0.01	0.030	0.014	0.091	<0.01	0.017	
				0.013	<0.01	0.010	0.036	0.017	0.11	<0.01	0.021	
			Mean	<0.0115[A]	<0.01	<0.01	0.033	0.0155	0.1005	<0.01	0.019	0.2190
			90	<0.01	<0.01	<0.01	0.033	0.017	0.12	<0.01	0.022	
				<0.01	<0.01	<0.01	0.039	0.018	0.12	<0.01	0.024	
			Mean	<0.01	<0.01	<0.01	0.036	0.0175	0.12	<0.01	0.023	0.2515
			94	0.012	<0.01	0.01[A]	0.042	0.019	0.13	<0.01	0.024	
				<0.01	<0.01	<0.01	0.036	0.014	0.10	<0.01	0.023	
			Mean	<0.011	<0.01	<0.01	0.039	0.0165	0.115	<0.01	0.0235	0.2424
			99	<0.01	<0.01	<0.01	0.040	0.012	0.11	<0.01	0.022	
				<0.01	<0.01	<0.01	0.032	0.012	0.093	<0.01	0.020	
			Mean	<0.01	<0.01	<0.01	0.036	0.012	0.1015	<0.01	0.021	0.2152
Germansville, PA, United States, 2015 Dark Red Norland	2 (13) banded in-furrow at plant, banded over row/pre-emergence	1.15 1.14	63	<0.01	<0.01	<0.01	0.034	0.017	0.078	<0.01	0.015	
				<0.01	<0.01	<0.01	0.024	0.014	0.061	<0.01	0.013	
			Mean	<0.01	<0.01	<0.01	0.029	0.0155	0.0695	<0.01	0.014	0.1722
			67	0.010	<0.01	<0.01	0.035	0.014	0.086	<0.01	0.018	
				<0.01	<0.01	<0.01	0.028	0.014	0.069	<0.01	0.015	
			Mean	<0.01	<0.01	<0.01	0.0315	0.014	0.0775	<0.01	0.0165	0.1820
			72	<0.01	<0.01	<0.01	0.033	0.013	0.093	<0.01	0.019	
				<0.01	<0.01	<0.01	0.036	0.014	0.094	<0.01	0.020	
			Mean	<0.01	<0.01	<0.01	0.0345	0.0135	0.0935	<0.01	0.0195	0.2054
			77	<0.01	<0.01	ND	0.032	0.01[A]	0.076	<0.01	0.018	
				<0.01	<0.01	<0.01	0.038	0.011	0.070	<0.01	0.017	
			Mean	<0.01	<0.01	<0.01	0.035	0.0105	0.073	<0.01	0.0175	0.1699
			81	<0.01	<0.01	ND	0.036	0.013	0.093	<0.01	0.021	
				<0.01	<0.01	<0.01	0.035	0.010	0.069	<0.01	0.016	
			Mean	<0.01	<0.01	<0.01	0.0355	0.0115	0.081	<0.01	0.0185	0.1835
			86	<0.01	<0.01	<0.01	0.040	0.011	0.094	<0.01	0.022	
				<0.01	<0.01	<0.01	0.048	0.011	0.095	<0.01	0.025	
			Mean	<0.01	<0.01	<0.01	0.044	0.011	0.0945	<0.01	0.0235	0.2031
Frenchtown, NJ, United States, 2015 Waneta	banded in-furrow at plant	2.29	71	0.016	0.014	0.020	0.040	0.022	0.15	<0.01	0.028	
				0.017	0.016	0.023	0.049	0.026	0.16	<0.01	0.034	
			Mean	0.0165	0.015	0.0215	0.0445	0.024	0.155	<0.01	0.031	0.3498
			77	0.010	0.013	0.014	0.033	0.017	0.14	<0.01	0.026	
				0.010	0.012	0.015	0.033	0.018	0.13	ND	0.025	
			Mean	0.010	0.0125	0.0145	0.033	0.0175	0.135	<0.01	0.0255	0.2893
			81	0.013	0.011	0.021	0.035	0.016	0.13	<0.01	0.024	
				0.012	0.014	0.025	0.037	0.020	0.15	<0.01	0.028	
			Mean	0.0125	0.0125	0.023	0.036	0.018	0.14	<0.01	0.026	0.3155
			86	0.014	0.015	0.030	0.045	0.022	0.16	<0.01	0.031	
				0.012	0.011	0.026	0.039	0.019	0.14	<0.01	0.027	
			Mean	0.013	0.013	0.028	0.042	0.0205	0.15	<0.01	0.029	0.3461
			91	0.014	0.015	0.024	0.044	0.025	0.18	<0.01	0.033	
				<0.01	0.014	0.020	0.039	0.021	0.16	<0.01	0.028	
			Mean	<0.012	0.0145	0.022	0.0415	0.023	0.17	<0.01	0.0305	0.3709
			96	<0.01	0.011	0.015	0.032	0.016	0.12	<0.01	0.025	
				0.011	0.011	0.018	0.038	0.018	0.15	<0.01	0.027	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	<0.0105	0.011	0.0165	0.035	0.017	0.135	<0.01	0.026	0.2894
Frenchtown, NJ, United States, 2015 Waneta	2 (14) banded in-furrow at plant, banded over row/pre-emergence	1.15 1.15	57	<0.01	0.01[A]	0.01[A]	0.021	0.010	0.078	ND	0.018	
				<0.01	<0.01	0.011	0.022	0.011	0.078	ND	0.016	
			Mean	<0.01	<0.01	0.0105	0.0215	0.0105	0.078	<0.01	0.017	0.1785
			63	<0.01	<0.01	<0.01	0.017	<0.01	0.067	ND	0.016	
				<0.01	<0.01	0.01[A]	0.021	0.010	0.087	ND	0.020	
			Mean	<0.01	<0.01	<0.01	0.019	0.01	0.077	<0.01	0.018	0.1752
			67	<0.01	<0.01	0.012	0.021	<0.01	0.068	ND	0.016	
				<0.01	<0.01	0.012	0.021	<0.01	0.065	ND	0.016	
			Mean	<0.01	<0.01	0.012	0.021	<0.01	0.0665	<0.01	0.016	0.1635
			72	<0.01	0.010	0.016	0.029	0.013	0.090	<0.01	0.020	
				<0.01	<0.01	0.014	0.028	0.013	0.10	<0.01	0.021	
			Mean	<0.01	<0.01[A]	0.015	0.0285	0.013	0.095	<0.01	0.0205	0.2175
			77	ND	<0.01	<0.01	0.020	0.011	0.076	<0.01	0.019	
				ND	<0.01	<0.01	0.023	0.011	0.093	<0.01	0.021	
			Mean	<0.01	<0.01	<0.01	0.0215	0.011	0.0845	<0.01	0.020	0.1880
			82	ND	<0.01	<0.01	0.019	0.011	0.083	ND	0.019	
				ND	<0.01	<0.01	0.022	0.011	0.086	ND	0.020	
			Mean	<0.01	<0.01	<0.01	0.0205	0.011	0.0845	<0.01	0.0195	0.1880
Oviedo, FL, United States, 2015 Red La Soda	1 banded spray at plant	2.23	100	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			105	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			110	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	0.01[A]	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			115	<0.01	ND	<0.01	0.012	ND	ND	ND	<0.01	
				<0.01	ND	<0.01	0.013	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.0125	<0.01	<0.01	<0.01	<0.01	0.0740
			121	<0.01	ND	<0.01	0.011	ND	ND	ND	<0.01	
				<0.01	ND	<0.01	0.011	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			127	<0.01	ND	ND	0.011	ND	ND	ND	<0.01	
				<0.01	ND	<0.01	0.010	ND	ND	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.0105	<0.01	<0.01	<0.01	<0.01	0.0740
Oviedo, FL, United States, 2015 Red La Soda	2 (14) banded spray at plant, directed spray over row BBCH07	1.12 1.12	86	ND	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			91	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			96	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			101	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			107	<0.01	ND	ND	<0.01	ND	ND	<0.01	ND	
				<0.01	ND	ND	<0.01	ND	ND	<0.01	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			113	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Geneva, MN, United States, 2015 Cascade	1 in-furrow at plant	2.27	94	0.014	ND	<0.01	0.010	ND	ND	ND	<0.01	
				0.011	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	0.0125	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			99	0.027	ND	0.021	0.021	<0.01	ND	ND	<0.01	
				0.034	ND	0.025	0.026	ND	ND	ND	<0.01	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
			Mean	0.0305	<0.01	0.023	0.0235	<0.01	<0.01	<0.01	<0.01	0.1014
			104	0.015	ND	0.013	0.012	ND	ND	ND	<0.01	
				0.018	ND	0.015	0.014	ND	ND	ND	ND	
			Mean	0.0165	<0.01	0.014	0.013	<0.01	<0.01	<0.01	<0.01	0.0824
			109	0.019	ND	0.013	0.018	ND	ND	ND	<0.01	
				0.013	ND	0.01[A]	0.013	ND	ND	ND	<0.01	
			Mean	0.016	<0.01	0.0115	0.0155	<0.01	<0.01	<0.01	<0.01	0.0772
			114	0.012	ND	0.010	0.013	<0.01	<0.01	ND	<0.01	
				0.018	ND	0.012	0.017	<0.01	<0.01	ND	<0.01	
			Mean	0.015	<0.01	0.011	0.015	<0.01	<0.01	<0.01	<0.01	0.0761
			119	0.017	ND	0.014	0.018	<0.01	<0.01	ND	<0.01	
				0.022	ND	0.017	0.019	<0.01	<0.01	ND	<0.01	
			Mean	0.0195	<0.01	0.0155	0.0185	<0.01	<0.01	<0.01	<0.01	0.0856
Geneva, MN, United States, 2015 Cascade	2 (14) in-furrow at plant, banded soil-directed 25 percent cracking	1.13	80	ND	ND	ND	ND	ND	ND	ND	<0.01	
		1.13		ND	ND	ND	ND	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Geneva, MN, United States, 2015 Cascade	2 (14) in-furrow at plant, banded soil-directed 25 percent cracking	1.13	80	0.013	ND	<0.01	0.01[A]	ND	ND	ND	ND	
		1.13		0.014	ND	<0.01	0.01[A]	ND	ND	ND	ND	
			Mean	0.0135	<0.01	<0.01	0.01[A]	<0.01	<0.01	<0.01	<0.01	0.0740
			85	0.021	ND	0.014	0.014	ND	ND	ND	ND	
				0.023	ND	0.012	0.015	ND	ND	ND	ND	
			Mean	0.022	<0.01	0.013	0.0145	<0.01	<0.01	<0.01	<0.01	0.0803
			90	0.023	ND	0.015	0.020	ND	ND	ND	<0.01	
				0.020	ND	0.014	0.018	ND	ND	ND	ND	
			Mean	0.0215	<0.01	0.0145	0.019	<0.01	<0.01	<0.01	<0.01	0.0835
			95	0.021	ND	0.013	0.020	ND	ND	ND	<0.01	
				0.014	ND	0.010	0.014	ND	ND	ND	<0.01	
			Mean	0.0175	<0.01	0.0115	0.017	<0.01	<0.01	<0.01	<0.01	0.0772
			100	0.014	ND	0.01[A]	0.011	ND	ND	ND	<0.01	
				0.023	ND	0.015	0.020	ND	ND	ND	<0.01	
			Mean	0.0185	<0.01	0.0125	0.0155	<0.01	<0.01	<0.01	<0.01	0.0793
			105	0.013	ND	<0.01	0.011	<0.01	ND	ND	<0.01	
				0.012	ND	<0.01	0.010	<0.01	ND	ND	<0.01	
			Mean	0.0125	<0.01	<0.01	0.0105	<0.01	<0.01	<0.01	<0.01	0.0740
Carlyle, IL, United States, 2015 Kennebec	1 in-furrow at plant	2.27	86	0.054	0.017	0.049	0.17	0.078	0.36	0.017	0.083	
				0.033	0.012	0.034	0.12	0.066	0.29	0.015	0.072	
			Mean	0.0435	0.0145	0.0415	0.145	0.072	0.335	0.016	0.0775	0.7356
			91	<0.01	0.013	0.020	0.12	0.045	0.25	0.011	0.072	
				<0.01	0.011	0.018	0.13	0.053	0.32	0.014	0.080	
			Mean	<0.01	0.012	0.019	0.125	0.049	0.285	0.0125	0.076	0.5720
			96	<0.01	<0.01	0.018	0.15	0.047	0.26	0.012	0.065	
				<0.01	0.016	0.019	0.16	0.069	0.37	0.018	0.088	
			Mean	<0.01	<0.013	0.0185	0.155	0.058	0.315	0.015	0.0765	0.6322
			100	<0.01	0.011	0.013	0.14	0.048	0.29	0.012	0.078	
				<0.01	0.016	0.018	0.17	0.067	0.38	0.015	0.095	
			Mean	<0.01	0.0135	0.0155	0.155	0.0575	0.335	0.0135	0.0865	0.6565
Carlyle, IL, United States, 2015 Kennebec	2 (14) in-furrow at plant, broadcast	1.13	72	0.011	0.010	0.015	0.099	0.033	0.17	<0.01	0.067	
		1.12		<0.01	0.011	0.014	0.090	0.032	0.17	<0.01	0.057	
			Mean	<0.0105	0.0105	0.0145	0.0945	0.0325	0.17	<0.01	0.062	0.3604
			77	ND	<0.01	<0.01	0.081	0.022	0.17	<0.01	0.046	
				ND	<0.01	0.011	0.068	0.024	0.16	<0.01	0.050	
			Mean	<0.01	<0.01	<0.0105	0.0745	0.023	0.165	<0.01	0.048	0.3289
			82	ND	0.011	<0.01	0.093	0.024	0.17	<0.01	0.053	
				ND	<0.01	0.010	0.086	0.029	0.17	<0.01	0.058	
			Mean	<0.01	<0.0105	<0.01	0.0895	0.0265	0.17	<0.01	0.0555	0.3418
			86	<0.01	0.011	0.010	0.099	0.032	0.19	<0.01	0.067	
				<0.01	0.011	<0.01	0.11	0.030	0.23	<0.01	0.067	
			Mean	<0.01	0.011	<0.01	0.1045	0.031	0.21	<0.01	0.067	0.4102
Jerome, ID,	1 in-furrow at	2.26	143	0.058	ND	0.027	0.042	0.015	<0.01	<0.01	<0.01	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
United States, 2015 Ranger Russet	plant			0.039	ND	0.017	0.030	0.010	<0.01	ND	<0.01	
			Mean	0.0485	<0.01	0.022	0.036	0.0125	<0.01	<0.01	<0.01	0.1031
Jerome, ID, United States, 2015 Ranger Russet	1 in-furrow at plant	2.26	143	0.12	ND	0.060	0.079	0.015	<0.01	ND	<0.01	
				0.088	ND	0.044	0.064	0.018	<0.01	<0.01	<0.01	
			Mean	0.104	<0.01	0.052	0.0715	0.0165	<0.01	<0.01	<0.01	0.1725
			147	0.040	ND	0.021	0.029	0.011	<0.01	ND	<0.01	
				0.10	ND	0.056	0.070	0.017	<0.01	<0.01	<0.01	
			Mean	0.07	<0.01	0.0385	0.0495	0.014	<0.01	<0.01	<0.01	0.1402
			152	0.095	<0.01	0.055	0.065	0.018	<0.01	<0.01	<0.01	
				0.082	ND	0.041	0.059	0.017	0.01[A]	<0.01	<0.01	
			Mean	0.0885	<0.01	0.048	0.062	0.0175	<0.01	<0.01	<0.01	0.1656
			157	0.090	ND	0.051	0.058	0.013	<0.01	<0.01	<0.01	
				0.040	ND	0.025	0.032	<0.01	ND	ND	<0.01	
			Mean	0.065	<0.01	0.038	0.045	<0.0115	<0.01	<0.01	<0.01	0.1354
			161	0.11	ND	0.073	0.079	0.024	0.011	<0.01	<0.01	
				0.036	ND	0.022	0.028	<0.01	ND	ND	<0.01	
			Mean	0.073	<0.01	0.0475	0.0535	<0.017	<0.0105	<0.01	<0.01	0.1645
			166	0.13	ND	0.074	0.086	0.014	<0.01	ND	<0.01	
				0.19	<0.01	0.13	0.13	0.026	0.013	<0.01	<0.01	
			Mean	0.16	<0.01	0.102	0.108	0.020	0.0115	<0.01	<0.01	0.2856
Jerome, ID, United States, 2015 Ranger Russet	2 (14) in-furrow at plant, soil-directed spray/pre-emergence	1.16 1.14	129	0.034	ND	0.016	0.026	<0.01	<0.01	ND	<0.01	
				0.023	ND	0.011	0.017	<0.01	<0.01	ND	<0.01	
			Mean	0.0285	<0.01	0.0135	0.0215	<0.01	<0.01	<0.01	<0.01	0.0814
Jerome, ID, United States, 2015 Ranger Russet	2 (14) in-furrow at plant, soil-directed spray/pre-emergence	1.16 1.14	129	0.046	ND	0.023	0.034	<0.01	ND	ND	<0.01	
				0.032	ND	0.018	0.025	<0.01	ND	ND	ND	
			Mean	0.039	<0.01	0.0205	0.0295	<0.01	<0.01	<0.01	<0.01	0.0962
			133	0.025	ND	0.012	0.022	<0.01	ND	ND	ND	
				0.055	ND	0.028	0.044	0.01[A]	<0.01	ND	<0.01	
			Mean	0.040	<0.01	0.020	0.033	<0.01	<0.01	<0.01	<0.01	0.0951
			138	0.060	ND	0.030	0.044	<0.01	<0.01	ND	<0.01	
				0.042	ND	0.020	0.030	<0.01	ND	ND	ND	
			Mean	0.051	<0.01	0.025	0.037	<0.01	<0.01	<0.01	<0.01	0.1057
			143	0.028	ND	0.014	0.025	<0.01	ND	ND	<0.01	
				0.078	ND	0.043	0.059	0.011	<0.01	<0.01	<0.01	
			Mean	0.053	<0.01	0.0285	0.042	<0.0105	<0.01	<0.01	<0.01	0.1138
			147	0.044	ND	0.028	0.034	0.010	<0.01	<0.01	<0.01	
				0.043	ND	0.023	0.033	<0.01	ND	ND	<0.01	
			Mean	0.0435	<0.01	0.0255	0.0335	<0.01	<0.01	<0.01	<0.01	0.1067
			152	0.063	ND	0.032	0.044	0.01[A]	<0.01	ND	<0.01	
				0.051	ND	0.032	0.039	<0.01	<0.01	ND	<0.01	
			Mean	0.057	<0.01	0.032	0.0415	<0.01	<0.01	<0.01	<0.01	0.1204
Hughson, CA, United States, 2015 Yukon Gold	1 in-furrow at plant	2.23	95	<0.01	ND	ND	0.030	ND	<0.01	ND	<0.01	
				<0.01	ND	ND	0.026	ND	0.011	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.028	<0.01	<0.0105	<0.01	<0.01	0.0748
			100	<0.01	ND	ND	0.040	<0.01	0.010	ND	0.010	
				<0.01	ND	ND	0.029	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	0.0345	<0.01	<0.01[A]	<0.01	<0.01	0.0740
			105	<0.01	ND	<0.01	0.036	ND	0.015	ND	0.013	
				<0.01	ND	ND	0.034	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.035	<0.01	<0.0125	<0.01	<0.0115	0.0778
			110	ND	ND	ND	0.038	ND	0.01[A]	ND	<0.01	
				ND	ND	ND	0.036	ND	<0.01	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.037	<0.01	<0.01	<0.01	<0.01	0.0740
			116	ND	ND	ND	0.055	ND	0.013	ND	0.014	
				ND	ND	ND	0.034	ND	0.011	ND	0.010	
			Mean	<0.01	<0.01	<0.01	0.0445	<0.01	0.012	<0.01	0.012	0.0770
			120	<0.01	ND	<0.01	0.029	ND	<0.01	ND	<0.01	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
				<0.01	ND	<0.01	0.029	ND	0.01[A]	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.029	<0.01	<0.01	<0.01	<0.01	0.0740
Hughson, CA, United States, 2015 Yukon Gold	2 (14) in-furrow at plant, broadcast crop not emerged	1.11 1.13	81	<0.01	ND	<0.01	0.14	<0.01	0.022	ND	0.023	
				0.01[A]	ND	<0.01	0.19	<0.01	0.034	ND	0.027	
			Mean	<0.01	<0.01	<0.01	0.165	<0.01	0.028	<0.01	0.025	0.1012
			86	ND	ND	ND	0.18	<0.01	0.027	ND	0.025	
				<0.01	ND	<0.01	0.15	<0.01	0.028	ND	0.024	
			Mean	<0.01	<0.01	<0.01	0.165	<0.01	0.0275	<0.01	0.0245	0.1004
			91	<0.01	ND	<0.01	0.17	<0.01	0.029	ND	0.026	
				ND	ND	<0.01	0.21	<0.01	0.031	ND	0.026	
			Mean	<0.01	<0.01	<0.01	0.19	<0.01	0.030	<0.01	0.026	0.1042
			96	<0.01	<0.01	<0.01	0.19	<0.01	0.033	ND	0.028	
				ND	<0.01	ND	0.16	<0.01	0.026	ND	0.024	
			Mean	<0.01	<0.01	<0.01	0.175	<0.01	0.0295	<0.01	0.026	0.1034
			102	<0.01	ND	ND	0.21	<0.01	0.039	ND	0.035	
				<0.01	<0.01	<0.01	0.15	<0.01	0.030	ND	0.025	
			Mean	<0.01	<0.01	<0.01	0.18	<0.01	0.0345	<0.01	0.030	0.1110
			106	<0.01	<0.01	<0.01	0.17	<0.01	0.043	ND	0.034	
				<0.01	<0.01	<0.01	0.17	<0.01	0.042	ND	0.034	
			Mean	<0.01	<0.01	<0.01	0.17	<0.01	0.0425	<0.01	0.034	0.1231
Tulelake, CA, United States, 2015 Russet Norkotah	1 banded in-furrow/ seed in-furrow	2.23	92	0.065	ND	0.032	0.046	ND	ND	ND	ND	
				0.071	ND	ND	ND	ND	ND	ND	ND	
			Mean	0.068	<0.01	<0.021	<0.028	<0.01	<0.01	<0.01	<0.01	0.0972
			98	0.053	ND	0.024	0.034	ND	ND	ND	ND	
				0.020	ND	<0.01	0.014	ND	ND	ND	ND	
			Mean	0.0365	<0.01	<0.017	0.024	<0.01	<0.01	<0.01	<0.01	0.0888
			102	0.037	ND	0.015	0.024	ND	ND	ND	ND	
				0.012	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	0.0245	<0.01	<0.0125	<0.017	<0.01	<0.01	<0.01	<0.01	0.0793
			106	0.039	ND	0.020	0.030	ND	ND	ND	ND	
				0.022	ND	0.012	0.017	ND	ND	ND	ND	
			Mean	0.0305	<0.01	0.016	0.0235	<0.01	<0.01	<0.01	<0.01	0.0867
			112	0.048	ND	0.023	0.038	ND	ND	ND	ND	
				0.037	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	0.0425	<0.01	<0.0165	0.024	<0.01	<0.01	<0.01	<0.01	0.0877
			116	0.042	ND	<0.01	<0.01	ND	ND	ND	ND	
				0.044	<0.01	0.021	0.036	ND	ND	ND	ND	
			Mean	0.043	<0.01	<0.0155	<0.023	<0.01	<0.01	<0.01	<0.01	0.0856
Tulelake, CA, United States, 2015 Russet Norkotah	2 (14) banded in-furrow/ seed in-furrow, banded over seed with seed not emerged	1.10 1.11	78	0.016	ND	0.032	0.044	ND	ND	ND	ND	
				0.021	ND	0.011	0.015	ND	ND	ND	ND	
			Mean	0.0185	<0.01	0.0215	0.0295	<0.01	<0.01	<0.01	<0.01	0.0983
			84	0.012	ND	<0.01	<0.01	ND	ND	ND	ND	
				0.019	ND	<0.01	0.013	ND	ND	ND	ND	
			Mean	0.0155	<0.01	<0.01	0.0115	<0.01	<0.01	<0.01	<0.01	0.0740
			88	0.012	ND	<0.01	<0.01	ND	ND	ND	ND	
				0.027	ND	0.011	0.018	ND	ND	ND	ND	
			Mean	0.0195	<0.01	<0.0105	0.014	<0.01	<0.01	<0.01	<0.01	0.0751
			92	0.026	ND	0.014	0.020	ND	ND	ND	ND	
				0.016	ND	<0.01	0.012	ND	ND	ND	ND	
			Mean	0.021	<0.01	<0.012	0.016	<0.01	<0.01	<0.01	<0.01	0.0782
			98	0.024	ND	<0.01	0.017	ND	ND	ND	ND	
				0.026	ND	0.013	0.019	ND	ND	ND	ND	
			Mean	0.025	<0.01	0.0115	0.018	<0.01	<0.01	<0.01	<0.01	0.0772
			102	0.018	ND	0.01[A]	0.013	ND	ND	ND	ND	
				0.015	ND	<0.01	0.012	ND	ND	ND	ND	
			Mean	0.0165	<0.01	<0.01	0.0125	<0.01	<0.01	<0.01	<0.01	0.0740
Ephrata, WA, United States, 2015 Umatilla	1 banded in-furrow at plant	2.24	124	<0.01	ND	ND	ND	<0.01	ND	ND	ND	
				<0.01	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Ephrata, WA,	1 banded in-	2.24	124	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
United States, 2015 Umatilla	furrow at plant			ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			129	0.022	ND	0.011	0.017	0.012	<0.01	ND	ND	
				<0.01	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	0.016	<0.01	<0.0105	<0.0135	0.011	<0.01	<0.01	<0.01	0.0766
			134	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			139	<0.01	ND	<0.01	<0.01	0.01[A]	ND	ND	ND	
				<0.01	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			144	ND	ND	ND	ND	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			150	<0.01	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Ephrata, WA, United States, 2015 Umatilla	2 (14) banded in-furrow at plant, broadcast	1.12 1.12	110	ND	ND	ND	ND	<0.01	ND	ND	ND	
				0.016	<0.01	<0.01	0.013	0.01[A]	ND	ND	ND	
			Mean	<0.013	<0.01	<0.01	<0.0115	<0.01	<0.01	<0.01	<0.01	0.0740
Ephrata, WA, United States, 2015 Umatilla	2 (14) banded in-furrow at plant, broadcast	1.12 1.12	110	ND	ND	ND	ND	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			115	ND	ND	ND	ND	<0.01	ND	ND	ND	
				0.044	ND	0.029	0.041	0.020	<0.01	<0.01	<0.01	
			Mean	<0.027	<0.01	<0.0195	<0.0255	0.015	<0.01	<0.01	<0.01	0.1016
			120	<0.01	ND	ND	<0.01	<0.01	ND	ND	ND	
				<0.01	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			125	<0.01	ND	ND	<0.01	<0.01	ND	ND	ND	
				ND	ND	ND	ND	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			130	ND	ND	ND	ND	<0.01	ND	ND	ND	
				<0.01	ND	ND	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			136	0.019	ND	0.012	0.015	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.0145	<0.01	<0.011	<0.0125	<0.01	<0.01	<0.01	<0.01	0.0761
Ephrata, WA, United States, 2015 Norland Dark Red	1 banded in-furrow bare soil	2.24	80	ND	ND	ND	ND	ND	<0.01	ND	ND	
				<0.01	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Ephrata, WA, United States, 2015 Norland Dark Red	1 banded in-furrow bare soil	2.24	80	0.013	ND	<0.01	0.013	<0.01	0.016	ND	<0.01	
				0.026	ND	0.013	0.022	<0.01	0.018	ND	<0.01	
			Mean	0.0195	<0.01	<0.0115	0.0175	<0.01	0.017	<0.01	<0.01	0.0877
			85	ND	ND	ND	<0.01	ND	<0.01	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			90	<0.01	ND	ND	<0.01	ND	0.01[A]	ND	ND	
				ND	ND	ND	ND	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			95	0.019	ND	<0.01	0.018	<0.01	0.020	ND	<0.01	
				0.011	ND	<0.01	0.013	<0.01	0.016	ND	<0.01	
			Mean	0.015	<0.01	<0.01	0.0155	<0.01	0.018	<0.01	<0.01	0.0861
			100	<0.01	ND	ND	<0.01	ND	<0.01	ND	ND	
				<0.01	ND	ND	<0.01	ND	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			105	0.052	ND	0.025	0.042	0.015	0.031	ND	<0.01	
				<0.01	ND	ND	<0.01	ND	0.01[A]	ND	ND	
			Mean	0.031	<0.01	<0.0175	<0.026	<0.0125	0.0205	<0.01	<0.01	0.1095

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
Ephrata, WA, United States, 2015 Norland Dark Red	2 (14) banded in-furrow bare soil, broadcast soil BBCH03-07 pre-emergence	1.12	66	ND	ND	ND	<0.01	<0.01	<0.01	ND	ND	
		1.12		ND	ND	ND	<0.01	<0.01	<0.01	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
Ephrata, WA, United States, 2015 Norland Dark Red	2 (14) banded in-furrow bare soil, broadcast soil BBCH03-07 pre-emergence	1.12	66	0.039	ND	0.018	0.034	0.01[A]	0.022	ND	<0.01	
		1.12		<0.01	ND	ND	0.011	<0.01	0.01[A]	ND	ND	
			Mean	<0.0245	<0.01	<0.014	0.0225	<0.01	0.016	<0.01	<0.01	0.0915
			71	<0.01	ND	ND	<0.01	ND	<0.01	ND	ND	
				<0.01	ND	ND	<0.01	<0.01	0.01[A]	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			76	<0.01	ND	ND	<0.01	<0.01	<0.01	ND	ND	
				ND	ND	ND	<0.01	<0.01	0.01[A]	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01[A]	<0.01	<0.01	0.0740
			81	0.014	ND	<0.01	0.019	<0.01	0.020	ND	<0.01	
				<0.01	ND	<0.01	0.014	<0.01	0.013	ND	<0.01	
			Mean	<0.012	<0.01	<0.01	0.0165	<0.01	0.0165	<0.01	<0.01	0.0838
			86	<0.01	ND	ND	0.011	<0.01	0.012	ND	<0.01	
				<0.01	ND	ND	0.012	<0.01	0.014	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.0115	<0.01	0.013	<0.01	<0.01	0.0785
			91	<0.01	ND	<0.01	0.015	<0.01	0.014	ND	<0.01	
				ND	ND	ND	0.011	ND	0.011	ND	<0.01	
			Mean	<0.01	<0.01	<0.01	0.013	<0.01	0.0125	<0.01	<0.01	0.0778
Rupert, ID, United States, 2015 Western Russet	1 banded in-furrow BBCH01	2.32	127	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
				0.046	<0.01	0.025	0.035	<0.01	ND	ND	<0.01	
			Mean	0.028	<0.01	0.0175	0.0225	<0.01	<0.01	<0.01	<0.01	0.0898
			132	0.036	<0.01	0.022	0.029	<0.01	ND	ND	ND	
				0.025	<0.01	0.013	0.020	<0.01	ND	ND	ND	
			Mean	0.0305	<0.01	0.0175	0.0245	<0.01	<0.01	<0.01	<0.01	0.0898
			137	0.045	ND	0.027	0.037	<0.01	ND	ND	ND	
				0.17	<0.01	0.095	0.12	<0.01	<0.01	ND	<0.01	
			Mean	0.1075	<0.01	0.061	0.0785	<0.01	<0.01	<0.01	<0.01	0.1816
			142	0.083	<0.01	0.045	0.062	<0.01	ND	ND	<0.01	
				0.010	ND	<0.01	0.010	<0.01	ND	ND	ND	
			Mean	0.0465	<0.01	<0.0275	0.036	<0.01	<0.01	<0.01	<0.01	0.1109
			147	0.083	<0.01	0.049	0.065	<0.01	ND	ND	<0.01	
				0.036	<0.01	0.019	0.027	<0.01	ND	ND	ND	
			Mean	0.0595	<0.01	0.034	0.046	<0.01	<0.01	<0.01	<0.01	0.1246
			152	0.030	ND	0.016	0.022	<0.01	ND	ND	ND	
				0.043	<0.01	0.023	0.034	<0.01	ND	ND	ND	
			Mean	0.0365	<0.01	0.0195	0.028	<0.01	<0.01	<0.01	<0.01	0.0940
Rupert, ID, United States, 2015 Western Russet	2 (15) banded in-furrow BBCH01, banded over row BBCH07-08	1.15	112	0.019	<0.01	0.013	0.018	<0.01	ND	ND	ND	
		1.11		0.029	<0.01	0.019	0.027	<0.01	ND	ND	ND	
			Mean	0.024	<0.01	0.016	0.024	<0.01	<0.01	<0.01	<0.01	0.0867
			117	0.024	<0.01	0.014	0.023	<0.01	ND	ND	<0.01	
				0.024	<0.01	0.016	0.024	<0.01	ND	ND	<0.01	
			Mean	0.024	<0.01	0.015	0.0235	<0.01	<0.01	<0.01	<0.01	0.0846
			122	0.021	<0.01	0.012	0.019	<0.01	ND	ND	<0.01	
				0.031	ND	0.018	0.025	<0.01	ND	ND	ND	
			Mean	0.026	<0.01	0.015	0.022	<0.01	<0.01	<0.01	<0.01	0.0846
			127	0.016	<0.01	0.010	0.015	<0.01	ND	ND	ND	
				0.027	<0.01	0.016	0.027	<0.01	ND	ND	<0.01	
			Mean	0.0215	<0.01	0.013	0.021	<0.01	<0.01	<0.01	<0.01	0.0803
			132	0.040	<0.01	0.022	0.032	<0.01	ND	ND	ND	
				0.020	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	0.030	<0.01	<0.016	<0.021	<0.01	<0.01	<0.01	<0.01	0.0867
			137	0.031	<0.01	0.019	0.028	<0.01	ND	ND	ND	
				0.021	<0.01	0.013	0.021	<0.01	ND	ND	ND	
			Mean	0.026	<0.01	0.016	0.0245	<0.01	<0.01	<0.01	<0.01	0.0867
Rupert, ID,	1 banded in-	2.23	121	0.011	ND	<0.01	<0.01	ND	ND	ND	ND	

Location Year variety	N (interval, days)	Rate (kg ai/ha)	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	SUM [B]
United States, 2015 Russet Burbank	furrow BBCH01			<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.0105	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			126	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				<0.01	ND	ND	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			131	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				0.01[A]	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			136	<0.01	ND	ND	<0.01	ND	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			141	ND	ND	ND	ND	ND	ND	ND	ND	
				0.022	ND	0.013	0.015	<0.01	ND	ND	ND	
			Mean	<0.016	<0.01	<0.0115	<0.0125	<0.01	<0.01	<0.01	<0.01	0.0772
			146	0.015	ND	<0.01	0.012	<0.01	ND	ND	ND	
				0.013	ND	<0.01	0.011	<0.01	ND	ND	ND	
			Mean	0.014	<0.01	<0.01	0.0115	<0.01	<0.01	<0.01	<0.01	0.0740
Rupert, ID, United States, 2015 Russet Burbank	2 (15) banded in-furrow BBCH01, banded over row BBCH07-08	1.11 1.12	107	0.012	ND	<0.01	0.010	<0.01	ND	ND	ND	
				0.017	ND	0.010	0.012	<0.01	ND	ND	ND	
			Mean	0.0145	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	<0.01	0.0740
			112	0.028	ND	0.015	0.022	<0.01	<0.01	ND	ND	
				0.016	ND	ND	ND	ND	ND	ND	ND	
			Mean	0.022	<0.01	<0.0125	<0.016	<0.01	<0.01	<0.01	<0.01	0.0793
			117	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				0.013	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.0115	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			122	<0.01	ND	<0.01	<0.01	<0.01	<0.01	ND	ND	
				<0.01	ND	<0.01	<0.01	ND	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			127	0.01[A]	ND	<0.01	<0.01	<0.01	ND	ND	ND	
				<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0740
			132	0.014	ND	<0.01	0.012	<0.01	ND	ND	ND	
				0.016	ND	<0.01	0.013	<0.01	<0.01	ND	ND	
			Mean	0.015	<0.01	<0.01	0.0125	<0.01	<0.01	<0.01	<0.01	0.0740

Notes:

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] SUM = 2.26×IN-A5760 + 2.11×IN-F4106 + 1.52×IN-QZY47 + 1.51×IN-TMQ01.

[C] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

ROTATIONAL CROPS

A number of studies investigated residues of fluazaindolizine and metabolites in follow crops. Doig (2020 DuPont-36790) conducted residue trials on wheat, lettuce, radish and bean in North (Alpicat, Catalunya) and South (Aguadulce, Andalucia) Spain in 2013/2014.

At each location, bare plots were treated with a fluazaindolizine SC formulation as either a single application at 1.25 kg ai/ha or two applications at 1.25 kg ai/ha and at an interval of 60±10 days and follow crops planted at target plant-back intervals of 7-30, 60-270, and 270-365 days. Duplicate field specimens were collected from each plot consisting of 50 percent mature lettuce, wheat (forage and hay), immature bean seeds, mature lettuce, mature radish (roots and tops), wheat (grain and straw) and mature beans (seeds, vines and whole plant).

The maximum interval of frozen storage before analysis was; wheat forage 15 months, wheat grain 29 months, wheat hay 30 months, wheat straw 33 months, immature lettuce 15 months, mature lettuce 15 months, bean hay 33 months, immature bean seeds 14 months, mature bean seeds 12 months, bean vines 14 months, radish roots 15 months and tops 15 months. Samples were analysed for residues of fluazaindolizine and its metabolites IN-A5760, IN-F4016, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12 and IN-UNS90 using method AP.224685 based on the analytical method DuPont-33861, rev. 3, with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg for each analyte. Average recovery values (±RSD) per analyte/matrix combination ranged from 73 ± 10 percent to 111 ± 5 percent for 4-17 fortifications per analyte/matrix combination from combined recoveries of all fortification levels. The RSD were below 20 percent with two exceptions. The RSD was 25 percent each for analysis of parent fluazaindolizine on legume hay and IN-UNS90 on radish tops.

In a separate study Doig (2020 DuPont-40828) conducted residue trials on oilseed rape, wheat, peas in South France (Charantonnay, Rhone Alpes), North Spain (Alpicat, Catalunya and Termens, Catalunya) and South Spain (Los Palacios, Andalucia and Aguadulce, Andalucia) Spain in 2014-2016.

At each location, bare plots were treated with a fluazaindolizine SC formulation as four applications at 0.825 kg ai/ha and at 14 day intervals and follow crops (oilseed rape, wheat, peas and maize) planted at target plant-back intervals of 7-10, 60-270, and 358-365 days. Duplicate field specimens were collected from each plot.

The maximum interval of frozen storage before analysis was; wheat forage 31 months, wheat grain 24 months, wheat hay 23 months, wheat straw 28 months, pea forage 25 months, dried peas 23 months, pea vines 29 months, pea hay 28 months, rape forage 26 months, rape seed 27 months, rape straw 20 months, maize forage 25 months, maize immature ears 20 months, maize grain 21 months and maize stover 18 months. Samples were analysed for residues of fluazaindolizine and its metabolites IN-A5760, IN-F4016, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12 and IN-UNS90 (IN-TQD54) using method AP.224685 based on the analytical method DuPont-33861, rev. 3, with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg for each analyte. Average recovery values (±RSD) per analyte/matrix combination ranged from 71±13 percent to 111±13 percent for 19–29 fortifications per analyte/matrix combination for combined data from all fortification levels. Average recoveries for the 304 analyte/matrix/fortification level combinations for pre-hydrolysis residues of fluazaindolizine and the 7 post-hydrolysis metabolites ranged from 51 to 115 percent. All but 10 of the 304 average recovery values were in the range of 70–110 percent. The 10 exceptions are:

- Fluazaindolizine on wheat straw and dried peas were 66 and 68 percent, respectively, for the 0.01 mg/kg level fortifications.
- IN-RSU03 (IN-TMQ01) average recoveries on wheat forage, dried peas and pea hay were 62, 57 and 56 percent, respectively, for the 1.0 mg/kg level fortifications.
- IN-QEK31 and IN-UNS90 average recoveries on pea hay were 68 and 65 percent, respectively, for the 1.0 mg/kg level fortifications.
- IN-QEK31 average recoveries on dried peas at 1.0 mg/kg had an average recovery of 51 percent.
- IN-QZY47 average recovery on wheat grain was 115 percent for the 0.10 mg/kg level fortifications.
- IN-F4106 average recovery on dried peas was 64 percent for the 1.0 mg/kg fortifications.

Standard deviations for the 304 analyte/matrix/fortification level combinations for pre-hydrolysis residues of fluazaindolizine and the seven post-hydrolysis metabolites ranged from 1 to 28 percent. All but nine of the 304 standard deviation values were 20 percent or less. The nine exceptions are:

- Fluazaindolizine standard deviation for wheat grain recoveries at the 0.01 mg/kg fortification level was 23 percent.
- IN-UNS90 (IN-TQD54) standard deviations for the 0.01 mg/kg fortification recoveries were 23 and 21 percent, respectively, for oilseed rape straw and wheat grain.
- IN-UNS90 (IN-TQD54) standard deviations for the 0.1 mg/kg fortification recoveries were 21 percent for pea vines.
- IN-UNS90 (IN-TQD54) standard deviation for wheat forage recoveries at the 0.1 mg/kg fortification level was 21 percent.
- IN-F4106 standard deviations for oilseed rape seed and wheat grain recoveries at the 0.01 mg/kg fortification level were 21 and 22 percent, respectively.
- IN-A5760 standard deviation for pea hay recoveries at the 0.10 mg/kg fortification level was 21 percent.
- IN-UJV12 standard deviation for maize immature ears recoveries at the 0.01 mg/kg fortification level was 28 percent.

In another study Doig (2020 DuPont-41762) conducted residue trials on tomato, strawberry, Swiss chard or celery, turnip, broccoli and lettuce in South France (Lucenay, Rhone Alpes), North Spain (Lleida, Catalunya and Termens, Catalunya) and South Spain (Los Palacios, Andalucia and Aguadulce, Andalucia) Spain in 2014-2016.

At each location, bare plots were treated with a fluazaindolizine SC formulation as four applications at 1.1 kg ai/ha and at 14-day intervals and follow crops (tomato, strawberry, Swiss chard or celery, turnip, broccoli and lettuce) planted at target plant-back intervals of 7–10, 60–270, and 358–365 days. Replicate field specimens were collected from each plot.

The maximum interval of frozen storage before analysis was 647 days (22 months). Samples were analysed for residues of fluazaindolizine and its metabolites IN-A5760, IN-F4016, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12 and IN-UNS90 (IN-TQD54) using method AP.224685 based on the analytical method DuPont-33861, rev. 3, with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg for each analyte. Average recoveries for the 104 analyte/matrix/fortification level combinations for pre-hydrolysis residues of fluazaindolizine and the 7 post-hydrolysis metabolites ranged from 67 to 110 percent. All but 1 of the 104 average recovery values were in the range of 70–110 percent. The average recovery of IN-UNS90 (IN-TQD54) on tomato fruit for the ca 0.01 mg/kg fortification level was 67 percent. Standard deviations for the 104 analyte/matrix/fortification level combinations for pre-hydrolysis residues of fluazaindolizine and the 7 post-hydrolysis metabolites ranged from 1 to 22 percent. All but 2 of the 104 standard deviation values were 20 percent or less. The standard deviation for recovery of IN-UNS90 (IN-TQD54) at ca 0.01 mg/kg level fortifications on celery/Swiss chard was 22 percent. The standard deviation for recovery of IN-QEK31 0.01 mg/kg level fortifications on strawberry fruit was 21 percent.

A series of studies conducted in Canada and the United States in 2013-2015 also investigated residues in follow crops. In the first by Shepard (2020 DuPont-36791 rev 1), bare soil was treated with an SC formulation of fluazaindolizine at either 1.25 kg ai/ha or as two application each of 1.25 kg ai/ha and at an interval of 2 months. Crops (spinach, lettuce, radish, wheat, sorghum or soya bean) were planted at PBIs in the following ranges: 7-30, 60-120, 60-270 and 270-365 days.

The maximum interval of frozen storage before analysis was 881 days except for two lettuce leaf samples which were re-extracted for IN-TMQ01 analysis at 1251 days (41 months). Samples were analysed for residues of fluazaindolizine and its metabolites IN-A5760, IN-F4016, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12 and IN-UNS90 (IN-TQD54) using the analytical method DuPont-33861, rev. 3, with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg for each analyte. Mean values per analyte/matrix

combination ranged from 78 to 107 percent for 4 to 18 fortifications per analyte/matrix combination. The standard deviations ranged from 2.9 to 27 percent per analyte/matrix combination.

In the second by Shepard (2020 DuPont-40012 rev 1), bare soil was treated with an SC formulation of fluazaindolizine as four applications of 1.12 kg ai/ha and at 7-day intervals. Crops (broccoli, leaf lettuce, carrot or radish, celery or Swiss chard, strawberry, and tomato) were planted at PBIs in the following ranges: 7-30, 60-270 and 270-365 days.

The maximum interval of frozen storage before analysis was 616 days (20 months). Samples were analysed for residues of fluazaindolizine and its metabolites IN-A5760, IN-F4016, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12 and IN-UNS90 (IN-TQD54) using the analytical method DuPont-33861, rev. 3, with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg for each analyte. Mean values per analyte/matrix combination ranged from 82 to 99 percent for 2 to 22 fortifications per analyte/matrix combination. The standard deviations ranged from 4.5 to 16 percent per analyte/matrix combination.

In the third study by Shepard (2020 DuPont-41070 rev 1), bare soil was treated with an SC formulation of fluazaindolizine as four applications of 1.12 kg ai/ha and at 7-day intervals. Crops (peas, soya bean, maize, wheat) were planted at PBIs in the following ranges: 7–30, 60–270 and 270–365 days.

The maximum interval of frozen storage before analysis was 1001 days (33 months). Samples were analysed for residues of fluazaindolizine and its metabolites IN-A5760, IN-F4016, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12 and IN-UNS90 (IN-TQD54) using the analytical method DuPont-33861, rev. 3, with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg for each analyte. Mean values per analyte/matrix combination ranged from 70 to 114 percent for 3 to 28 fortifications per analyte/matrix combination. The standard deviations ranged from 1.0 to 19 percent per analyte/matrix combination, with one exception. The standard deviation was 23 percent for analysis of IN-A5760 on soya bean seed.

Table 143 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in soil from rotational crop studies

Study Location	Rate (kg ai/ha)	DALA	Fluazaindolizine	IN-QEK31	IN-A5760	IN-F4106	IN-REG72	IN-RYC33	IN-VM862
Shepard 2020 DuPont-36791									
Richland IA	1.26	61	0.052	0.069	<0.01	0.052			
Richland IA	2.51	61	0.029	0.039	<0.01	0.040			
Richland IA	2.51	270	0.11	0.11	<0.01	0.098			
Porterville CA	2.51	361	0.052	0.042	<0.01	0.028			
Athens GA	2.51	361	<0.01	0.014	<0.01	0.018			
Doig 2020 DuPont-40828 rev 1									
Charantonnay, Rhone Alpes, France	0.83-0.84	56	0.12	0.015	<0.01	0.031	0.010	<0.01	0.012
Alpicat Catalunya Spain	0.83-0.84	62	0.14	0.047	ND	0.015	<0.01	<0.01	<0.01
Termens Catalunya Spain	0.83-0.84	63	0.24	0.024	ND	0.028	<0.01	<0.01	<0.01
Los Palacios Andalucía Spain	0.83-0.84	61	0.14	0.012	<0.01	0.017	<0.01	<0.01	<0.01
Aguadulce Andalucía Spain	0.83-0.84	57	0.42	0.013	ND	0.0248	<0.01	<0.01	<0.01
Doig 2020 DuPont 36790									
Alpicat, Catalunya, Spain	1.25	27	0.025	<0.01	ND	<0.01	ND	<0.01	ND
		127	0.18	0.017	ND	0.016	<0.01	<0.01	<0.01
		27	0.26	0.013	ND	0.011	<0.01	<0.01	<0.01
		141	0.30	0.069	<0.01	0.057	<0.01	<0.01	<0.01
		242	0.078	0.048	<0.01	0.035	<0.01	<0.01	<0.01
Aguadulce, Andalucia Spain	1.25	27	0.46	<0.01	ND	0.014	<0.01	<0.01	<0.01
		144	0.014	ND	ND	<0.01	ND	ND	ND
		26	0.029	ND	ND	<0.01	<0.01	ND	ND
		144	0.064	<0.01	ND	<0.01	<0.01	ND	<0.01
		241	0.096	<0.01	ND	0.011	<0.01	<0.01	<0.01

Table 144 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in strawberry rotational crops in the United States and Europe

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Lucenay, Rhone Alpes, France 2015 Lola la sucree⑤	4.50	7	Fruit	ND	ND	ND	ND	ND	ND	ND	ND
	4.51	60		ND	ND	ND	ND	ND	<0.01	ND	ND
	4.40	364		ND	ND	ND	ND	ND	ND	ND	ND
Lleida, Catalunya, Spain 2014 Sabrina⑤	4.42	7	Fruit	ND	ND	ND	<0.01	ND	0.013	ND	ND
	4.39	63		ND	ND	<0.01	<0.01	ND	0.015	ND	ND
Lleida, Catalunya, Spain 2014 Florida Fortuna⑤	4.40	361	Fruit	ND	ND	ND	<0.01	ND	0.011	ND	ND
Termens, Catalunya, Spain 2014 Sabrina⑤	4.45	10	Fruit	<0.01	ND	<0.01	<0.01	ND	0.014	ND	ND
Termens, Catalunya, Spain 2014 Florida Fortuna⑤	4.46	62	Fruit	ND	ND	<0.01	ND	ND	0.028	ND	ND
	4.40	367	Fruit	ND	ND	ND	<0.01	ND	0.017	ND	ND
Los Palacios, Andalucía, Spain 2014 Ventana⑤	4.46	8	Fruit	ND	ND	ND	<0.01	ND	0.024	ND	ND
	4.47	62	Fruit	ND	ND	ND	<0.01	ND	0.035	ND	ND
Los Palacios, Andalucía, Spain 2014 Fortuna⑤	4.50	363	Fruit	ND	ND	ND	ND	ND	<0.01	ND	ND
Aguadulce, Andalucía, Spain 2014 Ventana⑤	4.49	7	Fruit	ND	ND	ND	ND	ND	0.010	ND	ND
Aguadulce, Andalucía, Spain 2014 Fortuna⑤	4.50	63	Fruit	ND	ND	ND	ND	ND	<0.01	ND	ND
	4.49	365	Fruit	ND	ND	ND	ND	ND	<0.01	ND	ND
Athens, GA, United States, 2014-2016 Chandler②	4.48	8	Fruit	ND	ND	ND	<0.01	<0.01	0.13	ND	0.014
				ND	ND	ND	<0.01	<0.01	0.16	ND	0.019
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.145	<0.01	0.0165
	4.47	63		ND	ND	ND	<0.01	ND	0.079	ND	<0.01
				ND	ND	ND	<0.01	ND	0.086	ND	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0825	<0.01	<0.01
Athens, GA, United States, 2014-2016 Camarosa②	4.51	358	Fruit	ND	ND	ND	ND	ND	0.012	ND	ND
				ND	ND	ND	ND	ND	0.011	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0115	<0.01	<0.01
Oviedo, FL, United States, 2014/2015 Radiance Festival②	4.36	21	Fruit	ND	ND	0.018	<0.01	ND	0.094	ND	0.019
				ND	ND	ND	ND	ND	ND	ND	0.015
			Mean	<0.01	<0.01	<0.014	<0.01	<0.01	<0.052	<0.01	0.017
Oviedo, FL, United States, 2014/2015 Radiance ②	4.42	60	Fruit	ND	ND	ND	ND	ND	0.010	ND	ND
				ND	ND	ND	ND	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	4.40	373		ND	ND	ND	ND	ND	ND	ND	ND
				ND	ND	ND	ND	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Porterville, CA, United States, 2014/2015 Albion②	4.55	8	Fruit	ND	ND	<0.01	0.025	ND	0.11	ND	0.013
				ND	ND	<0.01	0.035	<0.01	0.14	ND	0.015
			Mean	<0.01	<0.01	<0.01	0.030	<0.01	0.125	<0.01	0.014
	4.51	60		ND	ND	<0.01	0.026	<0.01	0.13	ND	0.017
				ND	ND	<0.01	0.024	ND	0.11	ND	0.012
			Mean	<0.01	<0.01	<0.01	0.025	<0.01	0.12	<0.01	0.0145
	4.52	277		ND	ND	ND	0.021	<0.01	0.16	ND	0.017
				ND	ND	<0.01	0.015	ND	0.12	ND	0.012
			Mean	<0.01	<0.01	<0.01	0.018	<0.01	0.14	<0.01	0.0145
Sanger, CA, United	4.58	7	Fruit	ND	ND	ND	ND	ND	0.025	ND	ND

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
States, 2014-2016 Camarosa②				ND	ND	ND	<0.01	ND	0.030	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0275	<0.01	<0.01
	4.48	59		ND	ND	ND	ND	ND	0.023	ND	ND
				ND	ND	ND	ND	ND	0.019	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.021	<0.01	<0.01
	4.56	341		ND	ND	ND	ND	ND	ND	ND	ND
				ND	ND	ND	ND	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Fresno, CA, United States, 2014/2015 San Andreas②	4.45	7	Fruit	ND	ND	ND	ND	ND	0.01[A]	ND	ND
				ND	ND	ND	<0.01	ND	0.020	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.015	<0.01	<0.01
	4.43	63	Fruit	ND	ND	ND	<0.01	ND	0.010	ND	ND
				ND	ND	ND	<0.01	ND	0.012	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.011	<0.01	<0.01
Fresno, CA, United States, 2014/2015 Seascape②	4.46	365	Fruit	ND	ND	ND	ND	ND	0.011	ND	ND
				ND	ND	ND	ND	ND	0.01[A]	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0105	<0.01	<0.01

Notes:

② Shepard (2020 DuPont-40012 rev 1). A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

⑤ Doig 2020 DuPont-41762.

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] For trials conducted in the United States, a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-TQD54 to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 145 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in strawberry rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM [A] and 1.77×IN-QEK31
Athens, GA, United States, 2014-2016 Chandler	4.48	8	0.0050	0.0113	0.0182	0.0050	0.0050	0.0725	0.0050	0.0083	0.1466
	4.47	63	0.0050	0.0113	0.0183	0.0050	0.0050	0.0413	0.0050	0.0050	0.0996
Athens, GA, United States, 2014-2016 Camarosa	4.51	358	0.0050	0.0112	0.0181	0.0050	0.0050	0.0057	0.0050	0.0050	0.0455
Oviedo, FL, United States, 2014/2015 Festival Radiance	4.56	21	0.0049	0.0111	0.0251	0.0049	0.0049	0.0255	0.0049	0.0084	0.0822
Oviedo, FL, United States, 2014/2015 Radiance	4.42	60	0.0051	0.0115	0.0185	0.0051	0.0051	0.0051	0.0051	0.0051	0.0453
	4.4	373	0.0051	0.0115	0.0186	0.0051	0.0051	0.0051	0.0051	0.0051	0.0455
Oviedo, FL, United States, 2014/2015 Radiance	4.55	8	0.0049	0.0111	0.0179	0.0148	0.0049	0.0615	0.0049	0.0069	0.1295
	4.51	60	0.0050	0.0112	0.0181	0.0124	0.0050	0.0596	0.0050	0.0072	0.1269
	4.52	277	0.0050	0.0112	0.0181	0.0089	0.0050	0.0694	0.0050	0.0072	0.1416
Sanger, CA,	4.58	7	0.0049	0.0111	0.0178	0.0049	0.0049	0.0134	0.0049	0.0049	0.0566

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM [A] and 1.77×IN-QEK31
United States, 2014-2016 Camarosa	4.48	59	0.0050	0.0113	0.0182	0.0050	0.0050	0.0105	0.0050	0.0050	0.0530
	4.56	341	0.0049	0.0111	0.0179	0.0049	0.0049	0.0049	0.0049	0.0049	0.0439
Fresno, CA, United States, 2014/2015 San Andreas	4.45	7	0.0050	0.0114	0.0183	0.0050	0.0050	0.0076	0.0050	0.0050	0.0488
Fresno, CA, United States, 2014/2015 San Andreas	4.43	63	0.0051	0.0114	0.0184	0.0051	0.0051	0.0056	0.0051	0.0051	0.0459
Fresno, CA, United States, 2014/2015 Seascape	4.46	365	0.0050	0.0114	0.0183	0.0050	0.0050	0.0053	0.0050	0.0050	0.0453

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 146 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in tomato rotational crops in Europe and the United States

Location	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Lucenay, Rhone Alpes, France 2015 Gourmandia⑤	4.44	7	Fruit	ND	<0.01	ND	ND	ND	ND	ND	<0.01
	4.48	63	Fruit	ND	0.015	ND	0.011	ND	0.013	ND	0.021
	4.50	359	Fruit	ND	ND	ND	ND	ND	ND	ND	ND
Lleida, Catalunya, Spain 2014 Royesta⑤	4.41	7	Fruit	ND	ND	ND	ND	ND	ND	ND	ND
	4.47	63		ND	<0.01	ND	ND	ND	ND	ND	<0.01
Lleida, Catalunya, Spain 2014 Bodar⑤	4.31	358	Fruit	ND	ND	ND	ND	ND	ND	<0.01	ND
Termens, Catalunya, Spain 2014 Rio Grande⑤	4.46	10	Fruit	ND	<0.01	ND	ND	ND	ND	ND	ND
	4.44	60		ND	<0.01	ND	<0.01	ND	ND	ND	ND
Termens, Catalunya, Spain 2014 Incas⑤	4.47	359	Fruit	ND	<0.01	ND	ND	ND	ND	ND	<0.01
Los Palacios, Andalucía, Spain 2014 Matias⑤	4.55	8	Fruit	ND	<0.01	ND	ND	ND	ND	ND	ND
	4.47	62		ND	ND	ND	ND	ND	ND	ND	ND
Los Palacios, Andalucía, Spain 2014 Bodar⑤	4.29	363	Fruit	ND	<0.01	ND	ND	ND	ND	ND	ND
Aguadulce, Andalucía, Spain 2014 Tisey⑤	4.41	7	Fruit	<0.01	ND	ND	ND	ND	ND	ND	ND
	4.47	61		ND	ND	ND	ND	ND	ND	ND	ND
	4.40	361		ND	ND	ND	ND	ND	ND	ND	ND
Athens, GA, United States, 2014/2015 Tobago (large) ②	4.48	12	Fruit	ND	0.013	ND	0.016	ND	0.013	ND	<0.01
				ND	<0.01	ND	0.012	ND	0.010	ND	<0.01
			Mean	<0.01	<0.0115	<0.01	0.014	<0.01	0.0115	<0.01	<0.01
	4.49	260		ND	0.011	ND	<0.01	ND	<0.01	ND	<0.01
				ND	0.013	ND	<0.01	ND	<0.01	ND	<0.01
			Mean	<0.01	0.012	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	4.51	363		ND	<0.01	ND	ND	ND	ND	ND	ND

Location	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
				ND	<0.01	ND	ND	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Oviedo, FL, United States, 2014/2015Sweet 100 (small)②	4.51	7	Fruit	ND	0.012	ND	0.019	ND	0.016	ND	<0.01
				ND	0.014	ND	0.017	ND	0.017	ND	<0.01
			Mean	<0.01	0.013	<0.01	0.018	<0.01	0.0165	<0.01	<0.01
	4.57	60		ND	<0.01	ND	0.013	ND	0.011	ND	<0.01
				ND	<0.01	ND	<0.01	ND	<0.01	ND	<0.01
			Mean	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.0105	<0.01	<0.01
	4.52	365		ND	ND	ND	ND	ND	<0.01	ND	ND
				ND	ND	ND	ND	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Porterville, CA, United States, 2014/2015 Quality T23 (large) ②	4.50	7	Fruit	ND	0.026	<0.01	<0.01	ND	0.026	ND	0.014
				ND	0.023	<0.01	<0.01	ND	0.020	ND	0.014
			Mean	<0.01	0.0245	<0.01	<0.01	<0.01	0.023	<0.01	0.014
	4.55	60		ND	0.021	<0.01	<0.01	ND	0.020	ND	0.011
				ND	0.017	<0.01	<0.01	ND	0.015	ND	0.010
			Mean	<0.01	0.019	<0.01	<0.01	<0.01	0.0175	<0.01	0.0105
	4.54	365		ND	0.013	ND	<0.01	ND	0.014	ND	<0.01
				ND	0.011	ND	<0.01	ND	<0.01	ND	<0.01
			Mean	<0.01	0.012	<0.01	<0.01	<0.01	<0.012	<0.01	<0.01
Sanger, CA, United States, 2014/2015 Golden Gem (small) ②	4.44	7	Fruit	ND	0.019	ND	0.088	ND	0.053	ND	0.022
				ND	0.019	ND	0.082	ND	0.030	ND	0.019
			Mean	<0.01	0.019	<0.01	0.085	<0.01	0.0415	<0.01	0.0205
	4.40	61		ND	0.056	<0.01	0.18	<0.01	0.17	ND	0.053
				ND	0.051	<0.01	0.18	<0.01	0.093	ND	0.045
			Mean	<0.01	0.0535	<0.01	0.18	<0.01	0.1315	<0.01	0.049
	4.40	351		ND	0.015	ND	0.056	ND	0.046	ND	0.014
				ND	0.015	ND	0.045	ND	0.052	ND	0.014
			Mean	<0.01	0.015	<0.01	0.0505	<0.01	0.049	<0.01	0.014
Paso Robles, CA, United States, 2014/2015 Red Cherry Large (small) ②	4.50	7	Fruit	ND	<0.01	ND	ND	ND	<0.01	ND	ND
				ND	<0.01	ND	ND	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		63		ND	ND	ND	ND	ND	ND	ND	ND
				ND	ND	ND	ND	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		369		ND	<0.01	ND	ND	ND	<0.01	ND	<0.01
				ND	<0.01	ND	ND	ND	ND	ND	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Notes:

② Shepard (2020 DuPont-40012 rev 1).

⑤ Doig 2020 DuPont-41762.

[A] For trials conducted in the United States, a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 147 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in tomato rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Fluaza-indolizine	IN-A5760	IN-F4106	1.068×IN-A5760 + IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Lucenay, Rhone Alpes, France 2015	4.44	7	0.0050	0.0114	0.0184	0.0141	0.0050	0.0050	0.0050	0.0050	0.0050	0.0451
	4.48	63	0.0050	0.0170	0.0182	0.0166	0.0055	0.0050	0.0065	0.0050	0.0105	0.0526
	4.5	359	0.0050	0.0112	0.0181	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Fluaza-indolizine	IN-A5760	IN-F4106	1.068×IN-A5760 + IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Gourmandia												
Lleida, Catalunya, Spain 2014 Royesta	4.41	7	0.0051	0.0115	0.0185	0.0142	0.0051	0.0051	0.0051	0.0051	0.0051	0.0454
	4.47	63	0.0050	0.0113	0.0183	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0448
Lleida, Catalunya, Spain 2014 Bodar	4.31	358	0.0052	0.0117	0.0189	0.0145	0.0052	0.0052	0.0052	0.0052	0.0052	0.0464
Termens, Catalunya, Spain 2014 Rio Grande	4.46	10	0.0050	0.0114	0.0183	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0449
	4.44	60	0.0050	0.0114	0.0184	0.0141	0.0050	0.0050	0.0050	0.0050	0.0050	0.0451
Termens, Catalunya, Spain 2014 Incas	4.47	359	0.0050	0.0113	0.0183	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0448
Los Palacios, Andalucía, Spain 2014 Matias	4.55	8	0.0049	0.0111	0.0179	0.0138	0.0049	0.0049	0.0049	0.0049	0.0049	0.0440
	4.47	62	0.0050	0.0113	0.0183	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0448
Los Palacios, Andalucía, Spain 2014 Bodar	4.29	363	0.0052	0.0118	0.0190	0.0146	0.0052	0.0052	0.0052	0.0052	0.0052	0.0467
Aguadulce, Andalucía, Spain 2014 Tisey	4.41	7	0.0051	0.0115	0.0185	0.0142	0.0051	0.0051	0.0051	0.0051	0.0051	0.0454
	4.47	61	0.0050	0.0113	0.0183	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0448
	4.4	361	0.0051	0.0115	0.0186	0.0142	0.0051	0.0051	0.0051	0.0051	0.0051	0.0455
Athens, GA, United States, 2014/2015 Tobago (large)	4.48	12	0.0050	0.0130	0.0182	0.0148	0.0070	0.0050	0.0058	0.0050	0.0050	0.0475
	4.49	260	0.0050	0.0135	0.0182	0.0150	0.0050	0.0050	0.0050	0.0050	0.0050	0.0468
	4.51	363	0.0050	0.0112	0.0181	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0444
Oviedo, FL, United States, 2014/2015 Sweet 100 (small)	4.51	7	0.0050	0.0146	0.0181	0.0155	0.0089	0.0050	0.0082	0.0050	0.0050	0.0526
	4.57	60	0.0050	0.0111	0.0179	0.0137	0.0056	0.0049	0.0051	0.0049	0.0049	0.0442
	4.52	365	0.0050	0.0112	0.0181	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0443
Porterville, CA, United States, 2014/2015 Quality T23 (large)	4.5	7	0.0050	0.0276	0.0181	0.0216	0.0050	0.0050	0.0114	0.0050	0.0070	0.0706
	4.55	60	0.0049	0.0211	0.0179	0.0185	0.0049	0.0049	0.0086	0.0049	0.0052	0.0596
	4.54	365	0.0049	0.0134	0.0180	0.0148	0.0049	0.0049	0.0059	0.0049	0.0049	0.0478
Sanger, CA, United States, 2014/2015 Golden Gem (small)	4.44	7	0.0050	0.0217	0.0184	0.0190	0.0429	0.0050	0.0209	0.0050	0.0103	0.0793
	4.4	61	0.0051	0.0616	0.0186	0.0379	0.0916	0.0051	0.0669	0.0051	0.0249	0.1889
	4.4	351	0.0051	0.0173	0.0186	0.0170	0.0257	0.0051	0.0249	0.0051	0.0071	0.0812
Paso Robles, CA, United States, 2014/2015 Red Cherry Large (small)	4.5	7	0.0050	0.0112	0.0181	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445
	4.5	63	0.0050	0.0112	0.0181	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445
	4.5	369	0.0050	0.0112	0.0181	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 148 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in spinach rotational crops

Location Year Variety	Total Rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54

Location Year Variety	Total Rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Richland, IA, United States, 2013/2015 SC Raccoon F1①	1.26	30	50 % Mature	<0.01	0.023	ND	<0.01	0.13	<0.01	<0.01	ND
				<0.01	0.030	ND	<0.01	0.17	<0.01	0.011	<0.01
			Mean	<0.01	0.0265	<0.01	<0.01	0.15	<0.01	<0.0105	<0.01
		30	Mature	ND	<0.01	ND	ND	0.022	ND	<0.01	ND
				ND	<0.01	ND	<0.01	0.027	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.0245	<0.01	<0.01	<0.01
		95	50 % Mature	ND	0.010	ND	ND	0.058	<0.01	<0.01	ND
				ND	0.011	ND	ND	0.065	<0.01	<0.01	ND
			Mean	<0.01	0.0105	<0.01	<0.01	0.0615	<0.01	<0.01	<0.01
		95	Mature	ND	<0.01	ND	ND	0.014	ND	ND	ND
				ND	ND	ND	ND	0.011	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.0125	<0.01	<0.01	<0.01
Richland, IA, United States, 2013/2015 SC Raccoon F1①	2.51	30	50 % Mature	<0.01	0.031	ND	0.011	0.19	0.014	0.011	<0.01
				<0.01	0.026	ND	<0.01	0.16	0.013	0.01[A]	<0.01
			Mean	<0.01	0.0285	<0.01	<0.0105	0.175	0.0135	0.0105	<0.01
		30	Mature	ND	<0.01	ND	<0.01	0.035	<0.01	<0.01	ND
				ND	<0.01	ND	<0.01	0.041	<0.01	<0.01	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.038	<0.01	<0.01	<0.01
	2.60	95	50 % Mature	ND	0.022	ND	<0.01	0.14	<0.01	<0.01	ND
				ND	0.023	ND	<0.01	0.14	<0.01	<0.01	ND
			Mean	<0.01	0.0225	<0.01	<0.01	0.14	<0.01	<0.01	<0.01
		95	Mature	ND	<0.01	<0.01	ND	0.037	ND	ND	ND
				ND	<0.01	ND	ND	0.037	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.037	<0.01	<0.01	<0.01
	2.51	270	50 % Mature	0.015	0.037	0.022	0.032	0.23	0.024	0.016	<0.01
				0.017	0.035	0.020	0.031	0.25	0.023	0.013	<0.01
			Mean	0.016	0.036	0.021	0.0315	0.24	0.0235	0.0145	<0.01
		270	Mature	0.010	0.019	0.013	0.020	0.14	0.031	0.011	<0.01
				<0.01	0.024	0.014	0.018	0.14	0.033	0.012	<0.01
			Mean	<0.01	0.0215	0.0135	0.019	0.14	0.032	0.0115	<0.01

Notes:

① Shepard 2020 DuPont-36791 rev 1

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 149 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in spinach rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year Variety	Total Rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Richland, IA, United States, 2013/2015 Spinach Raccoon F1	1.26	30	50 % Mature	0.0178	0.0471	0.0307	0.0178	0.2667	0.0178	0.0187	0.0178	0.6035
	1.26	30	Mature Leaf	0.0178	0.0178	0.0307	0.0178	0.0436	0.0178	0.0178	0.0178	0.1980
	1.26	95	50 % Mature	0.0178	0.0187	0.0307	0.0178	0.1093	0.0178	0.0178	0.0178	0.3000
	1.26	95	Mature	0.0178	0.0178	0.0307	0.0178	0.0222	0.0178	0.0178	0.0178	0.1656
Richland, IA, United	2.51	30	50 % Mature	0.0089	0.0254	0.0154	0.0094	0.1562	0.0120	0.0094	0.0089	0.3456

Location Year Variety	Total Rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
States, 2013/2015 Spinach SC Raccoon F1	2.51	30	Mature	0.0089	0.0089	0.0154	0.0089	0.0339	0.0089	0.0089	0.0089	0.1177
	2.6	95	50 % Mature	0.0086	0.0194	0.0149	0.0086	0.1206	0.0086	0.0086	0.0086	0.2716
	2.6	95	Mature	0.0086	0.0086	0.0149	0.0086	0.0319	0.0086	0.0086	0.0086	0.1123
	2.51	270	50 % Mature	0.0143	0.0321	0.0324	0.0281	0.2142	0.0210	0.0129	0.0089	0.4982
	2.51	270	Mature	0.0089	0.0192	0.0208	0.0170	0.1249	0.0286	0.0103	0.0089	0.3203

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 150 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis[D])

Location	Total Rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Richland, IA, United States, 2013/2015 SC Crunchy Royale F1①	1.26	30	Top	ND	0.015[A]	<0.01	0.018[A]	0.086	0.078	0.016	0.070
				ND	0.019[A]	<0.01	0.019[A]	0.11	0.091	0.020	0.080
			Mean	<0.01	0.017	<0.01	0.0185	0.098	0.0845	0.018	0.075
			Root	ND	ND	ND	<0.01	0.054	0.018	<0.01	<0.01
				ND	ND	<0.01	<0.01	0.062	0.020	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	0.058	0.019	<0.01	<0.01
	1.26	95	Top	ND	0.012[A]	ND	0.011[A]	0.064	0.054	0.012	0.044
				ND	0.018[A]	ND	0.016[A]	0.10	0.085	0.018	0.063
			Mean	<0.01	0.015	<0.01	0.0135	0.082	0.0695	0.015	0.0535
			Root	ND	ND	ND	ND	0.030	0.010	<0.01	<0.01
				ND	ND	<0.01	<0.01	0.046	0.015	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	0.038	0.0125	<0.01	<0.01
Richland, IA, United States, 2013/2015 SC Crunchy Royale F1①	2.51	30	Top	ND	0.022[A]	<0.01	0.020[A]	0.13	0.12	0.023	0.089
				ND	0.026I	<0.01[A]	0.024B	0.15[A]	0.13[A]	0.027[A]	0.10[A]
			Mean	<0.01	0.024	<0.01	0.022	0.14	0.125	0.025	0.0945
			Root	<0.01	ND	<0.01	<0.01	0.072	0.025	<0.01	<0.01
				<0.01	ND	<0.01	<0.01	0.082	0.029	<0.01	0.01[C]
			Mean	<0.01	<0.01	<0.01	<0.01	0.077	0.027	<0.01	<0.01
	2.60	95	Top	ND	0.027B	<0.01[A]	0.028B	0.15[A]	0.14[A]	0.029[A]	0.12[A]
				ND	0.021[A]	<0.01	0.024[A]	0.12	0.12	0.024	0.11
			Mean	<0.01	0.024	<0.01	0.026	0.135	0.13	0.0265	0.115
			Root	ND	ND	<0.01	<0.01	0.076	0.028	<0.01	<0.01
				ND	ND	<0.01	<0.01	0.058	0.024	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	0.067	0.026	<0.01	<0.01
	2.51	270	Top	ND	0.022[A]	<0.01	0.028[A]	0.083	0.11	0.023	0.13
				ND	0.022[A]	<0.01	0.025[A]	0.081	0.11	0.023	0.11
			Mean	<0.01	0.022	<0.01	0.0265	0.082	0.11	0.023	0.12
			Root	<0.01	ND	<0.01	<0.01	0.046	0.033	<0.01	<0.01
				<0.01	ND	<0.01	<0.01	0.062	0.043	<0.01	0.01[C]
			Mean	<0.01	<0.01	<0.01	<0.01	0.054	0.038	<0.01	<0.01
Porterville, CA, United States, 2013/2014 Crimson Giant①	1.25	7	Top	0.019	0.015	0.024	0.026	0.14	0.14	0.012	0.062
				0.018	0.015	0.023	0.025	0.12	0.15	0.013	0.060
			Mean	0.0185	0.015	0.0235	0.0255	0.13	0.145	0.0125	0.061
			Root	0.023	ND	0.018	0.019	0.13[A]	0.067[A]	<0.01	0.019[A]
				0.023	ND	0.019	0.020	0.13[A]	0.074[A]	<0.01	0.020[A]
			Mean	0.023	<0.01	0.0185	0.0195	0.13	0.0705	<0.01	0.0195
	1.25	60	Top	<0.01	0.028	<0.01	0.014	0.13	0.23	0.032	0.19
				ND	0.019	<0.01	<0.01	0.097	0.14	0.018	0.10

Location	Total Rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Mean	<0.01	0.0235	<0.01	<0.012	0.1135	0.185	0.025	0.145
			Root	<0.01	ND	<0.01	<0.01	0.14[A]	0.11[A]	0.010	0.067[A]
				ND	ND	<0.01	<0.01	0.12[A]	0.082[A]	<0.01	0.037[A]
			Mean	<0.01	<0.01	<0.01	<0.01	0.13	0.096	<0.01	0.052
	2.51	7	Top	0.016	0.11	0.032	0.11	0.43	0.92	0.10	1.6
				0.016	0.11	0.030	0.10	0.42	1.0	0.10	1.5
			Mean	0.016	0.11	0.031	0.105	0.425	0.96	0.10	1.55
			Root	0.016	<0.01	0.026	0.049	0.52	0.68	0.044	0.46
				0.017	<0.01	0.026	0.059	0.53	0.76	0.045	0.57
			Mean	0.0165	<0.01	0.026	0.054	0.525	0.72	0.0445	0.515
		60	Top	<0.01	0.057	0.018	0.061	0.24	0.53	0.058	0.71
				0.011	0.064	0.031	0.087	0.27	0.55	0.076	0.81
			Mean	<0.0105	0.0605	0.0245	0.074	0.255	0.54	0.067	0.76
			Root	0.013	<0.01	0.015	0.035	0.24	0.31	0.019	0.16[A]
				0.014	<0.01	0.018	0.054	0.35	0.37	0.027	0.19[A]
			Mean	0.0135	<0.01	0.0165	0.0445	0.295	0.34	0.023	0.175
		368	Top	<0.01	0.053	<0.01	0.034	0.19	0.36	0.070	0.76
				<0.01	0.050	<0.01	0.042	0.15	0.41	0.065	0.76
			Mean	<0.01	0.0515	<0.01	0.038	0.17	0.385	0.0675	0.76
			Root	<0.01	<0.01	0.010	0.041	0.29	0.22[A]	0.031	0.29
				ND	<0.01	<0.01	0.029	0.20	0.19[A]	0.024	0.16[A]
			Mean	<0.01	<0.01	<0.01	0.035	0.245	0.205	0.0275	0.225
Oviedo, FL, United States, 2014/2015 Rudolph OG②	4.49	28	Tops	ND	0.027	<0.01	0.044	0.10	0.31	0.017	0.29
				<0.01	0.027	<0.01	0.045	0.094	0.28	0.017	0.27
			Mean	<0.01	0.027	0.01	0.0445	0.097	0.295	0.017	0.28
	4.50	65		<0.01	0.038	<0.01	0.029	0.12	0.21	0.035	0.29
				<0.01	0.041	<0.01	0.028	0.10	0.21	0.038	0.31
			Mean	<0.01	0.0395	<0.01	0.0285	0.11	0.21	0.0365	0.30
	4.51	365		ND	<0.01	ND	ND	0.047	0.037	<0.01	0.047
				ND	<0.01	ND	ND	0.046	0.040	<0.01	0.043
			Mean	<0.01	<0.01	<0.01	<0.01	0.0465	0.0385	<0.01	0.045
	4.49	28	Roots	0.010	<0.01	<0.01	0.023	0.086	0.10	<0.01	0.033
				0.01[C]	ND	<0.01	0.021	0.068	0.087	<0.01	0.028
			Mean	0.010	<0.01	<0.01	0.022	0.077	0.0935	<0.01	0.0305
	4.50	65		0.015	ND	0.01[C]	0.029	0.081	0.093	<0.01	0.028
				0.014	ND	0.010	0.030	0.075	0.084	<0.01	0.025
			Mean	0.0145	<0.01	0.010	0.0295	0.078	0.0885	<0.01	0.0265
	4.51	365		ND	ND	ND	ND	0.017	0.013	ND	<0.01
				ND	ND	ND	ND	0.017	0.013	ND	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	0.017	0.013	<0.01	<0.01
Branchton, ON, Canada, 2014/2015 Champion②	4.67	9	Tops	0.011	0.046	0.013	0.017	0.27	0.35	0.035	0.27
				0.011	0.040	0.015	0.015	0.25	0.33	0.032	0.22
			Mean	0.011	0.043	0.014	0.016	0.26	0.34	0.0335	0.245
		68		<0.01	0.034	0.011	0.016	0.16	0.36	0.027	0.24
				<0.01	0.042	0.012	0.017	0.16	0.34	0.034	0.25
			Mean	<0.01	0.038	0.0115	0.0165	0.16	0.35	0.0305	0.245
		379		ND	0.015	<0.01	0.014	0.094	0.13	0.013	0.077
				<0.01	0.013	<0.01	0.012	0.084	0.11	0.011	0.073
			Mean	<0.01	0.014	0.01	0.013	0.089	0.12	0.012	0.075
		9	Roots	0.018	<0.01	0.013	0.016	0.16	0.11	0.011	0.021
				0.015	ND	0.012	0.014	0.14	0.10	<0.01	0.017
			Mean	0.0165	<0.01	0.0125	0.015	0.15	0.105	<0.0105	0.019
		68		<0.01	ND	<0.01	<0.01	0.13	0.074	0.011	0.016
				<0.01	<0.01	<0.01	<0.01	0.11	0.065	<0.01	0.013
			Mean	<0.01	<0.01	<0.01	<0.01	0.12	0.0695	<0.01	0.0145
		379		<0.01	ND	ND	<0.01	0.044	0.030	<0.01	<0.01
				<0.01	ND	<0.01	<0.01	0.045	0.031	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	0.0445	0.0305	<0.01	<0.01

Location	Total Rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Alpicat, Catalunya, Spain 2013 Blanco cumbre④	1.25	28	Tops	ND	0.014	0.010	<0.01	0.042	0.033	<0.01	0.040
	1.25	128		ND	0.013	0.011	<0.01	0.030	0.036	<0.01	0.047
	2.50	28		ND	0.012	<0.01	<0.01	0.031	0.038	<0.01	0.042
	2.50	142		ND	0.021	0.019	<0.01	0.092	0.071	<0.01	0.066
	2.50	243		ND	0.014	0.015	0.010	0.076	0.052	<0.01	0.038
	1.25	28	Roots	ND	<0.01	<0.01	ND	0.034	0.013	ND	0.012
	1.25	128		ND	ND	<0.01	ND	0.036	0.016	ND	0.010
	2.50	28		ND	ND	<0.01	ND	0.041	0.017	ND	0.011
	2.50	142		ND	<0.01	<0.01	ND	0.078	0.021	<0.01	0.017
	2.50	243		ND	ND	ND	ND	0.030	0.010	ND	<0.01
Aguadulce, Andalucia Spain 2013 Largo Rojo④	1.25	28	Tops	<0.01	ND	<0.01	ND	0.012	<0.01	ND	<0.01
	1.25	145		ND	ND	ND	ND	<0.01	<0.01	ND	ND
	2.50	27		<0.01	ND	<0.01	<0.01	0.011	<0.01	ND	ND
	2.50	145		ND	ND	ND	ND	0.011	<0.01	ND	ND
	2.50	242		<0.01	<0.01	<0.01	<0.01	0.028	0.014	ND	<0.01
	1.25	28	Roots	<0.01	ND	<0.01	<0.01	<0.01	<0.01	ND	ND
	1.25	145		<0.01	ND	<0.01	<0.01	0.011	<0.01	ND	ND
	2.50	27		0.018	ND	0.010	0.010	0.028	0.012	ND	<0.01
	2.50	145		<0.01	ND	<0.01	<0.01	0.018	<0.01	ND	ND
	2.50	242		0.016	ND	<0.01	0.010	0.034	0.014	ND	ND

Notes:

① Shepard 2020 DuPont-36791 rev 1.

② Shepard 2020 DuPont-40012 rev 1.

④Doig 2020 DuPont-36790 rev 1.

[A] Average of duplicate analyses.

[B] Average of triplicate analyses.

[C] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[D] For trials conducted in the United States, a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 151 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in radish rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year variety	Total Rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Branchton, ON, Canada, 2014/2015 Champion	4.67	9	Tops	0.0053	0.0206	0.0116	0.0336	0.0077	0.1247	0.1631	0.0161	0.1175	0.5069
	4.67	68	Tops	0.0048	0.0182	0.0095	0.0290	0.0079	0.0767	0.1679	0.0146	0.1175	0.4315
	4.67	379	Tops	0.0048	0.0067	0.0083	0.0155	0.0062	0.0427	0.0576	0.0058	0.0360	0.1845
	4.67	9	Roots	0.0079	0.0048	0.0104	0.0155	0.0072	0.0719	0.0504	0.0050	0.0091	0.2181
	4.67	68	Roots	0.0048	0.0048	0.0083	0.0134	0.0048	0.0576	0.0333	0.0050	0.0070	0.1662
	4.67	379	Roots	0.0048	0.0048	0.0083	0.0134	0.0048	0.0213	0.0146	0.0048	0.0048	0.0829
Alpicat, Catalunya, Spain 2013 Blanco cumbre	1.25	28	Tops	0.0179	0.0251	0.0310	0.0578	0.0179	0.0753	0.0591	0.0179	0.0717	0.3257
	1.25	128		0.0179	0.0233	0.0341	0.0589	0.0179	0.0538	0.0645	0.0179	0.0842	0.3036
	2.5	28		0.0090	0.0108	0.0155	0.0270	0.0090	0.0278	0.0340	0.0090	0.0376	0.1506
	2.5	142		0.0090	0.0188	0.0294	0.0495	0.0090	0.0824	0.0636	0.0090	0.0591	0.3259
	2.5	243		0.0090	0.0125	0.0232	0.0366	0.0090	0.0681	0.0466	0.0090	0.0340	0.2512
	1.25	28	Roots	0.0179	0.0179	0.0310	0.0501	0.0179	0.0609	0.0233	0.0179	0.0215	0.2336
	1.25	128		0.0179	0.0179	0.0310	0.0501	0.0179	0.0645	0.0287	0.0179	0.0179	0.2472
	2.5	28		0.0090	0.0090	0.0155	0.0250	0.0090	0.0367	0.0152	0.0090	0.0099	0.1318
	2.5	142		0.0090	0.0090	0.0155	0.0250	0.0090	0.0699	0.0188	0.0090	0.0152	0.1876
	2.5	243		0.0090	0.0090	0.0155	0.0250	0.0090	0.0269	0.0090	0.0090	0.0090	0.1073

Location Year variety	Total Rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Aguadulce, Andalucia Spain 2013 Largo Rojo	1.25	28	Tops	0.0179	0.0179	0.0310	0.0501	0.0179	0.0215	0.0179	0.0179	0.0179	0.1656
	1.25	145		0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	2.5	27		0.0090	0.0090	0.0155	0.0250	0.0090	0.0099	0.0090	0.0090	0.0090	0.0814
	2.5	145		0.0090	0.0090	0.0155	0.0250	0.0090	0.0099	0.0090	0.0090	0.0090	0.0814
	2.5	242		0.0090	0.0090	0.0155	0.0250	0.0090	0.0251	0.0125	0.0090	0.0090	0.1100
	1.25	28	Roots	0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	1.25	145		0.0179	0.0179	0.0310	0.0501	0.0179	0.0197	0.0179	0.0179	0.0179	0.1628
	2.5	27		0.0161	0.0090	0.0155	0.0250	0.0090	0.0251	0.0108	0.0090	0.0090	0.1073
	2.5	145		0.0090	0.0090	0.0155	0.0250	0.0090	0.0161	0.0090	0.0090	0.0090	0.0910
	2.5	242		0.0143	0.0090	0.0155	0.0250	0.0090	0.0305	0.0125	0.0090	0.0090	0.1182
Richland, IA, United States, 2013/2015 Radish SC Crunchy Royale F1	1.26	30	tops	0.0178	0.0302	0.0307	0.0630	0.0329	0.1742	0.1502	0.0320	0.1333	0.6248
	1.26	30	Roots	0.0178	0.0178	0.0307	0.0497	0.0178	0.1031	0.0338	0.0178	0.0178	0.3127
	1.26	95	Tops	0.0178	0.0267	0.0307	0.0592	0.0240	0.1458	0.1236	0.0267	0.0951	0.5332
	1.26	95	Roots	0.0178	0.0178	0.0307	0.0497	0.0178	0.0676	0.0222	0.0178	0.0178	0.2412
	2.51	30	tops	0.0089	0.0214	0.0154	0.0383	0.0196	0.1249	0.1116	0.0223	0.0843	0.4393
	2.51	30	roots	0.0089	0.0089	0.0154	0.0249	0.0089	0.0687	0.0241	0.0089	0.0089	0.1935
	2.6	95	tops	0.0086	0.0207	0.0149	0.0370	0.0224	0.1163	0.1120	0.0228	0.0991	0.4240
	2.6	95	roots	0.0086	0.0086	0.0149	0.0241	0.0086	0.0577	0.0224	0.0086	0.0086	0.1724
	2.51	270	tops	0.0089	0.0196	0.0154	0.0364	0.0236	0.0732	0.0982	0.0205	0.1071	0.3364
	2.51	270	Roots	0.0089	0.0089	0.0154	0.0249	0.0089	0.0482	0.0339	0.0089	0.0089	0.1772
Porterville, CA, United States, 2013/2014 10 (Crimson Giant)	2.51	7	tops	0.0165	0.0134	0.0362	0.0505	0.0228	0.1160	0.1294	0.0112	0.0544	0.4784
	2.51	7	roots	0.0205	0.0089	0.0285	0.0381	0.0174	0.1160	0.0629	0.0089	0.0174	0.3517
	1.25	60	Tops	0.0179	0.0421	0.0310	0.0759	0.0215	0.2034	0.3315	0.0448	0.2598	0.9702
	1.25	60	Roots	0.0179	0.0179	0.0310	0.0501	0.0179	0.2330	0.1720	0.0179	0.0932	0.7197
	2.51	7	tops	0.0143	0.0982	0.0478	0.1526	0.0937	0.3793	0.8567	0.0892	1.383	2.193
	2.51	7	roots	0.0147	0.0089	0.0401	0.0496	0.0482	0.4685	0.6425	0.0397	0.4596	1.787
	2.51	60	tops	0.0094	0.0540	0.0378	0.0954	0.0660	0.2276	0.4819	0.0598	0.6782	1.2753
	2.51	60	roots	0.0120	0.0089	0.0254	0.0350	0.0397	0.2633	0.3034	0.0205	0.1562	0.9322
	2.51	368	tops	0.0089	0.0460	0.0154	0.0645	0.0339	0.1517	0.3436	0.0602	0.6782	0.8858
	2.51	368	Roots	0.0089	0.0089	0.0154	0.0249	0.0312	0.2186	0.1829	0.0245	0.2008	0.6613
Oviedo, FL, United States, 2014/2015 Rudolph OG	4.49	28	Tops	0.0050	0.0135	0.0086	0.0230	0.0222	0.0484	0.1472	0.0085	0.1397	0.3444
	4.5	65	Tops	0.0050	0.0197	0.0086	0.0296	0.0142	0.0548	0.1045	0.0182	0.1493	0.3037
	4.51	365	Tops	0.0050	0.0050	0.0086	0.0139	0.0050	0.0231	0.0191	0.0050	0.0224	0.0933
	4.49	28	Roots	0.0050	0.0050	0.0086	0.0139	0.0110	0.0384	0.0466	0.0050	0.0152	0.1583
	4.5	65	Roots	0.0072	0.0050	0.0086	0.0139	0.0147	0.0388	0.0441	0.0050	0.0132	0.1549
	4.51	365	Roots	0.0050	0.0050	0.0086	0.0139	0.0050	0.0084	0.0065	0.0050	0.0050	0.0519

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 152 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis[C]) in lettuce rotational crops

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Alpicat, Catalunya, Spain 2013 Maravilla de Verano④	1.25	28	Immature Whole Plant	ND	ND	ND	ND	ND	<0.01	ND	ND
	1.25	128		ND	ND	ND	ND	ND	ND	ND	ND
	2.50	28		ND	ND	0.011	ND	ND	0.054	ND	ND
	2.50	142		ND	ND	<0.01	ND	ND	0.023	ND	ND
	2.50	243		ND	ND	ND	ND	ND	ND	ND	ND
	1.25	28	Mature Whole Plant	ND	ND	ND	ND	ND	ND	ND	ND
	1.25	128		ND	ND	ND	ND	ND	<0.01	ND	ND
	2.50	28		<0.01	ND	ND	<0.01	ND	0.024	ND	ND
	2.50	142		ND	ND	ND	ND	ND	ND	ND	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
	2.50	243		ND	ND	ND	ND	ND	<0.01	ND	ND
Aguadulce, Andalucia Spain 2013 Isasa④	1.25	28	Immature Whole Plant	ND	ND	ND	ND	ND	ND	ND	ND
	1.25	127		ND	ND	ND	ND	ND	ND	ND	ND
	2.50	27		ND	ND	ND	ND	ND	<0.01	ND	ND
	2.50	145		ND	ND	ND	ND	ND	<0.01	ND	ND
	2.50	242		ND	<0.01	<0.01	<0.01	ND	<0.01	ND	ND
	1.25	28	Mature Whole Plant	ND	ND	ND	ND	ND	ND	ND	ND
	1.25	127		ND	ND	ND	ND	ND	ND	ND	ND
	2.50	27		ND	ND	ND	ND	ND	ND	ND	ND
	2.50	145		ND	ND	ND	ND	ND	<0.01	ND	ND
	2.50	242		ND	ND	ND	ND	ND	ND	ND	ND
Lucenay, Rhone Alpes, France 2015 Batavia Dedale⑤	4.49	7	Immature whole plant	ND	ND	ND	ND	ND	0.018	ND	ND
	4.44	66		ND	ND	ND	ND	ND	0.011	ND	ND
	4.48	361		ND	ND	ND	ND	ND	ND	ND	ND
	4.49	7	Mature whole plant	ND	ND	<0.01	ND	ND	0.023	ND	ND
	4.44	66		ND	ND	ND	ND	ND	<0.01	ND	ND
	4.48	361		ND	ND	ND	ND	ND	ND	ND	ND
Lleida, Catalunya, Spain 2014 Maravillo de Verano⑤	4.42	7	Immature whole plant	<0.01	ND	ND	<0.01	ND	0.068	ND	ND
	4.39	63		<0.01	ND	ND	<0.01	ND	0.046	ND	ND
	4.45	361		ND	ND	ND	<0.01	ND	0.012	ND	ND
	4.42	7	Mature whole plant	ND	ND	ND	ND	ND	0.014	ND	ND
	4.39	63		<0.01	ND	ND	ND	ND	0.010	ND	ND
	4.45	361		ND	ND	ND	ND	ND	<0.01	ND	ND
Termens, Catalunya, Spain 2014 Maravilla de Verano⑤	4.49	10	Immature whole plant	<0.01	ND	<0.01	<0.01	ND	0.011	ND	ND
	4.43	60		ND	ND	ND	ND	ND	0.021	ND	ND
Termens, Catalunya, Spain 2014 Teresa⑤	4.44	367	Immature whole plant	ND	ND	ND	ND	ND	0.023	ND	ND
Termens, Catalunya, Spain 2014 Maravilla de Verano⑤	4.49	10	Mature whole plant	<0.01	ND	ND	ND	ND	<0.01	ND	ND
	4.43	60		ND	ND	ND	ND	ND	<0.01	ND	ND
Termens, Catalunya, Spain 2014 Teresa⑤	4.44	367	Mature whole plant	ND	ND	ND	ND	ND	0.011	ND	ND
Los Palacios, Andalucía, Spain 2014 Filipus⑤	4.51	8	Immature whole plant	0.011	ND	<0.01	<0.01	ND	0.025	ND	ND
	4.40	62		0.012	ND	<0.01	<0.01	ND	0.035	ND	ND
	4.48	363		ND	ND	ND	ND	ND	0.013	ND	ND
	4.51	8	Mature whole plant	<0.01	ND	<0.01	ND	ND	<0.01	ND	ND
	4.40	62		<0.01	ND	<0.01	ND	ND	0.011	ND	ND
	4.48	363		ND	ND	ND	ND	ND	0.015	ND	ND
Aguadulce, Andalucía, Spain 2014 Issa⑤	4.50	7	Immature whole plant	<0.01	ND	ND	<0.01	ND	ND	ND	ND
	4.41	63		<0.01	ND	<0.01	0.020	ND	0.010	ND	ND
	4.44	365		<0.01	ND	0.012	ND	<0.01	0.040	ND	ND
	4.50	7	Mature whole plant	ND	ND	ND	ND	ND	ND	ND	ND
	4.41	63		ND	ND	ND	ND	ND	0.012	ND	ND
	4.44	365		ND	ND	<0.01	ND	ND	0.028	ND	ND
Oviedo, FL, United States, 2014/2015 Buttercrunch②	4.50	7	Leaf	<0.01	ND	<0.01	0.022	<0.01	0.15	ND	ND
				<0.01	ND	<0.01	0.027	<0.01	0.17	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0245	<0.01	0.16	<0.01	<0.01
	4.54	60	Leaf	<0.01	ND	<0.01	0.013	<0.01	0.21	ND	ND
				<0.01	<0.01	0.011	0.013	<0.01	0.18	ND	ND
			Mean	<0.01	<0.01	<0.0105	0.013	<0.01	0.195	<0.01	<0.01
	4.55	378	Leaf	ND	ND	ND	ND	ND	0.070	ND	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
				ND	ND	ND	ND	ND	0.066	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.068	<0.01	<0.01
Branchton, ON, Canada, 2014/2015 Butterhead②	4.56	9	Leaf	<0.01	ND	<0.01	<0.01	ND	0.15	ND	ND
				<0.01	ND	<0.01	ND	<0.01	0.17	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.16	<0.01	<0.01
		68	Leaf	<0.01	ND	<0.01	<0.01	<0.01	0.19	ND	ND
				<0.01	ND	<0.01	<0.01	ND	0.17	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.18	<0.01	<0.01
		365	Leaf	ND	ND	ND	ND	ND	0.10	ND	ND
				ND	ND	ND	ND	ND	0.11	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.105	<0.01	<0.01
Paso Robles, CA, United States, 2014/2015 Bergram's②	4.50	7	Leaf	<0.01	ND	<0.01	<0.01	ND	0.052	ND	ND
				<0.01	ND	<0.01	<0.01	ND	0.048	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.050	<0.01	<0.01
Paso Robles, CA, United States, 2014/2015 Imperial②		63	Leaf	ND	ND	ND	ND	ND	0.015	ND	ND
				ND	ND	<0.01	ND	ND	0.019	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.017	<0.01	<0.01
Paso Robles, CA, United States, 2014/2015 Heartbreaker②		369	Leaf	ND	ND	<0.01	<0.01	0.011	0.17	ND	ND
				ND	ND	<0.01	<0.01	0.013	0.17	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.012	0.17	<0.01	<0.01
Porterville, CA, United States, 2014/2015 Marin②	4.48	8	Leaf	0.013	ND	0.013	0.01[B]	<0.01	0.077	ND	ND
				0.017	ND	0.014	0.011	<0.01	0.11	ND	ND
			Mean	0.015	<0.01	0.0135	0.0105	<0.01	0.0935	<0.01	<0.01
		61	Leaf	<0.01	ND	<0.01	<0.01	<0.01	0.14	ND	ND
				<0.01	ND	<0.01	<0.01	<0.01	0.14	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.14	<0.01	<0.01
		203	Leaf	<0.01	ND	<0.01	<0.01	ND	0.16	ND	ND
				<0.01	ND	<0.01	<0.01	ND	0.18	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.17	<0.01	<0.01
Sanger, CA, United States, 2014/2015 Tropicana②	4.61	7	Leaf	ND	ND	ND	ND	<0.01	0.17	ND	ND
				ND	ND	<0.01	ND	<0.01	0.17	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.17	<0.01	<0.01
	4.60	59	Leaf	ND	ND	ND	ND	<0.01	0.11	ND	ND
				ND	ND	ND	ND	ND	0.10	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.105	<0.01	<0.01
	4.54	363	Leaf	ND	ND	ND	ND	ND	0.038	ND	ND
				ND	ND	ND	ND	<0.01	0.055	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0465	<0.01	<0.01
Porterville, CA, United States, 2013/2014 Red Sails①	1.26	7	50 % Mature	0.029	0.031	0.032	0.058	0.051	1.5	ND	<0.01
			Leaf	0.035	0.041	0.055	0.072	0.92	1.7[A]	ND	<0.01
			Mean	0.032	0.036	0.0435	0.065	0.04855	1.6	<0.01	<0.01
		7	Mature	0.012	0.01	0.014	0.018	0.020	0.57	ND	ND
			Leaf	0.018	0.015	0.029	0.032	0.028	0.96	ND	ND
			Mean	0.015	0.0125	0.0215	0.025	0.024	0.765	<0.01	<0.01
		60	50 %	ND	ND	ND	ND	ND	0.028	ND	ND
			Mature Leaf	ND	ND	ND	ND	ND	0.024	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.026	<0.01	<0.01
		60	Mature Leaf	ND	ND	ND	ND	ND	0.033	ND	ND
				ND	ND	ND	ND	ND	0.032	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0325	<0.01	<0.01
	2.51	7	50 % Mature	<0.01	0.013	0.015	0.011	0.015	0.97	ND	ND
			Leaf	<0.01	0.011	0.012	0.01	0.014	0.92	ND	ND
			Mean	<0.01	0.012	0.0135	0.0105	0.0145	0.945	<0.01	<0.01
		7	Mature	ND	<0.01	<0.01	<0.01	<0.01	0.23	ND	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Leaf	ND	<0.01	<0.01	ND	<0.01	0.26	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.245	<0.01	<0.01
		60	50 % Mature	<0.01	0.010	0.013	<0.01	0.027	0.77	ND	ND
			Leaf	<0.01	0.019	0.020	0.018	0.040	1.1	ND	ND
			Mean	<0.01	0.0145	0.0165	0.014	0.0335	0.935	<0.01	<0.01
		60	Mature	ND	ND	<0.01	ND	<0.01	0.15	ND	ND
			Leaf	ND	<0.01	<0.01	<0.01	0.013	0.31	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.0115	0.23	<0.01	<0.01
		361	50 % Mature	ND	ND	ND	ND	ND	0.031	ND	ND
			Leaf	ND	ND	ND	ND	<0.01	0.072	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0515	<0.01	<0.01
		361	Mature	ND	ND	ND	ND	<0.01	0.071	ND	ND
			Leaf	ND	ND	ND	ND	ND	0.033	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0515	<0.01	<0.01

Notes:

①Shepard 2020 DuPont-36791 rev 1.

②Shepard 2020 DuPont-40012 rev 1.

④Doig 2020 DuPont-36790 rev 1.

⑤Doig 2020 DuPont-41762.

[A] Average of duplicate analyses.

[B] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[C] For trials conducted in the United States, a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 153 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post hydrolysis) in lettuce rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location, Year, Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Branchton, ON, Canada,	4.56	9	Leaf	0.0049	0.0049	0.0085	0.0049	0.0049	0.0786	0.0049	0.0049	0.1552
2014/2015	4.56	68		0.0049	0.0049	0.0085	0.0049	0.0049	0.0884	0.0049	0.0049	0.1700
Butterhead	4.56	365		0.0049	0.0049	0.0085	0.0049	0.0049	0.0516	0.0049	0.0049	0.1144
Alpicat,	1.25	28	Lettuce/	0.0179	0.0179	0.0310	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
Catalunya,	1.25	128	Immature	0.0179	0.0179	0.0310	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
Spain 2013	2.5	28	Whole Plant	0.0090	0.0090	0.0170	0.0090	0.0090	0.0484	0.0090	0.0090	0.1429
Maravilla de	2.5	142		0.0090	0.0090	0.0155	0.0090	0.0090	0.0206	0.0090	0.0090	0.0976
Verano	2.5	243		0.0090	0.0090	0.0155	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	1.25	28	Mature Whole	0.0179	0.0179	0.0310	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	1.25	128	Plant	0.0179	0.0179	0.0310	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	2.5	28		0.0090	0.0090	0.0155	0.0090	0.0090	0.0215	0.0090	0.0090	0.0990
	2.5	142		0.0090	0.0090	0.0155	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	243		0.0090	0.0090	0.0155	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
Aguadulce,	1.25	28	Immature	0.0179	0.0179	0.0310	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
Andalucia	1.25	127	Whole Plant	0.0179	0.0179	0.0310	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
Spain 2013	2.5	27		0.0090	0.0090	0.0155	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
Isasa	2.5	145		0.0090	0.0090	0.0155	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	242		0.0090	0.0090	0.0155	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	1.25	28	Mature Whole	0.0179	0.0179	0.0310	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	1.25	127	Plant	0.0179	0.0179	0.0310	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601

Location, Year, Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
	2.5	27		0.0090	0.0090	0.0155	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	145		0.0090	0.0090	0.0155	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	242		0.0090	0.0090	0.0155	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
Lucenay, Rhone Alpes, France 2015 Batavia Dedale	4.49	7	Immature whole plant	0.0050	0.0050	0.0086	0.0050	0.0050	0.0090	0.0050	0.0050	0.0506
	4.44	66		0.0050	0.0050	0.0087	0.0050	0.0050	0.0055	0.0050	0.0050	0.0458
	4.48	361		0.0050	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0447
	4.49	7	Mature whole plant	0.0050	0.0050	0.0086	0.0050	0.0050	0.0115	0.0050	0.0050	0.0544
	4.44	66		0.0050	0.0050	0.0087	0.0050	0.0050	0.0050	0.0050	0.0050	0.0451
	4.48	361		0.0050	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0447
Lleida, Catalunya, Spain 2014 Maravillo de Verano	4.42	7	Immature whole plant	0.0051	0.0051	0.0088	0.0051	0.0051	0.0345	0.0051	0.0051	0.0897
	4.39	63		0.0051	0.0051	0.0088	0.0051	0.0051	0.0235	0.0051	0.0051	0.0733
	4.45	361		0.0050	0.0050	0.0087	0.0050	0.0050	0.0060	0.0050	0.0050	0.0465
	4.42	7	Mature whole plant	0.0051	0.0051	0.0088	0.0051	0.0051	0.0071	0.0051	0.0051	0.0483
	4.39	63		0.0051	0.0051	0.0088	0.0051	0.0051	0.0051	0.0051	0.0051	0.0456
	4.45	361		0.0050	0.0050	0.0087	0.0050	0.0050	0.0050	0.0050	0.0050	0.0450
Termens, Catalunya, Spain 2014 Maravilla de Verano	4.49	10	Immature whole plant	0.0050	0.0050	0.0086	0.0050	0.0050	0.0055	0.0050	0.0050	0.0453
	4.43	60		0.0051	0.0051	0.0087	0.0051	0.0051	0.0106	0.0051	0.0051	0.0536
Termens, Catalunya, Spain 2014 Teresa	4.44	367	Immature whole plant	0.0050	0.0050	0.0087	0.0050	0.0050	0.0116	0.0050	0.0050	0.0550
Termens, Catalunya, Spain 2014 Maravilla de Verano	4.49	10	Mature whole plant	0.0050	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0446
	4.43	60		0.0051	0.0051	0.0087	0.0051	0.0051	0.0051	0.0051	0.0051	0.0452
Termens, Catalunya, Spain 2014 Teresa	4.44	367	Mature whole plant	0.0050	0.0050	0.0087	0.0050	0.0050	0.0055	0.0050	0.0050	0.0458
Los Palacios, Andalucía, Spain 2014 Filipus	4.51	8	Immature whole plant	0.0055	0.0050	0.0086	0.0050	0.0050	0.0124	0.0050	0.0050	0.0556
	4.4	62		0.0061	0.0051	0.0088	0.0051	0.0051	0.0178	0.0051	0.0051	0.0647
	4.48	363		0.0050	0.0050	0.0086	0.0050	0.0050	0.0065	0.0050	0.0050	0.0469
	4.51	8	Mature whole plant	0.0050	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0444
	4.4	62		0.0051	0.0051	0.0088	0.0051	0.0051	0.0056	0.0051	0.0051	0.0463
	4.48	363		0.0050	0.0050	0.0086	0.0050	0.0050	0.0075	0.0050	0.0050	0.0485
Aguadulce, Andalucía, Spain 2014 Issa	4.5	7	Immature whole plant	0.0050	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445
	4.41	63		0.0051	0.0051	0.0088	0.0102	0.0051	0.0051	0.0051	0.0051	0.0454
	4.44	365		0.0050	0.0050	0.0105	0.0050	0.0050	0.0202	0.0050	0.0050	0.0716
	4.5	7	Mature whole plant	0.0050	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445
	4.41	63		0.0051	0.0051	0.0088	0.0051	0.0051	0.0061	0.0051	0.0051	0.0469
	4.44	365		0.0050	0.0050	0.0087	0.0050	0.0050	0.0141	0.0050	0.0050	0.0588
Oviedo, FL, United States, 2014/2015 Buttercrunch	4.5	7	Leaf	0.0050	0.0050	0.0086	0.0122	0.0050	0.0796	0.0050	0.0050	0.1572
	4.54	60	Leaf	0.0049	0.0049	0.0090	0.0064	0.0049	0.0962	0.0049	0.0049	0.1828
	4.55	378	Leaf	0.0049	0.0049	0.0085	0.0049	0.0049	0.0335	0.0049	0.0049	0.0871
		68		0.0049	0.0049	0.0085	0.0049	0.0049	0.0884	0.0049	0.0049	0.1700
		365		0.0049	0.0049	0.0085	0.0049	0.0049	0.0516	0.0049	0.0049	0.1144
Paso Robles, CA, United States, 2014/2015 Bergram's	4.5	7	Leaf	0.0050	0.0050	0.0086	0.0050	0.0050	0.0249	0.0050	0.0050	0.0745
Paso Robles, CA, United States,	4.5	63	Leaf	0.0050	0.0050	0.0086	0.0050	0.0050	0.0085	0.0050	0.0050	0.0497

Location, Year, Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
2014/2015 Imperial												
Paso Robles, CA, United States, 2014/2015 Heartbreaker	4.5	369	Leaf	0.0050	0.0050	0.0086	0.0050	0.0060	0.0846	0.0050	0.0050	0.1663
Porterville, CA, United States, 2014/2015 Marin	4.48	8	Leaf	0.0075	0.0050	0.0117	0.0053	0.0050	0.0468	0.0050	0.0050	0.1141
	4.48	61		0.0050	0.0050	0.0086	0.0050	0.0050	0.0700	0.0050	0.0050	0.1428
	4.48	203		0.0050	0.0050	0.0086	0.0050	0.0000	0.0850	0.0050	0.0050	0.1579
Sanger, CA, United States, 2014/2015 Tropicana	4.61	7	Leaf	0.0049	0.0049	0.0084	0.0049	0.0049	0.0826	0.0049	0.0049	0.1608
	4.6	59		0.0049	0.0049	0.0084	0.0049	0.0049	0.0511	0.0049	0.0049	0.1134
	4.54	363		0.0049	0.0049	0.0085	0.0049	0.0049	0.0229	0.0049	0.0049	0.0713
Porterville, CA, United States, 2013/2014 Red Sails	1.26	7	50 % mature leaf	0.0569	0.0640	0.1336	0.1156	0.8631	2.8444	0.0178	0.0178	6.0336
	1.26	7	Mature leaf	0.0267	0.0222	0.0660	0.0444	0.0427	1.3600	0.0178	0.0178	2.3080
	1.26	60	50 % mature Leaf	0.0178	0.0178	0.0307	0.0178	0.0178	0.0462	0.0178	0.0178	0.2018
	1.26	60	Mature Leaf	0.0178	0.0178	0.0307	0.0178	0.0178	0.0578	0.0178	0.0178	0.2193
	2.51	7	50 % mature Leaf	0.0089	0.0107	0.0208	0.0094	0.0129	0.8433	0.0089	0.0089	1.3612
	2.51	7	Mature Leaf	0.0089	0.0089	0.0154	0.0089	0.0089	0.2186	0.0089	0.0089	0.3964
	2.51	60	50 % mature Leaf	0.0089	0.0129	0.0254	0.0125	0.0229	0.8344	0.0089	0.0089	1.3883
	2.51	60	Mature Leaf	0.0089	0.0089	0.0154	0.0089	0.0103	0.2053	0.0089	0.0089	0.3782
	2.51	361	50 % mature Leaf	0.0089	0.0089	0.0154	0.00189	0.0089	0.0460	0.0089	0.0089	0.1357
	2.51	361	Mature Leaf	0.0089	0.0089	0.0154	0.0089	0.0089	0.0464	0.0089	0.0089	0.1363

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 154 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post hydrolysis[A]) in Swiss chard rotational crops

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Fluaza indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TDQ54
Branchton, ON, Canada, 2014/2015 Peppermint (Swiss chard) ②	4.57	9	0.016 0.017 (0.0165)	0.025 0.027 (0.026)	0.022 0.023 (0.0225)	0.015 0.017 0.016)	0.079 0.079 (0.079)	<0.01 0.010 <0.01)	0.020 0.021 (0.0205)	ND ND <0.01)
		68	<0.01 <0.01 (<0.01)	0.013 0.014 (0.0135)	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	0.038 0.041 (0.0395)	ND ND (<0.01)	0.012 0.013 (0.0125)	ND ND <0.01)
		366	ND ND (<0.01)	0.016 0.016 (0.016)	ND <0.01 (<0.01)	<0.01 <0.01 (<0.01)	0.041 0.034 (0.0375)	<0.01 <0.01 (<0.01)	0.014 0.014 (0.014)	ND ND <0.01)
Lucenay, Rhone Alpes, France 2015 Blanche 2 (Swiss chard) ⑤	4.44	7	ND	ND	ND	ND	ND	ND	ND	ND
	4446	65	ND	ND	ND	ND	ND	ND	ND	ND
	4.47	363	ND	ND	ND	ND	ND	ND	ND	ND
Lleida, Catalunya, Spain 2014 Amarilla de Lyon Selga (Swiss chard) ⑤	4.42	7	0.010	ND	<0.01	0.013	ND	ND	ND	<0.01
	4.39	63	<0.01	ND	<0.01	0.014	<0.01	ND	<0.01	<0.01
	4.44	361	ND	ND	ND	ND	<0.01	ND	<0.01	ND
Termens, Catalunya, Spain 2014 Amarillo de Lyon Selga	4.47	10	ND	ND	ND	<0.01	ND	ND	ND	ND
	4.43	60	ND	ND	<0.01	ND	<0.01	ND	<0.01	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Fluaza indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TDQ54
(Swiss chard) ⑤	4.47	367	ND	ND	ND	ND	ND	ND	ND	ND
Porterville, CA, United States, 2014/2015 Large white ribbed (Swiss chard) ②	4.51	67	<0.01 <0.01 (<0.01)	ND ND (<0.01)	ND ND (<0.01)	<0.01 ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)
		226	0.013 0.018 (0.0155)	0.016 0.017 (0.0165)	0.023 0.032 (0.0275)	0.014 0.017 (0.0155)	0.031 0.035 (0.033)	ND <0.01 (<0.01)	<0.01 ND (<0.01)	ND ND (<0.01)

Notes:

② Shepard (2020 DuPont-40012 rev 1.

⑤ Doig 2020 DuPont-41762.

[A] For trials conducted in the United States, a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 155 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post hydrolysis) and related compounds in Swiss chard stalk rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Branchton, ON, Canada, 2014/2015 Peppermint	4.57	9	0.0081	0.0127	0.0191	0.0078	0.0387	0.0049	0.0100	0.0049	0.1353
	4.57	68	0.0049	0.0066	0.0085	0.0049	0.0194	0.0049	0.0061	0.0049	0.0697
	4.57	366	0.0049	0.0078	0.0042	0.0049	0.0184	0.0049	0.0069	0.0049	0.0620
Porterville, CA, United States, 2014/2015 Large white ribbed	4.51	67	0.0050	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0444
	4.51	226	0.0077	0.0082	0.0236	0.0077	0.0164	0.0050	0.0050	0.0050	0.1007
Lucenay, Rhone Alpes, France 2015 Blanche 2	4.44	7	0.0050	0.0050	0.0087	0.0050	0.0050	0.0050	0.0050	0.0050	0.0451
	4.46	65	0.0050	0.0050	0.0087	0.0050	0.0050	0.0058	0.0050	0.0050	0.0460
	4.47	363	0.0050	0.0050	0.0087	0.0050	0.0050	0.0050	0.0050	0.0050	0.0448
Lleida, Catalunya, Spain 2014 Amarilla de Lyon Selga	4.42	7	0.0051	0.0051	0.0088	0.0066	0.0051	0.0051	0.0051	0.0051	0.0453
	4.39	63	0.0051	0.0051	0.0088	0.0071	0.0051	0.0051	0.0051	0.0051	0.0456
	4.44	361	0.0050	0.0050	0.0087	0.0050	0.0050	0.0050	0.0050	0.0050	0.0451
Termens, Catalunya, Spain 2014 Amarillo de Lyon Selga	4.47	10	0.0050	0.0050	0.0087	0.0050	0.0050	0.0050	0.0050	0.0050	0.0448
	4.43	60	0.0051	0.0051	0.0087	0.0051	0.0051	0.0051	0.0051	0.0051	0.0452
	4.47	367	0.0050	0.0050	0.0087	0.0050	0.0050	0.0050	0.0050	0.0050	0.0448

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 156 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post hydrolysis) in broccoli head rotational crops

Location Year, variety	Total rate (kg ai/ha)	PBI (days)	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Lucenay, Rhone Alpes, France 2015 Belstar⑤	4.35	7	ND	ND	ND	<0.01	ND	ND	ND	ND
	4.45	65	ND	ND	ND	<0.01	<0.01	<0.01	ND	ND
	4.50	361	ND	ND	ND	ND	ND	ND	ND	ND
Lleida, Catalunya, Spain 2014 Trevi⑤	4.42	7	<0.01	ND	<0.01	0.016	0.015	0.013	ND	ND
	4.39	63	0.011	ND	<0.01	0.017	0.016	0.018	ND	ND

Location Year, variety	Total rate (kg ai/ha)	PBI (days)	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
	4.46	361	ND	ND	ND	<0.01	<0.01	ND	ND	ND
Termens, Catalunya, Spain 2014 Trevi⑤	4.50	10	0.016	ND	<0.01	0.011	0.019	<0.01	ND	ND
	4.43	60	ND	ND	ND	ND	0.012	<0.01	ND	ND
Termens, Catalunya, Spain 2014 Verdz⑤	4.45	367	ND	ND	<0.01	ND	<0.01	ND	ND	ND
Los Palacios, Andalucía, Spain 2014 Marathon⑤	4.46	8	<0.01	ND	<0.01	<0.01	<0.01	<0.01	ND	ND
	4.41	62	<0.01	ND	<0.01	<0.01	<0.01	<0.01	ND	ND
	4.35	363	ND	ND	<0.01	ND	<0.01	ND	ND	ND
Aguadulce, Andalucía, Spain 2014 Parthenon⑤	4.46	7	0.017	ND	<0.01	<0.01	ND	ND	ND	ND
	4.44	63	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND
	4.52	365	ND	ND	ND	ND	ND	ND	ND	ND
Oviedo, FL, United States, 2014/2015 Packman②	4.49	7	0.014 0.033 (0.0235)	ND <0.01 (<0.01)	0.01[A] 0.020 (0.015)	0.058 0.11 (0.084)	0.025 0.032 (0.0285)	0.023 0.037 (0.030)	ND ND (<0.01)	0.015 0.023 (0.019)
	4.52	60	0.014 0.014 (0.014)	ND ND (<0.01)	0.01[A] <0.01 (<0.01)	0.028 0.024 (0.026)	0.019 0.016 (0.0175)	0.013 0.011 (0.012)	ND ND (<0.01)	<0.01 <0.01 (<0.01)
	4.48	365	ND ND (<0.01)	ND ND (<0.01	ND ND (<0.01	ND ND (<0.01	<0.01 <0.01 (<0.0)	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)
Fresno, CA, United States, 2014-2016 Imperial②	4.41	7	0.016 0.015 (0.0155)	ND ND (<0.01	0.015 0.012 (0.0135)	0.025 0.025 (0.025)	0.044 0.043 (0.0435)	0.024 0.027 (0.0255)	ND ND (<0.01)	<0.01 <0.01 (<0.01)
	4.39	63	ND ND (<0.01)	ND ND (<0.01	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01	0.014 0.013 (0.0135)	0.011 <0.01 (<0.0105)	ND ND (<0.01)	ND ND (<0.01)
Fresno, CA, United States, 2014-2016 Tradition②	4.42	365	ND ND (<0.01	ND ND (<0.01	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)
Paso Robles, CA, United States, 2014/2015 Heritage②	4.50	7	<0.01 <0.01 (<0.01)	ND ND (<0.01)	ND ND (<0.01)	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	0.01[A] <0.01 (<0.01)	ND ND (<0.01)	<0.01 <0.01 (<0.01)
Paso Robles, CA, United States, 2014/2015 Imperial②	4.50	63	<0.01 <0.01 (<0.01)	ND ND (<0.01	ND ND (<0.01)	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	ND ND (<0.01)	ND ND (<0.01)
		369	0.01[A] <0.01 (<0.01)	ND ND (<0.01	0.01[A] <0.01 (<0.01)	0.019 0.017 (0.018)	0.039 0.036 (0.0375)	0.019 0.015 (0.017)	ND ND (<0.01)	<0.01 <0.01 (<0.01)
Porterville, CA, United States, 2014/2015 Heritage②	4.49	7	0.01[A] <0.01 (<0.01)	ND ND (<0.01)	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	0.011 <0.01 <0.0105)	<0.01 <0.01 (<0.01)	ND ND (<0.01)	ND ND (<0.01)
		61	0.015 0.014 (0.0145)	ND ND (<0.01)	<0.01 <0.01 (<0.01)	0.010 0.010 (0.010)	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	ND ND (<0.01)	ND ND (<0.01)
		271	ND <0.01 (<0.01)	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	ND ND (<0.01)	ND ND (<0.01)
Sanger, CA, United States, 2014/2015 Green Magic②	4.54	7	<0.01 ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)	0.021 0.019 (0.020)	<0.01 <0.01 (<0.01)	<0.01 ND (<0.01)	ND ND (<0.01)	<0.01 <0.01 (<0.01)
	4.56	59	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)	0.012 0.013 (0.0125)	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	ND ND (<0.01)	<0.01 <0.01 (<0.01)
	4.52	341	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)	<0.01 <0.01 (<0.01)	<0.01 <0.01 (<0.01)	ND ND (<0.01)	ND ND (<0.01)	ND ND (<0.01)

Notes:

②Shepard (2020 DuPont-40012 rev 1).

⑤Doig 2020 DuPont-41762.

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] For trials conducted in the United States, a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 157 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in broccoli rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Fluaza-indolizine	IN-A5760	IN-F4106	1.068×IN-A5760 + IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Lucenay, Rhone Alpes, France 2015 Belstar	4.35	7	0.0051	0.0051	0.0089	0.0144	0.0051	0.0051	0.0051	0.0051	0.0051	0.0460
	4.45	65	0.0050	0.0050	0.0087	0.0141	0.0050	0.0050	0.0050	0.0050	0.0050	0.0450
	4.5	361	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445
Lleida, Catalunya, Spain 2014 Trevi	4.42	7	0.0051	0.0051	0.0088	0.0142	0.0081	0.0076	0.0066	0.0051	0.0051	0.0514
	4.39	63	0.0056	0.0051	0.0088	0.0143	0.0087	0.0082	0.0092	0.0051	0.0051	0.0564
	4.46	361	0.0050	0.0050	0.0087	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0449
Termens, Catalunya, Spain 2014 Trevi	4.5	10	0.0080	0.0050	0.0086	0.0139	0.0055	0.0095	0.0050	0.0050	0.0050	0.0513
	4.43	60	0.0051	0.0051	0.0087	0.0141	0.0051	0.0061	0.0051	0.0051	0.0051	0.0467
Termens, Catalunya, Spain 2014 Verdz	4.45	367	0.0050	0.0050	0.0087	0.0141	0.0050	0.0050	0.0050	0.0050	0.0050	0.0450
Los Palacios, Andalucía, Spain 2014 Marathon	4.46	8	0.0050	0.0050	0.0087	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0449
	4.41	62	0.0051	0.0051	0.0088	0.0142	0.0051	0.0051	0.0051	0.0051	0.0051	0.0454
	4.35	363	0.0051	0.0051	0.0089	0.0144	0.0051	0.0051	0.0051	0.0051	0.0051	0.0460
Aguadulce, Andalucía, Spain 2014 Parthenon	4.646	7	0.0082	0.0048	0.0083	0.0135	0.0048	0.0048	0.0048	0.0048	0.0048	0.0431
	4.44	63	0.0050	0.0050	0.0087	0.0141	0.0050	0.0050	0.0050	0.0050	0.0050	0.0451
	4.52	365	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0443
Oviedo, FL, United States, 2014/2015 Packman	4.49	7	0.0117	0.0050	0.0129	0.0183	0.0419	0.0142	0.0150	0.0050	0.0095	0.0828
	4.52	60	0.0069	0.0050	0.0086	0.0139	0.0129	0.0087	0.0059	0.0050	0.0050	0.0514
	4.48	365	0.0050	0.0050	0.0086	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0447
Fresno, CA, United States, 2014-2016 Imperial	4.41	7	0.0079	0.0051	0.0118	0.0173	0.0127	0.0221	0.0130	0.0051	0.0051	0.0896
	4.39	63	0.0051	0.0051	0.0088	0.0143	0.0051	0.0069	0.0054	0.0051	0.0051	0.0487
Fresno, CA, United States, 2014-2016 Tradition	4.42	365	0.0051	0.0051	0.0088	0.0142	0.0051	0.0051	0.0051	0.0051	0.0051	0.0453
Paso Robles, CA, United States, 2014/2015 Heritage	4.5	7	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445
Paso Robles, CA, United States, 2014/2015 Imperial	4.5	63	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445
	4.5	369	0.0050	0.0050	0.0086	0.0139	0.0090	0.0187	0.0085	0.0050	0.0050	0.0705
Porterville, CA, United States, 2014/2015 Heritage	4.49	7	0.0050	0.0050	0.0086	0.0139	0.0050	0.0052	0.0050	0.0050	0.0050	0.0450
	4.49	61	0.0072	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0446
	4.49	271	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0446
Sanger, CA, United States, 2014/2015 Green Magic	4.54	7	0.0049	0.0049	0.0085	0.0138	0.0099	0.0049	0.0049	0.0049	0.0049	0.0441
	4.56	59	0.0049	0.0049	0.0085	0.0137	0.0061	0.0049	0.0049	0.0049	0.0049	0.0439
	4.52	341	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0443

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 158 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis[D]) in soya bean rotational crops in the United States

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Chula, GA, 2014/2015 95Y60 (soya bean) ③	4.48	7	Forage	0.030	0.015	0.036	0.38	0.90	ND	0.14	<0.01
				0.039	0.016	0.037	0.39	0.85	ND	0.14	<0.01
			Mean	0.0345	0.0155	0.0365	0.385	0.875	<0.01	0.14	<0.01
		7	Imm.	<0.01	ND	<0.01	0.063	<0.01	ND	ND	ND
			Seed+Pod	<0.01	ND	<0.01	0.061	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.062	<0.01	<0.01	<0.01	<0.01
Chula, GA, 2014/2015 95Y60 (soya bean) ③	4.48	7	Hay	0.079	0.15[A]	0.16[A]	1.3[A]	2.2[A]	0.045[A]	0.41[A]	0.032[A]
				0.075	0.11	0.13	0.97	1.5	0.024	0.32	0.025
			Mean	0.077	0.13	0.145	1.135	1.85	0.0345	0.365	0.0285
		7	Seed	0.015	ND	<0.01	0.12	<0.01	ND	ND	ND
				0.016	ND	0.011	0.13	<0.01	ND	ND	ND
			Mean	0.0155	<0.01	<0.0105	0.125	<0.01	<0.01	<0.01	<0.01
Chula, GA, 2014/2015 Pioneer 95Y (soya bean) ③		61	Forage	<0.01	<0.01	<0.01	0.11	0.38	ND	0.091	ND
				<0.01	<0.01	<0.01	0.11	0.40	ND	0.091	<0.01
			Mean	<0.01	<0.01	<0.01	0.11	0.39	<0.01	0.091	<0.01
		61	Imm.	<0.01	ND	ND	0.032	0.013	ND	ND	ND
			Seed+Pod	<0.01	ND	ND	0.029	0.013	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0305	0.013	<0.01	<0.01	<0.01
		61	Hay	0.017	0.093A	0.15[A]	0.37[A]	0.60	0.20	0.15[A]	0.037[A]
				0.018	0.097[A]	0.17[A]	0.43[A]	0.58	0.26	0.13[A]	0.043[A]
			Mean	0.0175	0.095	0.16	0.40	0.59	0.23	0.14	0.040
		61	Seed	<0.01	ND	<0.01	0.064	0.01B	ND	ND	ND
				<0.01	ND	ND	0.070	0.011	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.067	0.0105	<0.01	<0.01	<0.01
Chula, GA, 2014/2015 AG4933 (soya bean) ③		351	Forage	<0.01	ND	<0.01	0.030	0.090	ND	0.020	ND
				0.01B	ND	<0.01	0.025	0.098	ND	0.022	ND
			Mean	<0.01B	<0.01	<0.01	0.0275	0.094	<0.01	0.021	<0.01
Chula, GA, 2014/2015 AG4933 (soya bean) ③	4.48	351	Imm.	ND	ND	ND	<0.01	ND	ND	ND	ND
			Seed+Pod	ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		351	Hay	0.026	0.020	0.040	0.16	0.39	ND	0.065	ND
				0.023	0.021	0.043	0.13	0.40	ND	0.064	ND
			Mean	0.0245	0.0205	0.0415	0.145	0.395	<0.01	0.0645	<0.01
		351	Seed	ND	ND	ND	0.029	ND	ND	ND	ND
				ND	ND	ND	ND	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0145	<0.01	<0.01	<0.01	<0.01
Lime Springs, IA, 2014/2015 S090247 (soya bean) ③	4.52	6	Forage	0.011	<0.01	0.017	0.23	0.46	ND	0.086	<0.01
				<0.01	<0.01	0.015	0.17	0.38	<0.01	0.070	<0.01
			Mean	<0.0105	<0.01	0.016	0.20	0.42	<0.01	0.078	<0.01
		6	Imm.	ND	ND	ND	0.042	0.015	ND	ND	ND
			Seed+Pod	ND	ND	ND	0.045	0.014	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0435	0.0145	<0.01	<0.01	<0.01
		6	Hay	0.032	0.045	0.078	0.87	1.6	0.045	0.26	0.030
				0.048	0.057	0.10	0.86	1.8	0.046	0.31	0.040
			Mean	0.040	0.051	0.089	0.865	1.7	0.0455	0.285	0.035
		6	Seed	<0.01	ND	ND	0.058	0.019	ND	ND	ND
				<0.01	<0.01	ND	0.082	0.016	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	0.070	0.0175	<0.01	<0.01	<0.01
Lime Springs, IA, 2014/2015 A1024341 (soya bean) ③	4.45	64	Forage	<0.01	ND	<0.01	0.070	0.13	ND	0.016	ND
				<0.01	ND	ND	0.065	0.12	ND	0.015	ND
			Mean	<0.01	<0.01	<0.01	0.0675	0.125	<0.01	0.0155	<0.01
		64	Imm.	ND	ND	ND	0.028	ND	ND	ND	ND
			Seed+Pod	ND	ND	ND	0.031	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0295	<0.01	<0.01	<0.01	<0.01
		64	Hay	<0.01	<0.01	0.013	0.21	0.39	<0.01	0.046	<0.01

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
				<0.01	0.011	0.01B	0.24	0.43	0.015	0.057	<0.01
			Mean	<0.01	<0.0105	0.0115	0.225	0.41	<0.0125	0.0515	<0.01
		64	Seed	<0.01	ND	ND	0.053	<0.01	ND	ND	ND
				<0.01	ND	ND	0.051	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.052	<0.01	<0.01	<0.01	<0.01
	4.44	365	Forage	ND	ND	ND	0.10	0.092	ND	0.011	ND
				ND	ND	ND	0.11	0.081	ND	0.010	ND
			Mean	<0.01	<0.01	<0.01	0.105	0.0865	<0.01	0.0105	<0.01
		365	Imm. Seed+Pod	ND	ND	ND	0.028	ND	ND	ND	ND
				ND	ND	ND	0.027	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0275	<0.01	<0.01	<0.01	<0.01
		365	Hay	ND	<0.01	<0.01	0.29	0.21	0.012	0.026	ND
				ND	<0.01	<0.01	0.36	0.20	0.011	0.028	ND
			Mean	<0.01	<0.01	<0.01	0.325	0.205	0.0115	0.027	<0.01
		365	Seed	ND	ND	ND	0.044	ND	ND	ND	ND
				ND	ND	ND	0.047	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0455	<0.01	<0.01	<0.01	<0.01
Richland, IA, 2014/2015 93Y75 (soya bean) ③	4.38	7	Forage	0.011	<0.01	0.011	0.032	0.042	ND	<0.01	ND
				0.011	<0.01	0.012	0.034	0.048	ND	0.01B	ND
			Mean	0.011	<0.01	0.0115	0.033	0.045	<0.01	<0.01	<0.01
		7	Imm. Seed+Pod	<0.01	ND	<0.01	0.031	<0.01	ND	ND	ND
				<0.01	ND	<0.01	0.032	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0315	<0.01	<0.01	<0.01	<0.01
		7	Hay	0.069[A]	0.056	0.083	0.28	0.39	ND	0.084	ND
				0.060[A]	0.055	0.077	0.28	0.32	ND	0.076	ND
			Mean	0.0645	0.0555	0.080	0.28	0.355	<0.01	0.080	<0.01
		7	Seed	0.027	ND	0.01[B]	0.10	<0.01	ND	ND	ND
				0.019	ND	<0.01	0.078	<0.01	ND	ND	ND
			Mean	0.023	<0.01	<0.01	0.089	<0.01	<0.01	<0.01	<0.01
			Pre-processed Seed	0.020	<0.01	<0.01	0.088	<0.01	ND	<0.01	ND
			AGF	0.023	<0.01	0.014	0.045	0.012	0.012	ND	<0.01
Richland, IA, 2014/2015 93Y75 (soya bean) ③	4.39	60	Forage	<0.01	ND	<0.01	0.030	0.057	ND	0.011	ND
				0.010	<0.01	<0.01	0.032	0.074	ND	0.014	ND
			Mean	<0.01	<0.01	<0.01	0.031	0.0655	<0.01	0.0125	<0.01
		60	Imm. Seed+Pod	<0.01	ND	ND	0.024	ND	ND	ND	ND
				<0.01	ND	ND	0.022	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.023	<0.01	<0.01	<0.01	<0.01
Richland, IA, 2014/2015 93Y75 (soya bean) ③	4.39	60	Hay	0.054	0.015	0.049	0.15	0.20	ND	0.056	ND
				0.085[A]	0.013	0.056	0.17	0.23	ND	0.063	ND
			Mean	0.0695	0.014	0.0525	0.16	0.215	<0.01	0.0595	<0.01
		60	Seed	<0.01	ND	ND	0.017	<0.01	ND	<0.01	ND
				0.01[B]	ND	ND	0.019	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.018	<0.01	<0.01	<0.01	<0.01
Richland, IA, 2014/2015 93Y84 (soya bean) ③	4.44	356	Forage	0.015	ND	<0.01	0.075	0.10	<0.01	0.023	ND
				0.014	ND	0.012	0.064	0.10	ND	0.022	ND
			Mean	0.0145	<0.01	<0.011	0.0695	0.10	<0.01	0.0225	<0.01
		356	Imm. Seed+Pod	ND	ND	ND	0.027	<0.01	ND	ND	ND
				ND	ND	ND	0.023	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.025	<0.01	<0.01	<0.01	<0.01
		356	Hay	0.043[A]	0.011	0.035	0.33	0.37	ND	0.098	ND
				0.049[A]	0.010	0.046	0.32	0.37	ND	0.087	ND
			Mean	0.046	0.0105	0.0405	0.325	0.37	<0.01	0.0925	<0.01
		356	Seed	<0.01	ND	ND	0.047	<0.01	ND	ND	ND
				<0.01	ND	ND	0.052	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0495	<0.01	<0.01	<0.01	<0.01
Stewardson, IL,	4.52	18	Forage	0.022	0.012	0.036	0.36	0.31	ND	0.054	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
2014/2015 Pioneer 93Y84 (soya bean) ③				0.021	0.013	0.033	0.33	0.31	ND	0.053	ND
			Mean	0.0215	0.0125	0.0345	0.345	0.31	<0.01	0.0535	<0.01
		18	Imm.	<0.01	ND	<0.01	0.15	<0.01	ND	ND	ND
			Seed+Pod	<0.01	ND	<0.01	0.13	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.14	<0.01	<0.01	<0.01	<0.01
		18	Hay	0.13	0.034	0.11	0.76	0.58	<0.01	0.13	<0.01
				0.12	0.022	0.10	0.65	0.49	<0.01	0.11	<0.01
			Mean	0.125	0.028	0.105	0.705	0.535	<0.01	0.12	<0.01
		18	Seed	0.01[B]	ND	ND	0.23	<0.01	ND	ND	ND
				0.01[B]	ND	<0.01	0.22	<0.01	ND	ND	ND
			Mean	0.01[B]	<0.01	<0.0105	0.225	<0.01	<0.01	<0.01	<0.01
		63	Forage	<0.01	<0.01	0.010	0.19	0.23	ND	0.039	ND
				<0.01	<0.01	<0.01	0.18	0.13	ND	0.031	ND
			Mean	<0.01	<0.01	<0.01[B]	0.185	0.18	<0.01	0.035	<0.01
Stewardson, IL, 2014/2015 Pioneer 93Y84 (soya bean) ③	4.52	63	Imm.	<0.01	ND	ND	0.080	<0.01	ND	ND	ND
			Seed+Pod	<0.01	ND	ND	0.091	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0855	<0.01	<0.01	<0.01	<0.01
		63	Hay	0.024	0.011	0.026	0.68	0.60	ND	0.13	<0.01
				0.027	0.012	0.025	0.68	0.73	ND	0.14	<0.01
			Mean	0.0255	0.0115	0.0255	0.68	0.665	<0.01	0.135	<0.01
		63	Seed	<0.01	ND	ND	0.16	<0.01	ND	ND	ND
				<0.01	ND	<0.01	0.15	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.155	<0.01	<0.01	<0.01	<0.01
		361	Forage	ND	ND	ND	0.032	0.056	ND	0.014	ND
				ND	ND	ND	0.035	0.054	ND	0.015	ND
			Mean	<0.01	<0.01	<0.01	0.0335	0.055	<0.01	0.0145	<0.01
		361	Imm.	ND	ND	ND	0.046	<0.01	ND	ND	ND
			Seed+Pod	ND	ND	ND	0.049	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0475	<0.01	<0.01	<0.01	<0.01
		361	Hay	<0.01	ND	<0.01	0.12	0.12	0.028	0.024	0.011
				<0.01	<0.01	<0.01	0.14	0.12	0.016	0.028	<0.01
			Mean	<0.01	<0.01	<0.01	0.13	0.12	0.022	0.026	<0.0105
		361	Seed	ND	ND	ND	0.061	<0.01	ND	ND	ND
				ND	ND	ND	0.062	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0615	<0.01	<0.01	<0.01	<0.01
Carlyle, IL, 2014/2015 5N431R2 (soya bean) ③	4.46	11	Forage	0.013	<0.01	0.016	0.23	0.82	ND	0.12	ND
				0.012	<0.01	0.013	0.25	0.69	ND	0.093	ND
			Mean	0.0125	<0.01	0.0145	0.24	0.755	<0.01	0.1065	<0.01
		11	Imm.	<0.01	ND	ND	0.038	<0.01	ND	ND	ND
			Seed+Pod	<0.01	ND	ND	0.038	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.038	<0.01	<0.01	<0.01	<0.01
		11	Hay	0.017	0.017	0.026	0.49	1.0	ND	0.18	<0.01
				0.013	0.013	0.024	0.38	0.82	ND	0.17	ND
			Mean	0.015	0.015	0.025	0.435	0.91	<0.01	0.175	<0.01
		11	Seed	<0.01	ND	ND	0.11	0.01[B]	ND	ND	ND
				<0.01	ND	ND	0.10	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.105	<0.01	<0.01	<0.01	<0.01
		60	Forage	0.025	<0.01	0.021	0.060	0.21	ND	0.033	ND
				0.019	ND	0.016	0.053	0.15	ND	0.025	ND
			Mean	0.022	<0.01	0.0185	0.0565	0.18	<0.01	0.029	<0.01
		60	Imm.	<0.01	ND	ND	0.014	<0.01	ND	ND	ND
			Seed+Pod	<0.01	ND	<0.01	0.020	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.017	<0.01	<0.01	<0.01	<0.01
		60	Hay	0.055	0.025	0.063	0.20	0.66	ND	0.098	<0.01
				0.054	0.027	0.057	0.18	0.70	ND	0.10	<0.01
			Mean	0.0545	0.026	0.060	0.19	0.68	<0.01	0.099	<0.01
Carlyle, IL, 2014/2015 5N431R2 (soya bean) ③	4.46	60	Seed	0.015	ND	<0.01	0.059	<0.01	ND	ND	ND
				0.014	ND	0.010	0.055	0.01[B]	ND	ND	ND
			Mean	0.0145	<0.01	<0.01[B]	0.057	<0.01	<0.01	<0.01	<0.01

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Carlyle, IL, 2014/2015 H43L15 (soya bean) ③		365	Forage	<0.01	ND	<0.01	0.018	0.045	ND	0.012	ND
				0.010	ND	<0.01	0.024	0.049	ND	0.013	ND
			Mean	<0.01	<0.01	<0.01	0.021	0.047	<0.01	0.0125	<0.01
		365	Imm. Seed+Pod	ND	ND	ND	0.014	ND	ND	ND	ND
				ND	ND	ND	0.014	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.014	<0.01	<0.01	<0.01	<0.01
		365	Hay	0.016	<0.01	0.016	0.050	0.098	ND	0.022	ND
				0.017	<0.01	0.016	0.050	0.10	ND	0.023	ND
			Mean	0.0165	<0.01	0.016	0.050	0.099	<0.01	0.0225	<0.01
		365	Seed	<0.01	ND	ND	0.030	ND	ND	ND	ND
				<0.01	ND	ND	0.026	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.028	<0.01	<0.01	<0.01	<0.01
Richland, IA, 2013/2015 93Y82① Soya bean	1.26	24	Forage	<0.01	ND	<0.01	0.019	0.023	ND	<0.01	ND
				<0.01	ND	<0.01	0.015	0.017	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	0.017	0.020	<0.01	<0.01	<0.01
		24	Hay	<0.01	ND	<0.01	0.031	0.021	ND	<0.01	ND
				<0.01	<0.01	0.010	0.036	0.037	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	0.0335	0.029	<0.01	<0.01	<0.01
		24	Imm. Seed	ND	ND	ND	<0.01	ND	ND	ND	ND
				ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		24	Seed	ND	ND	ND	<0.01	ND	ND	ND	ND
				ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		67	Forage	ND	ND	ND	0.010	<0.01	ND	ND	ND
				ND	ND	ND	<0.01	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		67	Hay	<0.01	ND	<0.01	0.039	0.024	ND	ND	ND
				<0.01	ND	<0.01	0.033	0.023	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	0.036	0.0235	<0.01	<0.01	<0.01
		67	Imm. Seed	ND	ND	ND	<0.01	ND	ND	ND	ND
				ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		67	Seed	ND	ND	ND	<0.01	ND	ND	ND	ND
				ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	2.52	7	Forage	<0.01	ND	ND	0.028	0.021	ND	<0.01	ND
				<0.01	ND	ND	0.021	0.016	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0245	0.0185	<0.01	<0.01	<0.01
		7	Hay	0.014	<0.01	0.011	0.12	0.056	ND	0.015	ND
				0.018	<0.01	0.018	0.15	0.077	ND	0.021	ND
			Mean	0.016	<0.01	0.0145	0.135	0.0665	<0.01	0.018	<0.01
		7	Imm. Seed	ND	ND	ND	<0.01	ND	ND	ND	ND
				ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		7	Seed	ND	ND	ND	0.013	ND	ND	ND	ND
				ND	ND	ND	0.016	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0145	<0.01	<0.01	<0.01	<0.01
Richland, IA, 2013/2015 91Y80①	2.53	63	Forage	<0.01	<0.01	0.022	0.078	0.13	ND	0.018	ND
				<0.01	<0.01	0.022	0.070	0.11	ND	0.016	ND
			Mean	<0.01	<0.01	0.022	0.074	0.12	<0.01	0.017	<0.01
		63	Hay	0.038	0.020	0.044	0.37	0.58	<0.01	0.096	<0.01
				0.026	0.012	0.032	0.26	0.37	ND	0.070	ND
			Mean	0.032	0.016	0.038	0.315	0.475	<0.01	0.083	<0.01
		63	Imm. Seed	<0.01	ND	ND	0.016	<0.01	ND	ND	ND
				<0.01	ND	ND	0.019	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0175	<0.01	<0.01	<0.01	<0.01
		63	Seed	0.014	ND	<0.01	0.068	<0.01	ND	ND	ND
				0.015	ND	0.011	0.064	<0.01	ND	ND	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Mean	0.0145	<0.01	<0.0105	0.066	<0.01	<0.01	<0.01	<0.01
Richland, IA, 2013/2015 91Y75①	2.53	303	Forage	<0.01	ND	0.011	0.078	0.10	ND	0.014	ND
				<0.01	ND	0.01G	0.061	0.069	ND	0.010	ND
			Mean	<0.01	<0.01	0.0105	0.0695	0.0845	<0.01	0.012	<0.01
		303	Hay	0.023	0.011	0.019	0.33	0.44	ND	0.089	ND
				0.025	0.010	0.019	0.29	0.42	ND	0.080	ND
			Mean	0.024	0.0105	0.019	0.31	0.43	<0.01	0.0845	<0.01
		303	Imm. Seed	ND	ND	ND	0.015	ND	ND	ND	ND
				ND	ND	ND	0.016	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0155	<0.01	<0.01	<0.01	<0.01
		303	Seed	<0.01	ND	ND	0.063	<0.01	ND	ND	ND
				<0.01	ND	ND	0.055	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.059	<0.01	<0.01	<0.01	<0.01
Athens, GA, 2013/2014 95Y70①	1.26	7	Forage	ND	ND	<0.01	0.047	0.083	ND	0.014	ND
				ND	ND	<0.01	0.036	0.059	ND	0.011	ND
			Mean	<0.01	<0.01	<0.01	0.0415	0.071	<0.01	0.0125	<0.01
		7	Hay	0.011	<0.01	0.034	0.054	0.042	ND	0.012	ND
				<0.01	0.011	0.028	0.052	0.047	ND	0.012	ND
			Mean	<0.0105	<0.0105	0.031	0.053	0.0445	<0.01	0.012	<0.01
		7	Imm. Seed	<0.01	ND	ND	<0.01	ND	ND	ND	ND
				ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		7	Seed	<0.01	ND	ND	0.029	ND	ND	ND	ND
				<0.01	ND	ND	0.029	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.029	<0.01	<0.01	<0.01	<0.01
		61	Forage	ND	ND	<0.01	0.032	0.091	ND	0.017	ND
				ND	ND	<0.01	0.050	0.16	ND	0.025	ND
			Mean	<0.01	<0.01	<0.01	0.041	0.1255	<0.01	0.021	<0.01
		61	Hay	ND	0.013	<0.01	0.10	0.37	<0.01	0.053	<0.01
				ND	<0.01	<0.01	0.091	0.31	<0.01	0.042	<0.01
			Mean	<0.01	<0.0115	<0.01	0.0955	0.34	<0.01	0.0475	<0.01
		61	Imm. Seed	ND	ND	ND	<0.01	ND	ND	ND	ND
				ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		61	Seed	ND	ND	ND	0.027	ND	ND	ND	ND
				ND	ND	ND	0.026	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0265	<0.01	<0.01	<0.01	<0.01
Athens, GA, 2013/2014 95Y70①	2.51	17	Forage	ND	ND	0.019	0.14	0.40	ND	0.072	ND
				ND	ND	0.018	0.12	0.36	ND	0.074	ND
			Mean	<0.01	<0.01	0.0185	0.13	0.38	<0.01	0.073	<0.01
		17	Hay	<0.01	0.028	0.025	0.38	1.4	<0.01	0.17	0.011
				<0.01	0.027	0.013	0.26	1.0	<0.01	0.14	<0.01
			Mean	<0.01	0.0275	0.019	0.32	1.2	<0.01	0.155	<0.0105
		17	Imm. Seed	ND	ND	ND	0.021	<0.01	ND	ND	ND
				ND	ND	ND	0.026	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0235	<0.01	<0.01	<0.01	<0.01
		17	Seed	ND	ND	ND	0.081	0.011	ND	<0.01	ND
				ND	ND	ND	0.079	0.011	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.080	0.011	<0.01	<0.01	<0.01
Athens, GA, 2013/2014 578-G6①	2.51	252	Forage	ND	ND	0.014	0.12	0.24	ND	0.064	ND
				ND	ND	0.016	0.096	0.26	ND	0.070	ND
			Mean	<0.01	<0.01	0.015	0.108	0.25	<0.01	0.067	<0.01
		252	Hay	<0.01	0.025	0.023	0.28	0.93	0.011	0.16	0.011
				<0.01	0.022	0.022	0.24	0.74	0.014	0.15	0.012
			Mean	<0.01	0.0235	0.0225	0.26	0.835	0.0125	0.155	0.0115
		252	Imm. Seed	ND	ND	ND	<0.01	<0.01	ND	ND	ND
				ND	ND	ND	0.015	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.0115	<0.01	<0.01	<0.01	<0.01
		252	Seed	ND	<0.01	ND	0.027	<0.01	ND	ND	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
				ND	ND	ND	0.029	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.028	<0.01	<0.01	<0.01	<0.01
Athens, GA, 2013/2014 578-G6①	2.51	361	Forage	ND	ND	<0.01	0.033	0.057	ND	0.014	ND
				ND	ND	<0.01	0.040	0.062	ND	0.016	ND
			Mean	<0.01	<0.01	<0.01	0.0365	0.0595	<0.01	0.015	<0.01
		361	Hay	0.010	<0.01	0.020	0.085	0.12	<0.01	0.029	<0.01
				<0.01	<0.01	0.016	0.081	0.13	<0.01	0.034	<0.01
			Mean	<0.01	<0.01	0.018	0.083	0.125	<0.01	0.0315	<0.01
		361	Imm. Seed	ND	ND	ND	<0.01	ND	ND	ND	ND
				ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		361	Seed	<0.01	ND	ND	0.017	<0.01	ND	ND	ND
				<0.01	ND	ND	0.017	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.017	<0.01	<0.01	<0.01	<0.01

Notes:

① Shepard 2020 DuPont-36791 rev 1.

③ Shepard 2020 DuPont-41070 rev 1. Residues were found of IN-UJV12 in a soya bean hay control sample @ 0.01 mg/kg.

④Doig DuPont-36790 rev 1.

⑥ Doig 2020 DuPont-40828.

[A] Average of duplicate analyses.

[B] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[C] Average of triplicate analyses.

[D] a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 159 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in soya bean rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds) in the United States

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Richland, IA, 2013/2015 93Y82	1.26	24	Forage	0.0178	0.0178	0.0307	0.0497	0.0302	0.0356	0.0178	0.0178	0.0178	
	1.26	24	Hay	0.0178	0.0178	0.0307	0.0497	0.0596	0.0516	0.0178	0.0178	0.0178	
	1.26	24	Imm. Seed	0.0178	0.0178	0.0307		0.0178	0.0178	0.0178	0.0178	0.0178	0.1589
	1.26	24	Seed	0.0178	0.0178	0.0307	0.0497	0.0178	0.0178	0.0178	0.0178	0.0178	0.1589
	1.26	67	Forage	0.0178	0.0178	0.0307	0.0497	0.0178	0.0178	0.0178	0.0178	0.0178	
	1.26	67	Hay	0.0178	0.0178	0.0307	0.0497	0.0640	0.0418	0.0178	0.0178	0.0178	
	1.26	67	Imm. Seed	0.0178	0.0178	0.0307		0.0178	0.0178	0.0178	0.0178	0.0178	0.1589
	1.26	67	Seed	0.0178	0.0178	0.0307	0.0497	0.0178	0.0178	0.0178	0.0178	0.0178	0.1589
	2.52	7	Forage	0.0089	0.0089	0.0154	0.0249	0.0218	0.0164	0.0089	0.0089	0.0089	
	2.52	7	Hay	0.0142	0.0089	0.0223	0.0318	0.1200	0.0591	0.0089	0.0160	0.0089	
	2.52	7	Imm. Seed	0.0089	0.0089	0.0154		0.0089	0.0089	0.0089	0.0089	0.0089	0.0794
	2.52	7	Seed	0.0089	0.0089	0.0154	0.0249	0.0129	0.0089	0.0089	0.0089	0.0089	0.0794
Richland, IA, 2013/2015 5 (91Y80)	2.53	63	Forage	0.0089	0.0089	0.0337	0.0431	0.0655	0.1062	0.0089	0.0151	0.0089	
	2.53	63	Hay	0.0283	0.0142	0.0581	0.0733	0.2789	0.4206	0.0089	0.0735	0.0089	
	2.53	63	Imm. seed	0.0089	0.0089	0.0153		0.0155	0.0089	0.0089	0.0089	0.0089	0.0791
	2.53	63	seed	0.0128	0.0089	0.0161	0.0255	0.0584	0.0089	0.0089	0.0089	0.0089	0.1034
Richland, IA, 2013/2015 5 (91Y75)	2.53	303	Forage	0.0089	0.0089	0.0161	0.0255	0.0615	0.0748	0.0089	0.0106	0.0089	
	2.53	303	Hay	0.0212	0.0093	0.0291	0.0390	0.2745	0.3807	0.0089	0.0748	0.0089	
	2.53	303	Imm. seed	0.0089	0.0089	0.0153		0.0137	0.0089	0.0089	0.0089	0.0089	0.0791
	2.53	303	Seed	0.0089	0.0089	0.0153	0.0248	0.0522	0.0089	0.0089	0.0089	0.0089	0.0925
Athens, GA,	1.26	7	Forage	0.0178	0.0178	0.0307	0.0497	0.0738	0.1262	0.0178	0.0222	0.0178	
	1.26	7	Hay	0.0187	0.0187	0.0952	0.1152	0.0942	0.0791	0.0178	0.0213	0.0178	

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
2013/2014 2 (95Y70)	1.26	7	Imm. Seed	0.0178	0.0178	0.0307		0.0178	0.0178	0.0178	0.0178	0.0178	0.1589
	1.26	7	Seed	0.0178	0.0178	0.0307	0.0497	0.0516	0.0178	0.0178	0.0178	0.0178	0.1589
	1.26	61	Forage	0.0178	0.0178	0.0307	0.0497	0.0729	0.2231	0.0178	0.0373	0.0178	
	1.26	61	Hay	0.0178	0.0204	0.0307	0.0525	0.1698	0.6044	0.0178	0.0844	0.0178	
	1.26	61	Imm. Seed	0.0178	0.0178	0.0307		0.0178	0.0178	0.0178	0.0178	0.0178	0.1589
	1.26	61	Seed	0.0178	0.0178	0.0307	0.0497	0.0471	0.0178	0.0178	0.0178	0.0178	0.1589
	2.51	17	Forage	0.0089	0.0089	0.0285	0.0381	0.1160	0.3391	0.0089	0.0651	0.0089	
	2.51	17	Hay	0.0089	0.0245	0.0293	0.0555	0.2856	1.0709	0.0089	0.1383	0.0094	
	2.51	17	Imm. Seed	0.0089	0.0089	0.0154		0.0210	0.0089	0.0089	0.0089	0.0089	0.0797
	2.51	17	Seed	0.0089	0.0089	0.0154	0.0249	0.0714	0.0098	0.0089	0.0089	0.0089	0.1264
Athens, GA, 2013/2014 2 (578-G6)	2.51	252	Forage	0.0089	0.0089	0.0231	0.0327	0.0964	0.2231	0.0089	0.0598	0.0089	
	2.51	252	Hay	0.0089	0.0210	0.0347	0.0571	0.2320	0.7452	0.0112	0.1383	0.0103	
	2.51	252	Imm. Seed	0.0089	0.0089	0.0154		0.0112	0.0089	0.0089	0.0089	0.0089	0.0797
	2.51	252	Seed	0.0089	0.0089	0.0154	0.0249	0.0250	0.0089	0.0089	0.0089	0.0089	0.0797
	2.51	361	Forage	0.0089	0.0089	0.0154	0.0249	0.0326	0.0531	0.0089	0.0134	0.0089	
	2.51	361	Hay	0.0089	0.0089	0.0278	0.0373	0.0741	0.1116	0.0089	0.0281	0.0089	
	2.51	361	Imm. Seed	0.0089	0.0089	0.0154		0.0089	0.0089	0.0089	0.0089	0.0089	0.0797
	2.51	361	Seed	0.0089	0.0089	0.0154	0.0249	0.0152	0.0089	0.0089	0.0089	0.0089	0.0797
Chula, GA, 2014/2015 95Y60 (soya bean)	4.48	7	Forage	0.0173	0.0078	0.0315	0.0398	0.1925	0.4375	0.0050	0.0700	0.0050	
	4.48	7	Imm. Seed+pod	0.0050	0.0050	0.0086		0.0310	0.0050	0.0050	0.0050	0.0050	0.0549
	4.48	7	Hay	0.0385	0.0650	0.1253	0.1947	0.5675	0.9250	0.0173	0.1825	0.0143	
	4.48	7	Seed	0.0078	0.0050	0.0091	0.0144	0.0625	0.0050	0.0050	0.0050	0.0050	0.1106
Chula, GA, 2014/2015 Pioneer 95Y (soya bean)	4.48	61	Forage	0.0050	0.0050	0.0086	0.0140	0.0550	0.1950	0.0050	0.0455	0.0050	
	4.48	61	Imm. Seed+pod	0.0050	0.0050	0.0086		0.0153	0.0065	0.0050	0.0050	0.0050	0.0470
	4.48	61	Hay	0.0088	0.0475	0.1382	0.1889	0.2000	0.2950	0.1150	0.0700	0.0200	
	4.48	61	Seed	0.0050	0.0050	0.0086	0.0140	0.0335	0.0053	0.0050	0.0050	0.0050	0.0593
Chula, GA, 2014/2015 AG4933	4.48	351	Forage	0.0050	0.0050	0.0086	0.0140	0.0138	0.0470	0.0050	0.0105	0.0050	
	4.48	351	Imm. Seed+pod	0.0050	0.0050	0.0086		0.0050	0.0050	0.0050	0.0050	0.0050	0.0447
	4.48	351	Hay	0.0123	0.0103	0.0358	0.0468	0.0725	0.1975	0.0050	0.0323	0.0050	
	4.48	351	Seed	0.0050	0.0050	0.0086	0.0140	0.0098	0.0050	0.0050	0.0050	0.0050	0.0447
Lime Springs, IA, 2014/2015 S090247	4.52	6	Forage	0.0052	0.0050	0.0137	0.0190	0.0991	0.2081	0.0050	0.0387	0.0050	
	4.52	6	Imm. Seed+pod	0.0050	0.0050	0.0086		0.0216	0.0072	0.0050	0.0050	0.0050	0.0477
	4.52	6	Hay	0.0198	0.0253	0.0762	0.1032	0.4287	0.8425	0.0225	0.1412	0.0173	
	4.52	6	Seed	0.0050	0.0050	0.0086	0.0139	0.0347	0.0087	0.0050	0.0050	0.0050	0.0614
Lime Springs, IA, 2014/2015 A1024341	4.45	64	Forage	0.0050	0.0050	0.0087	0.0141	0.0340	0.0629	0.0050	0.0078	0.0050	
	4.45	64	Imm. Seed+pod	0.0050	0.0050	0.0087		0.0148	0.0050	0.0050	0.0050	0.0050	0.0450
	4.45	64	Hay	0.0050	0.0053	0.0100	0.0156	0.1133	0.2064	0.0063	0.0259	0.0050	
	4.45	64	Seed	0.0050	0.0050	0.0087	0.0141	0.0262	0.0050	0.0050	0.0050	0.0050	0.0463
	4.44	365	Forage	0.0050	0.0050	0.0087	0.0141	0.0530	0.0436	0.0050	0.0053	0.0050	
	4.44	365	Imm. Seed+pod	0.0050	0.0050	0.0087		0.0139	0.0050	0.0050	0.0050	0.0050	0.0451
	4.44	365	Hay	0.0050	0.0050	0.0087	0.0141	0.1640	0.1034	0.0058	0.0136	0.0050	
	4.44	365	Seed	0.0050	0.0050	0.0087	0.0141	0.0230	0.0050	0.0050	0.0050	0.0050	0.0451
Richland, IA, 2014/2015 93Y75	4.38	7	Forage	0.0056	0.0051	0.0102	0.0156	0.0169	0.0230	0.0051	0.0051	0.0051	
	4.38	7	Imm. Seed+Pod	0.0051	0.0051	0.0088		0.0161	0.0051	0.0051	0.0051	0.0051	0.0457
	4.38	7	Hay	0.0330	0.0284	0.0707	0.1010	0.1432	0.0051	0.0051	0.0409	0.0051	
	4.38	7	Seed	0.0118	0.0051	0.0088	0.0143	0.0455	0.0051	0.0051	0.0051	0.0051	0.0806
	4.39	60	Forage	0.0051	0.0051	0.0088	0.0143	0.0158	0.0334	0.0051	0.0064	0.0051	
	4.39	60	Imm. Seed+pod	0.0051	0.0051	0.0088		0.0117	0.0051	0.0051	0.0051	0.0051	0.0456
	4.39	60	Hay	0.0355	0.0071	0.0463	0.0539	0.0816	0.1097	0.0051	0.0304	0.0051	
	4.39	60	Seed	0.0051	0.0051	0.0088	0.0143	0.0092	0.0051	0.0051	0.0051	0.0051	0.0456
Richland,	4.44	356	Forage	0.0073	0.0050	0.0096	0.0150	0.0351	0.0505	0.0050	0.0114	0.0050	

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
IA, 2014/2015 93Y84	4.44	356	Imm seed+pod	0.0050	0.0050	0.0087		0.0126	0.0050	0.0050	0.0050	0.0050	0.0451
	4.44	356	Hay	0.0232	0.0053	0.0353	0.0410	0.1640	0.1867	0.0050	0.0467	0.0050	
	4.44	356	Seed	0.0050	0.0050	0.0087	0.0141	0.0250	0.0050	0.0050	0.0050	0.0050	0.0451
Stewardson, IL, 2014/2015 Pioneer 93Y84	4.52	18	Forage	0.0107	0.0062	0.0295	0.0362	0.1710	0.1536	0.0050	0.0265	0.0050	
	4.52	18	Imm. Seed+pod	0.0050	0.0050	0.0086		0.0694	0.0050	0.0050	0.0050	0.0050	0.1228
	4.52	18	Hay	0.0619	0.0139	0.0899	0.1047	0.3494	0.2651	0.0050	0.0595	0.0050	
	4.52	18	Seed	0.0050	0.0050	0.0086	0.0139	0.1115	0.0050	0.0050	0.0050	0.0050	0.1974
	4.52	63	Forage	0.0050	0.0050	0.0086	0.0139	0.0917	0.0892	0.0050	0.0173	0.0050	
	4.52	63	Imm. Seed+pod	0.0050	0.0050	0.0086		0.0424	0.0050	0.0050	0.0050	0.0050	0.0750
	4.52	63	Hay	0.0126	0.0057	0.0218	0.0279	0.3370	0.3296	0.0050	0.0669	0.0050	
	4.52	63	Seed	0.0050	0.0050	0.0086	0.0139	0.0768	0.0050	0.0050	0.0050	0.0050	0.1360
	4.52	361	Forage	0.0050	0.0050	0.0086	0.0139	0.0166	0.0273	0.0050	0.0072	0.0050	
	4.52	361	Imm. Seed+pod	0.0050	0.0050	0.0086		0.0235	0.0050	0.0050	0.0050	0.0050	0.0443
	4.52	361	Hay	0.0050	0.0050	0.0086	0.0139	0.0644	0.0595	0.0109	0.0129	0.0052	
	4.52	361	Seed	0.0050	0.0050	0.0086	0.0139	0.0305	0.0050	0.0050	0.0050	0.0050	0.0539
Carlyle, IL, 2014/2015 5N431R2	4.46	11	Forage	0.0063	0.0050	0.0126	0.0179	0.1205	0.3792	0.0050	0.0535	0.0050	
	4.46	11	Imm. Seed+pod	0.0050	0.0050	0.0087		0.0191	0.0050	0.0050	0.0050	0.0050	0.0449
	4.46	11	Hay	0.0075	0.0075	0.0217	0.0297	0.2185	0.4570	0.0050	0.0879	0.0050	
	4.46	11	Seed	0.0050	0.0050	0.0087	0.0140	0.0527	0.0050	0.0050	0.0050	0.0050	0.0933
	4.46	60	Forage	0.0110	0.0050	0.0161	0.0214	0.0284	0.0904	0.0050	0.0146	0.0050	
	4.46	60	Imm. Seed+pod	0.0050	0.0050	0.0087		0.0085	0.0050	0.0050	0.0050	0.0050	0.0449
	4.46	60	Hay	0.0274	0.0131	0.0521	0.0660	0.0954	0.3415	0.0050	0.0497	0.0050	
	4.46	60	Seed	0.0073	0.0050	0.0087	0.0140	0.0286	0.0050	0.0050	0.0050	0.0050	0.0507
Carlyle, IL, 2014/2015 H43L15	4.46	365	Forage	0.0050	0.0050	0.0087	0.0140	0.0105	0.0236	0.0050	0.0063	0.0050	
	4.46	365	Imm. Seed + pod	0.0050	0.0050	0.0087		0.0070	0.0050	0.0050	0.0050	0.0050	0.0449
	4.46	365	Hay	0.0083	0.0050	0.0139	0.0192	0.0251	0.0497	0.0050	0.0113	0.0050	
	4.46	365	Seed	0.0050	0.0050	0.0087	0.0140	0.0141	0.0050	0.0050	0.0050	0.0050	0.0449

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 160 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in pea rotational crops

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Charantonnay, Rhone Alpes, France 2014 Kristelle® (pea)	3.35	7	Forage	<0.01	0.031	<0.01	<0.01	0.043	ND	0.010	ND
	3.33	60		<0.01	0.053	0.011	<0.01	0.069	ND	0.014	ND
	3.37	363		ND	0.013	ND	ND	0.026	ND	<0.01	ND
	3.35	7	Vines	<0.01	<0.01	<0.01	ND	0.015	ND	ND	ND
	3.33	60		0.010	0.014	<0.01	ND	0.026	ND	<0.01	ND
	3.37	363		ND	ND	ND	ND	0.010	ND	<0.01	ND
	3.35	7	Hay	0.059	0.085	0.043	0.026	0.093	0.012	0.028	<0.01
	3.33	60		0.073	0.12	0.071	0.041	0.17	0.017	0.045	0.010
	3.37	363		0.011	0.023	0.019	<0.01	0.030	0.011	<0.01	<0.01
	3.35	7	Dried pea	0.088	0.21	0.11	0.065	0.20	0.027	0.058	0.014
	3.33	60		0.084	0.27	0.14	0.080	0.44	0.035	0.11	0.015
	3.37	363		<0.01	0.020	0.012	<0.01	0.037	0.010	0.010	<0.01
Alpicat	3.33	10	Forage	ND	<0.01	ND	ND	0.047	ND	0.012	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Catalunya Spain 2014 Audit⑥ (pea)	3.05	62		ND	<0.01	ND	ND	0.029	ND	ND	ND
	3.34	364		ND	<0.01	ND	ND	0.036	ND	<0.01	ND
	3.33	10	Vines	ND	0.010	ND	ND	0.042	ND	<0.01	ND
	3.05	62		ND	<0.01	ND	ND	0.026	ND	<0.01	ND
	3.34	364		ND	<0.01	ND	ND	0.018	ND	<0.01	ND
	3.33	10	Hay	<0.01	0.015	ND	<0.01	0.12	ND	0.014	ND
	3.05	62		<0.01	0.013	ND	ND	0.038	ND	<0.01	ND
	3.34	364		<0.01	<0.01	<0.01	ND	0.037	ND	<0.01	ND
	3.33	10	Dried pea	<0.01	ND	ND	ND	<0.01	ND	ND	ND
	3.05	62		<0.01	ND	ND	ND	<0.01	ND	ND	ND
	3.34	364		ND	ND	ND	ND	ND	ND	ND	ND
Termens Catalunya Spain 2014 Audit⑥ (pea)	3.32	7	Forage	<0.01	0.012	<0.01	<0.01	0.055	ND	<0.01	ND
	3.05	63		<0.01	<0.01	<0.01	<0.01	0.036	ND	<0.01	ND
	3.33	364		ND	<0.01	ND	ND	0.025	ND	<0.01	ND
	3.32	7	Vines	ND	ND	ND	ND	0.025	ND	ND	ND
	3.05	63		ND	ND	ND	ND	0.015	ND	ND	ND
	3.33	364		ND	ND	ND	ND	<0.01	ND	ND	ND
	3.32	7	Hay	0.032	<0.01	0.011	0.012	0.071	ND	0.013	ND
	3.05	63		0.016	<0.01	<0.01	0.011	0.035	ND	<0.01	ND
	3.33	364		ND	<0.01	ND	ND	0.044	ND	<0.01	<0.01
	3.32	7	Dried pea	0.052	ND	0.014	0.021	<0.01	ND	ND	ND
	3.05	63		0.030	ND	<0.01	0.021	ND	ND	ND	ND
	3.33	364		<0.01	ND	ND	<0.01	ND	ND	ND	ND
Los Palacios Andalucía Spain 2014 Audit⑥ (pea)	3.30	10	Forage	<0.01	<0.01	<0.01	<0.01	0.031	ND	<0.01	ND
	3.17	61		ND	<0.01	<0.01	<0.01	0.022	ND	ND	ND
	3.33	357		ND	ND	ND	ND	0.010	ND	ND	ND
	3.30	10	Vines	0.012	<0.01	ND	0.011	ND	ND	<0.01	ND
	3.17	61		<0.01	ND	ND	<0.01	ND	ND	ND	ND
	3.33	357		ND	ND	ND	ND	<0.01	ND	<0.01	ND
	3.30	10	Hay	0.19	0.031	0.066	0.11	0.015	ND	ND	ND
	3.17	61		0.12	0.018	0.039	0.090	0.011	ND	ND	ND
	3.33	357		0.013	ND	<0.01	ND	<0.01	<0.01	<0.01	ND
	3.30	10	Dried pea	0.042	ND	0.013	0.021	ND	ND	ND	ND
	3.17	61		0.021	ND	<0.01	0.012	ND	ND	ND	ND
	3.33	357		<0.01	ND	ND	ND	ND	ND	ND	ND
Aguadulce Andalucía Spain 2014 Audit⑥ (pea)	3.28	10	Forage	0.034	<0.01	0.034	0.020	0.014	ND	ND	ND
	3.10	62		<0.01	ND	ND	ND	0.014	ND	ND	ND
	3.21	357		ND	ND	ND	ND	0.019	ND	ND	ND
	3.28	10	Vines	ND	ND	ND	ND	ND	ND	ND	ND
	3.10	62		ND	ND	ND	ND	ND	ND	ND	ND
	3.21	357		ND	ND	ND	ND	ND	ND	ND	ND
	3.28	10	Hay	0.041	<0.01	0.011	0.012	<0.01	ND	ND	ND
	3.10	62		0.026	<0.01	<0.01	0.020	<0.01	ND	ND	ND
	3.21	357		0.015	ND	ND	<0.01	<0.01	ND	ND	ND
	3.28	10	Dried pea	<0.01	ND	ND	<0.01	ND	ND	ND	ND
	3.10	62		<0.01	<0.01	ND	ND	ND	ND	ND	ND
	3.21	357		ND	ND	ND	ND	ND	ND	ND	ND
Richland, IA, United States, 2014/2015 Sienna (pea)③	4.40	9	Vines	0.015	0.12	0.028	0.020	0.23	ND	0.039	ND
				0.013	0.11	0.032	0.018	0.23	ND	0.037	ND
			Mean	0.014	0.115	0.030	0.019	0.23	<0.01	0.038	<0.01
			Hay	0.037	0.18	0.042	0.056	0.45[A]	ND	0.13	ND
				0.043	0.17	0.041	0.059	0.46	ND	0.14	ND
			Mean	0.040	0.175	0.0415	0.0575	0.455	<0.01	0.135	<0.01
			Imm. Seed+Pod	<0.01	0.011	<0.01	0.011	0.012	ND	<0.01	ND
				<0.01	0.011	<0.01	0.011	0.011	ND	<0.01	ND
			Mean	<0.01	0.011	<0.01	0.011	0.0115	<0.01	<0.01	<0.01
		9	Dried Seed	0.026	0.013	0.029	0.044	0.029	ND	0.010	ND
				0.027	0.018	0.030	0.042	0.027	ND	0.012	ND
			Mean	0.0265	0.0155	0.0295	0.043	0.028	<0.01	0.011	<0.01
	4.50	116	Vines	ND	0.023	ND	<0.01	0.10	ND	0.015	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
				ND	0.033	ND	<0.01	0.14	ND	0.022	ND
			Mean	<0.01	0.028	<0.01	<0.01	0.12	<0.01	0.0185	<0.01
			Hay	ND	0.17	0.082	0.016	0.52	<0.01	0.11	ND
				ND	0.21	0.13	0.018	0.69	<0.01	0.14	<0.01
			Mean	<0.01	0.19	0.106	0.017	0.605	<0.01	0.125	0.005
			Imm. Seed+Pod	ND	ND	ND	ND	<0.01	ND	ND	ND
				ND	ND	ND	ND	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			Dried Seed	NC							
Richland, IA, United States, 2014/2015 Sienna (pea) ③	4.42	344	Vines	ND	0.040	<0.01	ND	0.068	ND	0.017	ND
				ND	0.025	ND	ND	0.062	ND	0.011	ND
			Mean	<0.01	0.0325	<0.01	<0.01	0.065	<0.01	0.014	<0.01
			Hay	ND	0.17	ND	0.012	0.31[A]	ND	0.13	ND
				<0.01	0.17	<0.01	0.015	0.38	ND	0.15	ND
			Mean	<0.01	0.17	<0.01	0.0135	0.345	<0.01	0.14	<0.01
			Imm. Seed+Pod	ND	ND	ND	ND	<0.01	ND	ND	ND
				ND	ND	ND	ND	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		344	Dried Seed	ND	ND	ND	<0.01	0.017	ND	<0.01	ND
				ND	ND	ND	<0.01	0.016	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.0165	<0.01	<0.01	<0.01
Richland, IA, United States, 2016 Sienna (pea) ③	4.53	62	Vines	ND	0.031	<0.01	ND	0.17	ND	0.016	ND
				ND	0.023	<0.01	<0.01	0.12	ND	0.013	ND
			Mean	<0.01	0.027	<0.01	<0.01	0.145	<0.01	0.0145	<0.01
			Hay	<0.01	0.15	0.10	0.021	0.78[A]	<0.01	0.088	ND
				0.01[B]	0.17	0.095	0.021	0.84[A]	<0.01	0.099	ND
			Mean	<0.01	0.16	0.0975	0.021	0.81	<0.01	0.0935	<0.01
			Imm. Seed+Pod	ND	ND	ND	ND	<0.01	ND	ND	ND
				ND	ND	ND	ND	<0.01	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		62	Dried Seed	ND	ND	ND	<0.01	0.013	ND	<0.01	ND
				<0.01	ND	ND	<0.01	0.013	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01	<0.01
Stewardson, IL, United States, 2014/2015 Knight (pea) ③	4.44	18	Vines	ND	0.086	0.014	<0.01	0.25	<0.01	0.050	ND
				<0.01	0.094	0.016	<0.01	0.28	0.011	0.054	ND
			Mean	<0.01	0.090	0.015	<0.01	0.265	<0.0105	0.052	<0.01
			Hay	0.054	0.33	0.064	0.050	1.1	0.11	0.33	0.014
				<0.01	0.25	0.029	0.016	0.93	0.011	0.24	<0.01
			Mean	<0.032	0.29	0.0465	0.033	1.015	0.0605	0.285	<0.012
		18	Imm. Seed+Pod	ND	<0.01	ND	<0.01	0.045	ND	<0.01	ND
				ND	ND	ND	ND	0.035	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.040	<0.01	<0.01	<0.01
		18	Dried Seed	ND	ND	ND	<0.01	0.050	ND	0.015	ND
				<0.01	ND	ND	0.013	0.15	ND	0.035	ND
			Mean	<0.01	<0.01	<0.01	<0.0115	0.10	<0.01	0.025	<0.01
		63	Vines	ND	0.058	0.016	ND	0.20	ND	0.044	ND
				ND	0.013	<0.01	ND	0.037	ND	0.011	ND
			Mean	<0.01	0.0355	<0.013	<0.01	0.1185	<0.01	0.0275	<0.01
		63	Hay	<0.01	0.12	0.018	<0.01	0.51	0.013	0.13	<0.01
				0.062	0.20	0.055	0.041	0.79	0.021	0.21	<0.01
			Mean	<0.036	0.16	0.0365	<0.0255	0.65	0.017	0.17	<0.01
		63	Imm. Seed+Pod	ND	ND	ND	ND	0.017	ND	<0.01	ND
				ND	ND	ND	ND	0.014	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.0155	<0.01	<0.01	<0.01
		63	Dried Seed	ND	ND	ND	<0.01	0.049	ND	0.017	ND
				ND	ND	ND	<0.01	0.051	ND	0.018	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.050	<0.01	0.0175	<0.01
Stewardson, IL, United States,	4.44	361	Vines	ND	0.031	<0.01	0.010	0.088	<0.01	0.034	ND
				ND	0.020	<0.01	<0.01	0.055	<0.01	0.020	<0.01

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
2014/2015 Knight (pea) ③			Mean	<0.01	0.0255	<0.01	<0.01	0.0715	<0.01	0.027	<0.01
			Hay	<0.01	0.066	<0.01	0.021	0.26	0.050	0.10	0.011
				<0.01	0.075	0.013	0.037	0.29	0.026	0.11	0.01[B]
			Mean	<0.01	0.0705	<0.0115	0.029	0.275	0.038	0.105	0.0105
		361	Imm.	ND	ND	ND	<0.01	0.011	ND	<0.01	ND
			Seed+Pod	ND	ND	ND	<0.01	0.018	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.0145	<0.01	<0.01	<0.01
		361	Dried Seed	<0.01	ND	<0.01	0.022	0.024	ND	0.011	ND
				<0.01	ND	ND	0.015	0.016	ND	<0.01	ND
			Mean	<0.01	<0.01	<0.01	0.0185	0.020	<0.01	<0.0105	<0.01
Jerome, ID, United States, 2014/2015 Talbot (pea) ③	4.51	7	Vines	0.010	0.044	0.018	0.016	0.13	ND	0.025	ND
				0.014	0.052	0.021	0.022	0.16	ND	0.034	ND
			Mean	0.012	0.048	0.0195	0.019	0.145	<0.01	0.0295	<0.01
			Hay	0.061	0.26	0.067	0.12	0.76	<0.01	0.21G	<0.01
				0.069	0.31	0.075	0.12	0.93	<0.01	0.29	<0.01
			Mean	0.065	0.285	0.071	0.12	0.845	<0.01	0.25	<0.01
		7	Imm.	<0.01	<0.01	<0.01	0.010	0.031	ND	<0.01	ND
			Seed+Pod	0.011	<0.01	<0.01	0.011	0.038	ND	<0.01	ND
			Mean	<0.0105	<0.01	<0.01	0.0105	0.0345	<0.01	<0.01	<0.01
	4.54	7	Dried Seed	0.082	0.014	0.045	0.060	0.088	ND	0.031	ND
				0.076	0.019	0.054	0.067	0.097	ND	0.032	ND
			Mean	0.079	0.0165	0.0495	0.0635	0.0925	<0.01	0.0315	<0.01
Jerome, ID, United States, 2014/2015 Talbot (pea) ③	4.54	60	Vines	0.014	0.16	0.030	0.027	0.57	<0.01	0.12	ND
				0.017	0.18	0.031	0.033	0.66	<0.01	0.14	ND
			Mean	0.0155	0.17	0.0305	0.030	0.615	<0.01	0.13	<0.01
			Hay	0.062	0.68[A]	0.14	0.14	3.0	0.024	0.66[A]	0.012
				0.067	0.76[A]	0.13	0.13	3.5	0.024	0.75[A]	0.011
			Mean	0.0645	0.72	0.135	0.135	3.25	0.024	0.705	0.0115
			Imm.	0.014	0.015	0.015	0.021	0.11	ND	0.018	ND
			Seed+Pod	0.015	0.015	0.016	0.023	0.13	ND	0.020	ND
			Mean	0.0145	0.015	0.0155	0.022	0.12	<0.01	0.019	<0.01
Jerome, ID, United States, 2014/2015 Strike (pea) ③		60	Dried Seed	0.053	0.012	0.051	0.053	0.24	ND	0.058	ND
				0.054	0.013	0.042	0.048	0.21	ND	0.048	ND
			Mean	0.0535	0.0125	0.0465	0.0505	0.225	<0.01	0.053	<0.01
	4.51	344	Vines	ND	0.023	ND	ND	0.065	ND	0.016	ND
				ND	0.021	ND	ND	0.063	ND	0.014	ND
			Mean	<0.01	0.022	<0.01	<0.01	0.064	<0.01	0.015	<0.01
			Hay	<0.01	0.083	<0.01	0.012	0.24	ND	0.079	ND
				<0.01	0.086	0.01[B]	0.012	0.36	ND	0.087	ND
			Mean	<0.01	0.0845	<0.01	0.012	0.30	<0.01	0.083	<0.01
		344	Imm.	ND	ND	ND	ND	0.012	ND	<0.01	ND
			Seed+Pod	ND	ND	ND	ND	0.011	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.0115	<0.01	<0.01	<0.01
		344	Dried Seed	0.017	ND	0.011	0.014	0.040	ND	0.014	ND
				0.017	ND	<0.01	0.015	0.043	ND	0.015	ND
			Mean	0.017	<0.01	<0.0105	0.0145	0.0415	<0.01	0.0145	<0.01
Ephrata, WA, United States, 2014/2015 Lochsa (pea) ③	4.48	10	Vines	0.15	0.45	0.20	0.14	2.0	<0.01	0.24	ND
				0.18	0.54	0.25	0.16	2.3	0.01[B]	0.29	<0.01
			Mean	0.165	0.495	0.225	0.15	2.15	<0.01	0.265	<0.01
			Hay	0.82[C]	2.1[C]	1.3[C]	0.82[C]	9.4[C]	0.054[C]	1.4[C]	0.017[C]
				0.79[C]	2.5[C]	1.4[C]	0.78[C]	8.9[C]	0.057[C]	1.5[C]	0.018[C]
			Mean	0.805	2.3	1.35	0.80	9.15	0.0555	1.45	0.0175
	4.48	10	Imm.	0.19	0.071	0.15	0.16	0.30	ND	0.029	ND
			Seed+Pod	0.18	0.070	0.15	0.15	0.28	ND	0.026	ND
			Mean	0.185	0.0705	0.15	0.155	0.29	<0.01	0.0275	<0.01
		10	Dried Seed	1.5[C]	0.24[C]	0.86[C]	1.2[C]	0.74[C]	NDC	0.16[C]	NDC
				1.5[C]	0.25[C]	0.97[C]	1.4[C]	0.85[C]	<0.01[C]	0.17[C]	NDC
			Mean	1.5	0.245	0.915	1.3	0.795	<0.01	0.165	<0.01
	4.50	81	Vines	0.023	0.19	0.031	0.024	0.63	ND	0.11	ND
				0.019	0.15	0.024	0.015	0.44	ND	0.086	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Mean	0.021	0.17	0.0275	0.0195	0.535	<0.01	0.098	<0.01
			Hay	0.10[C] 0.12[C]	1.0[C] 1.2[C]	0.27[C] 0.31[C]	0.11[C] 0.13[C]	2.7[C] 3.3[C]	0.024[C] 0.020[C]	0.65[C] 0.78[C]	0.016[C] 0.012[C]
			Mean	0.11	1.1	0.29	0.12	3.0	0.022	0.715	0.014
		81	Imm. Seed+Pod	0.015 0.021	0.011 0.018	0.014 0.021	0.012 0.016	0.045 0.078	ND ND	0.01[B] 0.015	ND ND
			Mean	0.018	0.0145	0.0175	0.014	0.0615	<0.01	0.0125	<0.01
		81	Dried Seed	0.061 0.077	0.015 0.026	0.054 0.074	0.069 0.079	0.14 0.19	ND ND	0.038 0.051	ND ND
			Mean	0.069	0.0205	0.064	0.074	0.165	<0.01	0.0445	<0.01
Ephrata, WA, United States, 2014/2015 Lochsa (pea) ③	4.48	399	Vines	ND ND	0.039 0.033	ND ND	ND ND	0.058 0.045	ND ND	0.024 0.018	ND ND
			Mean	<0.01	0.036	<0.01	<0.01	0.0525	<0.01	0.021	<0.01
		399	Hay	<0.01 <0.01	0.064 0.069	0.017 0.024	<0.01 <0.01	0.25 0.23	ND <0.01	0.078 0.085	ND ND
			Mean	<0.01	0.0665	0.0205	<0.01	0.24	<0.01	0.0815	<0.01
		399	Imm. Seed+Pod	ND ND	ND ND	ND ND	ND ND	<0.01 ND	ND ND	ND ND	ND ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		399	Dried Seed	<0.01 <0.01	ND ND	<0.01 ND	<0.01 <0.01	<0.01 <0.01	ND ND	<0.01 <0.01	ND ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Payette, ID, United States, 2014/2015 Austrian winter (pea) ③	4.55	7	Vines	0.034 0.027	0.23 0.23	0.073 0.063	0.068 0.057	0.39 0.34	<0.01 <0.01	0.083 0.078	<0.01 <0.01
			Mean	0.0305	0.23	0.068	0.0625	0.365	<0.01	0.0805	<0.01
			Hay	0.14 0.15	0.68 0.98	0.26 0.29	0.24 0.31	1.2 1.9	0.023 0.027	0.36 0.61	0.014 0.014
			Mean	0.145	0.83	0.275	0.275	1.55	0.025	0.485	0.014
		7	Imm. Seed+Pod	<0.01 <0.01	0.014 0.013	<0.01 0.012	0.012 0.015	0.051 0.052	ND ND	0.011 0.013	ND ND
			Mean	<0.01	0.0135	<0.011	0.0135	0.0515	<0.01	0.012	<0.01
		7	Dried Seed	0.017 0.017	0.017 0.018	<0.01 <0.01	0.039 0.039	0.098 0.10	ND ND	0.030 0.031	ND ND
			Mean	0.017	0.0175	<0.01	0.039	0.099	<0.01	0.0305	<0.01
	4.55	336	Vines	0.01[B] 0.010	0.12 0.14	0.012 0.010	0.016 0.016	0.27 0.25	<0.01 <0.01	0.067 0.062	<0.01 ND
			Mean	0.01	0.13	0.011	0.016	0.26	<0.01	0.0645	<0.01
			Hay	0.041 0.038	0.55 0.46	0.082 0.078	0.090 0.094	1.2 1.1	0.022 0.025	0.32 0.27	0.010 0.014
			Mean	0.0395	0.505	0.080	0.092	1.15	0.0235	0.295	0.012
		336	Imm. Seed+Pod	<0.01 0.010	0.011 0.010	<0.01 0.01[B]	0.011 0.015	0.045 0.053	ND ND	0.012 0.015	ND ND
			Mean	<0.01	0.0105	<0.01	0.013	0.049	<0.01	0.0135	<0.01
		336	Dried Seed	0.019	0.016	<0.01	0.019	0.096	ND	0.025	ND

Notes:

① Shepard 2020 DuPont-36791 rev 1.

③ Shepard 2020 DuPont-41070 rev 1. Residues were found of IN-UJV12 in a soya bean hay control sample @ 0.01 mg/kg.

④Doig DuPont-36790 rev 1.

⑥ Doig 2020 DuPont-40828.

[A] Average of duplicate analyses.

[B] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[C] Average of triplicate analyses.

[D] For trials conducted in the a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 161 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in pea rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Charantonnay, Rhone Alpes, France 2014 Kristelle	3.35	7	Forage	0.0067	0.0207	0.0116	0.0337	0.0067	0.0288	0.0067	0.0067	0.0067	
	3.33	60		0.0067	0.0357	0.0128	0.0509	0.0067	0.0464	0.0067	0.0094	0.0067	
	3.37	363		0.0066	0.0086	0.0115	0.0207	0.0066	0.0173	0.0066	0.0066	0.0066	
	3.35	7	Vines	0.0067	0.0067	0.0116	0.0187	0.0067	0.0100	0.0067	0.0000	0.0067	
	3.33	60		0.0067	0.0094	0.0116	0.0217	0.0067	0.0175	0.0067	0.0067	0.0067	
	3.37	363		0.0066	0.0066	0.0115	0.0186	0.0066	0.0066	0.0066	0.0066	0.0066	
	3.35	7	Hay	0.0395	0.0568	0.0497	0.1104	0.0174	0.0622	0.0080	0.0187	0.0067	
	3.33	60		0.0491	0.0807	0.0825	0.1687	0.0276	0.1144	0.0114	0.0303	0.0067	
	3.37	363		0.0073	0.0153	0.0218	0.0381	0.0066	0.0199	0.0073	0.0066	0.0066	
	3.35	7	Dried pea	0.0588	0.1404	0.1271	0.2770	0.0435	0.1337	0.0181	0.0388	0.0094	0.8160
	3.33	60		0.0565	0.1816	0.1627	0.3567	0.0538	0.2960	0.0235	0.0740	0.0101	1.2392
	3.37	363		0.0066	0.0133	0.0138	0.0280	0.0066	0.0246	0.0066	0.0066	0.0066	0.1065
Alpicat Catalunya Spain 2014 Audit	3.33	10	Forage	0.0067	0.0067	0.0116	0.0188	0.0067	0.0316	0.0067	0.0081	0.0067	
	3.05	62		0.0073	0.0073	0.0127	0.0205	0.0073	0.0213	0.0073	0.0073	0.0073	
	3.34	364		0.0067	0.0067	0.0116	0.0187	0.0067	0.0241	0.0067	0.0067	0.0067	
	3.33	10	Vines	0.0067	0.0067	0.0116	0.0188	0.0067	0.0283	0.0067	0.0067	0.0067	
	3.05	62		0.0073	0.0073	0.0127	0.0205	0.0073	0.0191	0.0073	0.0073	0.0073	
	3.34	364		0.0067	0.0067	0.0116	0.0187	0.0067	0.0121	0.0067	0.0067	0.0067	
	3.33	10	Hay	0.0067	0.0101	0.0116	0.0224	0.0067	0.0807	0.0067	0.0094	0.0067	
	3.05	62		0.0073	0.0095	0.0127	0.0229	0.0073	0.0279	0.0073	0.0073	0.0073	
	3.34	364		0.0067	0.0067	0.0116	0.0187	0.0067	0.0248	0.0067	0.0067	0.0067	
	3.33	10	Dried pea	0.0067	0.0067	0.0116	0.0188	0.0067	0.0067	0.0067	0.0067	0.0067	0.0601
	3.05	62		0.0073	0.0073	0.0127	0.0205	0.0073	0.0073	0.0073	0.0073	0.0073	0.0656
	3.34	364		0.0067	0.0067	0.0116	0.0187	0.0067	0.0067	0.0067	0.0067	0.0067	0.0599
Termens Catalunya Spain 2014 Audit	3.32	7	Forage	0.0067	0.0081	0.0117	0.0203	0.0067	0.0371	0.0067	0.0067	0.0067	
	3.05	63		0.0073	0.0073	0.0127	0.0205	0.0073	0.0264	0.0073	0.0073	0.0073	
	3.33	364		0.0067	0.0067	0.0116	0.0188	0.0067	0.0168	0.0067	0.0067	0.0067	
	3.32	7	Vines	0.0067	0.0067	0.0117	0.0189	0.0067	0.0169	0.0067	0.0067	0.0067	
	3.05	63		0.0073	0.0073	0.0127	0.0205	0.0073	0.0110	0.0073	0.0073	0.0073	
	3.33	364		0.0067	0.0067	0.0116	0.0188	0.0067	0.0067	0.0067	0.0067	0.0067	
	3.32	7	Hay	0.0216	0.0067	0.0128	0.0200	0.0081	0.0479	0.0067	0.0088	0.0067	
	3.05	63		0.0118	0.0073	0.0127	0.0205	0.0081	0.0257	0.0073	0.0073	0.0073	
	3.33	364		0.0067	0.0067	0.0116	0.0188	0.0067	0.0296	0.0067	0.0067	0.0067	
	3.32	7	Dried pea	0.0351	0.0067	0.0163	0.0235	0.0142	0.0067	0.0067	0.0067	0.0067	0.0701
	3.05	63		0.0220	0.0073	0.0127	0.0205	0.0154	0.0073	0.0073	0.0073	0.0073	0.0656
	3.33	364		0.0067	0.0067	0.0116	0.0188	0.0067	0.0067	0.0067	0.0067	0.0067	0.0601
Los Palacios Andalucía Spain 2014 Audit	3.3	10	Forage	0.0068	0.0068	0.0117	0.0190	0.0068	0.0210	0.0068	0.0068	0.0068	
	3.17	61		0.0071	0.0071	0.0122	0.0198	0.0071	0.0155	0.0071	0.0071	0.0071	
	3.33	357		0.0067	0.0067	0.0116	0.0188	0.0067	0.0067	0.0067	0.0067	0.0067	
	3.3	10	Vines	0.0081	0.0068	0.0117	0.0190	0.0075	0.0068	0.0068	0.0068	0.0068	
	3.17	61		0.0071	0.0071	0.0122	0.0198	0.0071	0.0071	0.0071	0.0071	0.0071	
	3.33	357		0.0067	0.0067	0.0116	0.0188	0.0067	0.0067	0.0067	0.0067	0.0067	
	3.3	10	Hay	0.1290	0.0210	0.0774	0.0999	0.0747	0.0102	0.0068	0.0068	0.0068	
	3.17	61		0.0848	0.0127	0.0476	0.0612	0.0636	0.0078	0.0071	0.0071	0.0071	
	3.33	357		0.0087	0.0067	0.0116	0.0188	0.0067	0.0067	0.0067	0.0067	0.0067	
	3.3	10	Dried pea	0.0285	0.0068	0.0152	0.0225	0.0143	0.0068	0.0068	0.0068	0.0068	0.0681
	3.17	61		0.0148	0.0071	0.0122	0.0198	0.0085	0.0071	0.0071	0.0071	0.0071	0.0631
	3.33	357		0.0067	0.0067	0.0116	0.0188	0.0067	0.0067	0.0067	0.0067	0.0067	0.0601
Aguadulce Andalucía Spain 2014 Audit	3.28	10	Forage	0.0232	0.0068	0.0401	0.0474	0.0137	0.0096	0.0068	0.0068	0.0068	
	3.1	62		0.0072	0.0072	0.0125	0.0202	0.0072	0.0101	0.0072	0.0072	0.0072	
	3.21	357		0.0070	0.0070	0.0121	0.0195	0.0070	0.0133	0.0070	0.0070	0.0070	
	3.28	10	Vines	0.0068	0.0068	0.0118	0.0191	0.0068	0.0068	0.0068	0.0068	0.0068	
	3.1	62		0.0072	0.0072	0.0125	0.0202	0.0072	0.0072	0.0072	0.0072	0.0072	

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
	3.21	357		0.0070	0.0070	0.0121	0.0195	0.0070	0.0070	0.0070	0.0070	0.0070	
	3.28	10	Hay	0.0280	0.0068	0.0130	0.0203	0.0082	0.0068	0.0068	0.0068	0.0068	
	3.1	62		0.0188	0.0072	0.0125	0.0202	0.0145	0.0072	0.0072	0.0072	0.0072	
	3.21	357		0.0105	0.0070	0.0121	0.0195	0.0070	0.0070	0.0070	0.0070	0.0070	
	3.28	10	Dried pea	0.0068	0.0068	0.0118	0.0191	0.0068	0.0068	0.0068	0.0068	0.0068	0.0610
	3.1	62		0.0072	0.0072	0.0125	0.0202	0.0072	0.0072	0.0072	0.0072	0.0072	0.0646
	3.21	357		0.0070	0.0070	0.0121	0.0195	0.0070	0.0070	0.0070	0.0070	0.0070	0.0624
Richland, IA, United States 2014/2015 Sienna (pea)	4.4	9	Vines	0.0071	0.0585	0.0264	0.0889	0.0097	0.1171	0.0051	0.0193	0.0051	
	4.4	9	Hay	0.0204	0.0891	0.0365	0.1317	0.0293	0.2316	0.0051	0.0687	0.0051	
	4.4	9	Imm. Seed+Pod	0.0051	0.0056	0.0088		0.0056	0.0059	0.0051	0.0051	0.0051	0.0478
	4.4	9	Dried seed	0.0135	0.0079	0.0259	0.0344	0.0219	0.0143	0.0051	0.0056	0.0051	0.1019
	4.5	116	Vines	0.0050	0.0139	0.0086	0.0235	0.0050	0.0597	0.0050	0.0092	0.0050	
	4.5	116	Hay	0.0050	0.0946	0.0912	0.1922	0.0085	0.3012	0.0050	0.0622	0.0050	
	4.5	116	Imm. Seed+Pod	0.0050	0.0050	0.0086		0.0050	0.0050	0.0050	0.0050	0.0050	0.0445
	4.42	344	Vines	0.0051	0.0165	0.0088	0.0263	0.0051	0.0329	0.0051	0.0071	0.0051	
	4.42	344	Hay	0.0051	0.0862	0.0088	0.1008	0.0068	0.1748	0.0051	0.0710	0.0051	
	4.42	344	Imm. Seed+Pod	0.0051	0.0051	0.0088		0.0051	0.0051	0.0051	0.0051	0.0051	0.0453
	4.42	344	Dried seed	0.0051	0.0051	0.0088	0.0142	0.0051	0.0084	0.0051	0.0051	0.0051	0.0503
Richland, IA, United States 2016 Sienna (pea)	4.53	62	Vines	0.0049	0.0134	0.0085	0.0228	0.0049	0.0717	0.0049	0.0072	0.0049	
	4.53	62	Hay	0.0049	0.0791	0.0833	0.1678	0.0104	0.4005	0.0049	0.0462	0.0049	
	4.53	62	Imm. Seed+Pod	0.0049	0.0049	0.0085		0.0049	0.0049	0.0049	0.0049	0.0049	0.0442
	4.53	62	Dried seed	0.0049	0.0049	0.0085	0.0138	0.0049	0.0064	0.0049	0.0049	0.0049	0.0464
Stewardson, IL, United States 2014/2015 Knight (pea)	4.44	18	Vines	0.0050	0.0454	0.0131	0.0616	0.0050	0.1337	0.0053	0.0262	0.0050	
	4.44	18	Hay	0.0161	0.1463	0.0405	0.1968	0.0166	0.5121	0.0305	0.1438	0.0061	
	4.44	18	Imm. Seed+Pod	0.0050	0.0050	0.0087		0.0050	0.0202	0.0050	0.0050	0.0050	0.0681
	4.44	18	Dried seed	0.0050	0.0050	0.0087	0.0141	0.0058	0.0505	0.0050	0.0126	0.0050	0.1141
	4.44	63	Vines	0.0050	0.0179	0.0113	0.0305	0.0050	0.0598	0.0050	0.0139	0.0050	
	4.44	63	Hay	0.0182	0.0807	0.0318	0.1180	0.0129	0.3279	0.0086	0.0858	0.0050	
	4.44	63	Imm. Seed+Pod	0.0050	0.0050	0.0087		0.0050	0.0078	0.0050	0.0050	0.0050	0.0493
	4.44	63	Dried seed	0.0050	0.0050	0.0087	0.0141	0.0050	0.0252	0.0050	0.0088	0.0050	0.0758
	4.44	361	Vines	0.0050	0.0129	0.0087	0.0225	0.0050	0.0361	0.0050	0.0136	0.0050	
	4.44	361	Hay	0.0050	0.0356	0.0100	0.0480	0.0146	0.1387	0.0192	0.0530	0.0053	
	4.44	361	Imm. Seed+Pod	0.0050	0.0050	0.0087		0.0050	0.0073	0.0050	0.0050	0.0050	0.0485
	4.44	361	Dried seed	0.0050	0.0050	0.0087	0.0141	0.0093	0.0101	0.0050	0.0053	0.0050	0.0527
Jerome, ID, United States 2014/2015 Talbot (pea)	4.51	7	Vines	0.0060	0.0238	0.0167	0.0422	0.0094	0.0720	0.0050	0.0147	0.0050	
	4.51	7	Hay	0.0323	0.1416	0.0609	0.2121	0.0596	0.4197	0.0050	0.1242	0.0050	
	4.51	7	Imm. Seed+Pod	0.0052	0.0050	0.0086		0.0052	0.0171	0.0050	0.0050	0.0050	0.0629
	4.54	7	Dried seed	0.0390	0.0081	0.0422	0.0509	0.0313	0.0456	0.0049	0.0155	0.0049	0.1843
	4.54	60	Vines	0.0076	0.0839	0.0260	0.1156	0.0148	0.3034	0.0049	0.0641	0.0049	
	4.54	60	Hay	0.0318	0.3552	0.1151	0.4945	0.0666	1.6035	0.0118	0.3478	0.0057	
	4.54	60	Imm. Seed+Pod	0.0072	0.0074	0.0132		0.0109	0.0592	0.0049	0.0094	0.0049	0.1420
	4.54	60	Dried seed	0.0264	0.0062	0.0396	0.0462	0.0249	0.1110	0.0049	0.0261	0.0049	0.2738
	4.51	344	Vines	0.0050	0.0109	0.0086	0.0203	0.0050	0.0318	0.0050	0.0075	0.0050	
	4.51	344	Hay	0.0050	0.0420	0.0086	0.0534	0.0060	0.1490	0.0050	0.0412	0.0050	
	4.51	344	Imm. Seed+Pod	0.0050	0.0050	0.0086		0.0050	0.0057	0.0050	0.0050	0.0050	0.0455
	4.51	344	Dried seed	0.0084	0.0050	0.0090	0.0143	0.0072	0.0206	0.0050	0.0072	0.0050	0.0691
Ephrata, WA, United States	4.48	10	Vines	0.0825	0.2475	0.1944	0.4587	0.0750	1.0750	0.0050	0.1325	0.0050	
	4.48	10	Hay	0.4025	1.1500	1.1662	2.3944	0.4000	4.5750	0.0278	0.7250	0.0088	

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
2014/2015 Lochsa (pea)	4.48	10	Imm. Seed+Pod	0.0925	0.0353	0.1296		0.0775	0.1450	0.0050	0.0138	0.0050	0.5810
	4.48	10	Dried seed	0.7500	0.1225	0.7904	0.9212	0.6500	0.3975	0.0050	0.0825	0.0050	2.5564
	4.5	81	Vines	0.0105	0.0846	0.0237	0.1140	0.0097	0.2663	0.0050	0.0488	0.0050	
	4.5	81	Hay	0.0548	0.5476	0.2494	0.8342	0.0597	1.4933	0.0110	0.3559	0.0070	
	4.5	81	Imm. Seed+Pod	0.0090	0.0072	0.0151					0.0062	0.0050	0.1021
	4.5	81	Dried seed	0.0343	0.0102	0.0550	0.0659	0.0368	0.0821	0.0050	0.0222	0.0050	0.2716
	4.48	399	Vines	0.0050	0.0180	0.0086	0.0279	0.0050	0.0258	0.0050	0.0105	0.0050	
	4.48	399	Hay	0.0050	0.0333	0.0177	0.0532	0.0050	0.1200	0.0050	0.0408	0.0050	
	4.48	399	Imm. Seed+Pod	0.0050	0.0050	0.0086		0.0050	0.0050	0.0050	0.0050	0.0050	0.0447
	4.48	399	Dried seed	0.0050	0.0050	0.0086	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0447
Payette, ID, United States 2014/2015 Austrian winter (pea)	4.55	7	Vines	0.0150	0.1132	0.0578	0.1788	0.0308	0.1797	0.0049	0.0396	0.0049	
	4.55	7	Hay	0.0714	0.4086	0.2339	0.6703	0.1354	0.7631	0.0123	0.2388	0.0069	
	4.55	7	Imm. Seed+Pod	0.0049	0.0066	0.0094		0.0066	0.0254	0.0049	0.0059	0.0049	0.0807
	4.55	7	Dried seed	0.0084	0.0086	0.0085	0.0177	0.0192	0.0487	0.0049	0.0150	0.0049	0.1189
	4.55	336	Vines	0.0049	0.0640	0.0094	0.0777	0.0079	0.1280	0.0049	0.0318	0.0049	
	4.55	336	Hay	0.0194	0.2486	0.0680	0.3336	0.0453	0.5662	0.0116	0.1452	0.0059	
	4.55	336	Imm. Seed+Pod	0.0049	0.0052	0.0085					0.0066	0.0049	0.0737
	4.55	336	Dried seed	0.0094	0.0079	0.0085	0.0169	0.0094	0.0473	0.0049	0.0123	0.0049	0.1150

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 162 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in bean rotational crops in Spain

Location, year, variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Alpicat, Catalunya, Spain 2013 B.B. Lake 274④	1.25	28	Immature Seed	ND	ND	ND	ND	<0.01	ND	ND	ND
	1.25	128		ND	ND	ND	ND	<0.01	ND	ND	ND
	2.50	28		ND	ND	ND	ND	0.010	ND	ND	ND
	2.50	142		ND	ND	ND	<0.01	0.011	ND	<0.01	ND
	2.50	243		ND	ND	ND	<0.01	<0.01	ND	ND	ND
	1.25	28	Mature Seed	ND	ND	ND	<0.01	<0.01	ND	ND	ND
	1.25	128		ND	ND	ND	ND	ND	ND	ND	ND
	2.50	28		ND	ND	ND	<0.01	ND	ND	ND	ND
	2.50	142		ND	ND	ND	<0.01	<0.01	ND	ND	ND
	2.50	243		ND	ND	ND	<0.01	ND	ND	ND	ND
	1.25	28	Vines	<0.01	ND	ND	<0.01	0.15	ND	0.017	ND
	1.25	128		<0.01	<0.01	<0.01	0.015	0.16	ND	0.026	ND
	2.50	28		<0.01	<0.01	0.010	0.017	0.31	ND	0.031	ND
	2.50	142		ND	<0.01	<0.01	0.018	0.23	ND	0.030	ND
	2.50	243		ND	<0.01	ND	0.035	0.41	ND	0.045	ND
	1.25	28	Hay	0.011	<0.01	0.023	0.039	0.67	ND	0.064	<0.01
	1.25	128		0.017	0.014	0.026	0.056	1.2	<0.01	0.11	<0.01
	2.50	28		0.021	0.018	0.027	0.072	1.5	<0.01	0.13	0.011
	2.50	142		0.013	0.013	0.033	0.059	1.3	<0.01	0.14	0.016
	2.50	243		<0.01	0.010	0.014	0.073	0.99	<0.01	0.10	<0.01
Aguadulce, Andalucia Spain 2013 B.B. Lake	1.25	28	Immature Seed	ND	ND	ND	ND	ND	ND	ND	ND
	1.25	145		ND	ND	ND	ND	ND	ND	ND	ND
	2.50	27		ND	ND	ND	ND	<0.01	ND	ND	ND
	2.50	145		ND	ND	ND	ND	<0.01	ND	ND	ND

Location, year, variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
274④	2.50	242		ND	ND	ND	ND	<0.01	ND	ND	ND
	1.25	28	Mature Seed	ND	ND	ND	ND	ND	ND	ND	ND
	1.25	145		ND	ND	ND	ND	ND	ND	ND	ND
	2.50	27		<0.01	ND	ND	<0.01	ND	ND	ND	ND
	2.50	145		ND	ND	ND	<0.01	ND	ND	ND	ND
	2.50	242		<0.01	ND	ND	<0.01	<0.01	ND	ND	ND
	1.25	28	Vines	ND	ND	ND	ND	0.022	ND	ND	ND
	1.25	145		ND	ND	ND	ND	0.048	ND	<0.01	ND
	2.50	27		ND	ND	ND	ND	0.034	ND	ND	ND
	2.50	145		ND	ND	ND	ND	0.038	ND	ND	ND
	2.50	242		ND	ND	ND	ND	0.035	ND	<0.01	ND
	1.25	28	Hay	<0.01	<0.01	ND	ND	0.050	ND	<0.01	<0.01
	1.25	145		ND	ND	ND	ND	0.027	ND	ND	ND
	2.50	27		0.016	0.012	0.018	0.014	0.35	<0.01	0.036	<0.01
	2.50	145		0.012	<0.01	0.011	0.013	0.20	<0.01	0.021	<0.01
	2.50	242		0.015	<0.01	0.017	0.015	0.21	<0.01	0.022	<0.01

Notes:

④Doig DuPont-36790 rev 1.

Table 163 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in bean rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds) in Spain

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Alpicat, Catalunya, Spain 2013 B.B. Lake 274	1.25	28	Immature Seed	0.0179	0.0179	0.0310					0.0179	0.0179	0.1601
	1.25	128		0.0179	0.0179	0.0310					0.0179	0.0179	0.1601
	2.5	28		0.0090	0.0090	0.0155					0.0090	0.0090	0.0801
	2.5	142		0.0090	0.0090	0.0155					0.0090	0.0090	0.0814
	2.5	243		0.0090	0.0090	0.0155					0.0090	0.0090	0.0801
	1.25	28	Mature Seed	0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	1.25	128		0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	2.5	28		0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	142		0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	243		0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	1.25	28	Vines	0.0179	0.0179	0.0310	0.0501	0.0179	0.2688	0.0179	0.0305	0.0179	
	1.25	128		0.0179	0.0179	0.0310	0.0501	0.0269	0.2867	0.0179	0.0466	0.0179	
	2.5	28		0.0090	0.0090	0.0155	0.0250	0.0152	0.2778	0.0090	0.0278	0.0090	
	2.5	142		0.0090	0.0090	0.0155	0.0250	0.0161	0.2061	0.0090	0.0269	0.0090	
	2.5	243		0.0090	0.0090	0.0155	0.0250	0.0314	0.3674	0.0090	0.0403	0.0090	
	1.25	28	Hay	0.0197	0.0179	0.0712	0.0903	0.0699	1.2006	0.0179	0.1147	0.0179	
	1.25	128		0.0305	0.0251	0.0805	0.1073	0.1004	2.1504	0.0179	0.1971	0.0179	
	2.5	28		0.0188	0.0161	0.0418	0.0590	0.0645	1.3440	0.0090	0.1165	0.0099	
	2.5	142		0.0116	0.0116	0.0511	0.0635	0.0529	1.1648	0.0090	0.1254	0.0143	
	2.5	243		0.0090	0.0090	0.0217	0.0312	0.0654	0.8870	0.0090	0.0896	0.0090	
Aguadulce, Andalucia Spain 2013 B.B. Lake 274	1.25	28	Immature Seed	0.0179	0.0179	0.0310					0.0179	0.0179	0.1601
	1.25	145		0.0179	0.0179	0.0310					0.0179	0.0179	0.1601
	2.5	27		0.0090	0.0090	0.0155					0.0090	0.0090	0.0801
	2.5	145		0.0090	0.0090	0.0155					0.0090	0.0090	0.0801
	2.5	242		0.0090	0.0090	0.0155					0.0090	0.0090	0.0801
	1.25	28	Mature Seed	0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	1.25	145		0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	2.5	27		0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	145		0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	242		0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	1.25	28	Vines	0.0179	0.0179	0.0310	0.0501	0.0179	0.0394	0.0179	0.0179	0.0179	

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
	1.25	145		0.0179	0.0179	0.0310	0.0501	0.0179	0.0860	0.0179	0.0179	0.0179	
	2.5	27		0.0090	0.0090	0.0155	0.0250	0.0090	0.0305	0.0090	0.0090	0.0090	
	2.5	145		0.0090	0.0090	0.0155	0.0250	0.0090	0.0340	0.0090	0.0090	0.0090	
	2.5	242		0.0090	0.0090	0.0155	0.0250	0.0090	0.0314	0.0090	0.0090	0.0090	
	1.25	28	Hay	0.0179	0.0179	0.0310	0.0501	0.0179	0.0896	0.0179	0.0179	0.0179	
	1.25	145		0.0179	0.0179	0.0310	0.0501	0.0179	0.0484	0.0179	0.0179	0.0179	
	2.5	27		0.0143	0.0108	0.0279	0.0393	0.0125	0.3136	0.0090	0.0323	0.0090	
	2.5	145		0.0108	0.0090	0.0170	0.0266	0.0116	0.1792	0.0090	0.0188	0.0090	
	2.5	242		0.0134	0.0090	0.0263	0.0359	0.0134	0.1882	0.0090	0.0197	0.0090	

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 164 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis[D]) in carrot rotational crops in the United States

Location, year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Paso Robles, CA, 2014/2015 Bolero②	4.50	7	Tops	ND	ND	<0.01	<0.01	0.034	0.69	ND	0.021
				ND	ND	<0.01	<0.01	0.032	0.64	ND	0.019
			Mean	<0.01	<0.01	<0.01	<0.01	0.033	0.665	<0.01	0.020
		63		ND	ND	ND	<0.01	0.023	0.37	ND	0.013
				ND	ND	ND	<0.01	0.025	0.37	ND	0.014
			Mean	<0.01	<0.01	<0.01	<0.01	0.024	0.37	<0.01	0.0135
		369		ND	ND	ND	<0.01	0.038	0.71	ND	0.021
				ND	ND	ND	<0.01	0.040	0.69	ND	0.019
			Mean	<0.01	<0.01	<0.01	<0.01	0.039	0.70	<0.01	0.020
		7	Roots	ND	ND	ND	ND	<0.01	0.042	ND	<0.01
				ND	ND	ND	ND	0.01[A]	0.048	ND	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01A	0.045	<0.01	<0.01
		63		ND	ND	ND	ND	<0.01	0.029	ND	ND
				ND	ND	ND	ND	0.011	0.031	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.0105	0.030	<0.01	<0.01
		369		ND	ND	ND	ND	0.023	0.078	ND	<0.01
				ND	ND	ND	ND	0.028	0.084	ND	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	0.0255	0.081	<0.01	<0.01
Porterville, CA, 2014/2015 Danvers②	4.49	7	Tops	0.011[B]	<0.01[B]	0.028[B]	0.036[B]	0.048[B]	3.1[B]	ND[B]	0.091[B]
				<0.01[B,C]	<0.01[B,C]	0.024[B,C]	0.032[B,C]	0.046[B,C]	2.7[B,C]	ND[B,C]	0.085[B,C]
			Mean	<0.0105	<0.01	0.026	0.034	0.047	2.9	<0.01	0.088
		60		0.011[B]	0.01[AB]	0.028[B]	0.033[B]	0.050[B]	3.3	NDB	0.094B
				0.013[B]	0.011[B]	0.032[B]	0.039[B]	0.059[B]	4.0	NDB	0.15B
			Mean	0.012	0.0105	0.030	0.036	0.0545	3.65	<0.01	0.122
		270		ND	ND	ND	ND	ND	0.028	ND	ND
				ND	ND	ND	ND	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.0175	<0.01	<0.01
	4.49	7	Roots	<0.01	ND	ND	0.012	0.032	0.23	ND	0.013
				<0.01	ND	ND	0.012	0.025	0.13	ND	<0.01
			Mean	<0.01	<0.01	<0.01	0.012	0.0285	0.18	<0.01	<0.0115
		60		<0.01	ND	ND	0.011	0.029	0.20	ND	0.013
				<0.01	ND	ND	0.011	0.038	0.20	ND	0.011
			Mean	<0.01	<0.01	<0.01	0.011	0.0335	0.20	<0.01	0.012
		270		ND	ND	ND	ND	ND	ND	ND	ND
				ND	ND	ND	ND	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Yuma, AZ, 2014-2016 SCR2586②	4.50	7	Tops	ND	ND	<0.01	<0.01	<0.01	0.13	ND	<0.01
				ND	ND	<0.01	<0.01	ND	0.12	ND	<0.01

Location, year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.125	<0.01	<0.01
Yuma, AZ, 2014-2016 NUN85110 CAC②		60	Tops	ND	ND	ND	ND	<0.01	0.061	ND	ND
				<0.01	ND	<0.01	<0.01	<0.01	0.084	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0725	<0.01	<0.01
Yuma, AZ, 2014-2016 Bangor②		385	Tops	ND	ND	ND	ND	0.011	0.26	ND	<0.01
				ND	ND	ND	ND	0.01[A]	0.22	ND	0.01[A]
			Mean	<0.01	<0.01	<0.01	<0.01	0.0105	0.24	<0.01	<0.01
Yuma, AZ, 2014-2016 SCR2586②		7	Roots	ND	ND	ND	ND	<0.01	0.01[A]	ND	ND
				<0.01	ND	ND	ND	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Yuma, AZ, 2014-2016 NUN85110 CAC②		60	Roots	ND	ND	ND	ND	ND	<0.01	ND	ND
				ND	ND	ND	ND	<0.01	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Yuma, AZ, 2014-2016 Bangor②		385	Roots	ND	ND	ND	ND	ND	0.010	ND	ND
				ND	ND	ND	ND	ND	0.011	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0105	<0.01	<0.01
Yuma, AZ, 2014-2016 Bangor②	4.50	385	Immature Tops	ND	ND	<0.01	<0.01	<0.01	0.21	ND	<0.01
				ND	ND	<0.01	<0.01	<0.01	0.23	ND	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.22	<0.01	<0.01
		385	Immature Roots	ND	ND	ND	ND	ND	<0.01	ND	ND
				ND	ND	ND	ND	ND	0.01[A]	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Notes:

② Shepard (2020 DuPont-40012 rev 1).

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but round to 0.01 mg/kg.

[B] Average of duplicate analyses.

[C] Control inadvertently switched with treated sample, as evidenced by residues found after duplicate analyses of both samples.

[D] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 165 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in carrot rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds) in the United States

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	1.068×IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Paso Robles, CA, 2014/2015 Bolero	4.5	7	Tops	0.0050	0.0050	0.0086	0.0139	0.0050	0.0164	0.3310	0.0050	0.0100	0.5542
	4.5	63	Tops	0.0050	0.0050	0.0086	0.0139	0.0050	0.0119	0.1842	0.0050	0.0067	0.3257
	4.5	369	Tops	0.0050	0.0050	0.0086	0.0139	0.0050	0.0194	0.3484	0.0050	0.0100	0.5851
	4.5	7	Roots	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0224	0.0050	0.0050	0.0708
	4.5	63	Roots	0.0050	0.0050	0.0086	0.0139	0.0050	0.0052	0.0149	0.0050	0.0050	0.0599
	4.5	369	roots	0.0050	0.0050	0.0086	0.0139	0.0050	0.0127	0.0403	0.0050	0.0050	0.1096
Porterville, CA, 2014/2015 Danvers	4.49	7	Tops	0.0052	0.0050	0.0224	0.0277	0.0170	0.0234	1.4468	0.0050	0.0439	2.2788
	4.49	60		0.0060	0.0052	0.0259	0.0315	0.0180	0.0272	1.8209	0.0050	0.0609	2.8573
	4.49	270		0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0095	0.0050	0.0050	0.0514
	4.49	7	Roots	0.0050	0.0050	0.0086	0.0139	0.0060	0.0142	0.0898	0.0050	0.0057	0.1867
	4.49	60		0.0050	0.0050	0.0086	0.0139	0.0055	0.0167	0.0998	0.0050	0.0060	0.2055
	4.49	270		0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0446
Yuma, AZ, 2014-2016 SCR2586	4.5	7	Tops	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0622	0.0050	0.0050	0.1309
Yuma, AZ,	4.5	60	tops	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0361	0.0050	0.0050	0.0915

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	1.068×IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
2014-2016 NUN85110 CAC													
Yuma, AZ, 2014-2016 Bangor	4.5	385	Tops	0.0050	0.0050	0.0086	0.0139	0.0050	0.0052	0.1195	0.0050	0.0050	0.2177
Yuma, AZ, 2014-2016 SCR2586	4.5	7	Roots	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445
Yuma, AZ, 2014-2016 NUN85110 CAC	4.5	60	Roots	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445
Yuma, AZ, 2014-2016 Bangor	4.5	385	Roots	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0052	0.0050	0.0050	0.0449
Yuma, AZ, 2014-2016 Bangor	4.5	385	Immature Tops	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.1095	0.0050	0.0050	0.2023
	4.5	385	Immature roots	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 166 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in turnip rotational crops

Location	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Lucenay, Rhone Alpes, France 2015 Rave Tardive⑤	4.47	7	Tops	ND	0.018	ND	<0.01	0.032	0.014	<0.01	0.016
	4.51	65		ND	0.015	ND	<0.01	0.067	0.025	0.017	0.029
	4.48	361		ND	ND	ND	ND	ND	ND	ND	ND
	4.47	7	Roots	<0.01	ND	ND	ND	0.027	ND	ND	<0.01
	4.51	65		ND	ND	ND	ND	0.032	ND	ND	<0.01
	4.48	361		ND	ND	ND	ND	ND	ND	ND	ND
Lleida, Catalunya, Spain 2014 Virtudes Martillo⑤	4.42	7	Tops	<0.01	ND	<0.01	<0.01	0.021	<0.01	ND	ND
	4.39	63		<0.01	ND	<0.01	<0.01	0.034	<0.01	<0.01	<0.01
	4.43	361		ND	ND	ND	<0.01	<0.01	ND	ND	<0.01
	4.42	7	Roots	0.013	ND	<0.01	<0.01	0.029	ND	ND	ND
	4.39	63		0.015	ND	<0.01	<0.01	0.035	ND	ND	ND
	4.43	361		ND	ND	ND	ND	0.027	ND	ND	ND
Termens, Catalunya, Spain 2014 Virtudes Martillo⑤	4.53	10	Tops	<0.01	ND	<0.01	<0.01	0.031	<0.01	ND	ND
	4.43	60		ND	ND	<0.01	<0.01	0.067	<0.01	<0.01	<0.01
	4.50	367		ND	ND	ND	<0.01	0.029	<0.01	ND	ND
	4.53	10	Roots	0.013	ND	<0.01	0.011	0.078	ND	ND	ND
	4.43	60		ND	ND	ND	<0.01	0.093	ND	ND	ND
	4.50	367		<0.01	ND	ND	<0.01	0.085	ND	ND	ND
Los Palacios, Andalucía, Spain 2014 AR-2602 F1⑤	4.50	8	Tops	0.015	ND	<0.01	0.010	0.017	<0.01	<0.01	<0.01
	4.40	62		<0.01	<0.01	0.010	<0.01	0.043	0.015	<0.01	0.022
	4.42	363		<0.01	<0.01	<0.01	<0.01	0.084	0.023	<0.01	0.020
	4.50	8	Roots	0.017	ND	<0.01	0.011	0.027	ND	ND	<0.01
	4.41	62		0.018	ND	<0.01	<0.01	0.031	ND	ND	<0.01
	4.42	363		<0.01	ND	ND	<0.01	0.055	<0.01	ND	<0.01
Aguadulce, Andalucía, Spain 2014 AR-2602	4.43	7	Tops	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND
	4.45	63		<0.01	ND	<0.01	<0.01	0.017	ND	ND	ND
	4.41	365		ND	ND	<0.01	<0.01	0.014	<0.01	ND	ND

Location	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
F1⑤	4.43	7	Roots	0.012	ND	<0.01	<0.01	0.015	ND	ND	ND
	4.45	63		0.021	ND	<0.01	<0.01	0.019	ND	ND	ND
	4.41	365		<0.01	ND	ND	ND	0.021	ND	ND	ND

Notes:

⑤ Doig 2020 DuPont-41762.

Table 167 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in turnip rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Lucenay, Rhone Alpes, France 2015 Rave Tardive	4.47	7	Tops	0.0050	0.0090	0.0087	0.0183	0.0050	0.0160	0.0070	0.0050	0.0080	0.0736
	4.51	65		0.0050	0.0075	0.0086	0.0165	0.0050	0.0333	0.0124	0.0084	0.0144	0.1043
	4.48	361		0.0050	0.0050	0.0086	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0447
	4.47	7	Roots	0.0050	0.0050	0.0087	0.0140	0.0050	0.0135	0.0050	0.0050	0.0050	0.0577
	4.51	65		0.0050	0.0050	0.0086	0.0139	0.0050	0.0159	0.0050	0.0050	0.0050	0.0610
	4.48	361		0.0050	0.0050	0.0086	0.0140	0.0050	0.0050	0.0050	0.0050	0.0050	0.0447
Lleida, Catalunya, Spain 2014 Virtudes Martillo	4.42	7	Tops	0.0051	0.0051	0.0088	0.0142	0.0051	0.0106	0.0051	0.0051	0.0051	0.0538
	4.39	63		0.0051	0.0051	0.0088	0.0143	0.0051	0.0173	0.0051	0.0051	0.0051	0.0642
	4.43	361		0.0051	0.0051	0.0087	0.0141	0.0051	0.0051	0.0051	0.0051	0.0051	0.0452
	4.42	7	Roots	0.0066	0.0051	0.0088	0.0142	0.0051	0.0147	0.0051	0.0051	0.0051	0.0599
	4.39	63		0.0077	0.0051	0.0088	0.0143	0.0051	0.0179	0.0051	0.0051	0.0051	0.0650
	4.43	361		0.0051	0.0051	0.0087	0.0141	0.0051	0.0137	0.0051	0.0051	0.0051	0.0582
Termens, Catalunya, Spain 2014 Virtudes Martillo	4.53	10	Tops	0.0049	0.0049	0.0085	0.0138	0.0049	0.0153	0.0049	0.0049	0.0049	0.0600
	4.43	60		0.0051	0.0051	0.0087	0.0141	0.0051	0.0339	0.0051	0.0051	0.0051	0.0890
	4.5	367		0.0050	0.0050	0.0086	0.0139	0.0050	0.0144	0.0050	0.0050	0.0050	0.0589
	4.53	10	Roots	0.0064	0.0049	0.0085	0.0138	0.0054	0.0386	0.0049	0.0049	0.0049	0.0953
	4.43	60		0.0051	0.0051	0.0087	0.0141	0.0051	0.0470	0.0051	0.0051	0.0051	0.1090
	4.5	367		0.0050	0.0050	0.0086	0.0139	0.0050	0.0423	0.0050	0.0050	0.0050	0.1012
Los Palacios, Andalucía, Spain 2014 AR-2602 F1	4.5	8	Tops	0.0075	0.0050	0.0086	0.0139	0.0050	0.0085	0.0050	0.0050	0.0050	0.0498
	4.4	62		0.0051	0.0051	0.0088	0.0142	0.0051	0.0219	0.0076	0.0051	0.0112	0.0749
	4.42	363		0.0051	0.0051	0.0088	0.0142	0.0051	0.0426	0.0117	0.0051	0.0101	0.1122
	4.5	8	Roots	0.0085	0.0050	0.0086	0.0139	0.0055	0.0134	0.0050	0.0050	0.0050	0.0573
	4.41	62		0.0091	0.0051	0.0088	0.0142	0.0051	0.0157	0.0051	0.0051	0.0051	0.0616
	4.42	363		0.0051	0.0051	0.0088	0.0142	0.0051	0.0279	0.0051	0.0051	0.0051	0.0799
Aguadulce, Andalucía, Spain 2014 AR-2602 F1	4.43	7	Tops	0.0051	0.0051	0.0087	0.0141	0.0051	0.0051	0.0051	0.0051	0.0051	0.0452
	4.45	63		0.0050	0.0050	0.0087	0.0141	0.0050	0.0086	0.0050	0.0050	0.0050	0.0503
	4.41	365		0.0051	0.0051	0.0088	0.0142	0.0051	0.0071	0.0051	0.0051	0.0051	0.0485
	4.43	7	Roots	0.0061	0.0051	0.0087	0.0141	0.0051	0.0076	0.0051	0.0051	0.0051	0.0490
	4.45	63		0.0106	0.0050	0.0087	0.0141	0.0050	0.0096	0.0050	0.0050	0.0050	0.0519
	4.41	365		0.0051	0.0051	0.0088	0.0142	0.0051	0.0107	0.0051	0.0051	0.0051	0.0539

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 168 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis [A]) in celery rotational crops

Location, year variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Los Palacios, Andalucía, Spain	4.45	8	Stalks	0.028	ND	0.012	0.011	ND	0.014	ND	ND
	4.41	62		0.018	ND	0.014	0.011	<0.01	0.029	ND	ND

Location, year variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
2014 Blanc de Perpignan ⑤	4.32	363		<0.01	ND	<0.01	<0.01	<0.01	0.019	ND	ND
Aguadulce, Andalucía, Spain 2014 Utah Tall ⑤	4.50	7	Stalks	0.013	ND	<0.01	<0.01	ND	<0.01	ND	ND
	4.37	63		0.017	ND	<0.01	<0.01	ND	<0.01	ND	ND
	4.49	365		<0.01	ND	ND	ND	ND	<0.01	ND	ND
Oviedo, FL, 2014/2015 Utah ②	4.46	26	Untrimmed	<0.01	ND	<0.01	0.020	0.025	0.093	ND	ND
			Stalks	ND	<0.01	<0.01	0.018	0.023	0.091	ND	ND
			Mean	<0.01	<0.01	<0.01	0.019	0.024	0.092	<0.01	<0.01
	4.44	60	Untrimmed	0.034	0.028	0.045	0.053	0.027	0.85	0.016	0.022
			Stalks	0.044	0.030	0.056	0.062	0.026	0.94	0.019	0.026
			Mean	0.039	0.029	0.0505	0.0575	0.0265	0.895	0.0175	0.024
	4.49	378	Untrimmed	ND	ND	ND	ND	ND	<0.01	ND	ND
			Stalks	ND	ND	ND	ND	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Porterville, CA, United States 2014/2015 Sonova ②	4.51	7	Untrimmed	0.01F	ND	<0.01	<0.01	ND	<0.01	ND	ND
			Stalks	<0.01	ND	<0.01	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Yuma, AZ, United States 2014-2016 Command ②	4.50	7	Untrimmed	<0.01	ND	<0.01	<0.01	<0.01	0.058	ND	ND
			Stalks	<0.01	ND	<0.01	<0.01	<0.01	0.065	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0615	<0.01	<0.01
		67	Untrimmed	<0.01	ND	<0.01	<0.01	0.019	0.18	ND	ND
			Stalks	<0.01	ND	<0.01	<0.01	0.017	0.13	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	0.018	0.155	<0.01	<0.01
		363	Untrimmed	ND	ND	ND	ND	<0.01	0.041	ND	ND
			Stalks	ND	ND	ND	ND	<0.01	0.032	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0365	<0.01	<0.01
Fresno, CA, United States 2014-2016 Stix ②	4.48	7	Untrimmed	0.012	ND	<0.01	<0.01	0.014	0.071	ND	ND
			Stalks	0.013	ND	0.011	<0.01	0.016	0.065	ND	ND
			Mean	0.0125	<0.01	<0.0105	<0.01	0.015	0.068	<0.01	<0.01
	4.43	63	Untrimmed	<0.01	ND	<0.01	<0.01	<0.01	0.039	ND	ND
			Stalks	<0.01	ND	<0.01	<0.01	<0.01	0.037	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.038	<0.01	<0.01
	4.42	365	Untrimmed	ND	ND	ND	<0.01	<0.01	0.015	ND	ND
			Stalks	ND	ND	ND	<0.01	<0.01	0.011	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.013	<0.01	<0.01

Notes:

② Shepard (2020 DuPont-40012 rev 1).

⑤ Doig 2020 DuPont-41762.

[A] For trials conducted in the a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 169 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in celery stalks rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM [A] and 1.77×IN QEK31
Los Palacios, Andalucía, Spain 2014 Blanc de Perpignan	4.45	8	0.0141	0.0050	0.0104	0.0055	0.0050	0.0070	0.0050	0.0050	0.0517
	4.41	62	0.0091	0.0051	0.0123	0.0056	0.0051	0.0147	0.0051	0.0051	0.0674
	4.32	363	0.0052	0.0052	0.0090	0.0052	0.0052	0.0099	0.0052	0.0052	0.0534
Aguadulce,	4.5	7	0.0065	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0445

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM [A] and 1.77×IN QEK31
Andalucía, Spain 2014 Utah Tall	4.37	63	0.0087	0.0051	0.0089	0.0051	0.0051	0.0051	0.0051	0.0051	0.0458
	4.49	365	0.0050	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0446
Oviedo, FL, United States, 2014/2015 Utah	4.46	26	0.0050	0.0050	0.0087	0.0095	0.0121	0.0462	0.0050	0.0050	0.1178
	4.44	60	0.0197	0.0146	0.0440	0.0290	0.0134	0.4515	0.0088	0.0121	0.8281
	4.49	378	0.0050	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0446
Porterville, CA, United States, 2014/2015 Sonova	4.51	7	0.0050	0.0050	0.0086	0.0050	0.0050	0.0050	0.0050	0.0050	0.0444
Yuma, AZ, United States, 2014-2016 Command	4.5	7	0.0050	0.0050	0.0086	0.0050	0.0050	0.0306	0.0050	0.0050	0.0832
	4.5	67	0.0050	0.0050	0.0086	0.0050	0.0090	0.0772	0.0050	0.0050	0.1595
	4.5	363	0.0050	0.0050	0.0086	0.0050	0.0050	0.0182	0.0050	0.0050	0.0644
Fresno, CA, United States, 2014-2016 Stix	4.48	7	0.0063	0.0050	0.0091	0.0050	0.0075	0.0340	0.0050	0.0050	0.0932
	4.43	63	0.0051	0.0051	0.0087	0.0051	0.0051	0.0192	0.0051	0.0051	0.0666
	4.42	365	0.0051	0.0051	0.0088	0.0051	0.0051	0.0066	0.0051	0.0051	0.0476

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 170 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in corn/maize rotational crops

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Charantonnay, Rhone Alpes, France 2014 DK4012©	3.34	7	Forage	ND	0.017	0.027	0.011	0.016	0.10	ND	0.12
	3.33	60		ND	0.024	0.061	0.016	0.022	0.17	ND	0.20
	3.36	361		ND	0.016	0.038	<0.01	0.010	0.10	ND	0.14
	3.34	7	immature ears	ND	ND	ND	<0.01	ND	ND	ND	ND
	3.33	60		ND	ND	ND	ND	ND	ND	ND	ND
	3.36	361		ND	ND	ND	ND	ND	ND	ND	ND
	3.34	7	Grain	ND	ND	ND	0.015	ND	<0.01	ND	<0.01
	3.33	60		ND	ND	ND	0.017	ND	<0.01	ND	ND
	3.36	361		ND	ND	ND	ND	ND	ND	ND	ND
	3.34	7	Stover/ fodder	ND	<0.01	0.016	<0.01	ND	0.038	ND	0.028
	3.33	60		ND	<0.01	0.021	0.016	ND	0.050	ND	0.034
	3.36	361		ND	<0.01	0.059	ND	ND	0.14	ND	0.12
Alpicat Catalunya Spain 2014 DKC6340©	3.33	7	Forage	ND	0.023	0.054	0.066	0.024	0.11	<0.01	0.22
	3.34	60		ND	0.028	0.049	0.047	0.024	0.11	<0.01	0.28
	3.37	359		ND	0.014	0.043	0.046	0.025	0.089	<0.01	0.16
	3.33	7	immature ears	ND	ND	ND	0.020	<0.01	ND	ND	ND
	3.34	60		ND	ND	ND	0.024	<0.01	ND	ND	ND
	3.37	359		ND	ND	ND	0.021	<0.01	ND	ND	ND
	3.33	7	Grain	ND	ND	ND	0.016	ND	ND	ND	ND
	3.34	60		ND	ND	ND	0.019	ND	ND	ND	ND
	3.37	359		ND	ND	ND	0.020	ND	ND	ND	ND
	3.33	7	Stover/ fodder	ND	0.025	0.069	0.038	<0.01	0.14	ND	0.18
	3.34	60		ND	0.028	0.053	0.031	<0.01	0.11	<0.01	0.16
	3.37	359		ND	0.016	0.032	0.039	<0.01	0.052	<0.01	0.043
Termens Catalunya Spain 2014 DKC6340©	3.32	10	Forage	ND	0.027	0.069	0.028	0.030	0.15	<0.01	0.23
	3.35	60		ND	0.028	0.067	0.030	0.038	0.15	<0.01	0.23
	3.33	363		ND	0.012	0.031	0.020	<0.01	0.064	ND	0.11

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
	3.32	10	immature ears	ND	ND	ND	0.013	<0.01	ND	ND	<0.01
	3.35	60		ND	ND	ND	0.01	ND	ND	ND	<0.01
	3.33	363		ND	ND	ND	0.01	ND	ND	ND	ND
	3.32	10	Grain	ND	ND	ND	0.018	ND	<0.01	ND	<0.01
	3.35	60		ND	ND	ND	0.016	ND	<0.01	ND	<0.01
	3.33	363		ND	ND	ND	<0.01	ND	ND	ND	ND
	3.32	10	Stover/ fodder	ND	0.050	0.093	0.031	<0.01	0.17	ND	0.10
	3.35	60		ND	0.047	0.094	0.032	<0.01	0.18	<0.01	0.13
	3.33	363		ND	0.017	0.013	0.013	ND	0.020	ND	0.018
Los Palacios Andalucía Spain 2014 MAS71.B⑥	3.35	7	Forage	ND	0.025	0.014	0.036	0.012	0.046	ND	0.027
	3.36	89		ND	0.028	0.014	0.051	0.019	0.043	ND	0.027
	3.37	363		ND	0.016	0.014	<0.01	<0.01	0.039	ND	0.033
	3.35	7	immature ears	ND	ND	<0.01	0.023	ND	<0.01	ND	ND
	3.36	89		ND	ND	<0.01	0.031	<0.01	<0.01	ND	ND
	3.37	363		ND	ND	ND	ND	ND	ND	ND	ND
	3.35	7	Grain	ND	ND	ND	0.022	ND	<0.01	ND	<0.01
	3.36	89		ND	ND	ND	0.038	ND	0.010	ND	<0.01
	3.37	363		ND	ND	ND	ND	ND	ND	ND	ND
	3.35	7	Stover/ fodder	ND	0.054	0.084	0.067	<0.01	0.18	<0.01	0.21
	3.36	89		ND	0.081	0.18	0.065	0.014	0.31	<0.01	0.33
	3.37	363	Mean	ND	0.031	0.054	0.012	ND	0.11	ND	0.12
Aguadulce Andalucía Spain 2014 MAS71.B⑥	3.34	7	Forage	ND	<0.01	<0.01	<0.01	<0.01	0.013	ND	0.013
	3.39	61		ND	ND	ND	<0.01	ND	<0.01	ND	<0.01
	3.30	362	Mean	ND	<0.01	<0.01	ND	ND	0.012	ND	0.016
	3.34	7	immature ears	ND	ND	ND	ND	ND	ND	ND	ND
	3.39	61		ND	ND	ND	ND	ND	ND	ND	ND
	3.30	362	Mean	ND	ND	ND	ND	ND	ND	ND	ND
	3.34	7	Grain	ND	ND	ND	ND	ND	ND	ND	ND
	3.39	61		ND	ND	ND	<0.01	ND	ND	ND	ND
	3.30	362	Mean	ND	ND	ND	<0.01	ND	ND	ND	ND
	3.34	7	Stover/ fodder	ND	ND	<0.01	<0.01	ND	0.011	ND	0.021
	3.39	61		ND	ND	0.013	<0.01	<0.01	0.017	<0.01	0.025
	3.30	362		ND	<0.01	<0.01	<0.01	ND	0.013	ND	0.017
Chula, GA, United States 2014/2015 DKC 62-05③	4.49	7	Forage	ND	0.075	<0.01	0.11	0.012	0.18	0.021	0.10
				ND	0.083	<0.01	0.14	0.013	0.24	0.021	0.14
			Mean	<0.01	0.079	<0.01	0.125	0.0125	0.21	0.021	0.12
		7	Immature Ears	ND	ND	ND	0.060	<0.01	0.011	ND	ND
				ND	ND	ND	0.053	<0.01	0.010	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0565	<0.01	0.0105	<0.01	<0.01
Chula, GA, United States 2014/2015 DKC 62-05③	4.49	7	Stover	ND	0.23	0.046	0.24	<0.01	0.43	0.031	0.22
				ND	0.17	0.048	0.18	<0.01	0.35	0.032	0.19
			Mean	<0.01	0.20	0.047	0.21	<0.01	0.39	0.0315	0.205
			Grain	ND	<0.01	ND	0.35	<0.01	0.021	<0.01	0.012
				ND	<0.01	ND	0.38	<0.01	0.022	<0.01	0.014
			Mean	<0.01	<0.01	<0.01	0.365	<0.01	0.0215	<0.01	0.013
		61	Forage	ND	0.068	0.01[A]	0.055	0.013	0.15	0.023	0.087
				ND	0.065	<0.01	0.045	0.011	0.16	0.020	0.080
			Mean	<0.01	0.0665	<0.01	0.050	0.012	0.155	0.0215	0.0835
		61	Immature Ears	ND	ND	ND	0.039	<0.01	0.013	ND	<0.01
				ND	ND	ND	0.036	<0.01	0.012	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	0.0375	<0.01	0.0125	<0.01	<0.01
		61	Stover	ND	0.081	0.037	0.053	0.023	0.23	0.090	0.14
				ND	0.065	0.028	0.042	0.018	0.18	0.064	0.12
			Mean	<0.01	0.073	0.0325	0.0475	0.0205	0.205	0.077	0.13
			Grain	ND	<0.01	ND	0.12	ND	0.025	<0.01	0.016
				ND	<0.01	ND	0.11	ND	0.022	<0.01	0.015
			Mean	<0.01	<0.01	<0.01	0.115	<0.01	0.0235	<0.01	0.0155
Chula, GA, United States		351	Forage	ND	0.011	ND	0.014	ND	0.037	<0.01	0.018
				ND	<0.01	ND	0.013	ND	0.036	ND	0.017

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
2014/2015 DKC 62-08③			Mean	<0.01	<0.0105	<0.01	0.0135	<0.01	0.0365	<0.01	0.0175
Chula, GA, United States 2014/2015 DKC 62-05③	4.49	351	Immature Ears	ND	ND	ND	0.028	ND	<0.01	ND	ND
				ND	ND	ND	0.025	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0265	<0.01	<0.01	<0.01	<0.01
		351	Stover	ND	0.034	<0.01	0.022	ND	0.097	<0.01	0.045
				ND	0.042	0.010	0.025	ND	0.11	0.01[A]	0.054
			Mean	<0.01	0.038	<0.01[A]	0.0235	<0.01	0.1035	<0.01	0.0495
			Grain	ND	ND	ND	0.041	ND	<0.01	ND	ND
				ND	ND	ND	0.039	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.040	<0.01	<0.01	<0.01	<0.01
Richland, IA, United States 2014/2015 P1023AM③	4.52	7	Forage	ND	<0.01	ND	0.012	ND	0.032	ND	0.021
				ND	<0.01	ND	<0.01	ND	0.028	<0.01	0.022
			Mean	<0.01	<0.01	<0.01	<0.011	<0.01	0.030	<0.01	0.0215
		7	Immature Ears	ND	ND	ND	0.013	ND	<0.01	ND	ND
				ND	ND	ND	0.014	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0135	<0.01	<0.01	<0.01	<0.01
		7	Stover	ND	0.010	ND	<0.01	ND	0.016	<0.01	0.011
				ND	0.017	ND	0.011	ND	0.034	0.01[A]	0.023
			Mean	<0.01	0.0135	<0.01	<0.0105	<0.01	0.025	<0.01	0.017
			Grain	ND	ND	ND	0.012	ND	ND	ND	ND
				ND	ND	ND	0.020	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.016	<0.01	<0.01	<0.01	<0.01
Richland, IA, United States 2014/2015 P1023AM③	4.52	7	Pre-processed Grain	ND	ND	ND	0.029	ND	<0.01	ND	ND
			AGF	ND	ND	ND	0.011	ND	<0.01	ND	ND
Richland, IA, United States 2014/2015 P0506AM③	4.52	60	Forage	ND	ND	ND	<0.01	ND	0.031	ND	0.028
				ND	ND	ND	ND	ND	0.024	ND	0.016
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0275	<0.01	0.022
		60	Immature Ears	ND	ND	ND	<0.01	ND	ND	ND	ND
				ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		60	Stover	ND	<0.01	ND	<0.01	ND	0.11	ND	0.034
				ND	<0.01	ND	<0.01	ND	0.10	ND	0.032
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.105	<0.01	0.033
			Grain	ND	ND	ND	<0.01	ND	<0.01	ND	ND
				ND	ND	ND	<0.01	<0.01	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	4.45	351	Forage	ND	0.010	ND	0.022	ND	0.061	<0.01	ND
				ND	0.01[A]	ND	0.024	<0.01	0.058	<0.01	0.042
			Mean	<0.01	0.010	<0.01	0.023	<0.01	0.0595	<0.01	<0.026
		351	Immature Ears	ND	ND	ND	0.024	ND	<0.01	ND	ND
				ND	ND	ND	0.030	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.027	<0.01	<0.01	<0.01	<0.01
Richland, IA, United States 2014/2015 P0506AM③	4.45	351	Stover	ND	0.055	<0.01	0.028	<0.01	0.11	0.012	0.056
				ND	0.068	<0.01	0.035	<0.01	0.14	0.010	0.066
			Mean	<0.01	0.0615	<0.01	0.0315	<0.01	0.125	0.011	0.061
			Grain	ND	ND	ND	0.059	<0.01	<0.01	ND	<0.01
				ND	ND	ND	0.048	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0535	<0.01	<0.01	<0.01	<0.01
Stewardson, IL, United States 2014/2015 Pioneer P7443R③	4.46	18	Forage	ND	0.013	ND	0.018	0.020	0.14	<0.01	0.12
				ND	0.016	ND	0.019	0.018	0.17	<0.01	0.17
			Mean	<0.01	0.0145	<0.01	0.0185	0.019	0.155	<0.01	0.145
		18	Immature Ears	ND	ND	ND	0.027	0.017	0.014	ND	<0.01
				ND	ND	ND	0.017	0.019	0.014	ND	<0.01
			Mean	<0.01	<0.01	<0.01	0.022	0.018	0.014	<0.01	<0.01
		18	Stover	ND	0.045	0.035	0.033	<0.01	0.32	ND	0.14
				ND	0.081	0.043	0.039	<0.01	0.32	<0.01	0.18
			Mean	<0.01	0.063	0.039	0.036	<0.01	0.32	<0.01	0.16

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Grain	ND	<0.01	ND	0.041	0.012	0.035	<0.01	0.019
				ND	<0.01	ND	0.063	0.01[A]	0.037	<0.01	0.020
			Mean	<0.01	<0.01	<0.01	0.052	0.011	0.036	<0.01	0.0195
		63	Forage	ND	<0.01	ND	<0.01	0.010	0.060	<0.01	0.059
				ND	<0.01	ND	<0.01	0.011	0.050	<0.01	0.047
			Mean	<0.01	<0.01	<0.01	<0.01	0.0105	0.055	<0.01	0.053
Stewardson, IL, United States 2014/2015 Pioneer P7443R③	4.46	63	Immature Ears	ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01
				ND	ND	ND	<0.01	0.012	0.012	ND	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	<0.011	<0.011	<0.01	<0.01
		63	Stover	ND	0.035	0.013	0.012	0.022	0.11	0.019	0.099
				ND	0.037	0.013	0.011	0.023	0.13	0.024	0.10
			Mean	<0.01	0.036	0.013	0.0115	0.0225	0.12	0.0215	0.0995
			Grain	ND	ND	ND	0.025	0.012	0.014	<0.01	<0.01
				ND	ND	ND	0.027	0.013	0.013	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	0.026	0.0125	0.0135	<0.01	<0.01
		361	Forage	ND	ND	ND	<0.01	<0.01	0.056	ND	0.045
				ND	<0.01	ND	<0.01	<0.01	0.060	ND	0.058
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.058	<0.01	0.0515
		361	Immature Ears	ND	ND	ND	<0.01	<0.01	<0.01	ND	<0.01
				ND	ND	ND	<0.01	<0.01	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		361	Stover	ND	0.034	0.019	<0.01	ND	0.11	ND	0.045
				ND	0.040	0.010	<0.01	ND	0.11	ND	0.054
			Mean	<0.01	0.037	0.0145	<0.01	<0.01	0.11	<0.01	0.0495
			Grain	ND	ND	ND	0.012	ND	0.012	ND	<0.01
				ND	ND	ND	0.010	ND	0.013	ND	<0.01
			Mean	<0.01	<0.01	<0.01	0.011	<0.01	0.0125	<0.01	<0.01
Carlyle, IL, United States 2014/2015 FS 66JV1 RIB③	4.49	11	Forage	ND	0.021	<0.01	0.021	0.019	0.21	<0.01	0.094
				ND	0.021	ND	0.022	0.018	0.20	<0.01	0.096
			Mean	<0.01	0.021	<0.01	0.0215	0.0185	0.205	<0.01	0.095
		11	Immature Ears	ND	ND	ND	0.013	<0.01	0.01[A]	ND	ND
				ND	ND	ND	0.013	<0.01	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.013	<0.01	<0.01[A]	<0.01	<0.01
		11	Stover	ND	0.12	0.042	0.042	<0.01	0.27	ND	0.073
				ND	0.10	0.046	0.034	<0.01	0.30	ND	0.077
			Mean	<0.01	0.11	0.044	0.038	<0.01	0.285	<0.01	0.075
			Grain	ND	0.010	ND	0.035	ND	0.038	<0.01	0.018
				ND	<0.01	ND	0.028	ND	0.033	<0.01	0.015
			Mean	<0.01	<0.01	<0.01	0.0315	<0.01	0.0355	<0.01	0.0165
Carlyle, IL, United States 2014/2015 130670③		60	Forage	ND	0.025	ND	0.012	0.014	0.12	<0.01	0.060
				ND	0.018	ND	0.01[A]	0.012	0.11	<0.01	0.057
			Mean	<0.01	0.0215	<0.01	0.011	0.013	0.115	<0.01	0.0585
		60	Immature Ears	ND	ND	ND	0.011	<0.01	<0.01	ND	ND
				ND	ND	ND	0.011	<0.01	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	<0.01
		60	Stover	ND	0.072	<0.01	0.016	0.021	0.28	0.016	0.12
				ND	0.10	<0.01	0.018	0.020	0.38	0.019	0.16
			Mean	<0.01	0.086	<0.01	0.017	0.0205	0.33	0.0175	0.14
Carlyle, IL, 2014/2015 130670③	4.49	60	Grain	ND	ND	ND	0.023	0.011	<0.01	ND	<0.01
				ND	ND	ND	0.021	<0.01	<0.01	ND	<0.01
			Mean	<0.01	<0.01	<0.01	0.022	<0.0105	<0.01	<0.01	<0.01
Carlyle, IL, United States 2014/2015 FS 63SV1 RIB③		365	Forage	ND	0.012	ND	<0.01	<0.01	0.060	<0.01	0.034
				ND	0.016	ND	<0.01	ND	0.063	<0.01	0.033
			Mean	<0.01	0.014	<0.01	<0.01	<0.01	0.0615	<0.01	0.0335
		365	Immature Ears	ND	ND	ND	<0.01	<0.01	<0.01	ND	ND
				ND	ND	ND	<0.01	<0.01	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		365	Stover	ND	0.091	<0.01	0.011	ND	0.18	<0.01	0.070
				ND	0.080	<0.01	<0.01	ND	0.17	<0.01	0.062
			Mean	<0.01	0.0855	<0.01	<0.0105	<0.01	0.175	<0.01	0.066

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Grain	ND	ND	ND	<0.01	ND	0.012	ND	<0.01
				ND	ND	ND	<0.01	ND	0.011	ND	<0.01
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0115	<0.01	<0.01
Uvalde, TX, United States 2014/2015 DKC 69-43③	4.49	7	Forage	ND	0.064	ND	0.020	0.029	0.19	0.018	0.14
				ND	0.055	ND	0.021	0.031	0.22	0.016	0.16
			Mean	<0.01	0.0595	<0.01	0.0205	0.030	0.205	0.017	0.15
		7	Immature Ears	ND	ND	ND	<0.01	ND	<0.01	ND	ND
				ND	ND	ND	<0.01	<0.01	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Uvalde, TX, United States 2014/2015 DKC 69-43③	4.92	7	Stover	<0.01	0.17	0.040	0.050	0.01[A]	0.40	0.025	0.23
				0.018	0.19	0.049	0.064	0.01[A]	0.44	0.020	0.27
			Mean	0.014	0.18	0.0445	0.057	0.01[A]	0.42	0.0225	0.25
			Grain	ND	<0.01	ND	0.013	ND	0.018	ND	0.010
				ND	<0.01	ND	0.011	ND	0.022	ND	0.011
			Mean	<0.01	<0.01	<0.01	0.012	<0.01	0.020	<0.01	0.0105
Uvalde, TX, United States 2014/2015 DKC 64-69③	4.54	67	Forage	ND	0.021	ND	0.013	0.016	0.056	0.01[A]	0.024
				ND	0.020	ND	0.016	0.019	0.063	0.013	0.028
			Mean	<0.01	0.0205	<0.01	0.0145	0.0175	0.0595	0.0115	0.026
		67	Immature Ears	ND	ND	ND	0.010	<0.01	<0.01	ND	ND
				ND	ND	ND	0.012	<0.01	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.011	<0.01	<0.01	<0.01	<0.01
		67	Stover	ND	0.13	<0.01	0.038	0.013	0.23	0.031	0.16
				ND	0.12	<0.01	0.040	0.013	0.22	0.033	0.15
			Mean	<0.01	0.125	<0.01	0.039	0.013	0.225	0.032	0.155
			Grain	ND	<0.01	ND	0.035	<0.01	0.018	<0.01	<0.01
				ND	<0.01	ND	0.032	<0.01	0.017	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	0.0335	<0.01	0.0175	<0.01	<0.01
	4.49	317	Forage	ND	0.015	ND	0.014	0.013	0.073	0.011	0.034
				ND	0.014	ND	<0.01	0.013	0.071	0.011	0.032
			Mean	<0.01	0.0145	<0.01	<0.012	0.013	0.072	0.011	0.033
Uvalde, TX, United States 2014/2015 DKC 64-69③	4.49	317	Immature Ears	ND	ND	ND	0.015	<0.01	<0.01	ND	ND
				ND	ND	ND	0.012	<0.01	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0135	<0.01	<0.01	<0.01	<0.01
		317	Stover	ND	0.076	ND	0.029	0.01[A]	0.20	0.029	0.14
				ND	0.072	<0.01	0.027	0.011	0.20	0.031	0.15
			Mean	<0.01	0.074	<0.01	0.028	0.0105	0.20	0.030	0.145
			Grain	ND	ND	ND	0.027	ND	0.011	<0.01	<0.01
				ND	ND	ND	0.029	ND	0.012	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	0.028	<0.01	0.0115	<0.01	<0.01

Notes:

③ Shepard 2020 DuPont-41070 rev 1.

⑥ Doig 2020 DuPont-40828.

[A] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[B] For trials conducted in the a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 171 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in maize rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Charantonnay, Rhone	3.34	7-10	Forage	0.0067	0.0114	0.0313	0.0435	0.0074	0.0107	0.0671	0.0067	0.0805	
	3.33	60-	Forage	0.0067	0.0161	0.0709	0.0881	0.0108	0.0148	0.1144	0.0067	0.1345	

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Alpes, France 2014 DK4012		270											
	3.36	358-365	Forage	0.0067	0.0107	0.0438	0.0552	0.0067	0.0067	0.0667	0.0067	0.0933	
	3.34	7-10	immature ears	0.0067	0.0000	0.0116					0.0067	0.0000	0.0448
	3.33	60-270	immature ears	0.0067	0.0067	0.0279					0.0067	0.0471	0.1401
	3.36	358-365	immature ears	0.0067	0.0067	0.0115					0.0067	0.0067	0.0596
	3.34	7-10	Grain	0.0067	0.0067	0.0116	0.0187	0.0101	0.0067	0.0067	0.0067	0.0067	0.0599
	3.33	60-270	Grain	0.0067	0.0067	0.0116	0.0188	0.0114	0.0067	0.0067	0.0067	0.0067	0.0601
	3.36	358-365	Grain	0.0067	0.0067	0.0115	0.0186	0.0067	0.0067	0.0067	0.0067	0.0067	0.0596
	3.34	7-10	Stover	0.0067	0.0067	0.0185	0.0257	0.0067	0.0067	0.0255	0.0067	0.0188	
	3.33	60-270	Stover	0.0067	0.0067	0.0244	0.0316	0.0108	0.0067	0.0336	0.0067	0.0229	
	3.36	358-365	Stover	0.0067	0.0067	0.0680	0.0751	0.0067	0.0067	0.0933	0.0067	0.0800	
Alpicat Catalunya Spain 2014 DKC6340	3.33	7	Forage	0.0067	0.0155	0.0628	0.0793	0.0444	0.0161	0.0740	0.0067	0.1480	
	3.34	60	Forage	0.0067	0.0188	0.0568	0.0768	0.0315	0.0161	0.0738	0.0067	0.1878	
	3.37	359	Forage	0.0066	0.0093	0.0494	0.0593	0.0306	0.0166	0.0592	0.0066	0.1064	
	3.33	7	immature ears	0.0067	0.0067	0.0116					0.0067	0.0067	0.0601
	3.34	60	immature ears	0.0067	0.0067	0.0116					0.0067	0.0067	0.0599
	3.37	359	immature ears	0.0066	0.0066	0.0115					0.0066	0.0066	0.0594
	3.33	7	Grain	0.0067	0.0067	0.0116	0.0188	0.0108	0.0067	0.0067	0.0067	0.0067	0.0601
	3.34	60	Grain	0.0067	0.0067	0.0116	0.0187	0.0127	0.0067	0.0067	0.0067	0.0067	0.0599
	3.37	359	Grain	0.0066	0.0066	0.0115	0.0186	0.0133	0.0066	0.0066	0.0066	0.0066	0.0594
	3.33	7	Stover	0.0067	0.0168	0.0802	0.0981	0.0256	0.0067	0.0942	0.0067	0.1211	
	3.34	60	Stover	0.0067	0.0188	0.0614	0.0815	0.0208	0.0067	0.0738	0.0067	0.1073	
	3.37	359	Stover	0.0066	0.0106	0.0367	0.0481	0.0259	0.0066	0.0346	0.0066	0.0286	
Termens Catalunya Spain 2014 DKC6340	3.32	10	Forage	0.0067	0.0182	0.0804	0.0999	0.0189	0.0202	0.1012	0.0067	0.1552	
	3.35	60	Forage	0.0067	0.0187	0.0774	0.0974	0.0201	0.0254	0.1003	0.0067	0.1538	
	3.33	363	Forage	0.0067	0.0081	0.0360	0.0446	0.0135	0.0067	0.0431	0.0067	0.0740	
	3.32	10	immature ears	0.0067	0.0067	0.0117					0.0067	0.0067	0.0603
	3.35	60	immature ears	0.0067	0.0067	0.0116					0.0067	0.0067	0.0597
	3.33	363	immature ears	0.0067	0.0067	0.0116					0.0067	0.0067	0.0601
	3.32	10	Grain	0.0067	0.0067	0.0117	0.0189	0.0121	0.0067	0.0067	0.0067	0.0067	0.0603
	3.35	60	Grain	0.0067	0.0067	0.0116	0.0187	0.0107	0.0067	0.0067	0.0067	0.0067	0.0597
	3.33	363	Grain	0.0067	0.0067	0.0116	0.0188	0.0067	0.0067	0.0067	0.0067	0.0067	0.0601
	3.32	10	Stover	0.0067	0.0337	0.1084	0.1444	0.0209	0.0067	0.1147	0.0067	0.0675	
	3.35	60	Stover	0.0067	0.0314	0.1086	0.1422	0.0214	0.0067	0.1204	0.0067	0.0869	
	3.33	363	Stover	0.0067	0.0114	0.0151	0.0273	0.0087	0.0067	0.0135	0.0067	0.0121	
Los Palacios Andalucía Spain 2014 MAS71.B	3.35	7	Forage	0.0067	0.0167	0.0162	0.0340	0.0241	0.0080	0.0308	0.0067	0.0181	
	3.36	89	Forage	0.0067	0.0187	0.0161	0.0361	0.0340	0.0127	0.0287	0.0067	0.0180	
	3.37	363	Forage	0.0066	0.0106	0.0161	0.0274	0.0066	0.0066	0.0259	0.0066	0.0219	
	3.35	7	immature ears	0.0067	0.0067	0.0116					0.0067	0.0067	0.0597
	3.36	89	immature ears	0.0067	0.0067	0.0115					0.0067	0.0067	0.0596
	3.37	363	immature	0.0066	0.0066	0.0115					0.0066	0.0066	0.0594

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
			ears										
	3.35	7	Grain	0.0067	0.0067	0.0116	0.0187	0.0147	0.0067	0.0067	0.0067	0.0067	0.0597
	3.36	89	Grain	0.0067	0.0067	0.0115	0.0186	0.0253	0.0067	0.0067	0.0067	0.0067	0.0596
	3.37	363	Grain	0.0066	0.0066	0.0115	0.0186	0.0066	0.0066	0.0066	0.0066	0.0066	0.0594
	3.35	7	Stover	0.0067	0.0361	0.0970	0.1356	0.0448	0.0067	0.1204	0.0067	0.1404	
	3.36	89	Stover	0.0067	0.0540	0.2073	0.2650	0.0433	0.0093	0.2067	0.0067	0.2200	
	3.37	363	Stover	0.0066	0.0206	0.0620	0.0840	0.0080	0.0066	0.0731	0.0066	0.0798	
Aguadulce Andalucía Spain 2014 MAS71.B	3.34	7	Forage	0.0067	0.0067	0.0116	0.0187	0.0067	0.0067	0.0087	0.0067	0.0087	
	3.39	61	Forage	0.0066	0.0066	0.0114	0.0185	0.0066	0.0066	0.0066	0.0066	0.0066	
	3.3	362	Forage	0.0068	0.0068	0.0117	0.0190	0.0068	0.0068	0.0081	0.0068	0.0109	
	3.34	7	immature ears	0.0067	0.0067	0.0116					0.0067	0.0067	0.0599
	3.39	61	immature ears	0.0066	0.0066	0.0114					0.0066	0.0066	0.0590
	3.3	362	immature ears	0.0068	0.0068	0.0117					0.0068	0.0068	0.0607
	3.34	7	Grain	0.0067	0.0067	0.0116	0.0187	0.0067	0.0067	0.0067	0.0067	0.0067	0.0599
	3.39	61	Grain	0.0066	0.0066	0.0114	0.0185	0.0066	0.0066	0.0066	0.0066	0.0066	0.0590
	3.3	362	Grain	0.0068	0.0068	0.0117	0.0190	0.0068	0.0068	0.0068	0.0068	0.0068	0.0607
	3.34	7	Stover	0.0067	0.0067	0.0116	0.0187	0.0067	0.0067	0.0074	0.0067	0.0141	
	3.39	61	Stover	0.0066	0.0066	0.0148	0.0219	0.0066	0.0066	0.0112	0.0066	0.0165	
	3.3	362	Stover	0.0068	0.0068	0.0117	0.0190	0.0068	0.0068	0.0088	0.0068	0.0115	
United States trials													
Chula, GA, 2014/2015 DKC 62-05	4.49	7	Forage	0.0050	0.0394	0.0086	0.0507	0.0624	0.0062	0.1048	0.0105	0.0599	
	4.49	7	Immature ears	0.0050	0.0050	0.0086					0.0050	0.0050	0.0499
	4.49	7	Stover	0.0050	0.0998	0.0405	0.1471	0.1048	0.0050	0.1946	0.0157	0.1023	
	4.49	7	Grain	0.0050	0.0050	0.0086	0.0139	0.1821	0.0050	0.0107	0.0050	0.0065	0.3223
	4.49	61	Forage	0.0050	0.0332	0.0086	0.0441	0.0249	0.0060	0.0773	0.0107	0.0417	
	4.49	61	Immature ears	0.0050	0.0050	0.0086					0.0050	0.0050	0.0465
	4.49	61	Stover	0.0050	0.0364	0.0280	0.0669	0.0237	0.0102	0.1023	0.0384	0.0649	
	4.49	61	Grain	0.0050	0.0050	0.0086	0.0139	0.0574	0.0050	0.0117	0.0050	0.0077	0.1015
Chula, GA, 2014/2015 DKC 62-08	4.49	351	Forage	0.0050	0.0052	0.0086	0.0142	0.0067	0.0050	0.0182	0.0050	0.0087	
Chula, GA, 2014/2015 DKC 62-05	4.49	351	Immature ears	0.0050	0.0050	0.0086					0.0050	0.0050	0.0446
	4.49	351	Stover	0.0050	0.0190	0.0086	0.0289	0.0117	0.0050	0.0516	0.0050	0.0247	
	4.49	351	Grain	0.0050	0.0050	0.0086	0.0139	0.0200	0.0050	0.0050	0.0050	0.0050	0.0446
Richland, IA, 2014/2015 P1023AM	4.52	7	Forage	0.0050	0.0050	0.0086	0.0139	0.0055	0.0050	0.0149	0.0050	0.0107	
	4.52	7	Immature ears	0.0050	0.0050	0.0086					0.0050	0.0050	0.0443
	4.52	7	Stover	0.0050	0.0067	0.0086	0.0157	0.0052	0.0050	0.0124	0.0050	0.0084	
	4.52	7	Grain	0.0050	0.0050	0.0086	0.0139	0.0079	0.0050	0.0050	0.0050	0.0050	0.0443
Richland, IA, 2014/2015 P0506AM	4.52	60	Forage	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0136	0.0050	0.0109	
	4.52	60	Immature ears	0.0050	0.0050	0.0086					0.0050	0.0050	0.0443
	4.52	60	Stover	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0520	0.0050	0.0164	
	4.52	60	Grain	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0050	0.0050	0.0050	0.0443
	4.45	351	Forage	0.0050	0.0050	0.0087	0.0141	0.0116	0.0050	0.0300	0.0050	0.0131	
	4.45	351	Immature ears	0.0050	0.0050	0.0087					0.0050	0.0050	0.0450
	4.45	351	Stover	0.0050	0.0310	0.0087	0.0418	0.0159	0.0050	0.0629	0.0055	0.0307	
	4.45	351	Grain	0.0050	0.0050	0.0087	0.0141	0.0269	0.0050	0.0050	0.0050	0.0050	0.0477
Stewardson, IL, 2014/2015	4.46	18	Forage	0.0050	0.0073	0.0087	0.0165	0.0093	0.0095	0.0778	0.0050	0.0728	
	4.46	18	Immature ears	0.0050	0.0050	0.0087					0.0050	0.0050	0.0540

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Pioneer P7443R	4.46	18	Stover	0.0050	0.0316	0.0338	0.0676	0.0181	0.0050	0.1607	0.0050	0.0804	
	4.46	18	Grain	0.0050	0.0050	0.0087	0.0140	0.0261	0.0055	0.0181	0.0050	0.0098	0.0654
	4.46	63	Forage	0.0050	0.0050	0.0087	0.0140	0.0050	0.0053	0.0276	0.0050	0.0266	
	4.46	63	Immature ears	0.0050	0.0050	0.0087					0.0050	0.0050	0.0464
	4.46	63	Stover	0.0050	0.0181	0.0113	0.0306	0.0058	0.0113	0.0603	0.0108	0.0500	
	4.46	63	Grain	0.0050	0.0050	0.0087	0.0140	0.0131	0.0063	0.0068	0.0050	0.0050	0.0494
	4.46	361	Forage	0.0050	0.0050	0.0087	0.0140	0.0050	0.0050	0.0291	0.0050	0.0259	
	4.46	361	Immature ears	0.0050	0.0050	0.0087					0.0050	0.0025	0.0449
	4.46	361	Stover	0.0050	0.0186	0.0126	0.0324	0.0050	0.0050	0.0552	0.0050	0.0249	
	4.46	361	Grain	0.0050	0.0050	0.0087	0.0140	0.0055	0.0050	0.0063	0.0050	0.0050	0.0468
Carlyle, IL, 2014/2015 FS 66JV1 RIB	4.49	11	Forage	0.0050	0.0105	0.0086	0.0198	0.0107	0.0092	0.1023	0.0050	0.0474	
	4.49	11	Immature ears	0.0050	0.0050	0.0086					0.0050	0.0050	0.0446
	4.49	11	Stover	0.0050	0.0549	0.0379	0.0965	0.0190	0.0050	0.1422	0.0050	0.0374	
	4.49	11	Grain	0.0050	0.0050	0.0086	0.0139	0.0157	0.0050	0.0177	0.0050	0.0082	0.0638
Carlyle, IL, 2014/2015 130670	4.49	60	Forage	0.0050	0.0107	0.0086	0.0201	0.0055	0.0065	0.0574	0.0050	0.0292	
	4.49	60	Immature ears	0.0050	0.0050	0.0086					0.0050	0.0050	0.0446
	4.49	60	Stover	0.0050	0.0429	0.0086	0.0544	0.0085	0.0102	0.1646	0.0087	0.0698	
	4.49	60	Grain	0.0050	0.0050	0.0086	0.0139	0.0110	0.0052	0.0050	0.0050	0.0050	0.0450
Carlyle, IL, 2014/2015 FS 63SV1 RIB	4.49	365	Forage	0.0050	0.0070	0.0086	0.0161	0.0050	0.0050	0.0307	0.0050	0.0167	
	4.49	365	Immature ears	0.0050	0.0050	0.0086					0.0050	0.0050	0.0446
	4.49	365	Stover	0.0050	0.0427	0.0086	0.0542	0.0052	0.0050	0.0873	0.0050	0.0329	
	4.49	365	Grain	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0057	0.0050	0.0050	0.0457
Uvalde, TX, 2014/2015 DKC 69-43	4.49	7	Forage	0.0050	0.0297	0.0086	0.0403	0.0102	0.0150	0.1023	0.0085	0.0748	
	4.49	7	Immature ears	0.0050	0.0050	0.0086					0.0050	0.0050	0.0446
	4.92	7	Stover	0.0064	0.0820	0.0350	0.1225	0.0260	0.0046	0.1912	0.0102	0.1138	
	4.92	7	Grain	0.0046	0.0046	0.0079	0.0127	0.0055	0.0046	0.0091	0.0046	0.0048	0.0476
Uvalde, TX, 2014/2015 DKC 64-69	4.54	67	Forage	0.0049	0.0101	0.0085	0.0193	0.0072	0.0086	0.0294	0.0057	0.0128	
	4.54	67	Immature ears	0.0049	0.0049	0.0085					0.0049	0.0049	0.0441
	4.54	67	Stover	0.0049	0.0617	0.0085	0.0744	0.0192	0.0064	0.1110	0.0158	0.0765	
	4.54	67	Grain	0.0049	0.0049	0.0085	0.0138	0.0165	0.0049	0.0086	0.0049	0.0049	0.0497
	4.49	317	Forage	0.0050	0.0072	0.0086	0.0163	0.0060	0.0065	0.0359	0.0055	0.0165	
	4.49	317	Immature ears	0.0050	0.0050	0.0086					0.0050	0.0050	0.0446
	4.49	317	Stover	0.0050	0.0369	0.0086	0.0480	0.0140	0.0052	0.0998	0.0150	0.0723	
	4.49	317	Grain	0.0050	0.0050	0.0086	0.0139	0.0140	0.0050	0.0057	0.0050	0.0050	0.0457

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 172 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in sorghum rotational crops (duplicate samples from plot) in the United States

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Porterville, CA, 2013/2014	1.26	7	Forage	<0.01	0.021	<0.01	0.084	0.048	0.26	ND	0.023
				<0.01	0.01A	<0.01	0.039	0.020	0.14	ND	0.011
			Mean	<0.01	0.016	<0.01	0.062	0.034	0.20	<0.01	0.017

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
AF7401①		7	Stover	ND	0.046	ND	0.18	0.059	0.44	<0.01	0.093
				ND	0.035	ND	0.15	0.018	0.26	ND	0.039
			Mean	<0.01	0.041	<0.01	0.165	0.039	0.35	<0.01	0.066
		7	Grain	ND	ND	ND	0.044	<0.01	<0.01	ND	<0.01
				ND	ND	ND	0.033	<0.01	0.010	ND	<0.01
			Mean	<0.01	<0.01	<0.01	0.039	<0.01	<0.01	<0.01	<0.01
		60	Forage	ND	<0.01	ND	0.031	0.013	0.10	ND	0.011
				ND	<0.01	ND	0.032	0.012	0.088	ND	<0.01
			Mean	<0.01	<0.01	<0.01	0.032	0.012	0.094	<0.01	0.0105
		60	Stover	ND	0.014	ND	0.079	0.028	0.23	<0.01	0.036
				ND	0.016	ND	0.065	0.011	0.14	ND	0.015
			Mean	<0.01	0.015	<0.01	0.072	0.020	0.185	<0.01	0.026
		60	Grain	ND	ND	ND	0.022	ND	ND	ND	ND
				ND	ND	ND	0.017	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.020	<0.01	<0.01	<0.01	<0.01
Porterville, CA, 2013/2014 AF7401①	2.51	7	Forage	<0.01	0.014	<0.01	0.076	0.041	0.29	<0.01	0.030
				ND	0.012	<0.01	0.053	0.027	0.17	ND	0.021
			Mean	<0.01	0.013	<0.01	0.065	0.034	0.23	<0.01	0.0255
		7	Stover	ND	0.066	0.021	0.31	0.13	0.94	0.017	0.14
				ND	0.069	0.019	0.39	0.17	1.0	0.026	0.21
			Mean	<0.01	0.068	0.020	0.35	0.15	0.97	0.022	0.175
		7	Grain	ND	ND	ND	0.043	<0.01	<0.01	ND	ND
				ND	ND	ND	0.039	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.041	<0.01	<0.01	<0.01	<0.01
		60	Forage	<0.01	0.037	0.015	0.12	0.068	0.67	<0.01	0.058
				ND	0.016	<0.01	0.058	0.046	0.34	<0.01	0.032
			Mean	<0.01	0.027	<0.013	0.089	0.057	0.505	<0.01	0.045
		60	Stover	ND	0.028	<0.01	0.19	0.065	0.67	<0.01	0.084
				ND	0.035	0.011	0.20	0.063	0.75	<0.01	0.085
			Mean	<0.01	0.032	<0.0105	0.195	0.064	0.71	<0.01	0.085
		60	Grain	ND	ND	ND	0.042	<0.01	<0.01	ND	ND
				ND	ND	ND	0.036	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.039	<0.01	<0.01	<0.01	<0.01
		361	Forage	ND	<0.01	ND	0.015	<0.01	0.19	ND	0.020
				ND	<0.01	ND	0.020	<0.01	0.19	ND	0.023
			Mean	<0.01	<0.01	<0.01	0.018	<0.01	0.19	<0.01	0.022
		361	Stover	ND	0.020	ND	0.010	0.018	0.35	<0.01	0.037
				ND	0.013	ND	0.034	<0.01	0.21	<0.01	0.023
			Mean	<0.01	0.017	<0.01	0.022	0.014	0.28	<0.01	0.030
		361	Grain	ND	ND	ND	0.089	ND	0.011	ND	<0.01
				ND	ND	ND	0.051	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.070	<0.01	<0.0105	<0.01	<0.01

Notes:

① Shepard (2020 DuPont-36791 rev 1). A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 173 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in sorghum rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds) in the United States

Location	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Porterville,	1.26	7	Forage	0.0178	0.0276	0.0307	0.0601	0.1093	0.0604	0.3556	0.0178	0.0302	

Location	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-A5760+IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
CA, 2013/2014 AF7401	1.26	7	Stover	0.0178	0.0720	0.0307	0.1076	0.2933	0.0684	0.6222	0.0178	0.1173	
	1.26	7	Grain	0.0178	0.0178	0.0307	0.0497	0.0684	0.0178	0.0178	0.0178	0.0178	0.1589
	1.26	60	Forage	0.0178	0.0178	0.0307	0.0497	0.0560	0.0222	0.1671	0.0178	0.0187	
	1.26	60	Stover	0.0178	0.0267	0.0307	0.0592	0.1280	0.0347	0.3289	0.0178	0.0453	
	1.26	60	Grain	0.0178	0.0178	0.0307	0.0497	0.0347	0.0178	0.0178	0.0178	0.0178	0.1589
	2.51	7	Forage	0.0045	0.0054	0.0154	0.0211	0.0236	0.0120	0.0759	0.0045	0.0094	
	2.51	7	Stover	0.0089	0.0602	0.0308	0.0952	0.3124	0.1339	0.8657	0.0192	0.1562	
	2.51	7	Grain	0.0089	0.0089	0.0154	0.0249	0.0366	0.0089	0.0089	0.0089	0.0089	0.0797
	2.51	60	Forage	0.0089	0.0236	0.0193	0.0445	0.0794	0.0509	0.4507	0.0089	0.0402	
	2.51	60	Stover	0.0089	0.0281	0.0162	0.0462	0.1740	0.0571	0.6336	0.0089	0.0754	
	2.51	60	Grain	0.0089	0.0089	0.0154	0.0249	0.0348	0.0089	0.0089	0.0089	0.0089	0.0797
	2.51	361	Forage	0.0089	0.0089	0.0154	0.0249	0.0156	0.0089	0.1696	0.0089	0.0192	
	2.51	361	Stover	0.0089	0.0147	0.0154	0.0311	0.0196	0.0125	0.2499	0.0089	0.0268	
	2.51	361	Grain	0.0089	0.0089	0.0154	0.0249	0.0625	0.0089	0.0094	0.0089	0.0089	0.1106

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 174 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis [D]) in wheat rotational crops

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Charantonnay, Rhone Alpes, France 2014 Solehio⑥	3.34	7	Forage	ND	ND	ND	ND	ND	ND	ND	ND
	3.06	60		ND	0.020	0.011	0.038	ND	0.018	ND	0.11
	3.33	360		ND	0.024	0.016	0.016	ND	0.046	ND	0.32
	3.34	7	Hay	ND	ND	ND	ND	ND	ND	ND	ND
	3.06	60		<0.01	0.043	0.018	0.044	ND	0.033	<0.01	0.16
	3.33	360		ND	0.037	0.025	0.013	ND	0.053	ND	0.17
	3.34	7	Grain	ND	ND	ND	ND	ND	ND	ND	ND
	3.06	60		ND	ND	ND	0.052	ND	ND	ND	ND
	3.33	360		ND	ND	ND	0.010	ND	ND	ND	ND
	3.34	7	Straw	ND	ND	ND	ND	ND	ND	ND	ND
	3.06	60		ND	0.098	0.066	0.069	<0.01	0.14	<0.01	0.31
	3.33	360		ND	0.049	0.033	0.016	ND	0.062	ND	0.11
Alpicat Catalunya Spain 2014 Mecano⑥	3.33	10	Forage	<0.01	0.034	0.027	0.065	<0.01	0.051	ND	0.28
	3.07	62		<0.01	0.023	0.017	0.066	<0.01	0.024	ND	0.16
	3.33	364		ND	0.025	0.025	0.041	<0.01	0.049	ND	0.17
	3.33	10	Hay	<0.01	0.043	0.036	0.079	<0.01	0.056	ND	0.37
	3.07	62		<0.01	0.031	0.016	0.074	<0.01	0.024	ND	0.20
	3.33	364		ND	0.016	0.014	0.046	ND	0.033	ND	0.13
	3.33	10	Grain	ND	ND	ND	0.029	ND	ND	ND	ND
	3.07	62		ND	ND	ND	0.033	ND	ND	ND	ND
	3.33	364		ND	ND	ND	0.035	ND	ND	ND	ND
	3.33	10	Straw	<0.01	0.10	0.089	0.072	<0.01	0.18	ND	0.42
	3.07	62		<0.01	0.080	0.074	0.13	<0.01	0.13	ND	0.33
	3.33	364		ND	0.061	0.059	0.029	<0.01	0.12	ND	0.45
Termens Catalunya Spain 2014 Mecano⑥	3.27	7	Forage	ND	<0.01	ND	0.017	<0.01	<0.01	ND	0.068
	2.96	63		ND	<0.01	ND	0.015	ND	<0.01	ND	0.063
	3.33	364		ND	0.012	0.011	0.047	ND	0.028	ND	0.10
	3.27	7	Hay	0.020	0.019	0.016	0.042	<0.01	0.016	ND	0.10
	2.96	63		<0.01	0.013	<0.01	0.026	<0.01	0.012	ND	0.081
	3.33	364		ND	0.010	<0.01	0.024	ND	0.014	ND	0.088
	3.27	7	Grain	ND	<0.01	ND	0.016	ND	ND	ND	<0.01

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
	2.96	63		ND	ND	ND	0.012	ND	ND	ND	ND
	3.33	364		ND	ND	ND	0.030	ND	ND	ND	ND
	3.27	7	Straw	0.019	0.051	0.040	0.069	<0.01	0.073	ND	0.13
	2.96	63		<0.01	0.040	0.022	0.049	<0.01	0.047	ND	0.078
	3.33	364		ND	0.037	0.022	0.027	<0.01	0.048	ND	0.18
Los Palacios Andalucía Spain 2014 Avispa⑥	3.30	10	Forage	ND	0.012	ND	0.015	ND	<0.01	ND	0.053
	3.22	61		ND	<0.01	ND	0.012	ND	<0.01	ND	0.035
	3.31	357		ND	<0.01	ND	0.011	ND	<0.01	ND	0.039
	3.30	10	Hay	0.011	0.035	0.012	0.079	ND	0.018	ND	0.17
	3.22	61		<0.01	0.018	<0.01	0.034	ND	<0.01	ND	0.083
	3.31	357		ND	<0.01	<0.01	0.033	ND	<0.01	ND	0.053
	3.30	10	Grain	ND	ND	ND	0.053	ND	<0.01	ND	0.013
	3.22	61		ND	ND	ND	0.024	ND	ND	ND	ND
	3.31	357		ND	ND	ND	ND	ND	ND	ND	ND
	3.30	10	Straw	0.16	0.18	0.10	0.44	<0.01	0.14	0.012	0.62
	3.22	61		0.045	0.12	0.056	0.22	ND	0.098	<0.01	0.43
	3.31	357		<0.01	0.019	0.025	0.060	0.01	0.044	ND	0.10
Aguadulce Andalucía Spain 2014 Avispa⑥	3.27	10	Forage	ND	ND	ND	<0.01	ND	<0.01	ND	0.044
	3.12	62		ND	ND	ND	<0.01	ND	<0.01	ND	0.028
	3.26	358		ND	ND	ND	ND	ND	<0.01	ND	0.046
	3.27	10	Hay	ND	0.015	0.014	0.020	ND	0.021	ND	0.12
	3.12	62		ND	0.016	0.012	0.023	ND	0.022	ND	0.14
	3.26	358		ND	0.015	0.012	0.017	ND	0.025	ND	0.15
	3.27	10	Grain	ND	ND	ND	ND	ND	ND	ND	ND
	3.12	62		ND	ND	ND	ND	ND	ND	ND	ND
	3.26	358		ND	ND	ND	ND	ND	ND	ND	ND
	3.27	10	Straw	0.013	0.028	0.013	0.022	ND	0.031	0.018	0.093
	3.12	62		<0.01	0.018	<0.01	0.015	ND	0.021	0.019	0.068
	3.26	358		ND	0.020	0.012	0.014	ND	0.024	ND	0.034
Alpicat, Catalunya, Spain 2013 Califa sur④	1.25	28	Forage	ND	<0.01	ND	<0.01	ND	<0.01	ND	0.014
	1.25	128		ND	0.012	<0.01	0.010	ND	0.012	ND	0.040
	2.50	28		ND	0.022	<0.01	0.034	<0.01	0.019	ND	0.085
	2.50	142		ND	0.037	0.018	0.032	<0.01	0.039	ND	0.16
	2.50	243		ND	0.026	0.013	0.039	<0.01	0.034	ND	0.13
	1.25	28	Hay	ND	0.045	0.066	0.075	<0.01	0.12	<0.01	0.26
	1.25	128		ND	0.014	0.028	0.022	ND	0.046	ND	0.053
	2.50	28		ND	0.023	0.019	0.044	ND	0.027	ND	0.11
	2.50	142		ND	0.034	0.032	0.058	<0.01	0.069	<0.01	0.19
	2.50	243		ND	0.035	0.045	0.064	<0.01	0.086	ND	0.17
	1.25	28	Grain	ND	ND	ND	<0.01	ND	ND	ND	ND
	1.25	128		ND	ND	ND	ND	ND	ND	ND	ND
	2.50	28		ND	ND	ND	<0.01	ND	ND	ND	ND
	2.50	142		ND	ND	ND	<0.01	ND	ND	ND	ND
	2.50	243		ND	ND	ND	<0.01	ND	ND	ND	ND
	1.25	28	Straw	ND	0.026	0.018	0.040	ND	0.055	ND	0.15
	1.25	128		ND	0.017	0.029	0.022	ND	0.042	ND	0.077
	2.50	28		ND	0.051	0.035	0.065	<0.01	0.066	ND	0.21
	2.50	142		ND	0.066	0.045	0.062	<0.01	0.095	ND	0.28
	2.50	243		ND	0.043	0.028	0.058	<0.01	0.068	ND	0.21
Aguadulce, Andalucia Spain 2013 Avispa④	1.25	29	Forage	ND	<0.01	ND	<0.01	ND	<0.01	ND	0.043
	1.25	145		ND	0.018	<0.01	<0.01	ND	0.013	ND	0.096
	2.50	27		<0.01	0.034	0.010	0.018	<0.01	0.024	ND	0.18
	2.50	145		ND	0.018	<0.01	<0.01	ND	0.010	ND	0.089
	2.50	242		ND	0.018	<0.01	<0.01	ND	0.012	ND	0.098
	1.25	29	Hay	ND	<0.01	ND	ND	ND	ND	ND	0.025
	1.25	145		ND	0.031	0.018	0.020	ND	0.027	ND	0.26
	2.50	27		ND	0.044	0.012	0.026	ND	0.012	ND	0.27
	2.50	145		ND	0.099	0.044	0.069	<0.01	0.082	<0.01	0.77
	2.50	242		ND	0.034	0.021	0.025	ND	0.034	ND	0.33
	1.25	29	Grain	ND	ND	ND	ND	ND	ND	ND	ND

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
	1.25	145		ND	ND	ND	ND	ND	ND	ND	ND
	2.50	27		ND	ND	ND	<0.01	ND	ND	ND	ND
	2.50	145		ND	ND	ND	<0.01	ND	ND	ND	ND
	2.50	242		ND	ND	ND	<0.01	ND	ND	ND	ND
	1.25	29	Straw	ND	0.013	<0.01	0.012	ND	0.027	ND	0.077
	1.25	145		ND	0.012	<0.01	<0.01	ND	0.020	ND	0.071
	2.50	27		<0.01	0.053	0.034	0.030	0.011	0.096	ND	0.32
	2.50	145		0.014	0.12	0.091	0.069	<0.01	0.23	ND	0.84
	2.50	242		0.010	0.082	0.058	0.049	<0.01	0.14	ND	0.57
Gardner, ND, United Sates 2014/2015 Prosper③	4.50	11	Forage	<0.01	<0.01	<0.01	0.016	ND	0.022	ND	0.053
				<0.01	<0.01	ND	0.013	ND	0.019	ND	0.044
			Mean	<0.01	<0.01	<0.01	0.0145	<0.01	0.0205	<0.01	0.0485
		11	Hay	<0.01	0.018	ND	0.035	ND	0.039	ND	0.089
				<0.01	0.022	<0.01	0.039	<0.01	0.040	ND	0.093
			Mean	<0.01	0.020	<0.01	0.037	<0.01	0.0395	<0.01	0.091
		11	Straw	<0.01	0.029	<0.01	0.044	ND	0.055	ND	0.046
				<0.01	0.029	<0.01	0.044	ND	0.048	ND	0.042
			Mean	<0.01	0.029	<0.01	0.044	<0.01	0.0515	<0.01	0.044
			Grain	ND[A]	<0.01	ND	0.012	ND	ND	ND	ND
				ND[A]	ND	ND	0.011	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0115	<0.01	<0.01	<0.01	<0.01
Gardner, ND, United Sates 2014/2015 Prosper③	4.50	61	Forage	ND	<0.01	ND	<0.01	ND	0.026	ND	0.057
				ND	<0.01	<0.01	<0.01	ND	0.031	ND	0.075
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0285	<0.01	0.066
		61	Hay	<0.01	0.023	0.014	0.037	<0.01	0.098	ND	0.12
				<0.01	0.027	0.014	0.037	<0.01	0.097	ND	0.12
			Mean	<0.01	0.025	0.014	0.037	<0.01	0.0975	<0.01	0.12
		61	Straw	ND	0.021	ND	0.036	ND	0.099	ND	0.13
				ND	0.023	<0.01	0.039	ND	0.10	ND	0.14
			Mean	<0.01	0.022	<0.01	0.0375	<0.01	0.0995	<0.01	0.135
			Grain	ND[A]	ND	ND	0.018	ND	ND	ND	ND
				ND[A]	ND	ND	0.018	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.018	<0.01	<0.01	<0.01	<0.01
		367	Forage	ND	<0.01	ND	<0.01	ND	0.030	ND	0.074
				ND	<0.01	ND	0.01[B]	ND	0.034	ND	0.086
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.032	<0.01	0.080
		367	Hay	ND	0.012	ND	0.022	ND	0.056	ND	0.18
				ND	<0.01	ND	0.018	ND	0.043	ND	0.14
			Mean	<0.01	<0.011	<0.01	0.020	<0.01	0.0495	<0.01	0.16
		367	Straw	<0.01	0.017	<0.01	0.019	<0.01	0.025	ND	0.034
				ND	0.016	<0.01	0.019	ND	0.029	ND	0.033
			Mean	<0.01	0.0165	<0.01	0.019	<0.01	0.027	<0.01	0.0335
Gardner, ND, United Sates 2014/2015, Prosper③	4.50	367	Grain	ND	ND	ND	<0.01	ND	ND	ND	ND
				ND	ND	ND	0.011	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.0105	<0.01	<0.01	<0.01	<0.01
Richland, IA, United Sates 2014/2015 Briggs③	4.43	7	Forage	<0.01	0.017	<0.01	0.030	<0.01	0.049	ND	0.099
				<0.01	0.019	<0.01	0.033	<0.01	0.052	ND	0.11
			Mean	<0.01	0.018	<0.01	0.0315	<0.01	0.0505	<0.01	0.1045
		7	Hay	<0.01	0.15	0.020	0.20	0.028	0.34	0.026	0.74
				<0.01	0.12	0.017	0.16	0.033	0.27	0.023	0.60
			Mean	<0.01	0.135	0.0185	0.18	0.0305	0.305	0.0245	0.67
		7	Straw	<0.01	0.20	0.061	0.16	0.021	0.46	0.010	0.44
				<0.01	0.21	0.077	0.15	0.026	0.46	0.013	0.44
			Mean	<0.01	0.205	0.069	0.155	0.0235	0.46	0.0115	0.44
			Grain	ND	ND	ND	0.052	<0.01	<0.01	<0.01	<0.01
				ND	ND	ND	0.047	<0.01	<0.01	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	0.0495	<0.01	<0.01	<0.01	<0.01
			Pre-processed Grain	ND	ND	ND	0.057	<0.01	<0.01	<0.01	<0.01

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			AGF	ND	0.020	0.024	0.080	0.050	0.036	0.024	0.032
Richland, IA, United Sates 2014/2015 GV662③	4.49	69	Forage	ND	0.035	<0.01	0.064	ND	0.12	ND	0.16
				ND	0.027	ND	0.052	ND	0.094	ND	0.12
			Mean	<0.01	0.031	<0.01	0.058	<0.01	0.107	<0.01	0.14
		69	Hay	ND	0.072	0.011	0.21	ND	0.28	<0.01	0.38
				ND	0.083	0.014	0.22	ND	0.30	<0.01	0.42
			Mean	<0.01	0.0775	0.0125	0.215	<0.01	0.29	<0.01	0.40
		69	Straw	ND	0.10	0.041	0.12	<0.01	0.26	<0.01	0.23
				ND	0.095	0.036	0.12	<0.01	0.34	<0.01	0.25
			Mean	<0.01	0.0975	0.0385	0.12	<0.01	0.30	<0.01	0.24
			Grain	ND	ND	ND	0.11	ND	ND	ND	ND
				ND	ND	ND	0.13	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.12	<0.01	<0.01	<0.01	<0.01
Richland, IA, United Sates 2014/2015 RB07③	4.37	345	Forage	ND	0.013	ND	0.039	0.011	0.066	ND	0.14
				ND	0.010	ND	0.022	<0.01	0.027	ND	0.057
			Mean	<0.01	0.0115	<0.01	0.0305	0.0105	0.0465	<0.01	0.0985
		345	Hay	ND	0.040	ND	0.10	0.011	0.13	<0.01	0.22
				ND	0.043	ND	0.11	0.011	0.15	<0.01	0.25
			Mean	<0.01	0.0415	<0.01	0.105	0.011	0.14	<0.01	0.235
		345	Straw	ND	0.089	0.027	0.15	0.033	0.38	<0.01	0.44
				ND	0.064	0.020	0.11	0.023	0.33	<0.01	0.42
			Mean	<0.01	0.0765	0.0235	0.13	0.028	0.355	<0.01	0.43
Richland, IA, United Sates 2014/2015, RB07③	4.37	345	Grain	ND	ND	ND	0.026	ND	ND	ND	ND
				ND	ND	ND	<0.01	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	<0.018	<0.01	<0.01	<0.01	<0.01
Lime Springs, IA, United Sates 2014/2015 SY Soren③	4.48	6	Forage	<0.01	0.070	0.018	0.22	0.085	0.77	<0.01	2.0
				0.013	0.073	0.023	0.26	0.14	0.84	<0.01	2.3
			Mean	<0.0115	0.0715	0.0205	0.24	0.1125	0.805	<0.01	2.15
		6	Hay	<0.01	0.25[A]	0.061	0.60	0.046[A]	1.4	0.011	1.9
				ND	0.23[A]	0.077	0.78	0.060[A]	1.8	0.014	2.1
			Mean	<0.01	0.24	0.069	0.69	0.053	1.6	0.0125	2.0
		6	Straw	ND	0.064	0.028	0.21	0.17[A]	0.96	0.023	1.6
				ND	0.078	0.047	0.20	0.18[A]	1.1	0.017	1.5
			Mean	<0.01	0.071	0.0375	0.205	0.175	1.03	0.020	1.55
			Grain	ND	<0.01	ND	0.21	0.019	0.039	<0.01	0.048
				ND	<0.01	<0.01	0.20	0.023	0.035	<0.01	0.045
			Mean	<0.01	<0.01	<0.01	0.205	0.021	0.037	<0.01	0.0465
Lime Springs, IA, United Sates 2014/2015 Forefront③	4.45	64	Forage	ND	0.032	<0.01	0.12	0.01[B]	0.16	<0.01	0.25
				ND	0.036	ND	0.13	0.020	0.21	<0.01	0.32
			Mean	<0.01	0.034	0.005	0.125	0.015	0.185	<0.01	0.285
Lime Springs, IA, United Sates 2014/2015 Forefront③	4.45	64	Hay	ND	0.078	0.011	0.24	0.055	0.58	0.012	0.56
				ND	0.085	0.014	0.29	0.070	0.69	<0.01	0.63
			Mean	<0.01	0.0815	0.0125	0.265	0.0625	0.635	<0.011	0.595
		64	Straw	ND	0.081	0.059	0.11	0.039	0.46	<0.01	0.24
				ND	0.073	0.037	0.13	0.050	0.52	<0.01	0.25
			Mean	<0.01	0.077	0.048	0.12	0.0445	0.49	<0.01	0.245
			Grain	ND	<0.01	ND	0.10	ND	<0.01	<0.01	ND
				ND	<0.01	<0.01	0.13	<0.01	0.017	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	0.115	<0.01	<0.0135	<0.01	<0.01
	4.45	365	Forage	ND	0.01[B]	ND	0.060	<0.01	0.067	ND	0.10
				ND	0.013	ND	0.071	<0.01	0.074	ND	0.12
			Mean	<0.01	0.0115	<0.01	0.0655	<0.01	0.0705	<0.01	0.11
		365	Hay	ND	0.038	ND	0.084	<0.01	0.17	ND	0.22
				ND	0.023	ND	0.067	<0.01	0.12	ND	0.18
			Mean	<0.01	0.0305	<0.01	0.0755	<0.01	0.145	<0.01	0.20
		365	Straw	ND	0.026	<0.01	0.050	<0.01	0.16	ND	0.13
				ND	0.026	<0.01	0.054	<0.01	0.15	ND	0.15
			Mean	<0.01	0.026	<0.01	0.052	<0.01	0.155	<0.01	0.14

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Grain	ND	ND	ND	0.046	ND	ND	ND	ND
				ND	ND	ND	0.046	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.046	<0.01	<0.01	<0.01	<0.01
York, NE, United Sates 2014/2015 Prosper HS③	4.48	8	Forage	<0.01	0.028	0.011	0.047	0.011	0.077	<0.01	0.27
				<0.01	0.029	0.011	0.047	0.012	0.072	<0.01	0.28
			Mean	<0.01	0.0285	0.011	0.047	0.0115	0.0745	<0.01	0.275
		8	Hay	0.013C	0.052[A,c]	<0.01[A,c]	0.099[A,c]	<0.01[A,c]	0.12[A,c]	<0.01[A,c]	0.20[A,c]
				0.013	0.056	<0.01	0.10	<0.01	0.11	<0.01	0.33
			Mean	0.013	0.054	<0.01	0.0995	<0.01	0.115	<0.01	0.275
		8	Straw	ND	0.027	<0.01	0.033	<0.01	0.057	ND	0.086
				ND	0.031	0.010	0.039	<0.01	0.044	ND	0.088
			Mean	<0.01	0.029	<0.01[B]	0.036	<0.01	0.0505	<0.01	0.087
			Grain	ND	ND	ND	0.022	ND	<0.01	ND	ND
				ND	ND	ND	0.021	ND	<0.01	ND	ND
			Mean	<0.01	<0.01	<0.01	0.0215	<0.01	<0.01	<0.01	<0.01
York, NE, United Sates 2014/2015 Overland③		145	Forage	ND	<0.01	ND	0.037	ND	0.062	ND	0.11
				ND	<0.01	ND	0.036	ND	0.057	ND	0.10
			Mean	<0.01	<0.01	<0.01	0.0365	<0.01	0.0595	<0.01	0.105
		145	Hay	<0.01	0.032	ND	0.045	<0.01	0.073	<0.01	0.15
				<0.01	0.039	ND	0.062	<0.01	0.098	<0.01	0.19
			Mean	<0.01	0.0355	<0.01	0.0535	<0.01	0.0855	<0.01	0.17
		145	Straw	<0.01	0.044	0.029	0.053	<0.01	0.10	ND	0.14
				ND	0.031	0.017	0.040	<0.01	0.10	ND	0.12
			Mean	<0.01	0.0375	0.023	0.0465	<0.01	0.10	<0.01	0.13
York, NE, United Sates, 2014/2015 Overland③	4.48	145	Grain	ND[A]	ND	ND	0.043	<0.01	<0.01[A]	ND[A]	<0.01[A]
				ND[A]	ND	ND	0.031	ND	<0.01[A]	ND[A]	<0.01[A]
			Mean	<0.01	<0.01	<0.01	0.037	<0.01	<0.01	<0.01	<0.01
York, NE, United Sates 2014/2015 Prosper③		363	Forage	ND	0.014	<0.01	0.017	<0.01	0.030	<0.01	0.061
				ND	0.014	ND	0.017	<0.01	0.030	<0.01	0.061
			Mean	<0.01	0.014	<0.01	0.017	<0.01	0.030	<0.01	0.061
		363	Hay	ND	0.025	<0.01	0.042	ND	0.053	ND	0.097
				ND	0.029	<0.01	0.045	ND	0.051	ND	0.090
			Mean	<0.01	0.027	<0.01	0.0435	<0.01	0.052	<0.01	0.0935
		363	Straw	ND	0.034	0.015	0.045	0.01[B]	0.099	<0.01	0.13
				<0.01	0.034	0.023	0.046	<0.01	0.089	ND	0.11
			Mean	<0.01	0.034	0.019	0.0455	<0.01	0.094	<0.01	0.12
			Grain	ND[A]	ND[A]	ND[A]	0.054	ND	<0.01[A]	ND[A]	<0.01[A]
				ND[A]	ND[A]	ND[A]	0.054	<0.01	<0.01	ND[A]	<0.01[A]
			Mean	<0.01	<0.01	<0.01	0.054	<0.01	<0.01	<0.01	<0.01
Uvalde, TX, United Sates 2014/2015/2016 Greer-Winter③		10	Forage	0.022	0.042	0.019	0.088	0.033	0.64	<0.01	1.2
				0.017	0.027	0.014	0.058	0.025	0.36	<0.01	0.73
			Mean	0.0195	0.0345	0.0165	0.073	0.029	0.50	<0.01	0.965
		10	Hay	<0.01	0.14	0.017	0.23	0.017	0.29	0.014	0.88
				<0.01	0.12	0.022	0.21	0.016	0.24	0.011	0.56
			Mean	<0.01	0.13	0.0195	0.22	0.0165	0.265	0.0125	0.72
Uvalde, TX, United Sates 2014/2015/2016 Greer-Winter③	4.52	10	Straw	<0.01[A]	0.12[A]	0.096[A]	0.15[A]	0.012[A]	0.19[A]	<0.01[A]	0.073[A]
				0.011[A]	0.11[A]	0.11[A]	0.15[A]	0.064[A]	0.92[A]	ND[A]	0.10[A]
			Mean	<0.0105	0.115	0.103	0.15	0.038	0.555	<0.01	0.0865
			Grain	<0.01	0.025	0.049	0.064	0.014	1.2	ND	0.026
				<0.01	0.029	0.069	0.054	0.014	0.79	ND	0.026
			Mean	<0.01	0.027	0.059	0.059	0.014	0.995	<0.01	0.026
Uvalde, TX, United Sates 2014/2015/2016 Expresso-Spring③		65	Forage	0.017	0.075	0.031	0.12	0.044	0.47	<0.01	1.2
				<0.01	0.035	0.013	0.056	0.016	0.18	ND	0.56
			Mean	<0.0135	0.055	0.022	0.088	0.030	0.325	<0.01	0.88
		65	Hay	<0.01	0.11	0.018	0.19	0.034	0.78	0.014	1.7
				0.012	0.12	0.021	0.18	0.047	0.62	0.016	1.8
			Mean	<0.011	0.115	0.0195	0.185	0.0405	0.70	0.015	1.8

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
		65	Straw	0.073[A] 0.15[A]	0.30[A] 0.35[A]	0.37[A] 0.51[A]	0.20[A] 0.30[A]	0.039[A] 0.025[A]	1.0[A] 1.0[A]	<0.01[A] <0.01[A]	0.30[A] 0.40[A]
			Mean	0.1115	0.325	0.44	0.25	0.032	1.0	<0.01	0.35
			Grain	<0.01 <0.01	0.023 0.029	0.042 0.038	0.033 0.044	<0.01 0.017	0.65 0.87	ND ND	0.023 0.043
			Mean	<0.01	0.026	0.040	0.0385	<0.0135	0.76	<0.01	0.033
Uvalde, TX, United Sates, 2014-2016 Greer-Winter③		375	Forage	ND ND	<0.01 0.011	ND ND	0.035 0.050	0.01[B] 0.014	0.17 0.19	ND ND	0.30 0.38
			Mean	<0.01	<0.0105	<0.01	0.0425	0.012	0.18	<0.01	0.34
Uvalde, TX, United Sates 2014/2015/2016 Greer-Winter③	4.52	375	Hay	ND ND	0.063 0.044	0.011 ND	0.12 0.11	0.018 0.01[B]	0.36 0.26	0.01[B] <0.01	0.89 0.64
			Mean	<0.01	0.0535	<0.0105	0.115	0.014	0.31	<0.01	0.765
		375	Straw	ND ND	0.033 0.036	<0.01 <0.01	0.076 0.069	<0.01 <0.01	0.14 0.17	ND ND	0.23 0.26
			Mean	<0.01	0.0345	<0.01	0.0725	<0.01	0.155	<0.01	0.245
			Grain	ND ND	<0.01 ND	ND ND	0.049 0.052	ND ND	<0.01 0.011	ND ND	0.013 0.016
			Mean	<0.01	<0.01	<0.01	0.0505	<0.01	<0.0105[B]	<0.01	0.0145
Richland, IA, United Sates 2013/2015 Briggs①	1.34	21	Forage	ND ND	<0.01 0.01[B]	ND ND	<0.01 <0.01	ND ND	<0.01 0.011	ND ND	0.019 0.026
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.0105	<0.01	0.0225
		21	Hay	ND ND	0.020 0.026	ND ND	0.021 0.028	ND ND	0.024 0.030	ND ND	0.060 0.073
			Mean	<0.01	0.023	<0.01	0.0245	<0.01	0.027	<0.01	0.0665
		21	Straw	ND ND	0.012 0.020	ND ND	0.013 0.016	ND ND	0.017 0.027	ND ND	0.020 0.028
			Mean	<0.01	0.016	<0.01	0.0145	<0.01	0.022	<0.01	0.024
		21	Grain	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	1.26	57	Forage	ND ND	0.01[B] 0.013	ND ND	0.013 0.018	<0.01 <0.01	0.034 0.050	ND ND	0.052 0.074
			Mean	<0.01	0.0115	<0.01	0.0155	<0.01	0.042	<0.01	0.063
		57	Hay	ND ND	0.035 0.034	ND ND	0.084 0.086	0.010 0.01[B]	0.11 0.12	ND <0.01	0.26 0.27
			Mean	<0.01	0.0345	<0.01	0.085	0.01[B]	0.115	<0.01	0.265
		57	Straw	0.019 <0.01	0.12 0.15	0.070 0.067	0.13 0.14	<0.01 <0.01	0.33 0.36	ND ND	0.25 0.27
			Mean	<0.0145	0.135	0.0685	0.135	<0.01	0.345	<0.01	0.26
		57	Grain	ND ND	<0.01 ND	ND ND	0.071 0.089	ND ND	ND ND	ND ND	ND <0.01
			Mean	<0.01	<0.01	<0.01	0.080	<0.01	<0.01	<0.01	<0.01
Richland, IA, United Sates 2013/2015 Briggs①	2.53	21	Forage	ND ND	0.021 0.016	<0.01 <0.01	0.018 0.017	<0.01 <0.01	0.037 0.028	ND ND	0.065 0.054
			Mean	<0.01	0.0185	<0.01	0.0175	<0.01	0.0325	<0.01	0.0595
		21	Straw	0.01[B] <0.01	0.064 0.055	0.054 0.042	0.053 0.047	<0.01 <0.01	0.065 0.078	ND ND	0.060 0.065
			Mean	<0.01	0.0595	0.048	0.050	<0.01	0.0715	<0.01	0.0625
		21	Grain	ND ND	ND ND	ND ND	<0.01 <0.01	ND ND	ND ND	ND ND	ND ND
			Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		57	Forage	ND ND	0.013 0.015	ND ND	0.020 0.029	<0.01 0.01[B]	0.053 0.085	ND ND	0.09 0.15
			Mean	<0.01	0.014	<0.01	0.0245	<0.01	0.069	<0.01	0.12
		57	Hay	ND ND	0.041 0.040	0.016 <0.01	0.094 0.073	0.025 0.010	0.20 0.12	<0.01 <0.01	0.44 0.25
			Mean	<0.01	0.0405	<0.013	0.0835	0.0175	0.16	<0.01	0.345
		57	Straw	0.012 <0.01	0.043 0.046	0.020 0.019	0.067 0.069	ND ND	0.11 0.11	ND ND	0.085 0.094

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Mean	<0.011	0.0445	0.0195	0.068	<0.01	0.11	<0.01	0.0895
		57	Grain	ND	ND	ND	0.068	ND	ND	ND	ND
				ND	ND	ND	0.11	ND	ND	ND	ND
			Mean	<0.01	<0.01	<0.01	0.089	<0.01	<0.01	<0.01	<0.01
Richland, IA, United Sates 2013/2015 6V653①	2.60	313	Forage	ND	0.012	ND	0.029	<0.01	0.042	ND	0.089
				ND	0.013	<0.01	0.027	<0.01	0.036	ND	0.079
			Mean	<0.01	0.0125	<0.01	0.028	<0.01	0.039	<0.01	0.084
		313	Hay	<0.01	0.055	<0.01	0.12	0.020	0.20	<0.01	0.62
				<0.01	0.062	<0.01	0.15	0.016	0.24	<0.01	0.80
			Mean	<0.01	0.0585	<0.01	0.135	0.018	0.22	<0.01	0.71
		313	Straw	ND	0.071	0.010	0.047	0.019	0.17	<0.01	0.16
				ND	0.061	0.013	0.056	0.01[B]	0.18	<0.01	0.18
			Mean	<0.01	0.066	0.0115	0.0515	0.0145	0.175	<0.01	0.17
		313	Grain	ND	ND	ND	0.052	ND	ND	ND	ND
				ND	ND	ND	0.058	ND	ND	ND	<0.01
			Mean	<0.01	<0.01	<0.01	0.055	<0.01	<0.01	<0.01	<0.01

Notes:

① Shepard 2020 DuPont-36791 rev 1.

③ Shepard 2020 DuPont-41070 rev 1.

④ Doig 2020 DuPont-36790 rev 1.

⑥ Doig 2020 DuPont-40828.

[A] Average of duplicate analyses.

[B] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[C] Control inadvertently switched with treated sample, as evidenced by residues found after duplicate analyses of both samples.

[D] For trials conducted in the a molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Table 175 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in wheat rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluazaindolizine	IN-A5760	IN-F4106	IN - A5760+IN F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
Charantonnay, Rhone Alpes, France 2014 Solehio	3.34	7	Forage	0.0067	0.0067	0.0116	0.0187	0.0067	0.0067	0.0067	0.0067	0.0067	
	3.06	60	Forage	0.0073	0.0146	0.0139	0.0295	0.0278	0.0073	0.0132	0.0073	0.0805	
	3.33	360	Forage	0.0067	0.0161	0.0186	0.0358	0.0108	0.0067	0.0309	0.0067	0.2153	
	3.34	7	Hay	0.0067	0.0067	0.0116	0.0187	0.0067	0.0067	0.0067	0.0067	0.0067	
	3.06	60	Hay	0.0073	0.0315	0.0228	0.0564	0.0322	0.0073	0.0242	0.0044	0.1171	
	3.33	360	Hay	0.0067	0.0249	0.0291	0.0556	0.0087	0.0067	0.0357	0.0067	0.1144	
	3.34	7	Grain	0.0067	0.0067	0.0116	0.0187	0.0067	0.0067	0.0067	0.0067	0.0067	0.0599
	3.06	60	Grain	0.0073	0.0073	0.0126	0.0205	0.0381	0.0073	0.0073	0.0073	0.0073	0.0674
	3.33	360	Grain	0.0067	0.0067	0.0116	0.0188	0.0067	0.0067	0.0067	0.0067	0.0067	0.0601
	3.34	7	Straw	0.0067	0.0067	0.0116	0.0187	0.0067	0.0067	0.0067	0.0067	0.0067	
	3.06	60	Straw	0.0073	0.0717	0.0835	0.1601	0.0505	0.0073	0.1025	0.0073	0.2269	
	3.33	360	Straw	0.0067	0.0330	0.0384	0.0736	0.0108	0.0067	0.0417	0.0067	0.0740	
Alpicat Catalunya Spa 2014 Mecano	3.33	10	Forage	0.0067	0.0229	0.0314	0.0558	0.0437	0.0067	0.0343	0.0067	0.1883	
	3.07	62	Forage	0.0073	0.0168	0.0214	0.0394	0.0482	0.0073	0.0175	0.0073	0.1167	
	3.33	364	Forage	0.0067	0.0168	0.0291	0.0470	0.0276	0.0067	0.0330	0.0067	0.1144	
	3.33	10	Hay	0.0067	0.0289	0.0418	0.0727	0.0531	0.0067	0.0377	0.0067	0.2489	
	3.07	62	Hay	0.0073	0.0226	0.0202	0.0443	0.0540	0.0073	0.0175	0.0073	0.1459	
	3.33	364	Hay	0.0067	0.0108	0.0163	0.0278	0.0309	0.0067	0.0222	0.0067	0.0874	
	3.33	10	Grain	0.0067	0.0067	0.0116	0.0188	0.0195	0.0067	0.0067	0.0067	0.0067	0.0601
	3.07	62	Grain	0.0073	0.0073	0.0126	0.0204	0.0241	0.0073	0.0073	0.0073	0.0073	0.0652

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluazaindolizine	IN-A5760	IN-F4106	IN - A5760+INF4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
	3.33	364	Grain	0.0067	0.0067	0.0116	0.0188	0.0235	0.0067	0.0067	0.0067	0.0067	0.0601
	3.33	10	Straw	0.0067	0.0673	0.1034	0.1753	0.0484	0.0067	0.1211	0.0067	0.2825	
	3.07	62	Straw	0.0073	0.0584	0.0933	0.1556	0.0949	0.0073	0.0949	0.0073	0.2408	
	3.33	364	Straw	0.0067	0.0410	0.0686	0.1124	0.0195	0.0067	0.0807	0.0067	0.3027	
Termens Catalunya Spa 2014 Mecano	3.27	7	Forage	0.0069	0.0069	0.0118	0.0192	0.0116	0.0069	0.0069	0.0069	0.0466	
	2.96	63	Forage	0.0076	0.0076	0.0131	0.0212	0.0114	0.0076	0.0076	0.0076	0.0477	
	3.33	364	Forage	0.0067	0.0081	0.0128	0.0214	0.0316	0.0067	0.0188	0.0067	0.0673	
	3.27	7	Hay	0.0137	0.0130	0.0189	0.0328	0.0288	0.0069	0.0110	0.0069	0.0685	
	2.96	63	Hay	0.0076	0.0098	0.0131	0.0236	0.0197	0.0076	0.0091	0.0076	0.0613	
	3.33	364	Hay	0.0067	0.0067	0.0116	0.0188	0.0161	0.0067	0.0094	0.0067	0.0592	
	3.27	7	Grain	0.0069	0.0069	0.0000	0.0073	0.0110	0.0069	0.0069	0.0069	0.0069	0.0362
	2.96	63	Grain	0.0076	0.0076	0.0131	0.0212	0.0091	0.0076	0.0076	0.0076	0.0076	0.0676
	3.33	364	Grain	0.0067	0.0067	0.0116	0.0188	0.0202	0.0067	0.0067	0.0067	0.0067	0.0601
	3.27	7	Straw	0.0130	0.0349	0.0473	0.0847	0.0473	0.0069	0.0500	0.0069	0.0891	
	2.96	63	Straw	0.0076	0.0303	0.0288	0.0611	0.0371	0.0076	0.0356	0.0076	0.0590	
	3.33	364	Straw	0.0067	0.0249	0.0256	0.0521	0.0182	0.0067	0.0323	0.0067	0.1211	
Los Palacios Andalucía Spa 2014 Avispa	3.3	10	Forage	0.0068	0.0081	0.0117	0.0204	0.0102	0.0068	0.0068	0.0068	0.0360	
	3.22	61	Forage	0.0070	0.0070	0.0120	0.0194	0.0083	0.0070	0.0070	0.0070	0.0243	
	3.31	357	Forage	0.0068	0.0068	0.0117	0.0189	0.0074	0.0068	0.0068	0.0068	0.0264	
	3.3	10	Hay	0.0075	0.0238	0.0141	0.0394	0.0536	0.0068	0.0122	0.0068	0.1154	
	3.22	61	Hay	0.0070	0.0125	0.0120	0.0254	0.0237	0.0070	0.0070	0.0070	0.0577	
	3.31	357	Hay	0.0068	0.0068	0.0117	0.0189	0.0223	0.0068	0.0068	0.0068	0.0359	
	3.3	10	Grain	0.0068	0.0068	0.0117	0.0190	0.0360	0.0068	0.0068	0.0068	0.0088	0.0637
	3.22	61	Grain	0.0070	0.0070	0.0120	0.0194	0.0167	0.0070	0.0070	0.0070	0.0070	0.0622
	3.31	357	Grain	0.0068	0.0068	0.0117	0.0189	0.0068	0.0068	0.0068	0.0068	0.0068	0.0605
	3.3	10	Straw	0.1086	0.1222	0.1173	0.2478	0.2987	0.0068	0.0950	0.0081	0.4208	
	3.22	61	Straw	0.0313	0.0835	0.0673	0.1565	0.1530	0.0070	0.0682	0.0070	0.2991	
	3.31	357	Straw	0.0068	0.0129	0.0292	0.0430	0.0406	0.0068	0.0298	0.0068	0.0677	
Aguadulce Andalucía Spa 2014 Avispa	3.27	10	Forage	0.0069	0.0069	0.0118	0.0192	0.0069	0.0069	0.0069	0.0069	0.0301	
	3.12	62	Forage	0.0072	0.0072	0.0124	0.0201	0.0072	0.0072	0.0072	0.0072	0.0201	
	3.26	114	Forage	0.0069	0.0069	0.0119	0.0192	0.0069	0.0069	0.0069	0.0069	0.0316	
	3.27	10	Hay	0.0069	0.0103	0.0166	0.0275	0.0137	0.0069	0.0144	0.0069	0.0822	
	3.12	62	Hay	0.0072	0.0115	0.0149	0.0272	0.0165	0.0072	0.0158	0.0072	0.1005	
	3.26	114	Hay	0.0069	0.0103	0.0142	0.0253	0.0117	0.0069	0.0172	0.0069	0.1031	
	3.27	10	Grain	0.0069	0.0069	0.0118	0.0192	0.0069	0.0069	0.0069	0.0069	0.0069	0.0612
	3.12	62	Grain	0.0072	0.0072	0.0124	0.0201	0.0072	0.0072	0.0072	0.0072	0.0072	0.0642
	3.26	114	Grain	0.0069	0.0069	0.0119	0.0192	0.0069	0.0069	0.0069	0.0069	0.0069	0.0614
	3.27	10	Straw	0.0089	0.0192	0.0154	0.0359	0.0151	0.0069	0.0212	0.0123	0.0637	
	3.12	62	Straw	0.0072	0.0129	0.0124	0.0262	0.0108	0.0072	0.0151	0.0136	0.0488	
	3.26	114	Straw	0.0069	0.0137	0.0142	0.0289	0.0096	0.0069	0.0165	0.0069	0.0234	
Alpicat, Catalunya, Spa 2013 Califa su	1.25	28	Forage	0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0251	
	1.25	128	Forage	0.0179	0.0215	0.0310	0.0539	0.0179	0.0179	0.0215	0.0179	0.0717	
	2.5	28	Forage	0.0090	0.0197	0.0155	0.0365	0.0305	0.0090	0.0170	0.0090	0.0762	
	2.5	142	Forage	0.0090	0.0332	0.0279	0.0633	0.0287	0.0090	0.0349	0.0090	0.1434	
	2.5	243	Forage	0.0090	0.0233	0.0201	0.0450	0.0349	0.0090	0.0305	0.0090	0.1165	
	1.25	28	Hay	0.0179	0.0806	0.2043	0.2905	0.1344	0.0179	0.2150	0.0179	0.4659	
	1.25	128	Hay	0.0179	0.0251	0.0867	0.1135	0.0394	0.0179	0.0824	0.0179	0.0950	
	2.5	28	Hay	0.0090	0.0206	0.0294	0.0514	0.0394	0.0090	0.0242	0.0090	0.0986	
	2.5	142	Hay	0.0090	0.0305	0.0495	0.0821	0.0520	0.0090	0.0618	0.0090	0.1702	
	2.5	243	Hay	0.0090	0.0314	0.0697	0.1032	0.0573	0.0090	0.0771	0.0090	0.1523	
	1.25	28	Grain	0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	1.25	128	Grain	0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	2.5	28	Grain	0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	142	Grain	0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	243	Grain	0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	1.25	28	Straw	0.0179	0.0466	0.0557	0.1055	0.0717	0.0179	0.0986	0.0179	0.2688	
	1.25	128	Straw	0.0179	0.0305	0.0898	0.1223	0.0394	0.0179	0.0753	0.0179	0.1380	
	2.5	28	Straw	0.0090	0.0457	0.0542	0.1030	0.0582	0.0090	0.0591	0.0090	0.1882	

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluazaindolizine	IN-A5760	IN-F4106	IN - A5760+INF4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
	2.5	142	Straw	0.0090	0.0591	0.0697	0.1328	0.0556	0.0090	0.0851	0.0090	0.2509	
	2.5	243	Straw	0.0090	0.0385	0.0433	0.0845	0.0520	0.0090	0.0609	0.0090	0.1882	
Aguadulce, Andalucia Spa 2013 Avispa	1.25	29	Forage	0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0771	
	1.25	145	Forage	0.0179	0.0323	0.0310	0.0654	0.0179	0.0179	0.0233	0.0179	0.1720	
	2.5	27	Forage	0.0090	0.0305	0.0155	0.0480	0.0161	0.0090	0.0215	0.0090	0.1613	
	2.5	145	Forage	0.0090	0.0161	0.0155	0.0327	0.0090	0.0090	0.0090	0.0090	0.0797	
	2.5	242	Forage	0.0090	0.0161	0.0155	0.0327	0.0090	0.0090	0.0108	0.0090	0.0878	
	1.25	29	Hay	0.0179	0.0072	0.0310	0.0386	0.0179	0.0179	0.0179	0.0179	0.0448	
	1.25	145	Hay	0.0179	0.0556	0.0557	0.1151	0.0358	0.0179	0.0484	0.0179	0.4659	
	2.5	27	Hay	0.0090	0.0394	0.0186	0.0607	0.0233	0.0090	0.0108	0.0090	0.2419	
	2.5	145	Hay	0.0090	0.0887	0.0681	0.1628	0.0618	0.0090	0.0735	0.0090	0.6899	
	2.5	242	Hay	0.0090	0.0305	0.0325	0.0650	0.0224	0.0090	0.0305	0.0090	0.2957	
	1.25	29	Grain	0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	1.25	145	Grain	0.0179	0.0179	0.0310	0.0501	0.0179	0.0179	0.0179	0.0179	0.0179	0.1601
	2.5	27	Grain	0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	145	Grain	0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	2.5	242	Grain	0.0090	0.0090	0.0155	0.0250	0.0090	0.0090	0.0090	0.0090	0.0090	0.0801
	1.25	29	Straw	0.0179	0.0233	0.0310	0.0558	0.0215	0.0179	0.0484	0.0179	0.1380	
	1.25	145	Straw	0.0179	0.0215	0.0310	0.0539	0.0179	0.0179	0.0358	0.0179	0.1272	
	2.5	27	Straw	0.0090	0.0475	0.0526	0.1033	0.0269	0.0099	0.0860	0.0090	0.2867	
	2.5	145	Straw	0.0125	0.1075	0.1409	0.2557	0.0618	0.0090	0.2061	0.0090	0.7526	
	2.5	242	Straw	0.0090	0.0735	0.0898	0.1683	0.0439	0.0090	0.1254	0.0090	0.5107	
Richland, IA, United States, 2013/2015 Wheat Briggs	1.34	21	Forage	0.0167	0.0167	0.0289	0.0467	0.0167	0.0167	0.0176	0.0167	0.0376	
	1.34	21	Hay	0.0167	0.0384	0.0289	0.0699	0.0410	0.0167	0.0451	0.0167	0.1112	
	1.34	21	Straw	0.0167	0.0267	0.0289	0.0574	0.0242	0.0167	0.0368	0.0167	0.0401	
	1.34	21	Grain	0.0167	0.0167	0.0289	0.0467	0.0167	0.0167	0.0167	0.0167	0.0167	0.1494
	1.26	57	Forage	0.0178	0.0204	0.0307	0.0525	0.0276	0.0178	0.0747	0.0178	0.1120	
	1.26	57	Hay	0.0178	0.0613	0.0307	0.0962	0.1511	0.0178	0.2044	0.0178	0.4711	
	1.26	57	Straw	0.0258	0.2400	0.2104	0.4667	0.2400	0.0178	0.6133	0.0178	0.4622	
	1.26	57	Grain	0.0178	0.0178	0.0307	0.0497	0.1422	0.0178	0.0178	0.0178	0.0178	0.2517
	2.53	21	Forage	0.0089	0.0164	0.0153	0.0328	0.0155	0.0089	0.0288	0.0089	0.0527	
	2.53	21	Straw	0.0089	0.0527	0.0734	0.1297	0.0443	0.0089	0.0633	0.0089	0.0553	
	2.53	21	Grain	0.0089	0.0089	0.0153	0.0248	0.0089	0.0089	0.0089	0.0089	0.0089	0.0791
	2.53	57	Forage	0.0089	0.0124	0.0153	0.0285	0.0217	0.0089	0.0611	0.0089	0.1062	
	2.53	57	Hay	0.0089	0.0359	0.0199	0.0582	0.0739	0.0155	0.1417	0.0089	0.3055	
	2.53	57	Straw	0.0097	0.0394	0.0298	0.0719	0.0602	0.0089	0.0974	0.0089	0.0792	
	2.53	57	Grain	0.0089	0.0089	0.0153	0.0248	0.0788	0.0089	0.0089	0.0089	0.0089	0.1395
Richland, IA, United States, 2013/2015 5 (6V653)	2.6	313	Forage	0.0086	0.0108	0.0149	0.0264	0.0241	0.0086	0.0336	0.0086	0.0724	
	2.6	313	Hay	0.0086	0.0504	0.0149	0.0687	0.1163	0.0155	0.1895	0.0086	0.6117	
	2.6	313	Straw	0.0086	0.0569	0.0171	0.0778	0.0444	0.0125	0.1508	0.0086	0.1465	
	2.6	313	Grain	0.0086	0.0086	0.0149	0.0241	0.0474	0.0086	0.0086	0.0086	0.0086	0.0839
Gardner, ND, United States, 2014/2015 Prosper	4.5	11	Forage	0.0050	0.0050	0.0086	0.0139	0.0072	0.0050	0.0102	0.0050	0.0241	
	4.5	11	Hay	0.0050	0.0100	0.0086	0.0192	0.0184	0.0050	0.0197	0.0050	0.0453	
	4.5	11	Straw	0.0050	0.0144	0.0086	0.0240	0.0219	0.0050	0.0256	0.0050	0.0219	
	4.5	11	Grain	0.0050	0.0050	0.0086	0.0139	0.0057	0.0050	0.0050	0.0050	0.0050	0.0445
	4.5	61	Forage	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0142	0.0050	0.0329	
	4.5	61	Hay	0.0050	0.0124	0.0120	0.0253	0.0184	0.0050	0.0485	0.0050	0.0597	
	4.5	61	Straw	0.0050	0.0110	0.0086	0.0203	0.0187	0.0050	0.0495	0.0050	0.0672	
	4.5	61	Grain	0.0050	0.0050	0.0086	0.0139	0.0090	0.0050	0.0050	0.0050	0.0050	0.0445
	4.5	367	Forage	0.0050	0.0050	0.0086	0.0139	0.0050	0.0050	0.0159	0.0050	0.0398	
	4.5	367	Hay	0.0050	0.0055	0.0086	0.0144	0.0100	0.0050	0.0246	0.0050	0.0796	
	4.5	367	Straw	0.0050	0.0082	0.0086	0.0174	0.0095	0.0050	0.0134	0.0050	0.0167	
	4.5	367	Grain	0.0050	0.0050	0.0086	0.0139	0.0052	0.0050	0.0050	0.0050	0.0050	0.0445
Richland, IA, United States, 2014/2015 Briggs	4.43	7	Forage	0.0051	0.0091	0.0087	0.0185	0.0159	0.0051	0.0255	0.0051	0.0528	
	4.43	7	Hay	0.0051	0.0683	0.0162	0.0891	0.0910	0.0154	0.1542	0.0124	0.3388	
	4.43	7	Straw	0.0051	0.1037	0.0603	0.1710	0.0784	0.0119	0.2326	0.0058	0.2225	
	4.43	7	Grain	0.0051	0.0051	0.0087	0.0141	0.0250	0.0051	0.0051	0.0051	0.0051	0.0452
Richland, IA,	4.49	69	Forage	0.0050	0.0155	0.0086	0.0251	0.0289	0.0050	0.0534	0.0050	0.0698	

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluazaindolizine	IN-A5760	IN-F4106	IN - A5760+IINF4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54	MAX, SUM[A] and 1.77×IN-QEK31
United States,	4.49	69	Hay	0.0050	0.0387	0.0108	0.0521	0.1073	0.0050	0.1447	0.0050	0.1996	
2014/2015	4.49	69	Straw	0.0050	0.0486	0.0332	0.0851	0.0599	0.0050	0.1497	0.0050	0.1197	
GV662	4.49	69	Grain	0.0050	0.0050	0.0086	0.0139	0.0599	0.0050	0.0050	0.0050	0.0050	0.1060
Richland, IA,	4.37	345	Forage	0.0051	0.0059	0.0089	0.0152	0.0156	0.0054	0.0238	0.0051	0.0505	
United States,	4.37	345	Hay	0.0051	0.0213	0.0089	0.0316	0.0538	0.0056	0.0718	0.0051	0.1205	
2014/2015	4.37	345	Straw	0.0051	0.0392	0.0208	0.0627	0.0666	0.0144	0.1820	0.0051	0.2204	
RB07	4.37	345	Grain	0.0051	0.0051	0.0089	0.0143	0.0092	0.0051	0.0051	0.0051	0.0051	0.0458
Lime Springs,	4.48	6	Forage	0.0058	0.0358	0.0177	0.0559	0.1200	0.0563	0.4025	0.0050	1.0750	
United States,	4.48	6	Hay	0.0050	0.1200	0.0596	0.1878	0.3450	0.0265	0.8000	0.0063	1.0000	
2014/2015 SY	4.48	6	Straw	0.0050	0.0355	0.0324	0.0703	0.1025	0.0875	0.5150	0.0100	0.7750	
Soren	4.48	6	Grain	0.0050	0.0050	0.0086	0.0140	0.1025	0.0105	0.0185	0.0050	0.0233	0.1814
Lime Springs,	4.45	64	Forage	0.0050	0.0171	0.0087	0.0270	0.0629	0.0076	0.0931	0.0050	0.1435	
United States,	4.45	64	Hay	0.0050	0.0410	0.0109	0.0547	0.1334	0.0315	0.3196	0.0055	0.2995	
2014/2015	4.45	64	Straw	0.0050	0.0388	0.0417	0.0831	0.0604	0.0224	0.2467	0.0050	0.1233	
Forefront	4.45	64	Grain	0.0050	0.0050	0.0087	0.0141	0.0579	0.0050	0.0050	0.0050	0.0050	0.1025
	4.45	365	Forage	0.0050	0.0058	0.0087	0.0149	0.0330	0.0050	0.0355	0.0050	0.0554	
	4.45	365	Hay	0.0050	0.0154	0.0087	0.0251	0.0380	0.0050	0.0730	0.0050	0.1007	
	4.45	365	Straw	0.0050	0.0131	0.0087	0.0227	0.0262	0.0050	0.0780	0.0050	0.0705	
	4.45	365	Grain	0.0050	0.0050	0.0087	0.0141	0.0232	0.0050	0.0050	0.0050	0.0050	0.0450
York, NE, Unite	4.48	8	Forage	0.0050	0.0143	0.0095	0.0247	0.0235	0.0058	0.0373	0.0050	0.1375	
States,	4.48	8	Hay	0.0065	0.0270	0.0086	0.0375	0.0498	0.0050	0.0575	0.0050	0.1325	
2014/2015	4.48	8	Straw	0.0050	0.0145	0.0086	0.0241	0.0180	0.0050	0.0253	0.0050	0.0435	
Prosper HS	4.48	8	Grain	0.0050	0.0050	0.0086	0.0140	0.0108	0.0050	0.0050	0.0050	0.0050	0.0447
York, NE, Unite	4.48	145	Forage	0.0050	0.0050	0.0086	0.0140	0.0183	0.0050	0.0298	0.0050	0.0525	
States,	4.48	145	Hay	0.0050	0.0178	0.0086	0.0276	0.0268	0.0050	0.0428	0.0050	0.0850	
2014/2015	4.48	145	Straw	0.0050	0.0188	0.0199	0.0399	0.0233	0.0050	0.0500	0.0050	0.0650	
Overland	4.48	145	Grain	0.0050	0.0050	0.0086	0.0140	0.0185	0.0050	0.0050	0.0050	0.0050	0.0447
York, NE, Unite	4.48	363	Forage	0.0050	0.0070	0.0086	0.0161	0.0085	0.0050	0.0150	0.0050	0.0305	
States,	4.48	363	Hay	0.0050	0.0135	0.0086	0.0231	0.0218	0.0050	0.0260	0.0050	0.0468	
2014/2015	4.48	363	Straw	0.0050	0.0170	0.0164	0.0346	0.0228	0.0050	0.0470	0.0050	0.0600	
Prosper	4.48	363	Grain	0.0050	0.0050	0.0086	0.0140	0.0270	0.0050	0.0050	0.0050	0.0050	0.0478
Uvalde, TX,	4.52	10	Forage	0.0097	0.0171	0.0141	0.0324	0.0362	0.0144	0.2478	0.0050	0.4782	
United States,	4.52	10	Hay	0.0050	0.0644	0.0167	0.0855	0.1090	0.0082	0.1313	0.0062	0.3568	
2014-2016	4.52	10	Straw	0.0052	0.0570	0.0882	0.1491	0.0743	0.0188	0.2750	0.0050	0.0429	
Greer-Winter	4.52	10	Grain	0.0050	0.0134	0.0505	0.0648	0.0292	0.0069	0.4931	0.0050	0.0129	0.8920
Uvalde, TX,	4.52	65	Forage	0.0067	0.0273	0.0188	0.0479	0.0436	0.0149	0.1611	0.0050	0.4361	
United States,	4.52		Hay	0.0055	0.0570	0.0167	0.0776	0.0917	0.0201	0.3469	0.0074	0.8673	
2014-2016	4.52		Straw	0.0553	0.1611	0.3767	0.5487	0.1239	0.0159	0.4956	0.0050	0.1735	
Expresso-Sprii	4.52		Grain	0.0050	0.0129	0.0342	0.0480	0.0191	0.0064	0.3766	0.0050	0.0164	0.6799
Uvalde, TX,	4.52	375	Forage	0.0050	0.0052	0.0086	0.0141	0.0211	0.0059	0.0892	0.0050	0.1685	
United States,	4.52	375	Hay	0.0050	0.0265	0.0090	0.0373	0.0570	0.0069	0.1536	0.0050	0.3791	
2014-2016	4.52	375	Straw	0.0050	0.0171	0.0086	0.0268	0.0359	0.0050	0.0768	0.0050	0.1214	
Greer-Winter	4.52	375	Grain	0.0050	0.0050	0.0086	0.0139	0.0250	0.0050	0.0050	0.0050	0.0072	0.0443

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

Table 176 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydroliysis) in rape rotational crops (single samples from plot) in Spain

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Charantonnay,	3.01	7	Forage	<0.01	ND	<0.01	<0.01	0.017	<0.01	ND	0.014
Rhone Alpes,	3.10	60		ND	ND	<0.01	0.012	0.064	0.011	<0.01	0.018

Location Year Variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
France 2014 Exception⑥	2.99	363		ND	ND	ND	ND	ND	ND	ND	ND
	3.01	7	Seed	ND	ND	ND	ND	ND	ND	ND	ND
	3.10	60		ND	ND	ND	ND	ND	ND	ND	ND
	2.99	363		ND	ND	ND	ND	ND	ND	ND	ND
	3.01	7	Straw	<0.01	0.018	<0.01	0.022	0.017	0.012	<0.01	0.11
	3.10	60		<0.01	0.014	0.013	0.022	0.025	0.015	0.013	0.15
	2.99	363		ND	ND	0.028	0.011	0.018	0.028	ND	0.016
Alpicat Catalunya Spain 2014 Atenzo⑥	3.32	10	Forage	ND	<0.01	ND	<0.01	0.034	<0.01	<0.01	0.034
	3.05	62		ND	ND	ND	ND	0.014	ND	ND	0.013
	3.30	364		ND	<0.01	ND	ND	0.034	<0.01	<0.01	0.047
	3.32	10	Seed	ND	ND	ND	ND	ND	ND	ND	<0.01
	3.05	62		ND	ND	ND	ND	ND	ND	ND	ND
	3.30	364		ND	ND	ND	ND	ND	ND	ND	<0.01
	3.32	10	Straw	ND	0.017	<0.01	ND	ND	<0.01	ND	0.079
	3.05	62		ND	0.017	ND	<0.01	ND	<0.01	ND	0.077
	3.30	364		ND	0.021	ND	ND	<0.01	<0.01	0.010	0.15
Termens Catalunya Spain 2014 Atenzo⑥	3.33	7	Forage	ND	ND	ND	ND	0.018	ND	<0.01	0.012
	3.05	63		ND	ND	ND	ND	0.015	ND	ND	0.011
	3.37	363		ND	ND	ND	ND	0.012	ND	ND	<0.01
	3.33	7	Seed	<0.01	ND	ND	ND	ND	ND	ND	ND
	3.05	63		<0.01	ND	ND	ND	ND	ND	ND	ND
	3.37	363		ND	ND	ND	ND	ND	ND	ND	ND
	3.33	7	Straw	ND	0.012	<0.01	ND	<0.01	ND	<0.01	0.054
	3.05	63		<0.01	0.015	<0.01	<0.01	0.016	ND	0.012	0.072
	3.37	363		ND	0.011	ND	ND	0.033	<0.01	0.013	0.11
Los Palacios Andalucía Spain 2014 NXH213CLS⑥	3.26	10	Forage	<0.01	ND	<0.01	ND	0.012	<0.01	ND	0.014
	3.17	61		ND	ND	ND	ND	0.010	<0.01	ND	<0.01
	3.30	357		ND	ND	ND	ND	ND	<0.01	ND	0.015
	3.26	10	Seed	0.012	ND	ND	<0.01	ND	ND	ND	ND
	3.17	61		<0.01	ND	ND	ND	ND	ND	ND	ND
	3.30	357		ND	ND	ND	ND	ND	ND	ND	ND
	3.26	10	Straw	0.045	0.18	0.051	0.034	0.039	0.024	0.039	0.50
	3.17	61		0.024	0.21	0.061	0.024	0.11	0.020	0.086	0.42
	3.30	357		0.018	0.15	0.044	0.037	0.036	0.028	0.014	0.14
Aguadulce Andalucía Spain 2014 NXH213CLS⑥	3.26	10	Forage	ND	<0.01	<0.01	ND	<0.01	ND	ND	0.011
	3.12	62		ND	ND	<0.01	ND	<0.01	ND	ND	<0.01
	3.24	358		ND	ND	ND	ND	ND	ND	ND	ND
	3.26	10	Seed	0.022	ND	<0.01	<0.01	ND	ND	ND	ND
	3.12	62		<0.01	ND	<0.01	ND	ND	ND	ND	ND
	3.24	358		<0.01	ND	ND	ND	ND	ND	ND	ND
	3.26	10	Straw	0.089	0.058	0.039	0.029	0.038	0.013	0.036	0.14
	3.12	62		0.035	0.014	0.028	0.022	0.018	ND	<0.01	0.037
	3.24	358		0.022	<0.01	ND	0.010	<0.01	ND	ND	0.019

Notes:

⑥ Doig 2020 DuPont-40828.

Table 177 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydroliysis) in rape rotational crops scaled to soil plateau levels (3.87 kg ai/ha for IN-F4106 and 2.24 kg ai/ha for other compounds)

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluazaindolizine	IN-A5760	IN-F4106	IN-A5760+INF4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD 54	MAX, SUM and 1.77×IN-QEK31
Charantonnay, Rhone Alpes, France 2014	3.01	7	Forage	0.0074	0.0074	0.0129	0.0208	0.0074	0.0127	0.0074	0.0074	0.0104	
	3.1	60	Forage	0.0072	0.0072	0.0125	0.0202	0.0087	0.0462	0.0079	0.0072	0.0130	
	2.99	363	Forage	0.0075	0.0075	0.0129	0.0209	0.0075	0.0075	0.0075	0.0075	0.0075	

Location Year variety	Total rate (kg ai/ha)	PBI (days)	Matrix	Fluazaindolizine	IN-A5760	IN-F4106	IN-A5760+INF4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD 54	MAX, SUM and 1.77×IN-QEK31
Exception	3.01	7	Seed	0.0074	0.0074	0.0129	0.0208	0.0074	0.0074	0.0074	0.0074	0.0074	0.0665
	3.1	60	Seed	0.0072	0.0072	0.0125	0.0202	0.0072	0.0072	0.0072	0.0072	0.0072	0.0646
	2.99	363	Seed	0.0075	0.0075	0.0129	0.0209	0.0075	0.0075	0.0075	0.0075	0.0075	0.0669
	3.01	7	Straw	0.0074	0.0134	0.0129	0.0272	0.0164	0.0127	0.0089	0.0074	0.0819	
	3.1	60	Straw	0.0072	0.0101	0.0162	0.0270	0.0159	0.0181	0.0108	0.0094	0.1084	
	2.99	363	Straw	0.0075	0.0075	0.0362	0.0442	0.0082	0.0135	0.0210	0.0075	0.0120	
Alpicat Catalunya Spain 2014 Atenzo	3.32	10	Forage	0.0067	0.0067	0.0117	0.0189	0.0067	0.0229	0.0067	0.0067	0.0229	
	3.05	62	Forage	0.0073	0.0073	0.0127	0.0205	0.0073	0.0103	0.0073	0.0073	0.0095	
	3.3	364	Forage	0.0068	0.0068	0.0117	0.0190	0.0068	0.0231	0.0068	0.0068	0.0319	
	3.32	10	Seed	0.0067	0.0067	0.0117	0.0189	0.0067	0.0067	0.0067	0.0067	0.0067	0.0603
	3.05	62	Seed	0.0073	0.0073	0.0127	0.0205	0.0073	0.0073	0.0073	0.0073	0.0073	0.0656
	3.3	364	Seed	0.0068	0.0068	0.0117	0.0190	0.0068	0.0068	0.0068	0.0068	0.0068	0.0607
	3.32	10	Straw	0.0067	0.0115	0.0117	0.0239	0.0067	0.0067	0.0067	0.0067	0.0533	
	3.05	62	Straw	0.0073	0.0125	0.0127	0.0260	0.0073	0.0073	0.0073	0.0073	0.0566	
	3.3	364	Straw	0.0068	0.0143	0.0117	0.0270	0.0068	0.0068	0.0068	0.0068	0.1018	
Termens Catalunya Spain 2014 Atenzo	3.33	7	Forage	0.0067	0.0067	0.0116	0.0188	0.0067	0.0121	0.0067	0.0067	0.0081	
	3.05	63	Forage	0.0073	0.0073	0.0127	0.0205	0.0073	0.0110	0.0073	0.0073	0.0081	
	3.37	363	Forage	0.0066	0.0066	0.0115	0.0186	0.0066	0.0080	0.0066	0.0066	0.0066	
	3.33	7	Seed	0.0067	0.0067	0.0116	0.0188	0.0067	0.0067	0.0067	0.0067	0.0067	0.0601
	3.05	63	Seed	0.0073	0.0073	0.0127	0.0205	0.0073	0.0073	0.0073	0.0073	0.0073	0.0656
	3.37	363	Seed	0.0066	0.0066	0.0115	0.0186	0.0066	0.0066	0.0066	0.0066	0.0066	0.0594
	3.33	7	Straw	0.0067	0.0081	0.0116	0.0202	0.0067	0.0067	0.0067	0.0067	0.0363	
	3.05	63	Straw	0.0073	0.0110	0.0127	0.0245	0.0073	0.0118	0.0073	0.0088	0.0529	
	3.37	363	Straw	0.0066	0.0073	0.0115	0.0193	0.0066	0.0219	0.0066	0.0086	0.0731	
Los Palacios Andalucía Spain 2014 NXH213CLS	3.26	10	Forage	0.0069	0.0069	0.0119	0.0192	0.0069	0.0082	0.0069	0.0069	0.0096	
	3.17	61	Forage	0.0071	0.0071	0.0122	0.0198	0.0071	0.0071	0.0071	0.0071	0.0071	
	3.3	357	Forage	0.0068	0.0068	0.0117	0.0190	0.0068	0.0068	0.0068	0.0068	0.0102	
	3.26	10	Seed	0.0082	0.0069	0.0119	0.0192	0.0069	0.0069	0.0069	0.0069	0.0069	0.0614
	3.17	61	Seed	0.0071	0.0071	0.0122	0.0198	0.0071	0.0071	0.0071	0.0071	0.0071	0.0631
	3.3	357	Seed	0.0068	0.0068	0.0117	0.0190	0.0068	0.0068	0.0068	0.0068	0.0068	0.0607
	3.26	10	Straw	0.0309	0.1237	0.0605	0.1926	0.0234	0.0268	0.0165	0.0268	0.3436	
	3.17	61	Straw	0.0170	0.1484	0.0745	0.2330	0.0170	0.0777	0.0141	0.0608	0.2968	
	3.3	357	Straw	0.0122	0.1018	0.0516	0.1603	0.0251	0.0244	0.0190	0.0095	0.0950	
Aguadulce Andalucía Spain 2014 NXH213CLS	3.26	10	Forage	0.0069	0.0069	0.0119	0.0192	0.0069	0.0069	0.0069	0.0069	0.0076	
	3.12	62	Forage	0.0072	0.0072	0.0124	0.0201	0.0072	0.0072	0.0072	0.0072	0.0072	
	3.24	114	Forage	0.0069	0.0069	0.0119	0.0193	0.0069	0.0069	0.0069	0.0069	0.0069	
	3.26	10	Seed	0.0151	0.0069	0.0119	0.0192	0.0069	0.0069	0.0069	0.0069	0.0069	0.0614
	3.12	62	Seed	0.0072	0.0072	0.0124	0.0201	0.0072	0.0072	0.0072	0.0072	0.0072	0.0642
	3.24	114	Seed	0.0069	0.0069	0.0119	0.0193	0.0069	0.0069	0.0069	0.0069	0.0069	0.0618
	3.26	10	Straw	0.0612	0.0399	0.0463	0.0889	0.0199	0.0261	0.0089	0.0247	0.0962	
	3.12	62	Straw	0.0251	0.0101	0.0347	0.0455	0.0158	0.0129	0.0072	0.0072	0.0266	
	3.24	114	Straw	0.0152	0.0069	0.0119	0.0193	0.0069	0.0069	0.0069	0.0069	0.0131	

Notes:

[A] SUM (scaled) = (soil plateau IN-A5760×2.26×IN-A5760 + soil plateau IN-F4106×2.11×IN-F4106 + soil plateau IN-QZY47×1.52×IN-QZY47 + soil plateau IN-TMQ01×1.51×IN-TMQ01)/total rate.

FATE OF RESIDUES IN STORAGE AND PROCESSING

Anand (2013 DuPont-35131) studied the nature of fluazaindolizine residue following high-temperature hydrolysis simulating industrial processing and/or household preparation. There is no demonstrated significant degradation of fluazaindolizine at any pH or temperature for any duration studied. It is not expected that hydrolysis will be a significant route of fluazaindolizine degradation in the environment or in food processing.

High-temperature hydrolysis was investigated to address the potential for metabolites to be found during processing of crop commodities with the test solutions prepared in 0.01 M citrate buffer solutions of pH 4, 5 and 6 which were incubated at 90 °C for 20 minutes, 100 °C for 60 minutes and 120 °C at 103.4 kPa, for 20 minutes, respectively. All these experiments were conducted at a concentration of 10 mg/kg, which was less than one-half of the solubility of fluazaindolizine in water.

During high-temperature hydrolysis experiments, at the end of the incubation periods (pasteurization: pH 4 90 °C for 20 minutes., baking: pH 5 100 °C for 60 minutes and sterilization: pH 6, 120 °C at 103.4 kPa, for 20 minutes), samples were analysed directly by LSC to determine the quantity of radioactivity present in each sample. Radioactivity was quantitatively recovered from each test solution with mass balances 100.0 to 101.4 percent AR.

In all samples, the applied radioactivity was recovered as unchanged fluazaindolizine and no detectable levels of any degradation products. Fluazaindolizine is hydrolytically stable under conditions representative of pasteurization (pH 4, 90 °C, for 20 min.), baking (pH 5, 100 °C, for 60 min.) and sterilization (pH 6, 120 °C at 103.4 kPa, for 20 min.).

Strawberry

Shepard (2020 DuPont-43226, Revision No. 1) studied the distribution of fluazaindolizine residues upon processing of strawberries. The strawberries from trials 1 and 3 were received frozen. The samples were placed in frozen storage prior to processing, except the control sample from trial 3, which was placed in cool storage overnight prior to processing. Strawberries from trial 2 were received cool and placed in cool storage prior to processing.

Samples were removed from cooler or freezer storage and batch rinsed in stainless steel draining baskets using high-pressure cold-water spray at approximately 483-689 kPa for 30 seconds per batch. Any damaged or unacceptable fruit was discarded.

Washed fruit were crushed using a hammermill. The crushed strawberries were transferred to a 35 L steam jacketed swept surface kettle and heated to approximately 30 °C, followed by pressing with a fruit press. The recovered fresh juice was filtered using a US #20 screen to remove any coarse solids, centrifuged to remove fine particulate matter, with an aliquot was reserved for making syrup for the canned fruit fraction. The remainder was vacuum filtered with a Buchner filter (juice).

Washed fruit was trimmed, spread on trays, and placed in the freezer (about -20 to -12 °C) for 2–6 hours or until sufficiently frozen (frozen fruit). The aliquot of washed fruit reserved for drying was trimmed and sliced in half by hand using a paring knife. The sliced strawberries were placed on Teflon covered drying trays and placed in a dryer with a temperature set point of 57-60 °C. Trays were periodically removed and weighed until a target moisture of approximately 15–20 percent moisture was obtained. A representative sample of the dried (dried) strawberries was removed, packaged, labelled, and placed in frozen storage for the required sample fraction.

Washed strawberries were trimmed then crushed in a food processor using the pulse mode and placed in a pot on the electric stovetop and sugar added at 1.65 kg per 1 kg fruit. The mixture was brought to a boil and 3.5 percent by weight of liquid pectin was added. The mixture was returned to a boil and boiled for one minute. Jars were filled with the cooked jam mixture, excess foam skimmed off with a spoon, the rims and threads wiped, and lids placed on. The jars were inverted for several minutes on the counter then turned upright. The finished jam was labelled and then left to cool at room temperature for up to 24 hours to allow the pectin to thicken (jam).

Syrup for canning was produced by adding 2.02 kg sugar to 1 kg strawberry juice and boiled. Washed strawberries were trimmed, packed into #303 cans, and filled with syrup. The cans were sealed using the Dixie Can Sealer, heated in boiling water for approximately 15 minutes, and cooled in cold tap water (canned). Residues found in strawberry processed commodities are shown in Table 178.

Table 178 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in processed strawberry commodities

	N (int)	Rate (kg ai/ha)	Matrix	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Dana, IA, 2015 Seascape	4(8 6 8)	1.13	RAC	89, 93,	ND	ND	ND	ND	ND	0.027	ND	<0.01
		1.13		99, & 104	ND	ND	ND	ND	ND	0.025	ND	<0.01
		1.12			ND	ND	ND	ND	ND	0.026	ND	<0.01
		1.13		Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.026	<0.01	<0.01
			Juice		ND	ND	ND	ND	ND	0.014	ND	<0.01
			Canned		ND	ND	ND	ND	ND	<0.01	ND	ND
			Jam		ND	ND	ND	ND	ND	<0.01	ND	ND
			Frozen fruit		ND	ND	ND	ND	ND	0.016	ND	ND
			Dehydrated fruit		<0.01	ND	<0.01	0.014	<0.01	0.14	ND	0.019
Santa Maria, CA, 2015 San Andreas	4 (5 9 7)	1.12	RAC	170	ND	ND	ND	ND	ND	0.028	ND	ND
		1.12			ND	ND	ND	ND	ND	0.026	ND	ND
		1.12			ND	ND	ND	ND	ND	0.031	ND	<0.01
		1.12		Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.029	<0.01	<0.01
			Juice		ND	ND	ND	ND	ND	0.021	ND	ND
			Canned		ND	ND	ND	ND	ND	0.011	ND	ND
			Jam		ND	ND	ND	ND	ND	<0.01	ND	ND
			Frozen Berries		ND	ND	ND	ND	ND	0.019	ND	ND
			Dehydrated fruit		ND	ND	ND	<0.01	ND	0.11	ND	<0.01
Oregon City, OR, 2015 Albion	4 (7 7 7)	1.12	RAC	77 & 80	ND	ND	ND	<0.01	ND	0.097	ND	<0.01
		1.13			ND	ND	ND	<0.01	ND	0.094	ND	<0.01
		1.12			ND	ND	ND	<0.01	ND	0.095	ND	<0.01
		1.12		Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.095	<0.01	<0.01
			Juice		ND	ND	ND	<0.01	ND	0.075	ND	<0.01
			Canned		ND	ND	ND	ND	ND	0.042	ND	<0.01
			Jam		ND	ND	ND	ND	ND	0.023	ND	ND
			Frozen fruit		ND	ND	ND	<0.01	ND	0.059	ND	<0.01
			Dehydrated fruit		ND	ND	ND	0.023	<0.01	0.39	ND	0.026

Notes:

[A] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Tomato

Shepard (2020 DuPont-40345, Revision No. 1) conducted a study on the distribution of fluazaindolizine and metabolites residues on processing of tomatoes.

The fruit was inspected and sorted, the weight of the culls (cannery waste) was recorded, and the culls discarded. The fruit for juice, purée, and paste was batch soaked in a steam jacketed stainless steel kettle with ~91 kg of water and 454 grams of sodium hydroxide at ~52–60 °C for 3 minutes, batch rinsed using a high-pressure spray warm water rinse at 68–74 °C for 30 seconds per batch and fed into a hammermill assembly for crushing. The crushed tomatoes were transferred to a steam jacketed kettle, rapidly heated to 79–85 °C, and held within that temperature range for 30 seconds. The hot break juice

was hand fed into a pulper finisher for the separation of pomace and juice. The wet pomace recovered was pressed (wet pomace). The recovered press juice was weighed and returned to the finished juice. A representative sample of the wet pomace was removed.

For purée preparation, an aliquot of approximately 9.0 kg of the tomato juice for tomato purée was then transferred to a vacuum evaporator. The purée was removed from the evaporator when the desired Brix range was achieved. Distilled water and 1.0 percent salt were added to adjust the Brix to the desired range of 12.0–13.0°. The purée was heated to 82–88 °C and packed and sealed in cans. The sealed cans were then processed in a boiling water bath for ~15 minutes at 96–100 °C and then cooled under running cold tap water (purée).

For paste preparation, an aliquot of at least 11.0 kg of the tomato juice for tomato paste was transferred to a vacuum evaporator. Tomato juice in excess was weighed and discarded. The paste was removed from the evaporator when the desired Brix range was achieved. An aliquot of paste was reserved for juice from concentrate. Prior to canning the paste, 0.5 percent salt and if necessary, distilled water was added to adjust the Brix of the paste to the desired range of approximately 24.0–33.0°. The paste was heated to 82–88 °C, the heated paste packed and sealed in cans and the sealed cans were then processed in a boiling water bath for ~15 minutes at 96–100 °C and then cooled in cold tap water (paste).

For juice preparation, an aliquot of paste reserved for juice was mixed with water, targeting a Brix range of ~4.5–5.5°, 0.5 percent salt was added to the juice and the juice was heated to 85 °C. The heated juice from concentrate was packed and sealed in cans. The sealed cans were then processed in a boiling water bath for approxiamtely 15 minutes at 96–100 °C and then cooled in cold tap water (juice).

For preparation of peeled fruit and canned tomatoes, fruit were spray-washed and then placed in a water bath and boiled for ~1 minute to crack the skin prior to removal. The peel was then removed by hand with a paring knife and the core, stem, and peel of the fruit was discarded (peeled tomatoes). Whole peeled tomatoes were packaged in cans and 1 teaspoon of salt was added to each can. The cans were then placed in wire baskets, and the baskets placed in a steam cabinet and steam exhausted for ~10 minutes until ~80 °C temperature was achieved. After the steam exhaust, the cans were sealed and processed in a boiling water bath for ~15 minutes. The cans were cooled in cold tap water (canned tomatoes).

For preparation of dried tomatoes, fruit were spray-washed and placed on a table, and the cores and stems of the fruits were removed by hand with a paring knife. The fruit was then cut into quarters with a paring knife and placed on drying trays with the peel down, contacting the metal of the drying tray, and placed in the dryer. The fruit was removed from the dryer once the desired moisture (<16 percent) was achieved (sun-dried tomatoes). Residues found in tomato processed commodities are shown in Table 179.

Table 179 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydroliysis) in processed tomato commodities

Location	N (int)	Rate (kg ai/ha)	DALA	Matrix		Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Carlyle, IL, 2014 Roma	4 (12 15 13)	2.24 2.24 2.24 2.24	1	RAC		<0.01	<0.01	<0.01	<0.01	ND	ND	ND	<0.01
						ND	<0.01	ND	<0.01	ND	ND	ND	<0.01
						<0.01	<0.01	<0.01	0.01B	ND	ND	ND	<0.01
					Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Washed		ND	<0.01	ND	<0.01	ND	ND	ND	<0.01
				Peeled		ND	<0.01	ND	<0.01	ND	ND	ND	<0.01
				Dried		<0.01	0.078	0.011	0.035	ND	0.020	ND	0.091
				Canned		ND	0.01[B]	ND	<0.01	ND	ND	ND	<0.01
				Juice		ND	0.010	ND	<0.01	ND	<0.01	ND	0.010

Location	N (int)	Rate (kg ai/ha)	DALA	Matrix		Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
				Wet pomace		<0.01	<0.01	<0.01	<0.01	ND	ND	ND	<0.01
				Paste		<0.01	0.037	0.010	0.020	ND	0.012	ND	0.037
				Purée		<0.01	0.022	<0.01	<0.01	ND	<0.01	ND	0.018
Paso Robles, CA, 2014 Roma Galileo	4 (14 14 15)	2.27 2.24 2.24 2.24	1	RAC		ND	0.01[B]	<0.01	<0.01	ND	ND	ND	ND
						ND	<0.01	<0.01	<0.01	ND	ND	ND	ND
						ND	<0.01	ND	<0.01	ND	ND	ND	ND
					Mean	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Washed		ND	<0.01	ND	<0.01	ND	ND	ND	ND
				Peeled		ND	0.01[B]	ND	<0.01	ND	ND	ND	ND
				Dried		<0.01	0.13	0.036	0.094	<0.01	0.012	ND	0.029
				Canned		ND	<0.01	ND	<0.01	ND	ND	ND	ND
				Juice		ND	0.019	<0.01	0.010	ND	ND	ND	<0.01
				Wet pomace		<0.01	<0.01	<0.01	<0.01	ND	ND	ND	ND
				Paste		<0.01	0.052	0.016	0.034	<0.01	<0.01	ND	0.015
				Purée		ND	0.019	<0.01	0.013	ND	ND	ND	<0.01
Sanger, CA, 2014 UG19406	4 (13 15 13)	2.27 2.24 2.24 2.24	1	RAC		0.041	0.77	0.15	1.1	0.018	0.048	0.012	0.094
						0.051	0.80	0.18	1.3	0.020	0.065	0.013	0.13
						0.044	0.75	0.18	1.2	0.019	0.053	0.012	0.11
					Mean	0.045	0.77	0.17	1.2	0.019	0.055	0.012	0.11
				Washed		0.039	0.81	0.25	1.1	0.027	0.049	0.014	0.095
				Peeled		0.011	0.87	0.13	1.3	0.019	0.049	0.013	0.12
				Dried		0.22[C]	3.4	0.94	5.5	0.083	0.25	0.056	0.39
				Canned		0.010	0.88	0.14	1.2	0.023	0.047	0.015	0.12
				Juice		0.024	0.45	0.13	0.74	0.013	0.031	<0.01	0.063
				Wet pomace		0.11	0.71	0.32	1.7	0.014	0.044	<0.01	0.087
				Paste		0.057	2.0	0.42	3.0	0.051	0.13	0.034	0.25
				Purée		0.024	0.85	0.24	1.3	0.026	0.067	0.016	0.13

Notes:

Untreated control samples were collected and analysed. Residues in untreated controls were ND for fluazaindolizine IN-QZY47, IN-TMQ01, and IN-UJV12; and <LOQ for IN-F4106; however, controls from Sanger CA showed residues as high as 0.046 mg/kg for IN-A5760, 0.14 mg/kg for IN-QEK31, and 0.010 mg/kg for IN-UNS90.

[A] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

[B] residue was ≥LOD but <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

[C] average duplicate analysis.

Potato

Shepard (2020 DuPont-43188, Revision No. 1) studied the distribution of fluazaindolizine residues upon processing of potatoes. Potatoes were batch tub washed for 5 minutes to produce washed potatoes and a specific gravity analysis conducted on the washed potatoes. Culled potatoes were removed (culls).

For preparation of flakes, washed potatoes were batch steam peeled 45 seconds at 689–827 kPa (100–120 psi). The potatoes were batch scrubbed for 30 seconds and the peel collected from the steam peeling and scrubbing process (wet peel). The steam peeled potatoes (steam peeled potatoes) were inspected and hand trimmed to remove additional peel, green, rot or otherwise damaged potatoes and the trim waste retained. The collected peel was hydraulically pressed and blended with the cut trim waste (steam waste).

For preparation of flakes, steam peeled potatoes were cut into slabs using a slicer. The potato slabs were batch spray-washed in cold tap water for about 30 seconds to remove free starch and

precooked at about 70–77 °C (targeting 71–74 °C) for 20 minutes in a 150 L steam jacketed kettle and cooled for 20 minutes to less than 32°C (pre-cooked potatoes). The cooled potato slabs were steam-cooked at 94–l00 °C for 40 minutes, mashed using a meat grinder and the mash mixed for about 40 seconds with an emulsion of pre-weighed food additives. The cooked mash was fed into a drum dryer to dry into a thin sheet and initially broken into large flakes by hand. The flakes were then milled into uniform potato flakes.

Moisture analysis was conducted on the potato flakes and if required dried (not required as all <9 percent) – (flakes).

For preparation of chips and abrasive waste, washed potatoes were batch peeled for 30 seconds using an abrasive base plate in a restaurant style peeler. The peeled potatoes were inspected by hand, and trimmed if necessary to remove rot, green or otherwise damaged potato tissue. Any trim waste collected was weighed and added to the peel (abrasive waste fraction).

A sample of the abrasion peeled tubers was removed (abrasion peeled tuber fraction). The peeled potatoes were cut into thin, approximately 0.16 cm slices using a restaurant style slicer and placed in a tub of hot water to remove free starch. The slices were drained over a screen to remove excess water and were fried at 163–191 °C in oil for about 90 seconds, drained, salted and inspected (potato chips)

For preparation of cooked potato and fresh fry, unpeeled washed potatoes reserved were divided into three subsamples for processing into peeled boiled potatoes, unpeeled boiled potatoes, and unpeeled microwaved potatoes.

Unpeeled washed potatoes were cut into quarters using a paring knife and placed into boiling water and cooked until an internal temperature of 88–92 °C was reached and drained (unpeeled boiled potato).

Unpeeled washed potatoes were hand peeled with a vegetable peeler and then cut into quarters using a paring knife. The peeled quartered potatoes were placed into boiling water and cooked until an internal temperature of 88–92 °C was reached and drained (peeled boiled potato).

Unpeeled washed potatoes were cut into quarters using a paring knife and placed into a plastic container and microwaved until an internal temperature of 88–92 °C was reached (unpeeled microwaved potato).

For preparation of French fries, washed potatoes were sliced into 0.48 cm strips using a French fry cutter. After slicing, the strips were then fried in a deep fat fryer for 2.5 to 3.0 minutes at 177–191 °C, drained and cooled before being packaged, labelled and placed into freezer storage (frozen French fries).

For preparation of French fry/frozen French fry steam peeled potatoes were pre-cooked in approximately 54 °C water for about 40 minutes and then sliced into 0.48 cm strips using a French fry cutter. After cutting, the strips were spray-washed for 30 seconds to remove free starch from the pre-cooking and cutting processes. The spray washed strips were then blanched in 79–85 °C water for 5 minutes and then dipped in a solution of 0.5 percent sodium acid pyrophosphate and 0.25 percent dextrose for 30 seconds at 71–74 °C. After blanching and dipping, the strips were dried in a tray air dryer to reduce the moisture content of the strips by approximately 15 percent and the strips dried in two stages, the first with air flowing up through the trays containing the strips and the second with the air flowing down through the trays. The air temperature of the dryer was set to and Mean temperature of about 77 °C and each drying stage was about 8 minutes. The dried strips were tempered at room temperature for 5 minutes and then par-fried in vegetable oil for 45–50 seconds at 188–191 °C. After frying, the fried strips were drained to remove excess oil and placed in the freezer for rapid cooling. After cooling in the freezer for a minimum of 12 minutes, the par-fried strips were removed, packaged, labelled

and placed back into freezer storage (finished frozen French fries). Residues in potato processed commodities are shown in Table 180.

Table 180 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis)in processed potato commodities

Location	N (int)	Total rate (kg ai/ha)	Commodity	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Gardner, ND, 2015 Russet Norkotah	2 (13)	5.67 at plant 5.67 directed to top of row	RAC	103	0.034	ND	0.016	0.023	ND	ND	ND	ND
					0.037	ND	0.020	0.026	ND	ND	ND	ND
					0.026	ND	0.015	0.021	ND	ND	ND	ND
				Mean	0.033	<0.01	0.017	0.023	<0.01	<0.01	<0.01	<0.01
			Washed Tubers		0.031	ND	0.016	0.022	ND	ND	ND	ND
			Culls		0.024	ND	0.012	0.017	ND	ND	ND	ND
			Steam-Peeled Tubers		ND	ND	ND	ND	ND	ND	ND	ND
			Steam Waste		0.038	ND	0.017	0.026	ND	ND	ND	ND
			Abrasion-Peeled Tubers		<0.01	ND	ND	<0.01	ND	ND	ND	ND
			Abrasive Waste		0.29	<0.01	0.13	0.17	ND	ND	ND	ND
			Dried Flakes		ND	ND	ND	<0.01	<0.01	<0.01	ND	ND
			Potato Chips		<0.01	ND	<0.01	<0.01	ND	ND	ND	ND
			Peeled French Fries		ND	ND	ND	ND	ND	ND	ND	ND
			Unpeeled French Fries		0.030	<0.01	0.013	0.023	<0.01	<0.01	ND	ND
			Boiled Unpeeled		<0.01	ND	<0.01	<0.01	ND	ND	ND	ND
			Boiled Peeled		ND	ND	ND	ND	ND	ND	ND	ND
			Microwaved Unpeeled (Baked)		0.044	ND	0.021	0.033	ND	ND	ND	ND
Jerome, ID, 2015 Ranger Russet	2 (14)	5.60 at plant 5.68 soil directed drench	RAC	139	0.18	<0.01	0.10	0.15	0.046	0.024	<0.01	0.017
					0.28	<0.01	0.17	0.26	0.071	0.028	0.013	0.023
					0.22	<0.01	0.14	0.20	0.047	0.018	<0.01	0.016
				Mean	0.23	<0.01	0.14	0.20	0.055	0.023	0.011	0.019
			Washed Tubers		0.032	<0.01	0.017	0.036	0.031	<0.01	<0.01	0.010
			Culls		0.039	<0.01	0.025	0.040	0.048	0.017	<0.01	0.016
			Steam-Peeled Tubers		<0.01	ND	ND	0.019	0.036	<0.01	<0.01	<0.01
			Steam Waste		0.062	<0.01	0.037	0.056	0.076	0.024	<0.01	<0.01
			Abrasion-Peeled Tubers		0.011	ND	<0.01	0.028	0.043	0.016	<0.01	0.012
			Abrasive Waste		0.25	<0.01	0.12	0.16	0.052	0.028	<0.01	0.014
			Potato Dried Flakes		0.017	<0.01	0.014	0.069	0.096	0.023	0.016	0.022
			Potato Chips		<0.01	<0.01	<0.01	0.027	0.032	0.014	<0.01	0.012
			Peeled French Fries		<0.01	ND	<0.01	0.035	0.044	<0.01	<0.01	<0.01
			Unpeeled French Fries		0.019	<0.01	0.013	0.042	0.066	0.018	<0.01	0.016
			Boiled Unpeeled		0.015	ND	<0.01	0.034	0.065	0.028	0.015	0.022
			Boiled Peeled		<0.01	ND	<0.01	0.028	0.058	0.014	<0.01	0.013
			Microwaved Unpeeled (Baked)		0.086	<0.01	0.044	0.10	0.076	0.041	0.016	0.027
Payette, ID, 2015 Ranger Russet	2 (14)	5.59 at plant 5.68 broadcast	RAC	125	0.14	<0.01	0.063	0.12	0.14	0.068	0.027	0.035
					0.057	<0.01	0.033	0.065	0.096	0.038	0.015	0.025
					0.023	<0.01	0.016	0.039	0.075	0.045	0.015	0.020
				Mean	0.074	<0.01	0.037	0.076	0.10	0.050	0.019	0.027
			Washed Tubers		0.022	<0.01	0.015	0.050	0.12	0.069	0.026	0.033
			Culls		0.035	<0.01	0.021	0.074	0.12	0.073	0.031	0.030
			Steam-Peeled Tubers		ND	<0.01	<0.01	0.035	0.10	0.036	0.020	0.022
			Steam Waste		0.053	<0.01	0.026	0.053	0.11	0.054	0.014	0.016

Location	N (int)	Total rate (kg ai/ha)	Commodity	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Abrasion-Peeled Tubers		<0.01	<0.01	<0.01	0.035	0.11	0.046	0.021	0.027
			Abrasive Waste		0.14	<0.01	0.069	0.10	0.072	0.053	0.011	0.016
			Dried Flakes		0.012	0.010	0.013	0.11	0.37	0.087	0.058	0.060
			Chips		<0.01	<0.01	0.013	0.047	0.12	0.064	0.025	0.036
			Peeled French Fries		<0.01	<0.01	<0.01	0.042	0.12	0.028	0.021	0.020
			Unpeeled French Fries		0.022	0.013	0.015	0.089	0.21	0.10	0.038	0.057
			Boiled Unpeeled		<0.01	<0.01	ND	0.038	0.11	0.041	0.024	0.026
			Boiled Peeled		ND	<0.01	ND	0.034	0.10	0.022	0.020	0.022
			Microwaved Unpeeled (Baked)		0.098	<0.01	0.051	0.12	0.16	0.087	0.034	0.044

Notes:

[A] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

Soya bean

Shepard (2020 DuPont-43191, Revision No. 1) studied the distribution of fluazaindolizine residues upon processing of soya beans. Since the moisture content of the whole seed samples was greater than 13.5 percent, samples were dried in an oven at 54–71 °C until the moisture content was 10.0–13.0 percent. Samples from trial number 03 did not require drying. Following drying (if required), samples were cleaned by aspiration and screening. Light impurities were removed from the whole soya bean by aspiration. After aspiration, the samples were screened with a 'two screen' cleaner to separate large and small foreign particles (screenings) from the soya bean seed sample (cleaned whole soya bean samples). Due to sample condition, both samples from trial number 03 did not require screening.

Clean whole soya beans were fed into a roller mill to crack the hull and liberate the kernel. After cracking, the material was separated with the aspirator into hull and kernel material. For trial number 02, the aspirator and two screen cleaners were used to separate the material (hulls, kernels). Moisture content of the kernel samples was adjusted to 13.5 percent with water and the sample mixed for 13-17 minutes. Following mixing, samples were allowed to equilibrate/temper for a minimum of twelve hours.

Moisture adjusted kernel material was heated to 71–79 °C in a mixer and flaked in a flaking roll with a gap setting of 0.20–0.33 mm. Flakes were extruded in a continuous processor, where they were turned into collets by direct steam injection and compression. Collets exited the processor at 93–127 °C. After extrusion, the collets were ground in a disc mill and then dried in an oven at 66–82 °C for 30–40 minutes.

Ground collets were placed in a stainless-steel batch extractor and submerged in 49–60 °C hexane. After 30 minutes, the miscella (crude oil and hexane) was drained and hexane was added to repeat the cycle two more times. Final two washes were for 15 minutes each at the same temperature range.

After removal of crude oil, extracted material was toasted by injecting steam directly on the material until the product temperature reached 103–106 °C. Steam injection was stopped, and the material heated to 104–116 °C and held for 30–60 minutes. After toasting, the product was cooled to room temperature. Cooled material was hand screened with an 8-mesh sieve (toasted solvent extracted meal).

Miscella was passed through a laboratory vacuum evaporator unit to separate the crude oil and hexane. Crude oil was heated to 91–96 °C for hexane removal and filtered.
Cleaned soya bean samples were moisture adjusted to 16.0 percent and allowed to equilibrate overnight. Moisture adjusted seed was fed into a mechanical screw press to separate a portion of the crude oil from the presscake (mechanical meal). Resulting fractions were crude oil and presscake. Crude oil was filtered.

Crude oil samples from production of toasted meal and mechanical meal were processed separately by the following procedures.

Based on the free fatty acid content, a weighed amount of crude oil and 14° Baume sodium hydroxide was placed in a water bath at 20–24 °C and mixed for 90 minutes at high RPM, and then for 20 minutes at low RPM and 63–-67 °C. Neutralised oil was then centrifuged. Refined oil was decanted and filtered. Resulting fractions were alkali refined oil and soapstock.

Alkali refined oil was heated to 40–50 °C and an activated bleaching earth added (1.0 percent by weight of oil). The solution was stirred and placed under vacuum. Temperature was increased to 85–100 °C and held for 10 to 15 minutes. After the bleaching period, vacuum was broken, and an inert filter aid added to the mixture (1.0 percent by weight of the oil). Vacuum was resumed and the temperature reduced to 58–68 °C. Vacuum was broken and the bleached oil and spent bleaching earth/filter aid separated by vacuum filtration. Resulting fraction was bleached oil.

Bleached oil was steam bathed for 28–32 minutes under vacuum and temperature held between 220–230 °C. During the cooling period, a 0.5 percent citric acid solution was added (1 mL per 100 grams of oil deodorised). Resulting fraction was deodorised oil (RBD oil).

Table 181 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis [A]) in processed soya bean commodities

Location	N (int)	Rate (kg ai/ha)	Matrix	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Richland, IA, 2015 P35T58R	4 (8 7 6)	1.12 1.10 1.12 1.11	RAC	149	0.017	ND	<0.01	0.10	<0.01	ND	ND	ND
					0.019	ND	0.010	0.11	<0.01	ND	ND	ND
					0.017	ND	<0.01	0.10	<0.01	ND	ND	ND
				Mean	0.018	<0.01	0.01[B]	0.10	<0.01	<0.01	<0.01	<0.01
			Mechanically Extracted Meal		0.024	ND	0.012	0.19	0.011	ND	ND	ND
			Solvent Extracted Meal		0.023	ND	0.016	0.30	0.012	ND	ND	ND
			Hullsf		0.017	ND	<0.01	0.035	0.013	ND	<0.01	ND
			Mechanically Extracted Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND
			Solvent Extracted Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND
Carlyle, IL, 2015 H43L15	4 (7 7 6)	1.09 1.14 1.14 1.12	RAC	147	0.024	ND	0.015	0.21	0.013	ND	ND	ND
					0.025	ND	0.012	0.19	0.012	ND	ND	ND
					0.025	ND	0.014	0.22	0.013	ND	<0.01	ND
				Mean	0.024	<0.01	0.014	0.21	0.013	<0.01	<0.01	<0.01
			Mechanically Extracted Meal		0.039	ND	0.020	0.41	0.019	ND	<0.01	ND
			Solvent Extracted Meal		0.033	<0.01	0.019	0.41	0.017	ND	<0.01	ND
			Hulls		0.021	ND	0.012	0.070	0.024	ND	<0.01	<0.01

Location	N (int)	Rate (kg ai/ha)	Matrix	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
			Mechanically Extracted Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND
			Solvent Extracted Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND
Wyoming, IL, 2015 S35-A5	4 (8 6 8)	1.14 1.12 1.13 1.12	Processor Seed	139	<0.01	ND	<0.01	0.039	ND	ND	ND	ND
					<0.01	ND	<0.01	0.037	ND	ND	ND	ND
					<0.01	ND	<0.01	0.038	ND	ND	ND	ND
				Mean	<0.01	<0.01	<0.01	0.038	<0.01	<0.01	<0.01	<0.01
			Mechanically Extracted Meal		<0.01	ND	<0.01	0.069	ND	ND	ND	ND
			Solvent Extracted Meal		0.01[B]	ND	<0.01	0.077	ND	ND	ND	ND
			Hulls		0.012	ND	<0.01	0.045	ND	ND	ND	ND
			Mechanically Extracted Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND
			Solvent Extracted Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND

Notes:

[A] A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

[B] Residue found was ≥LOD and <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg

Maize

Shepard (2020 DuPont-43225, Revision No. 1) studied the distribution of fluazaindolizine residues upon processing of maize. Moisture was adjusted (54–71 °C in oven), if required, to a moisture content of 10.0–15.0 percent. Samples were cleaned by aspiration and screening. Light impurities were removed using an aspirator. After aspiration, samples were screened in a 2-screen cleaner to separate large and small foreign particles (screenings) from the cleaned grain.

Dry Milling Process

Cleaned whole corn grain were moisture conditioned to 20.0–22.0 percent and tempered for approximately 2 hours. After tempering, the samples were fed into a disc mill to crack the kernel and the resulting material dried in an oven for 30 minutes at 54–71 °C. Dried cornstock was screened with a 3.2 mm screen to separate bran, germ, and large grits from grits, meal and flour.

Material <3.2 mm (grits, meal, and flour) was sieved (two screens: 1.4 mm and 0.25 mm) sieves. The fraction on top of 1.4 mm sieve was “grits”; the fraction on top of the 0.25 mm sieve was “meal” and the fraction through the 0.25 mm sieve was “flour.”

Material on top of the 3.2 mm screen (bran, germ, and large grits) was screened again using a 4.8 mm screen (or similar size). Material above the screen was aspirated using the aspirator to remove bran (hull material) from the germ with attached hull and endosperm. Germ with attached hull and endosperm was passed through the disc mill and roller mill and screened. Material above screen was aspirated to remove hull from the germ. Material through the 3.2 mm screen was added to the large grit fraction weight for mass balance purposes.

All material through the 4.8 mm screen (large grits and detached germ) was passed over a gravity separator to separate germ and large grits. If necessary, germ was milled, screened, and/or aspirated to remove endosperm and hull material.

The two germ fractions were combined and dried at 54–71 °C to a final moisture of 14.0–16.0 percent. Control germ from trial 01 did not require drying. Requested grits, meal, and flour fractions were collected and placed into freezer storage.

Germ material was heated to 71–79 °C in a mixer and held for 10 minutes. Following heating, the material was flaked in a flaking roll with a gap setting of 0.18–0.25 mm. After flaking, the treated sample from trial 21 was sieved to remove remaining endosperm from the germ flakes. Flaked material was placed in stainless steel batch extractors and submerged in 49–60 °C solvent (hexane). After 30 minutes, the miscella (crude oil and hexane) was drained and hexane was added to repeat the cycle two more times. Final two washings were at the same temperature range and for 15–30 minutes each. Following the final draining, ambient or warm air was passed over spent flakes to remove residual hexane. Resulting fractions from solvent extraction were miscella and solvent-extracted germ flakes.

Miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. Crude oil was then heated to 91–96 °C for hexane removal, filtered, and collected for refining.

Based on the free fatty acid content, a weighed amount of sodium hydroxide was added to the crude oil. The mixture was placed in a water bath and mixed for 15 minutes at high RPM at 20–24 °C and then for 12 minutes at low RPM at 63–67 °C. Neutralised oil and soapstock were separated using centrifugation. Alkali refined oil samples were decanted and filtered prior to bleaching.

During bleaching, alkali refined oil was heated to 40–50 °C and activated bleaching earth added (1.0 percent by weight of oil). After addition, the solution was placed under vacuum and stirred. Temperature was increased to 85–100 °C and held for 10 to 15 minutes and allowed to cool before filtering (bleached oil).

Bleached oil was steam bathed at 220–230 °C for 28–32 minutes under vacuum. The oil was then allowed to cool. During the cooling period, a 0.5 percent citric acid solution was added (1 mL per 100 grams of oil deodorised). Resulting fractions were refined.

During bleaching, alkali refined oil was heated to 40–50 °C and activated bleaching earth added (1.0 percent by weight of oil). After addition, the solution was placed under vacuum and stirred. Temperature was increased to 85–100 °C and held for 10 to 15 minutes, then cooled and filtered (bleached oil).

Bleached oil was steam bathed at 220–230 °C for 28–32 minutes under vacuum and to cool during which, a 0.5 percent citric acid solution was added (1 mL per 100 grams of oil deodorised). Resulting fraction was refined bleached-deodorised oil (RBD oil).

Wet Milling Process

A representative sample of dried and cleaned corn grain was steeped in 49–54 °C water containing 0.1–0.2 percent sulfur dioxide (sulphurous acid) for 22–48 hours. At the end of the steeping period, steep water was drained and a representative fraction collected and placed into frozen storage. Steeped whole corn was passed through a bell disc mill and a majority of the germ and hull was removed using a hydroclone (water centrifuge). Germ and hull were dried at 74-91 °C to obtain a final moisture between 5-10 percent. After drying, the germ and hull were separated using aspiration and screening.

Cornstock (without germ and hull) ground in the disc mill was passed over a separator equipped with a 325 mesh (50 micron) screen. In commercial industry, only bran (hull material) remains on top of

the screen. Material on top of the screen was discarded. Process water (with starch and gluten) passing through the screen was separated into starch and gluten using batch centrifugation. Starch was dried in an oven at 54–71 °C until the moisture content was less than 15.0 percent (Starch).

Germ samples were moisture conditioned to 14–16 percent, heated to 88–104 °C in a mixer, flaked in a flaking roll with a gap setting of 0.2 mm, and pressed in an expeller to liberate part of the crude oil. Resulting fractions are expelled crude oil and presscake with residual crude oil.

Presscake was placed in stainless steel batch extractors and submerged in 49–0 °C hexane. After 30 minutes, the miscella was drained and fresh hexane was added to repeat the cycle two more times. Final two washings were for 15 minutes each. Following the final draining, the spent presscake was desolventised with ambient or warm air to remove residual hexane. Resulting fractions from solvent extraction were miscella and solvent extracted presscake (germ cake).

Miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. Crude oil was then heated to 91–96 °C for hexane removal. Crude oils from expelling and solvent extraction were filtered and combined for refining. Crude oil samples from the wet milling process were alkali refined, bleached, and deodorised utilizing the same methods used during the dry milling procedure to produce RBD oil.

Table 182 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in processed maize commodities

Location	Rate (kg ai/ha)	Matrix	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Richland, IA, 2015 P1023AM	0.117 0.112 0.112 0.112	RAC	152	ND	<0.01	ND	0.072	<0.01	0.013	<0.01	<0.01
				ND	<0.01	ND	0.075	<0.01	0.012	<0.01	<0.01
				ND	<0.01	ND	0.072	<0.01	0.014	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	0.073	<0.01	0.013	<0.01	<0.01
		Starch		ND	ND	ND	0.01[B]	ND	ND	ND	ND
		Grits		ND	ND	ND	0.037	<0.01	<0.01	ND	<0.01
		Flour		ND	ND	ND	0.068	<0.01	0.01[B]	ND	<0.01
		Meal		ND	ND	ND	0.096	<0.01	0.014	<0.01	0.010
		Wet Milled Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND
		Dry Milled Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND
Stewardson, IL, 2015 RK 110-10RR	0.111 0.112 0.112 0.111	RAC	155	ND	ND	ND	0.055	ND	0.01[B]	ND	0.011
				ND	<0.01	ND	0.058	ND	0.011	ND	0.012
				ND	<0.01	ND	0.056	ND	0.010	ND	0.011
			Mean	<0.01	<0.01	<0.01	0.056	<0.01	0.010	<0.01	0.011
		Starch		ND	ND	ND	<0.01	ND	ND	ND	ND
		Grits		ND	ND	ND	0.027	ND	<0.01	ND	<0.01
		Flour		ND	<0.01	ND	0.13	<0.01	0.017	<0.01	0.021
		Meal		ND	<0.01	ND	0.11	<0.01	0.017	<0.01	0.018
		Wet Milled Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND
		Dry Milled Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND
Uvalde, TX, 2015 DKC64-69	0.111 0.112 0.112 0.112	RAC	178	ND	<0.01	ND	0.033	ND	<0.01	<0.01	<0.01
				ND	<0.01	ND	0.030	<0.01	0.013	<0.01	<0.01
				ND	<0.01	ND	0.031	<0.01	0.011	<0.01	<0.01
			Mean	<0.01	<0.01	<0.01	0.031	<0.01	0.011	<0.01	<0.01
		Starch		ND	ND	ND	ND	ND	ND	ND	ND
		Grits		ND	ND	ND	0.019	<0.01	<0.01	<0.01	<0.01
		Flour		ND	<0.01	ND	0.058	<0.01	0.016	<0.01	0.013

Location	Rate (kg ai/ha)	Matrix	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
		Meal		ND	<0.01	ND	0.052	<0.01	0.015	<0.01	0.01[B]
		Wet Milled Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND
		Dry Milled Refined Oil		ND	ND	ND	ND	ND	ND	ND	ND

Notes:

[A] molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing.

[B] residue was ≥LOD but <LOQ (reported to one significant figure) but rounds to 0.01 mg/kg.

Wheat

Shepard (2020 DuPont-43224, Revision No. 1) studied the distribution of fluazaindolizine residues upon processing of wheat. If the moisture content of a sample was above 13.5 percent, the sample was dried in a tray oven at 54–71 °C until the moisture content was 11.0–13.5 percent moisture. Samples from trial 02 required drying. Following drying (if required), samples were cleaned by aspiration and screening. Light impurities were separated from the sample using an aspirator. After aspiration, the sample was screened using a two-screen cleaner to separate large and small foreign particles (screenings) from the cleaned wheat sample. Due to large plant material in the grain, the treated sample from trial 03 was screened before aspiration.

Cleaned wheat was moisture adjusted (tempered) to 16 percent. After tempering for 1–1.5 hours, the wheat was passed through a disc mill. The material was sifted with a sifter equipped with an 8, 14, and 30-mesh sieve. The material on top of the 30-mesh sieve was aspirated to remove bran from the germ fraction. The germ (with endosperm) was passed through a reduction mill. The germ and reduced endosperm were sifted with a sifter equipped with 20, 24, and 28-mesh sieves to separate the germ from the endosperm. The germ material was also aspirated again to remove additional bran and milled/sieved to remove additional endosperm (germ). A 0.5–1.0 percent recovery of germ is expected.

Cleaned wheat was moisture conditioned (tempered) depending on the physical property of the wheat. The physical property of the grain kernel varies depending on whether the grain has a floury or vitreous kernel. In a floury kernel, a cross-section of the grain reveals a grainy soft white structure. The cross-section of a vitreous grain is hard and amber coloured. There are grains with an intermediate structure (a floury and vitreous part). Samples were determined to be vitreous (trial 01), intermediate structure (trial 02), and floury (trial 03) and were moisture conditioned accordingly.

- floury grain wheat: 16.5 percent moisture conditioning with 24 hour ±30 minute resting period before milling
- completely vitreous grain wheat: 17.5 percent moisture conditioning with 48 hour ±30 minute resting period before milling
- intermediate structure: 17.5 percent moisture conditioning with 24 hour ±30 minute resting period before milling

Tempered wheat was milled. Breaking of the wheat was accomplished by three break rolls. After passing through the break rolls, the material was sifted using a screen sifter (two sizes, 140 micron and 800 micron). Material passing through the 120-micron screen is “Break Flour.” Material passing through the 800 micron screen is middlings. Material not passing through was conveyed to the end of the sifter. Material exiting the end is bran (coarse).

Middlings were passed through two reduction rolls followed by a sifter screen (160 micron). Material passing through the screen is reduction flour with the remainder shorts. The break and reduction flours were mixed to produce standard mill-run flour.

Bran exiting the break sieve is conveyed through beater bars over a 128-micron screen. Material passing through the screen is “Shorts” and is added to “Shorts” from the reduction mill. Material passing over the screen and exiting the end is “Bran.” Residues in wheat processed commodities are shown in Table 183.

Table 183 Residues (mg/kg) of fluazaindolizine (pre-hydrolysis) and related compounds (post-hydrolysis) in processed wheat commodities

Location	N (int)	Rate (kg ai/ha)	Matrix	DALA	Fluaza-indolizine	IN-A5760	IN-F4106	IN-QEK31	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
Northwood, ND, 2015 Faller	4 (6 7 8)	1.12 1.12 1.12 1.13	RAC	119	ND	ND	ND	0.028	ND	ND	ND	ND
					ND	ND	ND	0.024	ND	ND	ND	ND
					ND	ND	ND	0.025	ND	ND	ND	ND
				Mean	<0.01	<0.01	<0.01	0.026	<0.01	<0.01	<0.01	<0.01
			Bran		ND	ND	ND	0.039	ND	ND	ND	ND
			Flour		ND	ND	ND	0.012	ND	ND	ND	ND
			Middlings		ND	ND	ND	0.020	ND	ND	ND	ND
			Shorts		ND	ND	ND	0.022	ND	ND	ND	ND
			Germ		ND	---	---	0.045	ND	---	ND	ND
					ND	<0.01	ND	0.049	ND	ND	ND	ND
				Mean	<0.01	<0.01	<0.01	0.047	<0.01	<0.01	<0.01	<0.01
Gardner, ND, 2015 Elgin-ND	4 (7 7 6)	1.12 1.15 1.15 1.12	RAC	121	ND	ND	ND	0.027	ND	ND	ND	ND
					ND	ND	ND	0.025	ND	ND	ND	ND
					ND	ND	ND	0.024	ND	ND	ND	ND
				Mean	ND	ND	ND	0.025	ND	ND	ND	ND
			Bran		<0.01	ND	ND	0.038	ND	ND	ND	ND
			Flour		ND	ND	ND	<0.01	ND	ND	ND	ND
			Middlings		ND	ND	ND	0.010	ND	ND	ND	ND
			Shorts		ND	ND	ND	0.019	ND	ND	ND	ND
			Germ		<0.01	ND	ND	0.053	ND	ND	ND	ND
Uvalde, TX, 2015 Greer	4 (7 7 7)	1.12 1.11 1.12 1.12	RAC	212	ND	ND	ND	0.087	ND	ND	ND	ND
					ND	ND	ND	0.091	ND	ND	ND	ND
					ND	ND	ND	0.095	ND	ND	ND	ND
				Mean	<0.01	<0.01	<0.01	0.091	<0.01	<0.01	<0.01	<0.01
			Bran		ND	ND	ND	0.088	ND	ND	ND	ND
			Flour		ND	ND	ND	0.029	ND	ND	ND	ND
			Middlings		ND	ND	ND	0.041	ND	ND	ND	ND
			Shorts		ND	ND	ND	0.070	ND	ND	ND	ND
			Germ		ND	<0.01	ND	0.14	ND	ND	ND	ND

A molecular weight back-calculation was applied to residue values of IN-QZY47, IN-TMQ01, IN-UJV12, and IN-UNS90 (IN-TQD54) to account for reference standards which were supplied in salt form but not taken into consideration during weighing. The calculated processing factors are shown in Table 184.

Table 184 Processing factors (PF) for fluazaindolizine and related compounds

Processed commodity	Fluazaindolizine		IN-F4106+1.068×IN-A5760		IN-QZY47		IN-TMQ01		IN-UJV12		IN-TQD54	
	Individual values	Median or best estimate	Individual values	Median or best estimate	Individual values	Median or best estimate	Individual values	Median or best estimate	Individual values	Median or best estimate	Individual values	Median or best estimate
Strawberry juice							0.5 0.7 0.8	0.7			0.8 0.9	0.8
Strawberry canned							0.3 0.4 0.4	0.4			0 0.5	0.2

Processed commodity	Fluazaindolizine		IN-F4106+1.068×IN-A5760		IN-QZY47		IN-TMQ01		IN-UJV12		IN-TQD54	
	Individual values	Median or best estimate	Individual values	Median or best estimate	Individual values	Median or best estimate	Individual values	Median or best estimate	Individual values	Median or best estimate	Individual values	Median or best estimate
Strawberry jam							0.2 0.2 0.2	0.2			0 0	0
Strawberry frozen fruit							0.6 0.6 0.7	0.6			0 0.5	0.2
Strawberry dehydrated fruit							3.8 4.1 5.4	4.1			3.3 3.8	3.6
Tomato dried	1.8 4.9	3.35	4.6 7.5 14	7.5	4.4	4.4	4.5	4.5	4.7	4.7	3.5	3.5
Tomato canned	0 0.22	0.11	0.7 0.9 1.1	0.9	1.2	1.2	0.9	0.9	1.2	1.2	1.1	1.1
Tomato juice	0 0.53	0.265	0.6 0.9 1.9	0.9	0.7	0.7	0.6	0.6	0.7	0.7	0.6	0.6
Tomato wet pomace	1 2.4	1.7	1.1 1.1 1.2	1.1	0.7	0.7	0.8	0.8	0.8	0.8	0.8	0.8
Tomato paste	1.2 1.3	1.25	2.6 3.9 5.7	3.9	2.7	2.7	2.4	2.4	2.8	2.8	2.3	2.3
Tomato purée	0.53 0.8	0.665	1.2 2.0 2.4	2	1.4	1.4	1.2	1.2	1.3	1.3	1.2	1.2
Potato dried flakes	0 0.07	0.035	0 0.1 0.6	0.1	1.7 3.7	2.7	1.0 1.7	1.4	1.5 3.1	2.3	1.2 2.2	1.7
Potato crisps	0.03 0.18	0.105	0.08 0.3 0.5	0.3	0.6 1.2	0.9	0.6 1.3	1	0.5 1.3	0.9	0.6 1.3	1
French fries peeled	0 0.04	0.02	0 0.04 0.2	0.04	0.8 1.2	1	0.3 0.6	0.4	0.6 1.1	0.8	0.4 0.7	0.6
French fries unpeeled	0.08 0.91	0.495	0.2 0.7 1.0	0.7	1.2 2.1	1.7	0.8 2.0	1.4	0.8 2.0	1.4	0.8 2.1	1.5
Potato boiled unpeeled	0.07 0.15	0.11	0.06 0.07 0.2	0.07	1.1 1.2	1.2	0.8 1.2	1	1.3 1.4	1.4	1.0 1.2	1.1
Potato boiled peeled	0 0.02	0.01	0 0.03 0.08	0.03	1.0 1.1	1	0.4 0.6	0.5	0.7 1.1	0.9	0.7 0.8	0.8
Potato microwaved unpeeled	0.4 1.3	0.85	0.4 1.2 1.4	1.2	1.4 1.6	1.5	1.7 1.8	1.8	1.5 1.8	1.6	1.5 1.8	1.6
Soya bean meal (mechanically extracted)	1.3 1.3 1.6	1.3	1.2 1.4 1.5	1.4	1.5 1.6	1.6					0 0 0	0
Soya bean meal (solvent extracted)	1.3 1.4 1.4	1.4	1.5 1.6 1.6	1.6	1.3 1.7	1.5					0 0 0	0
Soya bean hulls	0.9 0.9 1.7	0.9	0.8 0.9 1.5	0.9	1.8 1.9	1.8					0 0 0.09	0
Soya bean refined oil	0 0 0	0	0 0 0	0	0 0 0	0					0 0 0	0
Maize starch			0 0	0	0 0	0	0 0 0	0	0 0	0	0 0 0	0
Maize grits			0 0	0	0.7 1.0	0.85	0.4 0.5 0.7	0.5	0 0.5	0.2	0.15 0.2 0.2	0.2
Maize flour			0 2	0.1	1.3 1.6	1.45	0.8 1.4 1.7	1.4	0 1.3	0.6	0.3 0.6 1.0	0.6
Maize meal			0 1.7	0.85	1.5 1.6	1.55	1.1 1.4 1.7	1.4	1 1.5	1.2	0 0.4 0.9	0.4
Maize refined oil			0 0	0	0 0	0	0 0 0	0	0 0	0	0 0 0	0

Livestock feeding studies

Dairy cow feeding study

The transfer of fluazaindolizine from feed to tissues and milk of dairy cows was studied by Dunlop et al (2019 DuPont-42563, Revision No 1). Three groups of three Holstein Friesian and Ayrshire cows (2.6–11.3 years old, 470–764 kg bw,) were dosed orally once daily via gelatine capsules with fluazaindolizine at doses equivalent to 2.3, 6.7, 20.3 and 19.6 (depuration) ppm in the diet (dry weight basis) for 28 days

(0.055, 0.157, 0.506 and 0.509 mg/kg bw/day). An additional three animals were dosed at the high rate and were used for the depuration phase of the study. Mean daily feed consumption during the dosing period was 15.8 kg DM/day comprising 4 kg of concentrate and hay ad libitum (2.3 ppm 15.0 kg/day, 6.7 ppm 15.8 kg/day, 20.3 ppm 16.9 kg/day, depuration 16.4 kg/day). Mean daily milk yields during dosing were 13.2, 13.3, 12.8 and 11.24 kg/cow/day respectively. Milk was collected twice daily (am and pm sampling) and pooled (am and previous day pm samples) in a ratio reflecting production. Muscle (loin, hind leg and diaphragm), liver, kidney and fat (perirenal, mesenteric and subcutaneous) were collected at sacrifice within 22–24 hours after the last dose. Milk, liver, kidney, fat and muscle were analysed for residues of fluazaindolizine, IN-A5760, IN-F4106, IN-QEK31, IN-R2W56, IN-REG72 and IN-RYC33 using Charles River Analytical Procedure AP.225144.02, a modified version of method DuPont-39226 (LOQ 0.01 mg/kg for each analyte). Procedural recoveries were within acceptable ranges (Table 185).

Table 185 Procedural recoveries for bovine matrices (DuPont-42563, Revision No 1)

Matrix	Analyte	Fortification level		
		0.01 mg/kg	0.1 mg/kg	0.3 mg/kg
Whole milk	fluazaindolizine	103±16 %	99±14 %	101±9 percent
	IN-REG72	99±13	100±10	
	IN-F4106	98±14	102±10	
	IN-A5760	102±13	103±12	
	IN-QEK31	101±11	104±10	96±2
	IN-RYC33	101±10	104±4	
	IN-R2W56	104±10	105±8	
Skimmed milk	fluazaindolizine	102±22	100±16	109±4
	IN-REG72	98±15	101±7	
	IN-F4106	108±18	105±5	
	IN-A5760	105±9	105±5	
	IN-QEK31	103±8	99±9	99±1
	IN-RYC33	105±7	103±6	
	IN-R2W56	111±8	108±7	
Cream	fluazaindolizine	99±16	96±13	103±16
	IN-REG72	117±11	107±10	
	IN-F4106	111±8	108±7	
	IN-A5760	110±9	103±12	
	IN-QEK31	102±11	99±10	97±2
	IN-RYC33	104±10	101±7	
	IN-R2W56	116±10	111±9	
Muscle	fluazaindolizine	116±10	120±10	
	IN-REG72	107±6	91±18	
	IN-F4106	107±7	104±3	
	IN-A5760	110±5	106±3	
	IN-QEK31	96±5	93±6	
	IN-RYC33	104±6	99±5	
	IN-R2W56	107±5	102±6	
Fat	fluazaindolizine	115±5	98±5	
	IN-REG72	114±8	102±1	
	IN-F4106	106±16	99±3	
	IN-A5760	104±11	99±3	
	IN-QEK31	92±3	89±8	
	IN-RYC33	90±4	84±7	
	IN-R2W56	101±5	95±8	
Kidney	fluazaindolizine	100±13	100±12	98±10
	IN-REG72	84±12	94±13	
	IN-F4106	102±14	97±10	
	IN-A5760	92±8	93±6	

Matrix	Analyte	Fortification level		
		0.01 mg/kg	0.1 mg/kg	0.3 mg/kg
	IN-QEK31	96±17	96±10	105±7
	IN-RYC33	91±11	94±9	
	IN-R2W56	89±12	92±9	
Liver	fluazaindolizine	96±18	97±8	
	IN-REG72	86±17	91±14	
	IN-F4106	99±17	97±10	
	IN-A5760	95±10	95±9	
	IN-QEK31	86±9	90±9	
	IN-RYC33	90±14	91±15	
	IN-R2W56	90±13	90±11	

Residues of IN-REG72, IN-F4106, IN-A5760, IN-RYC33, and IN-R2W56 were not detected in whole milk, skim milk, cream, and tissue samples, with the exception of IN-F4106 in cows dosed with fluazaindolizine–6.7 ppm dose level < 0.01 mg/kg in kidney, 20.3 ppm dose level < 0.01 mg/kg in liver; 0.01 mg/kg in kidney.

Residues of IN-REG72, IN-F4106, IN-A5760, IN RYC33, and IN-R2W56 were not detected in whole milk, skim milk, cream, at dose levels up to 20.3 ppm, and IN-QEK31 ≤ LOQ in all samples of milk, skim milk and cream from the fluazaindolizine dosed cows. Residues of fluazaindolizine were < LOQ for the 2.3 ppm dose group, with the exception of one day-24 sample with residues of 0.01 mg/kg; however, fluazaindolizine in milk was quantifiable in the 6.7 and 20.3 ppm dose groups. Fluazaindolizine residues in milk plateaued by day 3, with average residues of < 0.020 and 0.067 mg/kg, respectively, for the 6.7 and 20.3-ppm dose groups (including milk samples from day 3–28). Maximum residues of fluazaindolizine in milk were 0.032 mg/kg for the 6.7-ppm group on Day 17 and 0.101 mg/kg for the 20.3-ppm group on Day 28. Comparison of dose levels with the maximum residues in whole milk and skim milk across all three dose groups showed a linear correlation (r^2≥0.999) between fluazaindolizine levels in the feed and fluazaindolizine residues in these matrices. Comparison of the transfer factors (TF) for cream from the 6.7- and 20.3-ppm dose groups also indicates a linear relationship between feed levels and residues in cream. Fluazaindolizine residues in skim milk and cream were generally similar to, or slightly lower than, residue levels in whole milk. Compared to average fluazaindolizine residues in whole milk on Days 14 and 21 from the 6.7- and 20.3-ppm dose groups, residue levels were 0.7–1.1× in the related samples of cream and 0.8–1.1× in the related samples of skim milk (Table 186).

In tissue samples, residues of metabolites IN-A5760, IN-R2W56, IN-REG72, and IN-RYC33 were <LOD in all samples of liver, kidney, muscle, and fat at dose levels up to 20.3 ppm in the diet, and residues of metabolites IN-F4106 and IN-QEK31 were <LOD in all muscle and fat samples. Mean fluazaindolizine residues were 0.020 mg/kg in milk, <LOQ in muscle, 0.020 mg/kg in fat, 0.021 mg/kg in liver and 0.091 mg/kg in kidney for the 6.7 ppm dose group and 0.066 mg/kg in milk, < 0.01 mg/kg in muscle, 0.034 mg/kg in fat, 0.061 mg/kg in liver and 0.215 mg/kg in kidney for the 20.3 ppm dose group. Mean residues in tissues showed a linear relationship with dose. Once dosing stopped, residues declined with a DT_{50} of < 0.5 days (Figure 31).

Table 187 Summary of fluazaindolizine and metabolite residues (mg/kg) in tissues of animals dosed with fluazaindolizine

	2.3 ppm (0.05 mg/kg bw/day)	6.7 ppm (0.15 mg/kg bw/day)			20.3 ppm (0.50 mg/kg bw/day)		
	Fluazaindolizine	Fluazaindolizine	IN-F4106	IN-QEK31	Fluazaindolizine	IN-F4106	IN-QEK31
Fat	<0.01 ND <0.01	0.018 0.020 0.022	ND ND ND	ND ND ND	<0.01 0.054 0.042	ND ND ND	<0.01 <0.01 <0.01

	2.3 ppm (0.05 mg/kg bw/day)	6.7 ppm (0.15 mg/kg bw/day)			20.3 ppm (0.50 mg/kg bw/day)		
	Fluazaindolizine	Fluazaindolizine	IN-F4106	IN-QEK31	Fluazaindolizine	IN-F4106	IN-QEK31
+ 1 day					0.015	ND	ND
+ 2 days					<0.01	ND	ND
+ 5 days					ND	ND	ND
Muscle	ND ND ND	ND ND ND	ND ND ND	ND ND ND	<0.01 <0.01 <0.01	ND ND ND	ND ND ND
+ 1 day					<0.01	ND	ND
+ 2 days					ND	ND	ND
+ 5 days					ND	ND	ND
Liver	<0.01 <0.01 ND	0.023 0.022 0.018	ND ND ND	ND ND ND	0.049 0.058 0.078	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01
+ 1 day					0.011	ND	ND
+ 2 days					<0.01	ND	ND
+ 5 days					ND	ND	ND
Kidney	0.022 0.018 0.027	0.085 0.091 0.096	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.17 0.19 0.29	<0.01 0.012 <0.01	<0.01 0.016 0.015
+ 1 day					0.059	<0.01	<0.01
+ 2 days					0.019	ND	<0.01
+ 5 days					ND	ND	ND

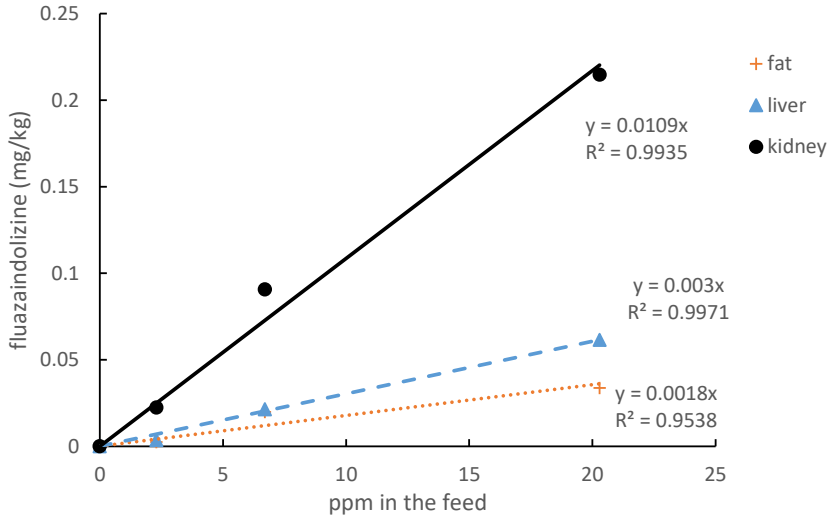


Figure 31 Mean residues of fluazaindolizine as a function of dose. Fat y=0.0018x, r2=0.954; liver y=0.003x, r2=0.997; kidney y=0.0109x, r2=0.9935.

Residues of fluazaindolizine reached a plateau in whole milk after approximately 3 days of dosing and did not appear to concentrate in the cream or skim milk fractions (Table 187).

Table 187 Summary of fluazaindolizine and metabolite residues (mg/kg) in milk of animals dosed with fluazaindolizine

	2.3 ppm (0.05 mg/kg bw/day)	6.7 ppm (0.15 mg/kg bw/day)		20.3 ppm (0.50 mg/kg bw/day)	
	Fluazaindolizine	Fluazaindolizine	IN-QEK31	Fluazaindolizine	IN-QEK31
Pre-dose	ND ND ND	ND ND ND	ND ND ND	ND ND ND	ND ND ND
1	ND ND ND	<0.01 0.013 ND	ND ND ND	0.031 0.018 0.031	ND ND ND
3	<0.01 <0.01 <0.01	0.024 0.020 <0.01	ND ND ND	0.083 0.054 0.074	ND ND ND
5	<0.01 <0.01 <0.01	0.019 0.022 0.012	ND ND ND	0.078 0.068 0.046	ND ND ND

	2.3 ppm (0.05 mg/kg bw/day)	6.7 ppm (0.15 mg/kg bw/day)		20.3 ppm (0.50 mg/kg bw/day)	
	Fluazaindolizine	Fluazaindolizine	IN-QEK31	Fluazaindolizine	IN-QEK31
7	<0.01 <0.01 <0.01	0.020 0.017 0.016	ND ND ND	0.062 0.050 0.065	ND ND ND
10	<0.01 <0.01 <0.01	0.020 0.021 0.013	ND ND ND	0.063 0.044 0.064	ND ND ND
14	<0.01 <0.01 <0.01	0.021 0.023 0.016	ND ND ND	0.067 0.027 0.089	<0.01 ND <0.01
17	<0.01 <0.01 <0.01	0.022 0.025 0.032	ND ND ND	0.071 0.050 0.068	ND ND ND
21	<0.01 <0.01 <0.01	0.024 0.024 0.018	ND ND ND	0.056 0.072 0.085	ND ND ND
24	0.011 <0.01 <0.01	0.023 0.025 0.015	ND ND ND	0.084 0.069 0.088	ND <0.01 <0.01
28	<0.01 <0.01 <0.01	0.022 0.022 0.014	ND ND <0.01	0.071 0.064 0.101 0.065 0.045 0.070	ND <0.01 <0.01 <0.01 <0.01 <0.0
29 (+1 day)				0.028 0.035	ND ND
30 (+2 days)				<0.01 0.013	ND ND
31 (+3 days)				<0.01	ND
32 (+4 days)				ND	ND
33 (+5 days)				ND	ND
Cream Day 14	<0.01 <0.01 0.077 (QEK ND ND <0.01)	0.020 0.19 0.12	ND ND ND	0.066 0.058 0.072	<0.01 ND ND
Cream Day 21	<0.01 <0.01 <0.01	0.034 0.020 0.012	ND ND <0.01	0.042 0.048 0.052	ND ND ND
Cream Day 30 (+2 days)				0.010 <0.01	ND ND
Cream Day 33 (+ 5 days)				ND	ND
Skimmed Milk Day 14	<0.01 <0.01 <0.01	0.020 0.017 0.012	ND ND ND	0.070 0.053 0.072	ND ND ND
Skimmed Milk Day 21	0.011 0.010 <0.01	0.028 <0.01 0.017	ND ND ND	0.052 0.090 0.037	ND ND <0.01
Skimmed Milk Day 30 (+2 days)				<0.01 0.013	ND ND
Skimmed Milk Day 33 (+5 days)				ND	ND

Notes:

IN-A5760, IN-F4106, IN-REG72, IN-RYC33 all <LOD.

The transfer of IN-QEK31from feed to tissues and milk of dairy cows was also studied by Dunlop *et al.* (2019 DuPont-42563, Revision No 1). IN QEK31 was administered orally to two groups of three Holstein Friesian and Ayrshire cattle (520-674 kg bw) by gelatine capsule for 28 days. The dosage was 0.506 mg/kg bw/day, equivalent to 19.5 ppm in the feed with an additional animal dosed at the equivalent of 18.5 ppm in the feed was slaughtered 5 days after the last dose. Mean daily feed consumption during the dosing period was 16.2 kg DM/day and 14.7 kg DM/day for the depuration group. Milk yields were 12.4 and 13.6 kg/cow/day.

Residues of IN-QEK31 reached a plateau in whole milk after approximately 3 days of dosing and did not appear to concentrate in the cream or skim milk fractions. Mean IN-QEK31 residues were 0.203 mg/kg in milk, <LOQ in muscle, <LOQ in fat, 0.013 mg/kg in liver and 0.128 mg/kg in kidney. Once dosing stopped, residues declined with a DT50 of <1.2 days (Tables 188 and 189).

Table 188 Summary of IN-QEK31 residues (mg/kg) in milk of animals dosed with IN-QEK31

Day	19.5 ppm (0.50 mg/kg bw/day)
Pre-dose	ND ND ND
1	0.16 0.13 0.21
3	0.24 0.20 0.21
5	0.18 0.18 0.25
7	0.14 0.17 0.34
10	0.21 0.16 0.22
14	0.18 0.15 0.18
17	0.21 0.22 0.27

Day	19.5 ppm (0.50 mg/kg bw/day)
21	0.20 0.18 0.18
24	0.22 0.21 0.22
28	0.21 0.18 0.22 0.15
29 (+1 days)	0.034
30 (+2 days)	ND
31 (+ 3days)	<0.01
32 (+4 days)	ND
33 (+5 days)	<0.01
Cream Day 14	0.15 0.14 0.19
Cream Day 21	0.11 0.11 0.096
Cream Day 30 (+2 days)	ND
Cream Day 33 (+5 days)	ND
Skimmed Milk Day 14	0.19 0.17 0.20
Skimmed Milk Day 21	0.17 0.20 0.19
Skimmed Milk Day 30 (+2 days)	ND
Skimmed Milk Day 33 (+5 days)	ND

Table 189 Summary of IN-QEK31 residues (mg/kg) in tissues of animals dosed with IN-QEK31

	19.5 ppm (0.50 mg/kg bw/day)	
	Residue (mg/kg)	Mean (mg/kg)
Fat	<0.01 <0.01 <0.01	<0.01
+5 days	ND	
Muscle	ND <0.01 ND	<0.01
+5 days	ND	
Liver	0.016 0.016 <0.01	0.0135
+5 days	ND	
Kidney	0.19 0.10 0.092	0.128
+5 days	<0.01	

Notes:

ND = Not detected. Response below the limit of detection (0.00333 mg/kg based on 100 percent recovery and no matrix effects).

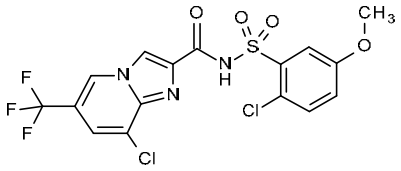
APPRAISAL

Fluazaindolizine is a new selective nematicide for the control of plant parasitic nematodes. At the Fifty-first Session of the CCPR, it was scheduled for the evaluation as a new compound in 2021 and rescheduled to the 2022 JMPR.

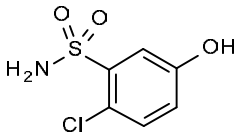
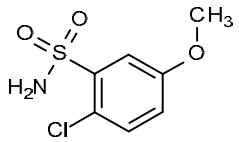
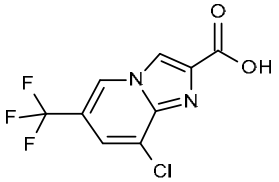
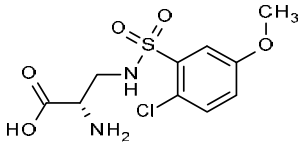
Fluazaindolizine is used for annual crops (e.g., fruiting vegetables, cucurbits, root vegetables, row crops) and certain perennial crops (e.g., citrus, tree nuts and stone fruits). Application methods include drip, drench, in furrow spray with or without soil incorporation either before or at planting, with the option for follow-up in crop treatment.

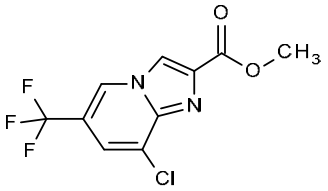
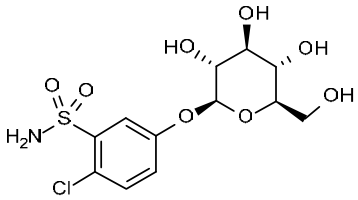
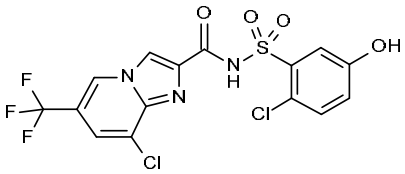
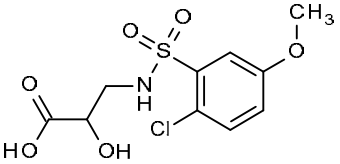
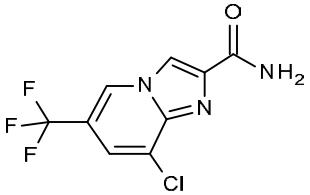
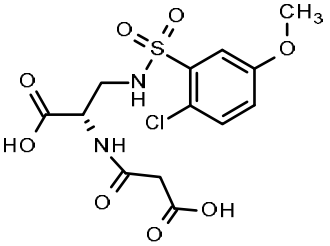
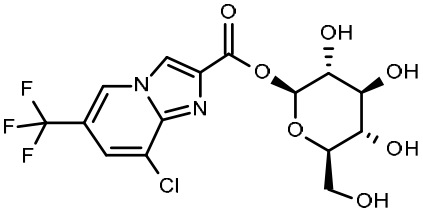
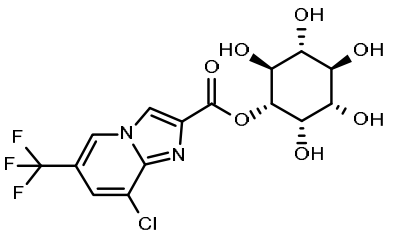
The Meeting received information on the metabolism of fluazaindolizine and a number of its metabolites in lactating goats and laying hens, the metabolism of fluazaindolizine in tomato, carrot, potato, soya bean and sugarcane and follow crops, methods of residue analysis, freezer storage stability, GAP information, supervised residue trials on a range of crops as well as livestock feeding studies (lactating cow).

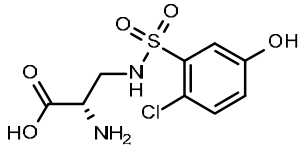
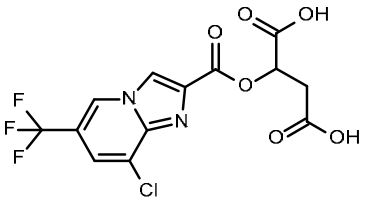
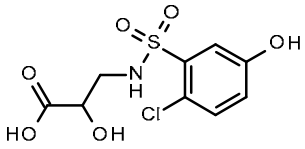
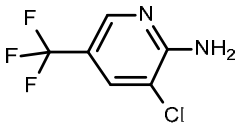
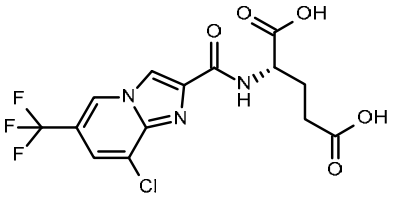
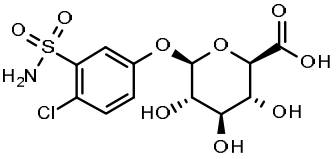
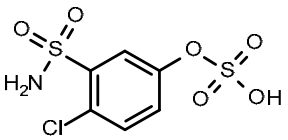
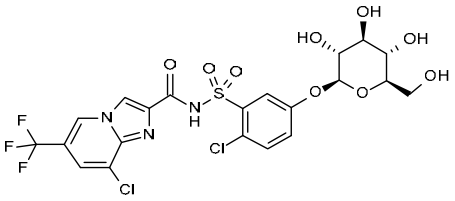
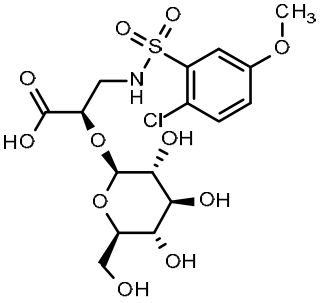
Fluazaindolizine (company code DPX-Q8U80).

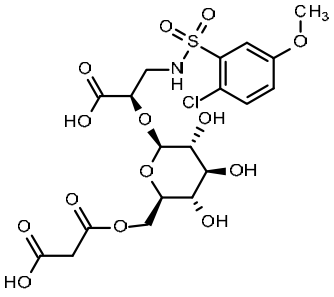
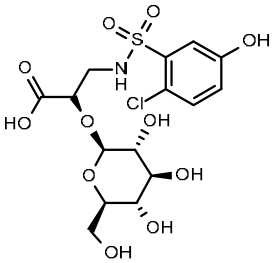
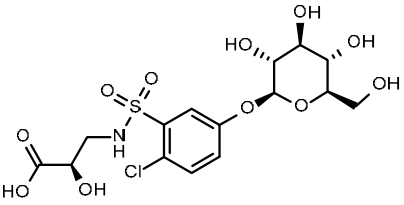
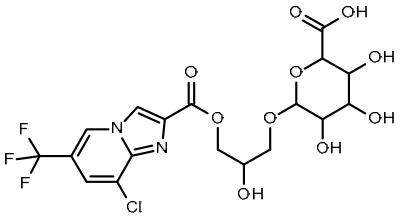
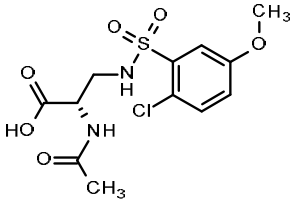
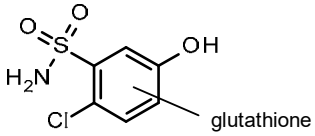


The following abbreviations are used for the major metabolites discussed below:

Code Names, MW	Chemical Name	Chemical Structure
IN-A5760	2-chloro-5-hydroxybenzenesulfonamide	
IN-F4106	2-chloro-5-methoxybenzenesulfonamide	
IN-QEK31	8-chloro-6-(trifluoromethyl) imidazo[1,2-a]pyridine-2-carboxylic acid	
IN-QZY47	3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-L-alanine	

Code Names, MW	Chemical Name	Chemical Structure
IN-R2W56 IN-QEK31 methyl ester	8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylic acid methyl ester	
IN-R3Z85 IN-A5760 glucose conjugate	2-chloro-5-(β-D-glucopyranosyloxy)benzenesulfonamide	
IN-REG72	8-chloro-N-[(2-chloro-5-hydroxyphenyl)sulfonyl]-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide	
IN-RSU03 (racemate) IN-TMQ01 (R-enantiomer)	3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-2-hydroxypropanoic acid	
IN-RYC33	8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide	
IN-TUT81 IN-QZY47 malonyl conjugate	N-(carboxyacetyl)-3-[[(2-chloro-5-methoxyphenyl)sulfonyl]-amino]-L-alanine	
IN-UGA20 IN-QEK31 glucose conjugate	β-D-glucopyranose 1-[8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylate]	
IN-UHD13 IN-QEK31 inositol conjugate	[(2S,3R,5S,6S)-2,3,4,5,6-pentahydroxycyclohexyl]1-[8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylate]	

Code Names, MW	Chemical Name	Chemical Structure
IN-UJV12 (S-enantiomer)	3-[[(2-chloro-5-hydroxyphenyl)sulfonyl]amino]-L-alanine	
IN-UJU44 QEK31 malic acid conjugate	2-[[[8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridin-2-yl]carbonyl]oxy]butanedioic acid	
IN-UNS90 (racemate) IN-TQD54 (R-enantiomer)	3-[[(2-chloro-5-hydroxyphenyl)sulfonyl]amino]-2-hydroxypropanoic acid	
IN-VM862	3-chloro-5-(trifluoromethyl)pyridin-2-amine	
IN-WUK12 Glutamic acid conjugate of IN QEK31	N-[[8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-yl]carbonyl]-L-glutamic acid	
IN-A5760 glucuronide conjugate	2-chloro-5-(β-L-glucopyranuronosyloxy)benzenesulfonamide	
IN-A5760 sulfate conjugate	2-chloro-5-(sulfooxy)benzenesulfonamide	
IN-REG72 glucose conjugate	8-chloro-N-[(2-chloro-5-(β -D-glucopyranosyl) phenyl) sulfonyl]-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide	
IN-RSU03 glucose conjugate	3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-(2R)-(β-D-glucopyranosyloxy)propanoic acid	

Code Names, MW	Chemical Name	Chemical Structure
IN-RSU03 malonyl conjugate	(2R)-[[6-O-(2-carboxyacetyl)-β-D-glucopyranosyl]oxy]-3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]- propanoic acid	
IN-UNS90 glucose conjugate	3-[[(2-chloro-5-hydroxyphenyl)sulfonyl]amino]-(2R)-(β-D-glucopyranosyloxy)-propanoic acid	
IN-UNS90 phenolic glucose conjugate	3-[[[2-chloro-5-(β-D-glucopyranosyloxy)phenyl]sulfonyl]amino]-(2R)-hydroxypropanoic acid	
IN-QEK31 glycerol glucuronide	imidazo[1,2-a]pyridine-2-carboxylic acid, 8-chloro-6-(trifluoromethyl)-, 3-(hexopyranuronosyloxy)-2-hydroxypropyl ester	
Acetylated IN-QZY47 IN-QZY47 acetyl conjugate	N-acetyl-3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-L-alanine	
IN-A5760 glutathione conjugate		

Based on the information on physical and chemical properties, fluazaindolizine is not volatile and is not lipid soluble with a log P_{ow} of around -0.16. Fluazaindolizine is hydrolytically stable at environmental pH. Aqueous photolysis is likely to be a major degradation pathway of fluazaindolizine in the environment.

Metabolism

The Meeting received studies on the metabolism of fluazaindolizine in plants, laboratory animals as well as lactating goats and laying hens. Metabolism studies for the metabolites IN-QEK31, IN-QZY47 and IN-

TMQ01 in lactating goat and IN-QEK31 in laying hen were also made available to the Meeting. The studies on laboratory animals were evaluated by the WHO Core Assessment Group.

Plant metabolism

Fluazaindolizine is typically applied to the soil pre- or at planting with additional soil directed applications during crop growth.

Plant metabolism studies with ^{14}C-fluazaindolizine following its use as a soil application were conducted on crops representative of fruiting vegetables (tomato), root and tuber vegetables (carrot, potato), cereals/grasses (sugarcane) and oilseeds (soya beans). Fluazaindolizine was applied either as [phenyl-^{14}C(U)]fluazaindolizine or [imidazo[1,2-a]pyridine-5,8a-^{14}C]fluazaindolizine. In all experiments, selected extracts were hydrolysed with acid or enzymes to release exocons from their conjugates.

Where conducted in the metabolism studies, chiral analysis confirmed compounds measured in the studies using the racemic standard IN-UNS90 were always present in the R-forms, designated IN-TQD54 IN-TMQ01 respectively. It is assumed these compounds are always present in their R-enantiomeric forms.

The pattern of metabolites formed in primary and rotated crops is similar with hydrolysis of fluazaindolizine at the amide bond to form IN-F4106 and IN-QEK31 and subsequent formation of conjugates, typically with sugars. The demethylated form of fluazaindolizine is also hydrolyzed at the amide bond, forming IN-A5760 and IN-QEK31 which may undergo conjugation.

In the following descriptions of the metabolism studies, the components are grouped into those "related to" various compounds. Here "related to" for IN-REG72, IN-A5760, IN-F4106, IN-UJV12, IN-TQD54, IN-TMQ01 and IN-QZY47 includes conjugates while for IN-QEK31 it includes conjugates as well as IN-R2W56 and IN-RYC33.

Soil treatments

Tomato

The metabolic fate of ^{14}C-fluazaindolizine in tomato plants maintained in a greenhouse was examined following planting seedlings into soil treated by soil drench with a SC-formulation at 1.5 kg ai/ha with a subsequent soil directed application at 0.5 kg ai/ha 30 days later. Samples of foliage and fruit were collected 30 to 62 days after the second application.

Total radioactive residues (TRR) were higher in leaves (0.44–5.7 mg eq/kg) than in fruit (0.029–0.079 mg eq/kg).

The extractability of ^{14}C with methanol:water (7:3) was good at 92–98 percent TRR for fruit and 80–94 percent TRR for foliage.

Parent fluazaindolizine was only detected at low levels in tomato fruit (≤ 0.9 percent TRR). The major components of ^{14}C identified in fruit from the [Ph-^{14}C]-experiment were those related to IN-A5760 (37–50 percent TRR), those related to IN-RSU03 (19–24 percent TRR), and those related to IN-TQD54 (2.5–13 percent TRR). The major components of ^{14}C identified in fruit from the [IP-5,8a-^{14}C]-experiment were those related to IN-QEK31 (53–72 percent TRR). Other components observed with either label were IN-REG72 (0.7–0.8 percent TRR), and IN-F4106 (3–8 percent TRR).

In foliage, fluazaindolizine parent accounted for ≤ 3.7 percent TRR. The major components of ^{14}C identified in foliage from the [Ph-^{14}C]-experiment were those related to IN-RSU03 (51–57 percent TRR), while in the [IP-5,8a-^{14}C]-experiment the major components were those related to IN-QEK31 (34–50

percent TRR). Other components observed with either label were those related to IN-A5760 (10–14 percent TRR), those related to IN-QZY47 (9–10 percent TRR), those related to IN-TQD54 (8–9 percent TRR), IN-REG72 (0.9–3 percent TRR), and IN-F4106 (4–6 percent TRR).

Soya beans

Soya bean seeds were sown into soil treated with a drench application of an SC formulation of ^{14}C-fluazaindolizine at 1 kg ai/ha. Forage was collected 48 days after sowing, fodder (hay) at 75 days and grain at maturity at 112 days.

Residue levels of ^{14}C were highest in hay (0.66 mg eq/kg) and forage (0.44 mg eq/kg) and lowest in seeds (0.27 mg eq/kg) for the [Ph-^{14}C]-experiment and highest in seeds (2.0 mg eq/kg) and lowest in hay (1.0 mg eq/kg) and forage (0.76 mg eq/kg) for the [IP-5,8a-^{14}C]-experiment.

The extractability of ^{14}C with methanol/water was good for forage (> 90 percent TRR), hay (> 87 percent TRR) and seeds (> 78 percent TRR). A further 7.0–22 percent TRR was released with additional treatments.

In forage from the [Ph-^{14}C]-experiment, fluazaindolizine accounted for 7.2 percent TRR in extracts (7.0 percent TRR in methanol:water; 0.2 percent TRR further treatments). The principal component of the ^{14}C were related to IN-QZY47 (64.2 percent TRR). Other identified metabolites included IN-F4106 (11 percent TRR) with IN-REG72 (0.7 percent TRR) and IN-RSU03 (IN-TMQ01, 1 percent TRR) at low-levels. A polar metabolite accounting for 4.2 percent TRR was tentatively assigned as a conjugate of IN-UJV12. Multiple unidentified metabolites were also detected accounting for an aggregate total of 17.6 percent TRR but each individually ≤ 2.6 percent TRR, ≤ 0.011 mg eq/kg.

In forage from the [IP-5,8a-^{14}C]-experiment, fluazaindolizine accounted for 6.9 percent TRR (0.053 mg/kg). The major component of ^{14}C was compounds related to IN-QEK31 (75 percent TRR). Other identified metabolites included IN-REG72 (1.4 percent TRR). Multiple unidentified metabolites were also detected; all ≤ 2.0 percent TRR, ≤ 0.015 mg eq/kg. A minor volatile metabolite tentatively identified as IN-VM862 accounted for 0.6 percent TRR.

In hay from the [Ph-^{14}C]-experiment, fluazaindolizine accounted for 6.1 percent TRR. The principal residue was those related to IN-QZY47 (64.8 percentT RR) those related to IN-F4106 (18 percent TRR), IN-REG72 (1.2 percent TRR), and IN-RSU03 (IN-TMQ01 1.2 percent TRR). A polar metabolite was observed accounting for 2.4 percent TRR and was tentatively assigned as a conjugate of IN-UJV12. Multiple unidentified metabolites were also detected; all ≤ 3.4 percent TRR, ≤ 0.022 mg eq/kg.

In hay from the [IP-5,8a-^{14}C]-experiment, fluazaindolizine accounted for 4.8 percent TRR. The major residue component was compounds related to IN-QEK31 (69.6 percent TRR). IN-REG72 (0.9 percent TRR) was identified at low levels.

The principal residue identified in seed in the [Ph-^{14}C]-experiment was parent fluazaindolizine (47 percent TRR) with compounds related to IN-F4106 (54 percent TRR), those related to IN-QZY47 (15 percent TRR), those related to IN-A5760 (6 percent TRR) with IN-REG72 (9.4 percent TRR). Multiple unidentified metabolites were detected; none >1.3 percent TRR.

In seed from the [IP-5,8a-^{14}C]-experiment, parent fluazaindolizine accounted for 8.3 percent TRR (0.167 mg/kg). Compounds related to IN-QEK31 accounting for 95 percent TRR. IN-REG72 was present at 1.2 percent TRR.

Carrot

Carrot seeds were sown into treated sandy loam soil about one hour after a soil drench application with an SC formulation of [^{14}C]-fluazaindolizine at a nominal application rate of 1.5 kg ai/ha. A further application was 30 days later at a nominal application rate of 0.5 kg ai/ha. Samples of foliage (30DAA1; BBCH 42), immature root and foliage sample (43DAA2, BBCH 45) and root and foliage sample at crop maturity (63DAA2, BBCH 49) were collected.

Extractability of ^{14}C using methanol:water was good for roots (86–93 percent TRR) and for foliage (67–87 percent TRR). Additional extraction using acetonitrile:water at 50°C with sonication, released a further 4.2–7.3 percent TRR while sequential treatment with driselase (a cell wall degrading enzyme), 0.1M HCl, 1M HCl and 0.1M NaOH released a further 5.2–10.4 percent TRR.

In foliage from the [Ph-^{14}C]-experiment, fluazaindolizine accounted for 20.9 percent TRR, (0.926 mg/kg) decreasing as days after application increased. The principal components were compounds related to IN-RSU03 (32–68 percent TRR), those related to IN-QZY47 (6.5–16 percent TRR). Other identified components were those related to IN-TQD54 (1.4–3.1 percent TRR), IN-F4106 (0.3–4.2 percent TRR) and IN-REG72 (0.4–1.3 percent TRR).

In foliage from the [IP-5,8a-^{14}C]-experiment, fluazaindolizine accounted for 40.6 percent TRR at 30DAA1 decreasing to 13.4 percent TRR in the mature foliage. The components identified were those related to IN-QEK31 (21–45 percent TRR). Other identified metabolites were free and conjugated IN-REG72 (2.9–5.4 percent TRR), and IN-VM862 (0.3 percent TRR).

In roots from the [Ph-^{14}C]-experiment, fluazaindolizine accounted for 1.7–8.4 percent. The principal residues were those related to IN-RSU03 (51–63 percent TRR) and those related to IN-QZY47 (20–27 percent TRR). Other identified metabolites were those related to IN-TQD54 (3.6–3.7 percent TRR). Multiple unidentified metabolites were also detected accounting for an aggregate total of 2.4–4.9 percent TRR in each sample but each individually was ≤ 2.6 percent TRR (≤ 0.003 mg eq/kg).

The major residue in the [IP-5,8a-^{14}C] experiment carrot roots was related to IN-QEK31 (62–68 percent TRR). Fluazaindolizine accounted for 12–13 percent TRR. A volatile metabolite was also detected in the mature carrot root extracts and accounted for 3.8 percent TRR. Multiple unidentified metabolites were detected accounting for an aggregate total of 1.4–6.1 percent TRR but each individually was ≤ 3.4 percent TRR (≤ 0.002 mg eq/kg).

Potato

Seed potatoes were sown into treated sandy loam soil about 2 hours prior to a soil drench application with an SC formulation of [Ph-^{14}C]- or [IP-5,8a-^{14}C]-fluazaindolizine, at a nominal application rate of 1.0 kg ai/ha and maintained in a glasshouse. A further application at a nominal rate of 1.0 kg ai/ha was made to the same plots 30 days after the initial application. Foliage samples were collected 15DAA2 and tuber and foliage 35DAA2 (immature) and 70DAA2 (mature).

Extractability of ^{14}C in tubers using methanol:water was good (> 75 percent TRR). Further treatments released an additional 11–15 percent TRR. Extractability of ^{14}C in foliage using methanol:water was also good (> 73 percent percent TRR).

In tubers from the [Ph-^{14}C]-experiment, fluazaindolizine accounted for 6.8 percent TRR, (0.006 mg/kg) in early tubers but was not detected in mature tubers. The principal residue was compounds related to IN-QZY47 (21–23 percent TRR), those related to IN-TQD54 (10–20 percent TRR), and those related to IN-RSU03 (8–12 percent TRR). Other identified metabolites were those related to IN-

F4106 (6–6.6 percent TRR), those related to IN-A5760 (3.4–3.9 percent TRR), IN-REG72 (0.4 percent TRR) and those related to IN-UJV12 (1.4–6.3 percent TRR).

In tubers from the [IP-5,8a-^{14}C]-experiment, fluazaindolizine was accounted for 9.3 percent TRR, (0.004 mg/kg) in early tubers and was not detected in mature tubers. The major residue was compounds related to IN-QEK31 (64–69 percent TRR). Multiple unidentified metabolites were also detected accounting for an aggregate total of 3.6–8.2 percent TRR in each sample but each individually was ≤ 1.7 percent TRR (≤ 0.001 mg eq/kg).

In foliage from the [Ph-^{14}C]-experiment, fluazaindolizine was present in low quantities (0.2 percent TRR; 0.010 mg/kg). The principal residues were compounds related to IN-RSU03 (12 percent TRR), those related to IN-QZY47 (16 percent TRR), those related to IN-TQD54 (14 percent TRR), and those related to IN-A5760 (12 percent TRR). Other identified metabolites were IN-F4106 (4.2 percent TRR), and those related to UJV12 (5.2 percent TRR).

In foliage from the [IP-5,8a-^{14}C]-experiment, fluazaindolizine accounted for 1.9 percent TRR, (0.015 mg/kg) with IN-REG72 (0.7 percent TRR). The principal residue was compounds related to IN-QEK31 (27–50 percent TRR). Multiple unidentified metabolites were also detected accounting for an aggregate total of 37.7 percent TRR but each individually was ≤ 3.5 percent TRR (≤ 0.027 mg eq/kg).

Sugarcane

Mature sugar cane sets (cv. NC0310) at the 2–3 leaf stage (BBCH 12) were transplanted into soil and within 2 hours of transplant, the soil was treated with [^{14}C]-fluazaindolizine applied as soil drench of an SC formulation at a nominal rate of 1.0 kg ai/ha. Samples were taken at BBCH 32, 51 days after application and whole plants above soil level at maturity (BBCH 39) which was 231 days after application.

The extractability of ^{14}C with methanol/water was good for foliage (> 69 percent TRR) and mature cane (> 80 percent TRR).

The major residue identified in [Ph-^{14}C] experiment sugarcane foliage was compounds related to IN-RSU03 (IN-TMQ01, 44–54 percent TRR), those related to IN-TQD54 (22–27 percent TRR) and those related to IN-A5760 (3.8–7.4 percent TRR). Multiple unidentified metabolites were also detected accounting for an aggregate total of 6.6–7.1 percent TRR in each sample but individually none > 2.3 percent TRR (0.004 mg eq/kg).

The principal residue in [IP-5-8a-^{14}C] experiment sugarcane foliage was compounds related to IN-QEK31 (28–46 percent TRR). IN-REG72 and its glucose conjugate were also detected at low levels (≤ 4.6 percent TRR, ≤ 0.004 mg eq/kg). Multiple unidentified metabolites were detected accounting for an aggregate total of 25.5–36.7 percent TRR in each sample but individually none > 6.2 percent TRR (0.007 mg eq/kg).

The principal residue identified in [Ph-^{14}C] mature sugarcane was compounds related to IN-RSU03 (26 percent TRR), those related to IN-A5760 (23 percent TRR), those related to IN-TQD54 (12 percent TRR), those related to IN-QZY47 (4.9 percent TRR) and those related to IN-UJV12 (8.6 percent TRR). Multiple unidentified metabolites were also detected accounting for an aggregate total of 19.0 percent TRR (< 0.005 mg eq/kg) but individually none > 7.0 percent TRR (0.001 mg eq/kg).

The principal residue identified in [IP-5-8a-^{14}C] mature sugarcane was compounds related to IN-QEK31 (65 percent TRR). The glucose conjugate of IN-REG72 was also detected at low levels (3.9 percent TRR). Multiple unidentified metabolites were also detected accounting for an aggregate total of 19.5 percent TRR but individually none > 3.1 percent TRR (0.002 mg eq/kg).

Rotational crop metabolism

The residue profile in follow crops grown in soil treated with fluazaindolizine is expected to be similar to that of primary crops as in both, plants are exposed following application to the soil.

Confined rotational crop studies

In the confined rotational crop study conducted in a glasshouse with spring wheat, spinach and radish, bare sandy loam soil was treated with ^{14}C-fluazaindolizine at the equivalent of ≈1.95 kg ai/ha (0.9× maximum seasonal rate). Fluazaindolizine was applied either as [phenyl-^{14}C(U)]fluazaindolizine or [imidazo[1,2-a]pyridine-5,8a-^{14}C]fluazaindolizine. Crops were sown 30, 120 and 300 days after soil application.

Radioactivity was >0.01 mg eq/kg in all rotated crops and at all plant-back intervals (PBIs).

Fluazaindolizine, IN-F4106, and IN-QEK31 were the major residues extracted from soil.

In general, ^{14}C residues in rotated crops were lower with longer PBIs apart from wheat commodities from the [IP-5,8a-^{14}C]-experiment soil application, where TRR levels in the various commodities at the 30- and 300-day PBIs were comparable.

The majority of residues were readily extracted across all commodities using a methanol:water mixture (70:30). In cases where residues were more extensively incorporated into the crop matrix, such as wheat straw or grain samples, additional enzymatic and acid treatments allowed for recovery of > 90 percent TRR.

Spinach

Extractability of ^{14}C in spinach using methanol:water was good (>81 percent TRR).

In the [Ph-^{14}C]-experiment, fluazaindolizine was found at all PBIs (0.4–14.1 percent TRR, ≤ 0.091 mg/kg) decreasing in later samples. The principal residue was compounds related to IN-QZY47 (42–76 percent TRR). Other identified metabolites were IN-REG72 (free and conjugated, 0.3–5.3 percent TRR), those related to IN-RSU03 (1.8–9.2 percent TRR), those related to IN-TQD54 (0.5–8.2 percent TRR), those related to IN-A5760 (1.1–7.1 percent TRR), those related to IN-UJV12 (0.5–3.1 percent TRR), and those related to IN-F4106 (0.5–21 percent TRR). Multiple unidentified metabolites were also detected accounting for an aggregate total of 6.6–14.2 percent TRR in each sample but each individually was ≤ 2.8 percent TRR (≤ 0.013 mg eq/kg).

In the [IP-5,8a-^{14}C] experiment, fluazaindolizine was identified in all spinach samples (2.9–29.0 percent TRR, ≤ 0.114 mg/kg) decreasing in concentration in later samples. The principal residue was compounds related to IN-QEK31 (48–72 percent TRR). Other identified metabolites were the IN-REG72 (free and conjugated, 1.4–11.6 percent TRR). Multiple unidentified metabolites accounted for an aggregate of 10.7–29.4 percent TRR in each sample but each individually was ≤ 5.3 percent TRR (≤ 0.011 mg eq/kg).

Radish

Extractability of ^{14}C in radish foliage using methanol:water was good (> 84 percent TRR).

In the [Ph-^{14}C] experiment, fluazaindolizine was identified in radish foliage at all PBIs (0.2–11.8 percent TRR, ≤ 0.039 mg/kg) decreasing in later samples. The principal residue was compounds related to IN-QZY47 (31–37 percent TRR), those related to IN-RSU03 (14–36 percent TRR), those related to IN-TQD54 (12–22 percent TRR), those related to IN-A5760 (5.2–6.2 percent TRR), those related to IN-UJV12

(0.8–1.6 percent TRR), those related to IN-F4106 (1.2–20 percent TRR), and IN-REG72 (free and conjugated 0.2–2.9 percent TRR).

In the [IP-5,8a-^{14}C] experiment, fluazaindolizine was found in all samples (1.0–6.8 percent TRR, ≤ 0.036 mg/kg) decreasing in concentrations at later PBIs. The major residue identified was compounds related to IN-QEK31 (65–75 percent TRR) together with IN-REG72 (free and conjugated ≤ 2.7 percent TRR).

In the [Ph-^{14}C]-experiment, fluazaindolizine was identified in radish roots at all PBIs (1.7–12.2 percent TRR, ≤ 0.047 mg/kg) decreasing in later samples. The principal residue identified was compounds related to IN-QZY47 (37–40 percent TRR), those related to IN-RSU03 (20–37 percent TRR), those related to IN-TQD54, (6.1–7.6 percent TRR), those related to IN-A5760 (1.1–6.6 percent TRR), those related to IN-F4106 (1.6–16.3 percent TRR), those related to In-UJV12 (2.5 percent TRR), and IN-REG72 (free and conjugated, 0.6–5.9 percent TRR).

In the [IP-5,8a-^{14}C]-experiment, fluazaindolizine was found in all samples (6.3–17.3 percent TRR, ≤ 0.048 mg/kg) decreasing in concentration in later samples. The principal residue identified was compounds related to IN-QEK31 (36–89 percent TRR). Other identified metabolites were IN-REG72 (free and conjugated 7.0–9.3 percent TRR).

Wheat

The highest levels of total radioactivity were found in straw from wheat grown in the [Ph-^{14}C]-fluazaindolizine treated soil. Radioactive residues in grain were considerably higher in wheat grown in soil treated with [IP-5,8a-^{14}C]-fluazaindolizine as compared to the [Ph-^{14}C]-experiment, indicating cleavage of fluazaindolizine molecule.

Extractability of ^{14}C in forage using methanol:water was good (> 83 percent TRR).

In the [Ph-^{14}C]-experiment, fluazaindolizine was only identified in wheat forage in small quantities at the 30DAA PBI (0.7 percent TRR, 0.009 mg/kg); it was not found in later PBIs. The principal residue identified in forage were compounds related to IN-TQD54 (53–67 percent TRR), those related to IN-UN-RSU03 (11–16 percent TRR), those related to IN-QZY47 (4.1–4.7 percent TRR), those related to IN-A5760 (3.3–4.1 percent TRR), and those related to IN-F4106 (1.3–2.2 percent TRR). Other identified metabolites were IN-REG72 (free and conjugated 1.3–1.7 percent TRR).

In the [IP-5,8a-^{14}C]-experiment, fluazaindolizine was identified in forage at all PBIs (0.6–3.9 percent TRR, ≤ 0.016 mg/kg) decreasing in later samples. The major residue identified in forage was compounds related to IN-QEK31 (43–76 percent TRR), IN-REG72 (free and conjugated ≤ 2.8 percent TRR).

Extractability of ^{14}C in hay using methanol:water was good (> 76 percent TRR).

In the [Ph-^{14}C]-experiment, fluazaindolizine was only identified in small quantities in wheat hay at the 30DAA PBI (0.5 percent TRR, 0.008 mg/kg) and not in later samples. The major residue was related to IN-TQD54 (46–54 percent TRR), those related to IN-RSU03 (11–17 percent TRR), those related to IN-A5760 (3.9–5.4 percent TRR), those related to IN-QZY47 (1.5–2.3 percent TRR), and those related to IN-F4106 (2.2–2.7 percent TRR). Other identified metabolites were IN-REG72 (free and conjugated 1.4–2.7 percent TRR). Chiral HPLC analysis was conducted on the isolated IN-RSU03 and it was demonstrated that only the *R*-enantiomer, IN-TMQ01, was present.

In the [IP-5,8a-^{14}C]-experiment, fluazaindolizine was identified only in the 30 and 120DAA samples at low concentrations (0.7–1.4 percent TRR, ≤ 0.016 mg/kg). The principal residues identified were compounds related to IN-QEK31 (32–71 percent TRR), with IN-REG72 (free and conjugated ≤ 8.0 percent TRR, ≤ 0.091 mg/kg).

Extractability of ^{14}C in straw using methanol:water was good for the [Ph-^{14}C]-experiment (> 72 percent TRR) and poor for the [IP-5,8a-^{14}C] experiment (< 65 percent TRR). Further treatments of [IP-5,8a-^{14}C]-experiment straw samples released an additional 27.7–38.9 percent TRR with terminal residues remaining in solids accounting for ≤ 8.3 percent TRR.

In the [Ph-^{14}C]-experiment, fluazaindolizine was only identified in wheat straw in small quantities at the 30-day PBI (0.7 percent TRR, 0.041 mg/kg) and was not found in later PBIs. The principal residue identified in [Ph-^{14}C] straw was compounds related to IN-TQD54 (37–43 percent TRR), those related to IN-RSU03 (15–17 percent TRR), those related to IN-A5760 (3.8–7.3 percent TRR), and those related to IN-F4106 (2.7–3.3 percent TRR). Other identified metabolites were IN-REG72 (free and conjugated 0.9–3.2 percent TRR) and IN-UJV12 (< 0.1–0.5 percent TRR).

In the [IP-5,8a-^{14}C]-experiment, fluazaindolizine was found in straw only in the 30 and 120-day PBIs (1.4–4.3 percent TRR, ≤ 0.153 mg/kg). The principal residue was compounds related to IN-QEK31 (26–40 percent TRR) with IN-REG72 (free and conjugated 0.2–7.2 percent TRR).

Extractability of ^{14}C in grain using methanol:water was poor for the [Ph-^{14}C]- (16–34 percent TRR) and [IP-5,8a-^{14}C]-experiments (54–63 percent TRR). Selected samples were subjected to further treatments which released an additional 63–84 percent TRR for the [Ph-^{14}C]- 300 DAA grain sample with terminal residues remaining in solids accounting for 2.8 percent TRR and an additional 36–46 percent TRR released for the [IP-5,8a-^{14}C] sample with terminal residues remaining in solids accounting for 0.7 percent TRR.

In the [Ph-^{14}C]-experiment, it was not possible to obtain accurate profiles from the 120 and 300-day PBI samples due to the large quantity of endogenous materials and large volumes of sample extract required to release the ^{14}C residue. The profiles obtained demonstrated that the residue was comprised of multiple metabolites although it was not possible to accurately determine their identity. The majority of the ^{14}C residues were released by enzyme and/or acidic extractions which may alter the nature of the residue in the extraction process.

In the [Ph-^{14}C]-experiment, fluazaindolizine was found in small quantities at the 30-day PBI (3.3 percent TRR, 0.003 mg/kg). The major residue identified in [Ph-^{14}C] grain was IN-A5760 (3.5–6.1 percent TRR, 0.002–0.005 mg eq/kg). Other identified metabolites were compounds related to IN-TQD54 (4.4 percent TRR), those related to IN-RSU03 (0.9–8.4 percent TRR), and IN-F4106 (≤ 3.3 percent TRR) and those related to IN-QZY47 (≤ 2.3 percent TRR). IN-REG72 was tentatively observed in the 300DAA grain at 7.9 percent TRR.

In the [IP-5,8a-^{14}C]-experiment, fluazaindolizine was not found in the grain samples. The principal residue identified in grain was compounds related to IN-QEK31 (69–76 percent TRR) with a glucose conjugate of IN-REG72 (≤ 2.6 percent TRR, ≤ 0.040 mg eq/kg).

In summary, the metabolism of fluazaindolizine and various metabolites taken up from the soil (such as major metabolites IN-F4106 and IN-QEK31) was consistent across all crops with differences mainly in the degree and complexity of conjugation that occurs in the various crops. The major metabolic route in primary and rotated crops was the hydrolysis of fluazaindolizine at the amide bond, resulting in IN-F4106 and IN-QEK31. Fluazaindolizine was also *O*-demethylated to form IN-REG72, which also was hydrolyzed to IN-A5760 and IN-QEK31. A less prominent pathway was hydrolysis of fluazaindolizine at the sulfonamide bond, resulting in IN-RYC33 or the further degradation of IN-QEK31 to IN-VM862. Several conjugates were also formed, typically with various sugars.

Table 190 Residue profiles for fluazaindolizine and metabolites in different plant matrices

	Percent TRR								
Component	Fluaza-indolizine	IN-REG72	IN-F4106	IN-A5760	IN-QEK31	IN-QZY47	IN-TQD54	IN-RSU03	IN-UJV12
Tomato fruit	< 0.9	< 0.8	3-8	35-50	53-72		2.5-13	19-24	
Tomato foliage	<3.7	0.9-3	4-6	10-14	34-50	9-10	8-9	51-57	
Soya forage	7.2	0.7-1.4	11		75	64		1	
Soya hay	6.1	0.9-1.2	18		70	65		1.2	2.4
Soya seed	8-47	1-9	54	6	95	15			
Carrot tops	21-41	0.4-5	0.3-4		21-45	6-16	1-3	32-68	
Carrot root	1.7-13				62-68	20-27	4	51-63	
Potato tuber	0-9	0.4	6-7	3-4	64-69	21-23	10-20	8-12	1-6
Potato foliage	0.2-1.9	0.7	4	12	27-50	16	14	12	5
Sugarcane foliage		4.6		4-7	28-46		22-27	44-54	
Sugarcane		3.9		23	65	5	12	26	9
Spinach	0.4-29	0.3-12	0.5-21	1-7	48-72	42-76	0.5-8	2-9	0.5-3
Radish foliage	0.2-12	0.2-3	1-20	5-6	65-75	31-37	12-22	14-36	1-2
Radish root	2-17	0.6-9	2-16	1-7	36-89	37-40	6-8	20-37	2
Wheat forage	0.6-4	1.3-2.8	1-2	3-4	43-76	4-5	53-67	11-16	
Wheat hay	0.5-1.4	1.4-8	2-3	4-5	32-71	2	46-54	11-17	
Wheat straw	0.7-4.3	0.9-7	3	4-7	26-40		37-43	15-17	0.5
Wheat grain	3	8		4-6	69-76	2	4	1-8	

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens dosed with fluazaindolizine and separate studies following dosing with IN-QEK31 (lactating goats, laying hens) and IN-QZY47 and IN-TMQ01 (lactating goats). Lactating goats and laying hens were dosed with [Ph-^{14}C(U)]-fluazaindolizine and [imidazo-(1,2-α)-pyridine-2-^{14}C]-fluazaindolizine.

Rats

Metabolism of fluazaindolizine in rats was evaluated by the WHO Core Assessment Group of the 2021 JMPR. Metabolites identified in rats included: IN-REG72, IN-UHD20, IN-UHD21, IN-F4106, IN-QEK31, IN-A5760 and sulfate or glucuronide conjugates of IN-A5760.

On dosing of laboratory animals with IN-F4106, IN-A5760 and conjugates were identified. Following dosing with IN-QZY47, acetylated IN-QZY47 was the predominant metabolite and following dosing with IN-TMQ01, IN-F4106 and IN-TQD54 were identified.

Lactating goats

Lactating goats were orally dosed by gavage once daily for seven consecutive days with ^{14}C-fluazaindolizine at doses equivalent to 12 ppm in the diet and sacrificed within 6 hours of the last dose. Milk samples were collected daily.

By 6 hours after the last dose, the majority of the ^{14}C was recovered in faeces (51–52 percent of the administered dose (AD)) and urine (21–33 percent AD). Milk accounted for < 0.1 to 0.1 percent AD while tissues accounted for 0.8–0.9 percent AD. The material balance was 97–108 percent AD.

Residues in milk reached a plateau by 5 days of dosing.

TRR levels in edible tissues from the [Ph-^{14}C]/[IP-2-^{14}C]-fluazaindolizine dosed goats were 0.223/0.275 mg eq/kg in liver, 0.358/0.357 mg eq/kg in kidney, 0.011/0.010 mg eq/kg in muscle, 0.015/0.008 mg eq/kg in omental fat, 0.028/0.014 mg eq/kg in renal fat and 0.024/0.013 mg eq/kg in subcutaneous fat. Radioactive residues did not selectively partition into skim milk or cream or into the various fat types.

Extractability from tissues with acetonitrile:0.1 M ammonium formate, pH 7 (9:1) was good for liver, kidney, muscle and fat (> 85 percent TRR) and Day 4–6 milk (> 96.9 percent TRR). Digestion of liver PES with protease released additional residues, with overall ≥ 96 percent TRR extracted.

In the [Ph-^{14}C] experiment, fluazaindolizine accounted for 17.5–84.6 percent TRR in tissues and milk. The phenyl-derived metabolites included IN-A5760 (maximum 4.1 percent TRR), IN-F4106 (maximum 38.4 percent TRR) and IN-REG72 (maximum 7.0 percent TRR). Several minor unidentified metabolites were also detected, none of which individually were greater than 7.3 percent TRR, which combined accounted for 5.5–12.8 percent TRR in milk and tissues.

In the [IP-2-^{14}C] experiment, fluazaindolizine accounted for 25.0–83.2 percent TRR in tissues and milk. The imidazopyridine-derived metabolites included IN-QEK31 (maximum 42.8 percent TRR), IN-REG72 (maximum 11.6 percent TRR) and IN-R2W56 (maximum 0.6 percent TRR). Several minor unidentified metabolites were also detected, none of which individually were greater than 4.0 percent TRR, which combined accounted for 4.0–8.3 percent TRR in milk and tissues.

Laying hens

The metabolism of ^{14}C-fluazaindolizine was studied in laying hens. Hens were dosed orally via capsules, once a day for a total of 14 days, with ^{14}C-fluazaindolizine at doses equivalent to 13 ppm in the diet. Hens were sacrificed 6 hours after the final dose.

The recovery of the administered dose was 94.6 percent for [Ph-^{14}C]-fluazaindolizine and 93.5 percent for the [IP-2-^{14}C]-fluazaindolizine. For both [^{14}C]-labels, 92.9–94 percent of the dose was recovered from the excreta and cage wash. For both [^{14}C]-labels, < 0.1 percent of the dose was recovered in eggs, muscle and fat with 0.6 percent of the dose recovered in liver.

Total radioactive residue levels in edible tissues were 0.732 mg eq/kg in liver, 0.043 in muscle and 0.020 mg eq/kg in abdominal fat from the [Ph-^{14}C]-fluazaindolizine dosed hens, and 0.701 mg eq/kg in liver, 0.047 in muscle and 0.027 mg eq/kg in abdominal fat from the [IP-2-^{14}C]-fluazaindolizine dosed hens. Radioactivity plateaued in whole eggs within 10 days from the start of dosing at *ca.* 0.017 mg eq/kg in the [Ph-^{14}C]-fluazaindolizine and within 8 days at *ca.* 0.018 mg eq/kg in the [IP-2-^{14}C]-fluazaindolizine.

Extractability of eggs, liver, muscle, and fat with acetonitrile/buffer extractions > 87.3 percent TRR.

Most of the radioactive residues (82.4–98.0 percent TRR) was identified and/or characterised in liver and eggs from the [Ph-^{14}C]-fluazaindolizine dosed hens. Fluazaindolizine was a major residue, accounting for 67.5–96.5 percent TRR in liver and eggs at levels of 0.013 mg/kg in eggs, 0.680 mg/kg in liver, 0.041 mg/kg in muscle and 0.014 mg/kg in fat. IN-F4106 (maximum 5.7 percent TRR) and IN-REG72 (maximum 1.1 percent TRR) were also detected. Several minor unidentified metabolites were also detected, none of which individually were greater than 11.3 percent TRR, which combined accounted for 0.9–11.3 percent TRR in eggs and tissues.

The majority of radioactive residues (73.1–97.9 percent TRR) was identified and/or characterised in tissues and eggs from the [IP-2-^{14}C]-fluazaindolizine dosed hens. Fluazaindolizine was a major residue, accounting for 66.2–97.1 percent TRR, levels of 0.012 mg/kg in eggs, 0.639 mg/kg in liver, 0.046 mg/kg in muscle and 0.018 mg/kg in fat. In eggs and tissues, IN-QEK31 (maximum 4.9 percent TRR), IN-REG72 (maximum 1.1 percent TRR), IN-RYC33 (maximum 11.0 percent TRR) and IN-R2W56 (maximum 1.9 percent TRR) were detected. An unidentified metabolite was detected in abdominal fat which accounted for 2.5 percent TRR (0.001 mg/kg).

In summary, the major residue in tissues, milk and eggs following dosing with fluazaindolizine is the parent compound.

Metabolism of IN-QEK31

A lactating goat received repeated oral administration of [IP-2-^{14}C]-IN-QEK31 by gelatine capsule once daily on five consecutive days at a dose level equivalent to 12.5 ppm in the feed. Animals were euthanized approximately 6 hours after the last dose.

The majority of the ^{14}C was recovered in urine (57 percent AD) and faeces (14 percent AD). Milk accounted for 2.1 percent AD while tissues for < 0.1 percent AD. Residues in milk reached a plateau by 1 to 4 days of dosing.

TRR in edible tissues were 0.035 mg eq/kg in liver, 0.282 in kidney, < 0.001 in muscle, 0.005 in omental fat, 0.046 in renal fat and 0.002 mg eq/kg in subcutaneous fat.

Extractability of milk and tissues with acetonitrile:(100 mM ammonium formate) (9:1) was good with > 90 percent TRR released, the exception being liver for which (79.7 percent TRR). Extraction of the bound liver residues with more polar and acidic extraction methods did not release any further radioactivity; however, PES were low (0.007 mg eq/kg).

Unchanged IN-QEK31 accounted for 74.3-95.4 percent TRR in tissues and milk. A single metabolite was present at greater than 10 percent TRR which was identified as IN-R2W56 and detected only in renal fat. Several minor unidentified metabolites were also detected, none of which individually were greater than 5.0 percent TRR, which combined accounted for 6.2 percent TRR in milk and tissues.

Hens were dosed orally *via* capsule with [IP-2-^{14}C]-IN-QEK31 once a day for 14 days, at a dose equivalent to 10 ppm in the diet and sacrificed 6 hours after the final dose-IN-QEK31 at a dose equivalent to 10 ppm in the diet. Hens were sacrificed 6 hours after the final dose.

IN-QEK31 was rapidly eliminated from hens into the excreta (approximately 93.2 percent of the dose). Edible tissues and eggs contained negligible amounts of radioactivity (< 0.1 percent) of the administered total dose. Radioactivity in whole eggs reached plateau within 5 days post first dose (*ca.* 0.005 mg/kg).

TRR were 0.014 mg eq/kg in liver and 0.002 mg eq/kg in abdominal fat and < 0.001 mg eq/kg in muscle. Most of the radioactive residues (≥ 71.1 percent TRR) were extracted with acetonitrile:(100 mM

ammonium formate). Unchanged IN-QEK31 accounted for 71 percent TRR in liver. As TRR in egg, muscle and fat samples were very low (< 0.01 mg eq/kg), metabolite profiling was not conducted.

In summary, the major residue in tissues, milk and eggs following dosing with IN-QEK31 is IN-QEK31.

Metabolism of IN-QZY47

The metabolism of [Phenyl-^{14}C(U)]-IN-QZY47 was investigated in a lactating goat following repeated oral administration by gelatine capsule once daily for five consecutive days at a dose level equivalent to 20 ppm in the feed. Animals were euthanized approximately 6 hours after the last dose

The majority of the ^{14}C was recovered in urine (75 percent AD) and faeces (7.2 percent AD). Milk accounted for 20.2 percent AD while tissues for < 0.1 percent AD. Residues in milk reached a plateau by 1 to 4 days of dosing.

TRR were 0.344 mg eq/kg in liver, 0.824 mg eq/kg in kidney, 0.057 mg eq/kg in muscle, 0.034 mg eq/kg in omental fat, 0.044 mg eq/kg in renal fat and 0.050 mg eq/kg in subcutaneous fat. There was no selective partitioning of residues (less than 2-fold) in the milk fractions.

Extractability of milk and tissues with acetonitrile:(100 mM ammonium formate) (9:1) was good (> 81 percent TRR). The majority of radioactive residues (74.6-98.6 percent TRR) were identified and/or characterised. In milk and tissues, metabolites included IN-F4106 (maximum 81.4 percent TRR), IN-A5760 (maximum 11.4 percent TRR) and IN-A5760 conjugates (maximum 41.0 percent TRR). IN-QZY47 was detected in milk (5.3 percent TRR) but not in tissues. Several minor unidentified metabolites were also detected, none of which individually were greater than 8.3 percent TRR or 0.017 mg eq/kg

Metabolism of IN-TMQ01

The metabolism of [Phenyl-^{14}C(U)]-IN-TMQ01 was investigated in a lactating goat following oral administration by gelatine capsule, once daily for five consecutive days at a dose level equivalent to 11 ppm in the feed. Animals were euthanized approximately 6 hours after the last dose.

Most of the residues was recovered in faeces (44 percent AD) and urine (34 percent AD). Milk and tissues accounted each for < 0.1 percent AD. Residues in milk reached a plateau by 1 to 4 days of dosing.

In milk and tissues containing residues greater than 0.01 mg eq/kg, the radioactive residues (from 66.1 percent for liver to 100.0 percent TRR for muscle and fat) were extracted with acetonitrile: 100 mM ammonium formate. Digestion with protease released the remaining radioactive residues in liver.

The majority of radioactive residues (66.1–100 percent TRR) were identified and/or characterised. Unchanged IN-TMQ01 accounted for 42.7–86.7 percent TRR in tissues. IN-F4106 accounted for 1.0–43.6 percent TRR in tissues and essentially all of the residue in milk (97.5 percent TRR; 0.007 mg eq/kg). Several minor unidentified metabolites were also detected, none of which individually exceeded 4.8 percent TRR, with the exception of muscle and subcutaneous fat in which a single unknown component accounting for 13.7–22.7 percent TRR was detected, however, these unknowns only equated to a low concentration of ≤ 0.001 mg eq/kg.

Environmental fate

The Meeting received aqueous and soil photolysis, aqueous hydrolysis and aerobic soil studies for fluazaindolizine.

Fluazaindolizine and metabolites (IN-F4106, IN-QEK31) are stable to hydrolysis (aqueous and soil) at environmental pHs, however aqueous photolysis is fast and may be a significant route of degradation with DT_{50} values ≤ 1.6 days for fluazaindolizine.

Fluazaindolizine does not undergo significant photolytic degradation on moist soil when exposed to artificial sunlight (DT_{50} 138 days). Soil metabolite IN-QEK31 degrades readily on moist soil surface in the presence of light while IN-F4106 was stable.

In laboratory aerobic soil degradation studies on fluazaindolizine the major soil degradates were IN-F4106, IN-QEK31 and IN-VM862. The laboratory DT_{50} values for fluazaindolizine in different soils were 3.4–241 days (geometric mean 28.1 days). DT_{50} were 177–461 days (geometric mean 216 days) for IN-F4106, 57.2-182 days (geometric mean 89 days) for IN-QEK31, 347-452 days for aerobic soil degradation and 42–57 days overall dissipation including volatilisation for IN-VM862, 28.4–117 days (geometric mean 58 days) for IN-REG72 and 3.7–88.6 days (geometric mean 27 days) for IN-A5760.

In field dissipation studies, the DT_{50} values for fluazaindolizine degradation ranged from 5.0 to 171 days (geometric mean of 28.1 days), indicating non-persistency. Soil metabolites show limited potential to accumulate following application on consecutive years, with the exception of IN-F4106 which may accumulate.

Field rotational crop studies

The persistence in soil and potential uptake of fluazaindolizine and degradates by plants was further evaluated in field studies at locations in Europe and the United States.

Fluazaindolizine (SC formulation) was applied as 1×1.25 kg ai/ha, 2×1.25 kg ai/ha (total 2.5 kg ai/ha), 4×0.82 kg ai/ha applications (total 3.3 kg ai/ha) or 4×1.12 kg ai/ha applications (total 4.48 kg). Three PBIs were targeted at each site, 7–30, 60–270 and 270–365 days.

Fluazaindolizine residues were observed at levels above the LOQ (0.01 mg/kg) in a variety of crops: up to 0.018 mg/kg in leafy vegetables (radish tops), 0.023 mg/kg in broccoli, 0.039 mg/kg in celery, 0.023 mg/kg in root crops (radish roots), immature pea seed 0.18 mg/kg, pulses 1.5 mg/kg (pea seed), rape seed 0.022 mg/kg, forages 0.035 mg/kg (soya bean), and fodders 0.8 mg/kg (pea hay).

Compounds hydrolyzed by acid to IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12, IN-TQD54 were observed in all commodities and generally at levels greater than parent fluazaindolizine with residues up to 2.3 mg/kg (pea hay) for IN-A5760, 1.3 mg/kg (pea ay) for IN-F4106, 1.3 mg/kg (pea mature seed) for IN-QEK31, 9.2 mg/kg (pea hay) for IN-QZY47, 3.7 mg/kg (carrot tops) for IN-TMQ01, 1.4 mg/kg (pea hay) for IN-UJV12 and 0.62 mg/kg (corn stover) for IN-TQD54.

In summary, at the maximum use pattern considered by the Meeting, soil application at up to 2.24 kg ai/ha, residues of fluazaindolizine and metabolites are expected in rotated crops. Residues associated with follow crops will be assessed in the section on supervised trials.

Methods of analysis

The Meeting received information on analytical methods for fluazaindolizine and components of interest in plant and animal matrices.

The methods for plants involve two parts. Part 1 is analysis without hydrolysis (fluazaindolizine and free IN-F4106, IN-QEK31, IN-QZY47, IN-R2W56, IN-REG72, IN-TEQ01, IN-RYC33) and part 2 with hydrolysis of compounds to IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12, IN-TQD54. Hydrolysis of fluazaindolizine and IN-REG72 results in cleavage of the amide bond and formation of IN-F4106 plus IN-QEK31 and IN-A5760 plus IN-QEK31 respectively, as shown in the Figure 32 below.

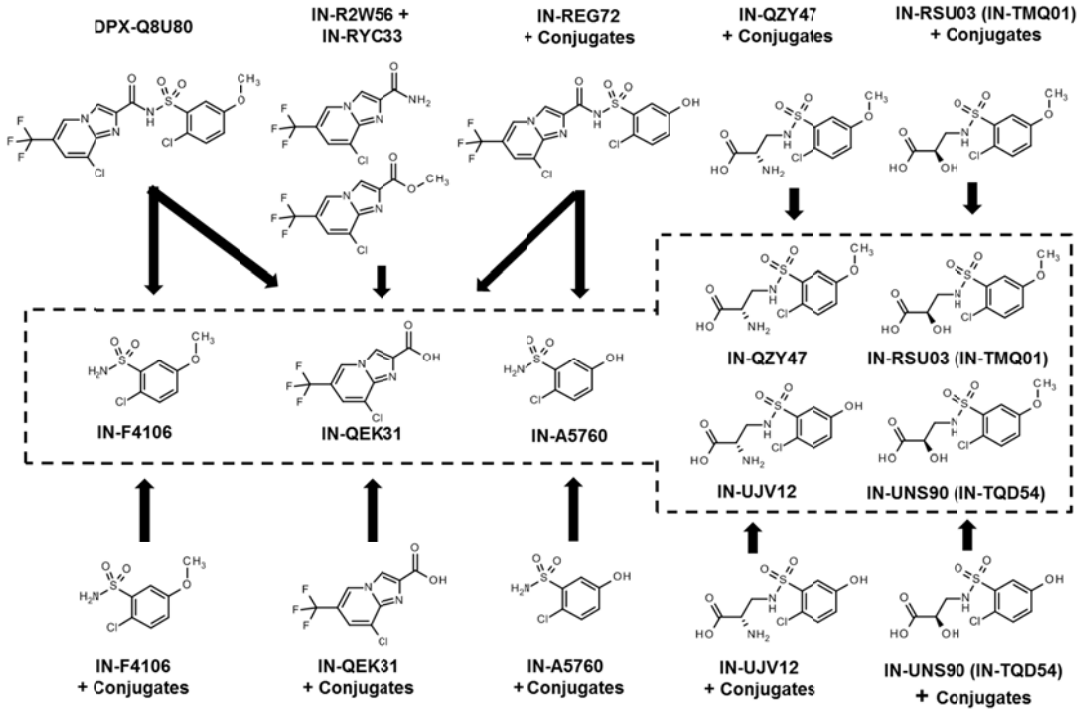


Figure 32 Metabolites of fluazaindolizine determined in the analytical methods

Residues are reported in terms of the analytes but may be converted to parent equivalents using molecular weight conversion factors: 2.26 for IN-A5760, 2.11 for IN-F4106, 1.77 for IN-QEK31, 1.52 for IN-QZY47, 1.51 for IN-TMQ01, 1.59 for IN-UJV12 and 1.58 for IN-TQD54.

In methods for plants, fluazaindolizine and its metabolites (free IN-F4106, IN-QEK31, IN-QZY47, IN-R2W56, IN-REG72, IN-TEQ01, IN-RYC33) are extracted using methanol:water. For oily crops the extract is partitioned against hexane, and the hexane discarded. The final determination is by LC-MS/MS, with validated LOQs typically of 0.01 mg/kg for each compound. This method is used to measure parent fluazaindolizine residues.

To measure compounds hydrolysed to IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12, IN-TQD54, a separate aliquot of the methanol:water extract is hydrolysed with concentrated HCl (1.2-2 M HCl, 80°C overnight), followed by clean-up on a SAX SPE column and analysis by LC-MS/MS. The LOQs for plant commodities are typically 0.01 mg/kg for each compound. In the hydrolysis step, exocons are released from their conjugates, fluazaindolizine is converted to IN-F4106 and IN-QEK31, IN-REG72 to IN-A5760 and IN-QEK31 and IN-RYC33 to IN-QEK31.

In the method for animal commodities residues of fluazaindolizine and metabolites (free IN-A5760, IN-F4106, IN-REG72, IN-QEK31, IN-R2W56 and IN-RYC33) are extracted with acetonitrile:water followed by clean-up on strong cation exchange and strong anion exchange columns and analysis by LC-MS/MS. The validated LOQ is 0.01 mg/kg for tissues and milk for the individual compounds.

The Meeting concluded that the presented methods were sufficiently validated and are suitable to measure fluazaindolizine and metabolites in plant and animal commodities.

The multi-residue method, DFG S19 is not suitable for analysis of fluazaindolizine, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12, and IN-TQD54 in crops, but is considered valid for the determination of residues of IN-F4106, IN-A5760, and IN-RYC33 in tomato, soya bean and grapefruit (but not wheat straw).

The multi-residue method, DFG S19 is not suitable for analysis of fluazaindolizine, IN-REG72 and IN-QEK31 in animal commodities but is considered valid for the determination of residues of IN-F4106, IN-A5760, and IN-RYC33 in milk, egg, bovine meat, fat, and liver.

Stability of pesticide residues in stored analytical samples

The Meeting received information on storage stability of fluazaindolizine and metabolites (IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-R2W56, IN-REG72, IN-RYC33, IN-TMQ01, IN-UJV12, IN-TQD54) in raw/processed plant commodities.

Fluazaindolizine and metabolites are stable on frozen storage in a high-acid commodities (orange at least 24 months), high-water commodities (tomato at least 34 months), high-oil (soya bean seed at least 33 months), and high-starch (wheat grain at least 24 months), high-protein commodities (dry pea seed at least 24 months), dry crop commodities (field corn stover at least 24 months, pea hay at least 23 months).

The demonstrated stability intervals on frozen storage generally encompass the duration of storage in the residue trials evaluated by the Meeting.

Fluazaindolizine and metabolites (IN-A5760, IN-F4106, IN-QEK31, IN-R2W56, IN-REG72, IN-RYC33) are stable on frozen storage in milk for at least 6.8 months, in muscle for at least 6.7 months, in fat for at least 8.5 months and for analytes other than IN-R2W56 in kidney for at least 8.3 months. IN-R2W56 was stable in kidney for 7 days but not in a sample stored for 250 days. Fluazaindolizine, IN-QEK31 and IN-R2W56 were stable in liver for at least 0.77 months (23 days), IN-RYC33 for 0.47 months (14 days) and IN-REG72, IN-F4106 and IN-A5760 for 0.23 months (7 days).

Most of the samples in the livestock feeding study were analysed within 14 days.

Definition of the residue

Plant commodities

The metabolism of fluazaindolizine was similar in the primary treated crops (tomato, carrot, potato, sugarcane, soya bean) and in rotational crops (spinach, radish, wheat).

Parent fluazaindolizine was a minor component of the ^{14}C residue, detected in all crops, often at low levels (tomato fruit ≤ 0.9 percent TRR, tomato foliage ≤ 3.7 percent TRR, soya bean seed ≤ 46 percent TRR, soya bean forage ≤ 7 percent TRR, soya bean hay ≤ 6.1 percent TRR, carrot mature root ≤ 12 percent TRR, carrot foliage ≤ 41 percent TRR, potato tuber ≤ 9 percent TRR, potato foliage ≤ 1.9 percent TRR).

The main components of the ^{14}C residues were free and conjugated IN-RSU03, IN-TQD54, IN-QZY47, IN-A5760, IN-F4106 and IN-QEK31.

In the [Ph-^{14}C]-experiments the sum of IN-RSU03 and its conjugates accounted for; tomato fruit ≤2 4 percent TRR, tomato foliage 49–57 percent TRR, carrot root ≤ 63 percent TRR, carrot foliage ≤ 68 percent TRR, potato tuber/foliage ≤ 12 percent TRR and in sugarcane foliage/cane ≤ 54 percent TRR, the sum of IN-TQD54 and its conjugates accounted for; tomato fruit ≤ 13 percent TRR, potato tuber/foliage ≤ 20 percent TRR, sugarcane foliage/cane ≤ 32 percent TRR, the sum of IN-QZY47 and its conjugates accounted for; tomato foliage ≤ 10 percent TRR, soya bean forage/hay ≤ 62 percent TRR, carrot root ≤ 27 percent TRR, carrot foliage ≤ 16 percent TRR, potato tuber/foliage ≤ 23 percent TRR, the sum of IN-A5760 and its conjugates accounted for; tomato fruit ≤ 50 percent TRR, tomato foliage ≤ 14 percent TRR, potato tuber 12 percent TRR, sugarcane ≤ 23 percent TRR, the sum of IN-F4106 and its conjugates accounted for; soya bean forage/hay ≤ 17.8 percent TRR, soya bean seed 53.5 percent TRR.

In the [IP-5,8a-^{14}C]-experiments the sum of IN-QEK31 and its conjugates accounted for; tomato fruit 53–72 percent TRR, tomato foliage ≤ 50 percent TRR, soya bean forage/hay/seed ≤ 95 percent TRR, carrot root ≤ 66 percent TRR, carrot foliage ≤ 45 percent TRR, potato tubers ≤ 69 percent TRR, potato foliage ≤ 50 percent TRR, sugarcane foliage/cane ≤ 65 percent TRR.

In studies on rotational crops, also reflecting uptake from soil, residues were detected at levels above the LOQ for a number of compounds and residues may occur in rotational (follow) crops. The compounds were fluazaindolizine as well as free and sometimes conjugated forms of the metabolites IN-REG72, IN-RSU03, IN-QZY47, IN-TQD54, INA5760, IN-F4106, IN-UJV12, IN-R3Z85 for the [Ph-^{14}C]-experiments and free and conjugated forms of IN-REG72, IN-RYC33, IN-UJU44, IN-R2W56, IN-QEK31, IN-UGA20 for the [IP-5,8a-^{14}C]-experiments.

Parent fluazaindolizine was detected at low levels or <LOD in rotated crops (wheat forage/hay/straw ≤ 0.7 percent TRR, spinach ≤ 29 percent TRR, radish foliage/roots ≤ 17 percent TRR).

In the [Ph-^{14}C]-experiments the sum of IN-RSU03 and its conjugates accounted for; wheat forage/hay/straw ≤ 17 percent TRR, radish foliage/roots ≤ 37 percent TRR, the sum of IN-TQD54 and its conjugates accounted for; wheat forage/hay/straw ≤ 67 percent TRR, radish foliage ≤ 22 percent TRR, the sum of IN-QZY47 and its conjugates accounted for; spinach ≤ 76 percent TRR, radish foliage/roots ≤ 40 percent TRR, the sum of IN-F4106 and its conjugates accounted for; spinach ≤ 27 percent TRR, radish roots ≤ 16 percent TRR.

In the [IP-5,8a-^{14}C]-experiments the sum of IN-QEK31 and its conjugates accounted for; wheat forage/hay/straw ≤ 71 percent TRR, wheat grain ≤ 76 percent TRR, spinach ≤ 72 percent TRR, radish foliage ≤ 75 percent TRR, radish root ≤ 89 percent TRR. IN-UGA20 was also a significant metabolite (wheat hay ≤17 percent TRR, radish roots ≤ 14 percent TRR).

Supervised field trials monitored fluazaindolizine, as well as compounds hydrolysed with acid to IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-TMQ01, IN-UJV12, and IN-TQD54 with residues of all analytes detected.

As fluazaindolizine occurs in most crops that have detectable residues and provides a pragmatic option as analysis of other compounds involves an intensive hydrolysis step, the Meeting decided the residue definition for compliance with MRLs in plants should be fluazaindolizine.

In deciding which compounds should be included in the residue definition for risk assessment for plant commodities the Meeting considered the likely occurrence of the compounds and the toxicological properties of the candidates. Compounds considered were fluazaindolizine, IN-REG72, IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-RYC33, IN-TMQ01, IN-UJV12, and IN-TQD54 and their conjugates.

Based on toxicological properties the following compounds are assumed to be covered by the fluazaindolizine HBGVs: fluazaindolizine, and free and conjugated forms of IN-REG72, IN-A5760, IN-F4106, IN-QEK31, IN-QZY47, IN-RYC33 and IN-TMQ01.

Each of the compounds is variously the predominant or a significant residue in primary and rotated crops. The Meeting agreed that the residue definition for dietary risk assessment for plant commodities should account for residues of fluazaindolizine, and free and conjugated forms of the following compounds: IN-REG72, IN-QEK31, IN-A5760, IN-F4106, IN-QZY47, IN-RYC33, and IN-TMQ01.

It is noted the analytical method utilising acid hydrolysis converts conjugates to their free form and also hydrolyses fluazaindolizine to IN-QEK31 and IN-F4106, IN-REG72 to IN-QEK31 and IN-A5760 and converts IN-RYC33 to IN-QEK31. To avoid double counting when expressing all residues in terms of fluazaindolizine, the Meeting considered the maximum of the sum of IN-A5760, IN-F4106, IN-QZY47 and

IN-TMQ01 or IN-QEK31 measured after hydrolysis to provide the best measure of the compounds included in the residue definition.

For example, for hydrolysis products containing the imidazopyridine ring

- IN-A5760 when expressed in parent equivalents would account for IN-A5760 and its conjugates as well as IN-REG72 and its conjugates and IN-QEK31,
- IN-F4106 would account for IN-F4106 and its conjugates as well as fluazaindolizine and IN-QEK31,
- IN-QZY47 would account for IN-QZY47 and its conjugates and IN-QEK31 and
- IN-TMQ01 for IN-TMQ01 and its conjugates and IN-QEK31.

For the hydrolysis products containing the phenyl ring,

- IN-QEK31, when expressed in parent equivalents accounts for IN-A5760, IN-F4106, IN-QZY47, IN-TMQ01, fluazaindolizine, IN-REG72 as well as IN-RYC33 and IN-R2W56.

To implement the residue definition for dietary risk assessment for plant commodities the maximum concentration of the sum of compounds containing the imidazopyridine ring and hydrolysed using acid to IN-A5760, IN-F4106, IN-QZY47 and IN-TMQ01 (expressed as fluazaindolizine) or the compounds containing the phenyl ring and hydrolysed to IN-QEK31 (expressed as fluazaindolizine) should be used.

Insufficient toxicological data was available for IN-TDQ54 and IN-UJV12 and the Meeting considered they could be assessed using the threshold of toxicological concern (TTC) approach Cramer class III (1.5 µg/kg bw/day).

Animal commodities

Livestock will be exposed to residues in feed, both from treated and rotated crops.

The metabolism of fluazaindolizine in lactating goats and laying hens was qualitatively similar. Fluazaindolizine was a major component of the ^{14}C residue in both the lactating goat and laying hen metabolism studies (goat: muscle > 62 percent TRR, fat > 67 percent TRR, kidney > 65 percent TRR, liver > 18 percent TRR, milk > 72 percent TRR; hen: egg > 76 percent TRR, fat > 66 percent TRR, liver > 91 percent TRR). The predominant metabolite was IN-F4106 (goat: muscle 26 percent TRR, fat 7 percent TRR, kidney 7 percent TRR, liver 39 percent TRR; hen: egg 6 percent TRR, fat 4 percent TRR, liver 3 percent TRR) together with small amounts of IN-A5760 (goat: muscle 3.4 percent TRR, liver 4.8 percent TRR, kidney 0.8 percent TRR), IN-REG72 (goat: liver 12 percent TRR, kidney 3.3 percent TRR, fat 5 percent TRR, milk 5 percent TRR; hen: liver 1.1 percent TRR) and IN-RYC33 (hen: egg 11 percent TRR, fat 2.5 percent TRR, liver 0.7 percent TRR).

In studies on the metabolism of IN-QEK31, limited metabolism occurred with IN-QEK31 the major component of the ^{14}C residue (goat: fat 74 percent TRR, liver 69 percent TRR, kidney 94 percent TRR, milk 95 percent TRR; hen: liver 71 percent TRR).

In a study on the metabolism on IN-QZY47, only low levels of IN-QZY47 were detected in milk (3.1 percent TRR) with no residues detected in tissues. The predominant residue in tissues was IN-F4106 (milk 23 percent TRR, liver 41 percent TRR, kidney 15 percent TRR, muscle 81 percent TRR, fat >60 percent TRR) with lower levels of IN-A5760 (milk 2.3 percent TRR, liver 10 percent TRR, kidney 5.3 percent TRR, fat 5 percent TRR).

In a metabolism study on IN-TMQ01 in a lactating goat, IN-TMQ01 was the predominant ^{14}C residue in liver (48 percent TRR), kidney (87 percent TRR), muscle (43 percent TRR) and fat (50 percent TRR) with IN-F4106 the predominant ^{14}C residue in milk (98 percent TRR) and muscle (44 percent TRR).

Fluazaindolizine is present in milk, eggs and all tissues in the lactating goat and most tissues in laying hen metabolism studies and would be suitable for monitoring compliance.

Methods are available for the determination of fluazaindolizine and IN-A5760, IN-F4106, IN-REG72, IN-QEK31, IN-R2W56 and IN-RYC33 in tissues, milk and eggs.

The Meeting agreed the residue for compliance monitoring for tissues, milk and eggs should be fluazaindolizine.

In deciding which compounds should be included in the residue definition for risk assessment, the Meeting considered the likely occurrence of the compounds and the toxicological properties of the candidates. The compounds fluazaindolizine, IN-A5760, IN-F4106, IN-REG72, IN-QEK31, IN-QZY47, IN-RYC33, and IN-TMQ01 are considered to be covered by the HBGV for fluazaindolizine.

The predominant residues livestock are exposed to are IN-A5760, IN-F4106, IN-QEK31, IN-QZY47 and IN-TMQ01 at similar levels and these compounds, together with fluazaindolizine will comprise the majority of residues. The metabolism study with IN-QZY47 suggests no residues of IN-QZY47 are expected in tissues, and only very low residues in milk. Residues of IN-QZY47 in poultry commodities are also expected to be insignificant. IN-REG72 and IN-RYC33 are only found at low levels in livestock feed items and the Meeting considered IN-REG72 and IN-RYC33 would not be found at significant levels in livestock tissues, milk, or eggs. When expressed in terms of fluazaindolizine, IN-QEK31 residues are accounted for by the sum of IN-A5760, IN-F4106 and IN-TMQ01 and noting that metabolism and feeding studies with IN-QEK31 suggest only low residues are expected, the Meeting agreed it was not necessary to include IN-QZY47, IN-REG72 or IN-QEK31 in the residue definition for risk assessment.

The Meeting agreed the residue definition for risk assessment for animal commodities should be the sum of fluazaindolizine, IN-A5760, IN-F4106, and IN-TMQ01 (expressed as fluazaindolizine).

Consideration of metabolites using TTC approach

The Meeting also considered that IN-TDQ54 and IN-UJV12, metabolites relevant for livestock dietary burden, could be assessed using the threshold of toxicological concern for Cramer Class III compounds of 1.5 µg/kg bw per day.

The Meeting recommended the following residue definitions for fluazaindolizine.

Definition of the residue for compliance with MRL for plant and animal commodities: *fluazaindolizine.*

Definition of the residue for dietary risk assessment for plant commodities:

fluazaindolizine, and free and conjugated forms of the following compounds: 2-chloro-5-hydroxybenzenesulfonamide (IN-A5760), 2-chloro-5-methoxybenzenesulfonamide (IN-F4106), 8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylic acid (IN-QEK31), 3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-L-alanine (IN-QZY47), 8-chloro-N-[(2-chloro-5-hydroxyphenyl)sulfonyl]-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide (IN-REG72), 8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide (IN-RYC33) and 3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-(2R)-hydroxypropanoic acid (IN-TMQ01) (expressed as fluazaindolizine). This can be implemented by taking the maximum of the sum of compounds containing the imidazopyridine ring and hydrolysed using acid to IN-A5760, IN-F4106, IN-QZY47 and IN-TMQ01 (expressed as fluazaindolizine) ***OR*** *compounds containing the*

phenyl ring and hydrolysed to 8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylic acid (IN-QEK31) (expressed as fluazaindolizine).

Definition of the residue for dietary risk assessment for animal commodities:

Sum of fluazaindolizine, 2-chloro-5-hydroxybenzenesulfonamide (IN-A5760), 2-chloro-5-methoxybenzenesulfonamide (IN-F4106), and 3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-(2R)-hydroxypropanoic acid (IN-TMQ01) (expressed as fluazaindolizine).

In deciding whether the residue for compliance is regarded as fat-soluble, the Meeting noted mean residues at the highest dose level (20.3 ppm) in the lactating cow study according to the compliance residue definition were 0.0066 mg/kg in muscle and 0.034 mg/kg in fat while residues in milk were similar in milk fat compared to whole milk (day-14 whole milk 0.061 mg/kg, cream 0.065 mg/kg, skim milk 0.07 mg/kg). In the laying hen metabolism study, residues of fluazaindolizine were 0.041 mg/kg in muscle and 0.0135 mg/kg in skin+fat.

The Meeting considers overall the residue for compliance is not fat-soluble.

To estimate livestock dietary burdens, the Meeting noted residues of fluazaindolizine, IN-A5760, IN-F4106 and IN-TMQ01 in feeds are required. In addition, IN-QZY47 is transformed into IN-F4106 and IN-A5760 in livestock and residue levels in feed are also required for IN-QZY47.

Results of supervised residue trials on crops

Supervised trials were available for the use of fluazaindolizine on a range of crops with product labels available from Australia and Canada.

The residue concentrations in the evaluation tables are expressed in terms of the individual compounds and not as fluazaindolizine equivalents.

In evaluating the crop residue data, a range of values are required to be derived. Estimates are made for residues of:

- fluazaindolizine for estimation of maximum residue levels and livestock dietary burden
- Maximum of 2.26×IN-A5760+2.11×IN-F4106+1.52×IN-QZY47+1.51×IN-TMQ01 or 1.77×IN-QEK31 for estimation of STMR and HR values
- IN-F4106+1.068×IN-A5760 for estimation of livestock dietary burden
- IN-QZY47 for estimation of livestock dietary burden
- IN-TMQ01 for estimation of livestock dietary burden
- Inputs required for compounds being assessed using the TTC approach
- IN-UJV12 for estimation of livestock dietary burden and median and highest values
- IN-TDQ54 for estimation of livestock dietary burden and median and highest values

In calculating sums, residues present at < 0.01 mg/kg are assumed present at 0.01 mg/kg.

	Residue (mg/kg)	Factor to convert to fluazaindolizine equivalents	Converted residue (mg/kg)
IN-A5760	0.01	2.26	0.0226
IN-F4106	0.05	2.11	0.1055
IN-QZY47	0.01	1.52	0.0152
IN-TMQ01	0.01	1.51	0.0151
Sum			0.1584

Fruiting vegetables, cucurbits (cucumber, melon, squash)

In Canada, cGAP for cucurbits consists of four soil applications at pre-plant or broadcast followed by soil incorporation/chemigation at 0.56–2.24 kg ai/ha and at 14-day intervals with a PHI of 1 day with a maximum application of 2.24 kg ai/ha/year.

The Meeting received supervised residue trials on cucumber and summer squash conducted in Canada and the United States.

In nine trials on cucumber approximating cGAP residues: < 0.01 (4), 0.0105, 0.012, 0.0155, 0.0535, 0.0755 mg/kg for fluazaindolizine.

In nine trials approximating cGAP residues in squash were: < 0.01 (6), 0.011, 0.0455, 0.089 mg/kg for fluazaindolizine.

The Meeting noted that residues in cucumber and summer squash are similar, confirmed by a Mann-Whitney U test, and decided the combine the data sets for mutual support. The combined data is: < 0.01 (10), 0.0105, 0.011, 0.012, 0.0155, 0.0455, 0.0535, 0.0755, 0.089 mg/kg.

Residues of the sum 2.26×IN-A5760+2.11×IN-F4106+1.52×IN-QZY47+1.51×IN-TMQ01: 0.0740, 0.0740, 0.0740, 0.0740, 0.0755, 0.0763, 0.0808, 0.0909, 0.1078, 0.1105, 0.1261, 0.1310, 0.1331, 0.1416, 0.1648, 0.1720, 0.2729, 0.3674 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg, an STMR of 0.1092 mg/kg and an HR of 0.3674 for fluazaindolizine in the fruiting vegetable cucurbit subgroup of cucumbers and summer squashes.

Residues of IN-UJV12 in cucumber and squash were: < 0.01 (11), 0.0105, 0.0105, 0.0135, 0.0155, 0.0165, 0.0165, 0.0205 mg/kg.

Residues of IN-TQD54 in cucumber and squash were: < 0.01 (18) mg/kg.

The Meeting received supervised residue trials on melon conducted in Canada and the United States.

In 10 trials approximating cGAP residues in whole melons were: < 0.01 (5), 0.011, 0.012, 0.012, 0.041, 0.056 mg/kg for fluazaindolizine, maximum individual analytical result 0.089 mg/kg.

In 10 trials approximating cGAP residues of 2.26×IN-A5760+2.11×IN-F4106+1.52×IN-QZY47+1.51×IN-TMQ01 in pulp were: 0.0740, 0.0763, 0.0900, 0.0960, 0.1340, 0.1355, 0.1835, 0.2144, 0.3380, 0.3937 mg/kg for fluazaindolizine.

The Meeting estimated a maximum residue level of 0.1 mg/kg (OECD calculator estimate 0.07 mg/kg but highest individual whole melon residue 0.089 mg/kg), an STMR of 0.1348 mg/kg and an HR of 0.3937 mg/kg for melon pulp.

Residues of IN-UJV12 in pulp were: < 0.01 (9), 0.0125 mg/kg

Residues of IN-TQD54 in pulp were: < 0.01 (9), 0.0105 mg/kg.

Fruiting vegetables, other than cucurbits (tomato, peppers including chili)

In Canada cGAP for fruiting vegetables is for three soil applications at pre-plant or broadcast followed by soil incorporation or chemigation at 0.56–2.24 kg ai/ha and at 14-day intervals with a PHI of 1 day with a maximum application of 2.24 kg ai/ha/year.

The Meeting received supervised residue trials on tomato conducted in Canada and the United States.

In 17 trials approximating cGAP residues in tomato were: < 0.01 (15), 0.025, 0.0665 mg/kg for fluazaindolizine.

In 17 trials approximating cGAP residues of 2.26×IN-A5760+2.11×IN-F4106+1.52×IN-QZY47+1.51×IN-TMQ01 in tomatoes were: 0.0740 (8), 0.0748, 0.0751, 0.0751, 0.0797, 0.0808, 0.0842, 0.0967, 0.1204, 0.9630 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg (OECD calculator estimate 0.07 mg/kg but highest individual analytical result 0.11 mg/kg), a STMR of 0.0748 mg/kg and an HR of 0.9630 mg/kg for fluazaindolizine in tomato.

Residues of the sum of IN-F4106+1.068×IN-A5760 were: 0.0207 (10), 0.0212, 0.0212, 0.0234, 0.0239, 0.0255, 0.0427, 0.3385 mg/kg.

Residues of IN-QZY47 in tomatoes were: < 0.01 (15), 0.0125, 0.058 mg/kg.

Residues of IN-TMQ01 in tomatoes were: < 0.01 (13), 0.0105, 0.0135, 0.025, 0.14 mg/kg.

Residues of IN-UJV12 in tomatoes were: < 0.01 (17) mg/kg

Residues of IN-TQD54 in tomatoes were: < 0.01 (15), 0.011, 0.061 mg/kg.

The Meeting agreed to extrapolate the results for tomato to eggplant and estimated a maximum residue level of 0.15 mg/kg, a STMR of 0.0748 mg/kg and an HR of 0.9630 mg/kg for fluazaindolizine in eggplant.

The Meeting received supervised residue trials on pepper, including chili peppers, conducted in Canada and the United States.

In 13 trials approximating cGAP residues in peppers were: < 0.01 (12), 0.0265 mg/kg for fluazaindolizine.

In 13 trials approximating cGAP residues of 2.26×IN-A5760+2.11×IN-F4106+1.52×IN-QZY47+1.51×IN-TMQ01 in peppers were: 0.0740 (9), 0.0755, 0.0909, 0.1209, 0.3102 mg/kg.

The Meeting estimated a maximum residue level of 0.03 mg/kg, a STMR of 0.0740 mg/kg and an HR of 0.3102 mg/kg for fluazaindolizine in peppers.

Residues of IN-UJV12 in peppers were: < 0.01 (13) mg/kg

Residues of IN-TQD54 in peppers were: < 0.01 (11), 0.0205, 0.0375 mg/kg.

Using a default concentration factor of 10, the Meeting estimated a maximum residue level of 0.3 mg/kg for fluazaindolizine in dried chili pepper.

Root and tuber vegetables

Carrot

In Canada cGAP for carrot is for two soil applications at pre-plant or broadcast followed by soil incorporation or chemigation at 0.56–2.24 kg ai/ha and at 14-day intervals with a PHI of 65 days with a maximum application of 2.24 kg ai/ha/year.

The Meeting received supervised residue trials on carrots conducted in Canada and the United States.

In 11 trials on carrots conducted in Canada and the United States approximating cGAP, residues in carrots were: < 0.01 (6), 0.02, 0.0235, 0.04, 0.0995, 0.265 mg/kg for fluazaindolizine.

Total residues for dietary risk assessment were: 0.0740, 0.0740, 0.1034, 0.1271, 0.1329, 0.1485, 0.1503, 0.4375, 0.5862, 0.7679, 1.973 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg, a STMR of 0.1485 mg/kg and an HR of 1.973 mg/kg for fluazaindolizine in carrots.

For livestock dietary burden, residues in carrot were (n=11):

[IN-F4106+1.068×IN-A5760]: 0.0207 (6), 0.0232, 0.0287, 0.0332, 0.0737, 0.1489 mg/kg, with a median of 0.0297 mg/kg and a highest residue of 0.1489 mg/kg

IN-QZY47: <0.01 (3), 0.014, 0.0205, 0.031, 0.0335, 0.089, 0.1095, 0.115, 0.5 mg/kg, with a median of 0.031 and a highest residue of 0.5 mg/kg

IN-TMQ01: <0.01 (2), 0.012, 0.024, 0.0245, 0.045, 0.0535, 0.0765, 0.24, 0.39, 0.595 mg/kg, with a median of 0.01 mg/kg and a highest residue of 0.595 mg/kg

For the TTC approach, residues were (n=11):

IN-UJV12: <0.01 (11) mg/kg, with a median and a highest residue of 0.01 mg/kg

IN-TQD54: <0.01 (8), 0.025, 0.042, 0.049 mg/kg, with a median of 0.01 mg/kg and a highest residue of 0.049 mg/kg

Tuberous and corm vegetables

In Canada cGAP for tuberous and corm vegetables is for two soil applications at pre-plant or broadcast followed by soil incorporation or chemigation at 0.56–2.24 kg ai/ha and at 14-day intervals with a PHI of 40 days with a maximum application of 2.24 kg ai/ha/year.

In 19 trials on potatoes conducted in Canada and the United States approximating cGAP, residues in were: < 0.01 (4), 0.012, 0.014, 0.0165, 0.0165, 0.021, 0.028, 0.0305, 0.031, 0.0415, 0.0435, 0.0465, 0.0515, 0.068, 0.1075, 0.16 mg/kg for fluazaindolizine.

Total residues for dietary risk assessment were: 0.0740 (3), 0.0751, 0.0983, 0.1014, 0.1057, 0.1095, 0.1182, 0.1231, 0.1558, 0.1816, 0.2089, 0.2272, 0.2515, 0.2856, 0.3709, 0.4116, 0.7356 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg, a STMR of 0.1231 mg/kg and an HR of 0.7356 mg/kg for fluazaindolizine in tuberous and corm vegetables.

For livestock dietary burden, residues in potatoes were (n=19):

[IN-F4106+1.068×IN-A5760]: 0.0207 (4), 0.0212, 0.0247, 0.0302, 0.0317, 0.0322, 0.0337 (2), 0.0357, 0.0372, 0.0419, 0.0437, 0.0452, 0.0570, 0.0717, 0.1127 mg/kg, with a median of 0.0337 mg/kg and a highest residue of 0.1127 mg/kg.

IN-QZY47: <0.01 (9), 0.011, 0.012, 0.015, 0.02, 0.022, 0.024, 0.038, 0.0425, 0.0455, 0.072 mg/kg , with a median of 0.011 mg/kg and a highest residue of 0.072 mg/kg.

IN-TMQ01: <0.01 (8), 0.0115, 0.0205, 0.024, 0.031, 0.0425, 0.046, 0.0475, 0.12, 0.17, 0.19, 0.335 mg/kg, with a median of 0.0205 mg/kg and a highest residue of 0.335 mg/kg.

For the TTC approach, residues were:

IN-UJV12: <0.01 (15), 0.011, 0.0115, 0.012, 0.016 mg/kg, with a median of 0.01 mg/kg and a highest residue of 0.016 mg/kg.

IN-TQD54: <0.01 (11), 0.0105, 0.0115, 0.0235, 0.0305, 0.031, 0.034, 0.044, 0.0865 mg/kg, with a median of 0.01 mg/kg and a highest residue of 0.0865 mg/kg.

Residues in rotational crops

Fluazaindolizine and metabolites are moderately persistent to persistent in the environment and may contribute to residues in follow/rotational crops through uptake from soil.

In assessing the potential uptake of residues, the Meeting considered the maximal season rate to be 2.24 kg ai/ha as detailed for all crops on the Canadian label. Application rates relevant for plateau concentrations in soil for the various compounds of interest can be calculated using the compound aerobic soil degradation DT_{50} (median) values for the various compounds of interest. Geometric mean DT_{50} value of 28.1 days for fluazaindolizine, 89 days for IN-QEK31, 293 days for IN-F4106 and 27 days for IN-A5760. For compounds not formed in soil, the concentration for fluazaindolizine is used (IN-QZY47, IN-TMQ01, IN-UJV12 and IN-TQD54). The estimated application rate for plateau residues in soil for fluazaindolizine, IN-A5760 and IN-QEK31 is 2.24 kg ai/ha, for IN-F4106 3.87 kg ai/ha.

Field rotational crop studies were used to derive estimates of residues in various commodities if the field were treated at the maximal seasonal rate. In combining residues from different trials to derive the various inputs required, individual analyte residues were scaled to the maximum seasonal rate.

The commodity groups studied in the field crop rotational studies were:

Fruit (strawberry)

Fruiting vegetables (tomato)

Leafy vegetables/Brassicas (lettuce, spinach, radish tops, turnip tops, broccoli, Swiss chard)

Root and tuber - tops (carrot tops, radish tops, turnip tops)

Root and tuber (carrots, radish, turnip)

Cereals (corn/maize, sorghum, wheat) – forage, straw or stover, hay, grain

Oilseeds/pulses (rape, beans, peas, soya beans) – forage, hay, grain

Bulb and stem vegetable (celery)

For each trial location/year, the highest residue from the PBIs that were longer than the PBI on the Canada label were used: 0 days for carrots, 14 days for root vegetables, except sugar beets (except carrot roots), leaves of root and tuber vegetables, bulb vegetables, leafy vegetables, brassica head and stem vegetable, legume vegetables, succulent or dried, foliage of legume vegetables, low growing berries, cereal grain, forage, fodder, and straw of cereal grains, grass forage, fodder, and hay, oilseeds, stalk, stem, and leaf petioles, and 365 days for all other crops.

Table 191 Example of scaled residues (mg/kg) in radish tops in one trial

kg ai/ha	PBI (days)	Fluazaindolizine	IN-A5760	IN-F4106	IN-QZY47	IN-TMQ01	IN-UJV12	IN-TQD54
4.67	68	0.01	0.038	0.0115	0.16	0.35	0.0305	0.245
Scaling factor		2.24/4.67	2.24/4.67	3.87/4.67	2.24/4.67	2.24/4.67	2.24/4.67	2.24/4.67
Scaled residues		0.0048	0.0182	0.0095	0.0767	0.1679	0.0146	0.1175

The sum of IN-F4106+1.068×IN-A5760 was 0.0289 mg/kg (expressed as IN-F4106).

When there were multiple crops within a rotational crop category, for example leafy vegetables, where residue data were available for lettuce, spinach, radish tops, turnip tops and Swiss chard, the highest of the STMR/median residues and HR/highest residues for the individual crops were selected for the STMR/median and HR/highest residue for the crop grouping.

Strawberry (Canada PBI 14 days, n=9)

Residues of fluazaindolizine: < 0.005 (4), < 0.0051 (5) mg/kg.

Total residues for dietary risk assessment were: 0.0446, 0.0455, 0.0459, 0.0494, 0.0530, 0.0585, 0.0637, 0.0996, 0.1416 mg/kg.

The Meeting estimated a maximum residue level of 0.015 mg/kg for strawberries, an STMR of 0.0530 mg/kg and an HR of 0.1416 mg/kg.

Residues of IN-UJV12 were: 0.0050 (4), 0.0051 (5) mg/kg, with a median of 0.005 mg/kg and a highest residue of 0.0051 mg/kg.

Residues of IN-TQD54 were: 0.0050 (4), 0.0051 (4), 0.0084 mg/kg, with a median of 0.0051 mg/kg and a highest residue of 0.0084 mg/kg.

Tomato (Canada PBI not relevant, n=10)

Residues of fluazaindolizine: 0.0050 (6), 0.0051, 0.0051, 0.0052, 0.0052 mg/kg.

Total residues for dietary risk assessment were: 0.0445, 0.0451, 0.0455, 0.0464, 0.0467, 0.0475, 0.0526, 0.0526, 0.0706, 0.1889 mg/kg.

Residues in tomatoes for livestock dietary burden were (n=10):

[IN-F4106+1.068×IN-A5760]: 0.0139, 0.0141, 0.0142, 0.0145, 0.0146, 0.0150, 0.0155, 0.0166, 0.0216, 0.0379 mg/kg

IN-QZY47: 0.0050 (6), 0.0051 (2), 0.0052 (2) mg/kg

IN-TMQ01: 0.0050 (2), 0.0051, 0.0052 (2), 0.0058, 0.0065, 0.0082, 0.0114, 0.0669 mg/kg,

Residues for the TTC approach were:

IN-UJV12: 0.0050 (6), 0.0051 (2), 0.0052 (2) mg/kg

IN-TQD54: 0.0050 (4), 0.0051, 0.0052 (2), 0.0070, 0.0105, 0.0249 mg/kg

Residues arising from rotational crops are lower than the ones found in treated tomatoes, peppers and eggplant and the Meeting confirms it previous estimations based on trials conducted with the primary crops.

Group 010 Brassica vegetables (except Brassica leafy vegetables)

Broccoli (Canada PBI 14 days, n=10)

Residues of fluazaindolizine: 0.0050 (4), 0.0051 (3), 0.0056, 0.0069, 0.0072 mg/kg.

Total residues for dietary intake assessment were: 0.0443, 0.0446, 0.0450, 0.0451, 0.0460, 0.0467, 0.0487, 0.0514, 0.0564, 0.0705 mg/kg.

The Meeting estimated a maximum residue level of 0.02 mg/kg, an STMR of 0.04335 mg/kg and a HR of 0.0705 mg/kg for fluazaindolizine in the Group 010 Brassica vegetables (except Brassica leafy vegetables).

Residues for livestock dietary burden were:

[IN-F4106+1.068×IN-A5760]: 0.0139 (3), 0.0140, 0.0141 (3), 0.0143 (2), 0.0144 mg/kg, with a median of 0.0141 mg/kg and a highest residue of 0.0144 mg/kg.

IN-QZY47: 0.0050 (4), 0.0051, 0.0061, 0.0069, 0.0082, 0.0087, 0.0187 mg/kg, with a median of 0.0056 mg/kg and a highest residue of 0.0187 mg/kg.

IN-TMQ01: 0.0050 (4), 0.0051 (2), 0.0054, 0.0059, 0.0085, 0.0092 mg/kg, with a median of 0.0051 mg/kg and a highest residue of 0.0092 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050 (6), 0.0051 (4) mg/kg, with a median of 0.0050 mg/kg and a highest residue of 0.0051 mg/kg.

IN-TQD54: 0.0050 (6), 0.0051 (4) mg/kg, with a median of 0.0050 mg/kg and a highest residue of 0.0051 mg/kg.

Group 013 Leafy vegetables (including Brassica leafy vegetables)

Spinach (Canada PBI 14 days, n=2)

Residues of fluazaindolizine in spinach: 0.0143, 0.0178 mg/kg.

Total residues for dietary risk assessment: 0.4982, 0.6035 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0129, 0.0187 mg/kg.

IN-TQD54: 0.0089, 0.0179 mg/kg.

Radish tops (Canada PBI 14 days, n=6)

Residues of fluazaindolizine in radish tops were: 0.0048, 0.0050, 0.0178, 0.0179 (3) mg/kg.

Total residues for dietary risk assessment: 0.1656, 0.3259, 0.3444, 0.4315, 0.6248, 1.275 mg/kg.

Residues for livestock dietary burden:

[IN-F4106+1.068×IN-A5760]: 0.0290, 0.0296, 0.0501, 0.0589, 0.0630, 0.0954 mg/kg.

IN-QZY47: 0.0251, 0.0548, 0.0767, 0.0824, 0.1742, 0.2276 mg/kg.

IN-TMQ01: 0.0179, 0.0645, 0.1472, 0.1502, 0.1679, 0.4819 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0146, 0.0179, 0.0179, 0.0182, 0.0320, 0.0602 mg/kg.

IN-TQD54: 0.0179, 0.0842, 0.1175, 0.1333, 0.1493, 0.6782 mg/kg.

Turnip tops (Canada PBI 14 days, n=5)

Residues of fluazaindolizine in turnip tops were: 0.005, 0.0051, 0.0051, 0.0051, 0.0051 mg/kg.

Total residues for dietary risk assessment : 0.0503, 0.0642, 0.0890, 0.1043, 0.1122 mg/kg.

Residues for livestock dietary burden:

[IN-F4106+1.068×IN-A5760]: 0.0141, 0.0142, 0.0142, 0.0143, 0.0165 mg/kg.

IN-QZY47: 0.0086, 0.0173, 0.0333, 0.0339, 0.0426 mg/kg.

IN-TMQ01: 0.0051, 0.0051, 0.0051, 0.0117, 0.0124 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0051, 0.0051, 0.0051, 0.0051, 0.0084 mg/kg.

IN-TQD54: 0.0051, 0.0051, 0.0051, 0.0112, 0.0144 mg/kg.

Carrot tops (Canada PBI 0 days, n=3)

Residues of fluazaindolizine in carrot tops: 0.0050, 0.0050, 0.0060 mg/kg.

Total residues for dietary risk assessment : 0.2177, 0.5851, 2.857 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050 (3) mg/kg.

IN-TQD54: 0.0050, 0.0100, 0.0609 mg/kg.

Swiss chard (Canada PBI 14 days, n=5):

Residues of fluazaindolizine in Swiss chard: 0.0049, 0.0050, 0.0051, 0.0051, 0.0077mg/kg.

Total residues for dietary risk assessment: 0.0452, 0.0456, 0.0460, 0.0697, 0.1007 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050, 0.0050, 0.0051, 0.0051, 0.0069 mg/kg.

IN-TQD54: 0.0049, 0.0050, 0.0050, 0.0051, 0.0051 mg/kg.

Lettuce (Canada PBI 14 days, n=13)

Residues of fluazaindolizine in lettuce: 0.0049 (3), 0.0050 (3), 0.0051 (3), 0.0061, 0.0178, 0.0179 (2) mg/kg.

Total residues for dietary risk assessment: 0.0458, 0.0536, 0.0647, 0.0716, 0.0773, 0.1134, 0.1579, 0.1601 (2), 0.1663, 0.1700, 0.1828, 1.388 mg/kg.

IN-UJV12: 0.0049, 0.0049, 0.0049, 0.0050, 0.0050, 0.0050, 0.0051, 0.0051, 0.0051, 0.0051, 0.0178, 0.0179, 0.0179 mg/kg.

IN-TQD54: 0.0049, 0.0049, 0.0049, 0.0050, 0.0050, 0.0050, 0.0051, 0.0051, 0.0051, 0.0051, 0.0178, 0.0179, 0.0179 mg/kg.

Of the leafy vegetable datasets available (spinach, lettuce, Swiss chard, carrot tops, radish tops and turnip tops) with five or more residue trials on rotational crops, radish tops had the highest fluazaindolizine residues, while residues of the other analytes of interest were sometimes highest in lettuce. The Meeting agreed to use the radish tops data to estimate a maximum residue level for leafy vegetables and the highest of the radish and lettuce data to estimate STMR and HR, respectively.

The Meeting estimated a maximum residue level of 0.04 mg/kg, an STMR of 0.3880 mg/kg (radish) and a HR of 1.388 mg/kg (lettuce) for fluazaindolizine in Group 013 Leafy vegetables (including Brassica leafy vegetables).

For livestock dietary burden, the Meeting estimated based on radish tops data:

[IN-F4106+1.068×IN-A5760]: median and highest residues of 0.0501 and 0.0954 mg/kg, respectively

IN-QZY47: median and highest residues of 0.07955 and 0.2276 mg/kg, respectively.

IN-TMQ01: median and highest residues for 0.1472) and 0.4819 mg/kg, respectively

For TTC the approach, also based on radish top data,

IN-UJV12: median and highest residues of 0.0182 and 0.0602 mg/kg, respectively

IN-TQD54: median and highest residues of 0.1175 and 0.6782 mg/kg, respectively

Group 14 Legume vegetables

Soya bean immature seed = seed+pod (Canada PBI 14 days, n=7)

Residues of fluazaindolizine: 0.0050, 0.0050, 0.0050, 0.0050, 0.0051, 0.0178, 0.0178 mg/kg.

Total residues for dietary risk assessment: 0.0449, 0.0451, 0.0456, 0.0470, 0.1228, 0.1589, 0.1589 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050 (4), 0.0051, 0.0178, 0.0178 mg/kg.

IN-TQD54: 0.0050 (4), 0.0051, 0.0178, 0.0178 mg/kg.

Pea immature, seed plus pod (Canada PBI 14 days, n=6)

Residues of fluazaindolizine were: 0.0049 (2), 0.0050, 0.0051, 0.0072, 0.0090 mg/kg.

Total residues for dietary risk assessment: 0.0442, 0.0453, 0.0681, 0.0737, 0.1021, 0.1420 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0049, 0.0050, 0.0051, 0.0062, 0.0066, 0.0094 mg/kg.

IN-TQD54: 0.0049, 0.0049, 0.0050, 0.0050, 0.0050, 0.0051 mg/kg.

Bean immature seed (Canada PBI 14 days, n=2)

Residues of fluazaindolizine were: 0.0179 (2) mg/kg.

Total residues for dietary risk assessment were: 0.1601 (2) mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0179 (2) mg/kg.

IN-TQD54: 0.0179 (2) mg/kg.

The Meeting agreed to use the highest of the soya bean and pea data (both seed with pods) to estimate STMR and HR values.

The Meeting estimated a maximum residue level of 0.04 mg/kg, an STMR of 0.0709 mg/kg (pea) and an HR of 0.1589 (soya) mg/kg for fluazaindolizine in Group 014 Legume vegetables [immature seed with pod]

For TTC approach, the Meeting estimated:

IN-UJV12: median and highest residues of 0.00565 (pea) and 0.0178 (soya) mg/kg, respectively.

IN-TQD54: median and highest residues of 0.0050 (soya) and 0.0178 (soya) mg/kg, respectively.

Group 15 Pulses

Soya bean seed (dry) (Canada PBI 14 days, n=7)

Residues of fluazaindolizine: 0.0050 (3), 0.0051, 0.0073, 0.0178, 0.0178 mg/kg.

Total residues for dietary risk assessment were: 0.0456, 0.0463, 0.0507, 0.0593, 0.1589, 0.1589, 0.1974 mg/kg.

Residues for livestock dietary burden were:

IN-F4106+1.068×IN-A5760: 0.0139, 0.0140, 0.0140, 0.0141, 0.0143, 0.0497, 0.0497 mg/kg.

IN-QZY47: 0.0050, 0.0050, 0.0050, 0.0051, 0.0053, 0.0178, 0.0178 mg/kg.

IN-TMQ01: 0.0050, 0.0050, 0.0050, 0.0050, 0.0051 0.0178, 0.0178 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050, 0.0050, 0.0050, 0.0050, 0.0051, 0.0178, 0.0178 mg/kg.

IN-TQD54: 0.0050, 0.0050, 0.0050, 0.0050, 0.0051, 0.0178, 0.0178 mg/kg.

Pea seed (dry) (Canada PBI 14 days, n=11)

Residues of fluazaindolizine: 0.0049, 0.0050, 0.0051, 0.0072, 0.0073, 0.0094, 0.0148, 0.0220, 0.0264, 0.0343, 0.0565 mg/kg.

Total residues for dietary risk assessment: 0.0464, 0.0503, 0.0631, 0.0646, 0.0656 (2), 0.1141, 0.1150, 0.2716, 0.2738, 1.2392 mg/kg.

Residues for livestock dietary burden:

[IN-F4106+1.068×IN-A5760]: 0.0138, 0.0141, 0.0142, 0.0169, 0.0198, 0.0202, 0.0205 (2), 0.0462, 0.0659, 0.3567 mg/kg.

IN-QZY47: 0.0064, 0.0071, 0.0072, 0.0073 (2), 0.0084, 0.0473, 0.0505, 0.0821, 0.1110, 0.2960 mg/kg.

IN-TMQ01: 0.0049 (2), 0.0050 (3), 0.0051, 0.0071, 0.0072, 0.0073 (2), 0.0235 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0049, 0.0051, 0.0071, 0.0072, 0.0073 (2), 0.0123, 0.0126, 0.0222, 0.0261, 0.0740 mg/kg.

IN-TQD54: 0.0049 (2), 0.0050 (3), 0.0051, 0.0071, 0.0072, 0.0073 (2), 0.0101 mg/kg.

Bean seed (dry) (Canada PBI 14 days, n=2)

Residues of fluazaindolizine: 0.0179 (2) mg/kg.

Total residues for dietary risk assessment: 0.1601 (2) mg/kg.

Residues for livestock dietary burden:

[IN-F4106+1.068×IN-A5760]: 0.0501 (2) mg/kg.

IN-QZY47: 0.0179 (2) mg/kg.

IN-TMQ01: 0.0179 (2) mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0179 (2) mg/kg.

IN-TQD54: 0.0179 (2) mg/kg.

Residues were highest in peas dry and the Meeting agreed to use the peas dry dataset to make the estimations.

The Meeting estimated a maximum residue level of 0.09 mg/kg and an STMR of 0.0656 mg/kg for fluazaindolizine in Group 015 Pulses.

For livestock dietary burden, the Meeting estimated median residues of 0.0202 mg/kg for [IN-F4106+1.068×IN-A5760], 0.0084 mg/kg for IN-QZY47 and 0.0051 mg/kg for IN-TMQ01.

For TTC approach, the Meeting estimated a median residue of 0.0073 mg/kg for IN-UJV12 and of 0.0051 mg/kg for IN-TQD54Group 16 Root and tuber vegetables

Carrot roots (Canada PBI 0 days, n=3)

Residues of fluazaindolizine were: 0.0050 (3) mg/kg.

Total residues for dietary risk assessment : 0.0449, 0.1096, 0.2055 mg/kg.

Residues for livestock dietary burden:

[IN-F4106+1.068×IN-A5760]: 0.0139 (3) mg/kg.

IN-TMQ01: 0.0052, 0.0403, 0.0998 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050 (3) mg/kg.

IN-TQD54: 0.0050 (2), 0.0060 mg/kg.

Radish root (Canada PBI 14 days, n=6)

Residues of fluazaindolizine were: 0.0048, 0.0072, 0.0178, 0.0179 (3) mg/kg.

Total residues for dietary risk assessment: 0.1583, 0.1628, 0.1662, 0.2472, 0.3127, 0.9322 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050, 0.0050, 0.0178, 0.0179, 0.0179, 0.0245 mg/kg.

IN-TQD54: 0.0070, 0.0152, 0.0178, 0.0179, 0.0215, 0.2008 mg/kg.

Turnip roots (Canada PBI 14 days, n=5)

Residues of fluazaindolizine were: 0.0050, 0.0051, 0.0077, 0.0091, 0.0106 mg/kg.

Total residues for dietary risk assessment: 0.0539, 0.0610, 0.0650, 0.0799, 0.1090 mg/kg.

Residues for livestock dietary burden

[IN-F4106+1.068×IN-A5760]: 0.0140, 0.0141, 0.0142 (2), 0.0143 mg/kg.

IN-QZY47: 0.0107, 0.0159, 0.0179, 0.0279, 0.0470 mg/kg.

IN-TMQ01: 0.0050, 0.0051 (4) mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050, 0.0051 (4) mg/kg.

IN-TQD54: 0.0050, 0.0051 (4) mg/kg.

Radish and turnip roots had the highest fluazaindolizine residues of the root and tuber vegetable datasets available.

Based on radish data, the Meeting estimated a maximum residue level of 0.04 mg/kg, an STMR of 0.1935 mg/kg and a HR of 0.9322 mg/kg for fluazaindolizine in Group 16 root vegetables (except carrot)

For livestock dietary burden, the Meeting estimated, based on turnip data:

[IN-F4106+1.068×IN-A5760]: median and highest residues of 0.0142 and 0.0143 mg/kg, respectively.

IN-QZY47: median and highest residues of 0.0179 and 0.0470 mg/kg, repectively.

IN-TMQ01: median and highest residues of 0.0051 and 0.0051 mg/kg, respectively.

For TTC approach, the Meeting estimated based on radish data:

IN-UJV12: median and highest residues of 0.0178 and 0.0245 mg/kg, respectively.

IN-TQD54: median and highest residues of 0.0178 and 0.2008 (radish) mg/kg, respectively.

Group 17 Stalk and stem vegetables

Celery (Canada PBI 14 days, n=5):

Residues of fluazaindolizine were: 0.0050, 0.0051, 0.0087, 0.0091, 0.0197 mg/kg.

Total residues for dietary risk assessment: 0.0458, 0.0666, 0.0674, 0.1595, 0.8281 mg/kg.

The Meeting estimated a maximum residue level of 0.04 mg/kg, an STMR of 0.0674 mg/kg and a HR of 0.8281 mg/kg for fluazaindolizine in Group 17 Stalk and stem vegetables.

The Meeting agreed to extrapolate the conclusions to bulb vegetables and estimated a maximum residue level of 0.04 mg/kg, an STMR and HR of 0.0674 and 0.8281 mg/kg, respectively for Bulb vegetables.

For the TTC approach the Meeting also estimated:

IN-UJV12: 0.0050, 0.0051 (2), 0.0052, 0.0088 mg/kg, median of 0.0051 mg/kg and highest residues of 0.0088 mg/kg.

IN-TQD54: 0.0050, 0.0051 (2), 0.0052, 0.0121 mg/kg, median of 0.0051 mg/kg and highest residues of 0.0121 mg/kg.

Group 020 Cereal Grains

Field corn grain (Canada PBI 14 days, n=10)

Residues of fluazaindolizine were: 0.0050 (5), 0.0067 (4), 0.0068 mg/kg.

Total residues for dietary risk assessment: 0.0457, 0.0477, 0.0497, 0.0596, 0.0599, 0.0601 (2), 0.0607, 0.0654, 0.1015 mg/kg.

Residues for livestock dietary burden

[IN-F4106+1.068×IN-A5760]: 0.0139 (3), 0.0140, 0.0141, 0.0186, 0.0187, 0.0188 (2), 0.0190 mg/kg.

IN-QZY47: 0.0050 (3), 0.0052, 0.0063, 0.0067 (4), 0.0068, mg/kg.

IN-TMQ01: 0.0050, 0.0057, 0.0067 (4), 0.0068, 0.0086, 0.0117, 0.0181 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050 (5), 0.0067 (4), 0.0068 mg/kg.

IN-TQD54: 0.0050 (3), 0.0067 (4), 0.0068, 0.0077, 0.0098 mg/kg.

Sorghum grain (Canada PBI 14 days, n=1)

Residues of fluazaindolizine were: 0.0178 mg/kg.

Total residues for dietary risk assessment: 0.1589 mg/kg.

Residues for livestock dietary burden

[IN-F4106+1.068×IN-A5760]: 0.0497 mg/kg.

IN-QZY47: 0.0178 mg/kg.

IN-TMQ01: 0.0178 mg/kg.

Residues for the TTC approach were.

IN-UJV12: 0.0178 mg/kg.

IN-TQD54: 0.0178 mg/kg.

Wheat grain (Canada PBI 14 days, n=13)

Residues of fluazaindolizine were: 0.0050 (4), 0.0051, 0.0070, 0.0072, 0.0073, 0.0073, 0.0076, 0.0178, 0.0179, 0.0179 mg/kg.

Total residues for dietary risk assessment: 0.0445, 0.0478, 0.0622, 0.0642, 0.0652, 0.0674, 0.0676, 0.1025, 0.1060, 0.1601 (2), 0.2517, 0.6799 mg/kg.

Residues for livestock dietary burden

[IN-F4106+1.068×IN-A5760]: 0.0139, 0.0140, 0.0141, 0.0143, 0.0194, 0.0201, 0.0204, 0.0205, 0.0212, 0.0480, 0.0497, 0.0501 (2) mg/kg.

IN-QZY47: 0.0050 (3), 0.0051, 0.0064, 0.0070, 0.0072, 0.0073 (2), 0.0076, 0.0178, 0.0179 (2) mg/kg.

IN-TMQ01: 0.0050 (3), 0.0051, 0.0070, 0.0072, 0.0073 (2), 0.0076, 0.0178, 0.0179 (2), 0.3766 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050 (4), 0.0051, 0.0070, 0.0072, 0.0073, 0.0073, 0.0076, 0.0178, 0.0179, 0.0179 mg/kg.

IN-TQD54: 0.0050 (3), 0.0051, 0.0070, 0.0072, 0.0073, 0.0073, 0.0076, 0.0164, 0.0178, 0.0179, 0.0179 mg/kg.

Sweet corn (Canada PBI 14 days, n=10) Field corn, immature

Residues of fluazaindolizine were: 0.0050 (5), 0.0067 (4), 0.0068 mg/kg.

Total residues for dietary risk assessment: 0.0446 (2), 0.0450, 0.0465, 0.0540, 0.0596, 0.0599, 0.0601, 0.0607, 0.1401 mg/kg.

Residues for livestock dietary burden

[IN-F4106+1.068×IN-A5760]: 0.0139 (3), 0.0140, 0.0141, 0.0186, 0.0187, 0.0188, 0.0190, 0.0351 mg/kg.

IN-TMQ01: 0.0050 (3), 0.0062, 0.0067 (3), 0.0068, 0.0070, 0.0370 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050 (5), 0.0067 (4), 0.0068 mg/kg.

IN-TQD54: 0.0050 (5), 0.0067 (3), 0.0068, 0.0471 mg/kg.

As there was only one trial on sorghum, the Meeting considered the wheat data to make the estimations for cereal grains

The Meeting estimated a maximum residue level of 0.03 mg/kg, and an STMR of 0.0676 mg/kg, for fluazaindolizine in Group 20 Cereal Grains

For livestock dietary burden, the Meeting estimated median residues of 0.0204 mg/kg for [IN-F4106+1.068×IN-A5760], of 0.0072 mg/kg for IN-QZY47 and of 0.0073 mg/kg for IN-TMQ01.

For TTC approach, the Meeting estimated median residues for IN-UJV12 of 0.0072 mg/kg and of 0.0073 mg/kg for IN-TQD54.

Group 023 Oilseeds

Rape seed (Canada PBI 14 days, n=5)

Residues of fluazaindolizine were: 0.0071, 0.0072, 0.0073, 0.0073, 0.0075 mg/kg.

Total residues for dietary risk assessment: 0.0631, 0.0642, 0.0656 (2), 0.0669 mg/kg.

Residues for livestock dietary burden.

[IN-F4106+1.068×IN-A5760]: 0.0198, 0.0201, 0.0205 (2), 0.0209 mg/kg.

IN-QZY47: 0.0071, 0.0072, 0.0073 (2), 0.0075 mg/kg.

IN-TMQ01: 0.0071, 0.0072, 0.0073 (2), 0.0075 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0071, 0.0072, 0.0073, 0.0073, 0.0075 mg/kg.

IN-TQD54: 0.0071, 0.0072, 0.0073, 0.0073, 0.0075 mg/kg.

Soya bean seed (dry) (Canada PBI 14 days, n=7)

Residues of fluazaindolizine were: 0.0050, 0.0050, 0.0050, 0.0051, 0.0073, 0.0178, 0.0178 mg/kg.

Total residues for dietary intake assessment: 0.0456, 0.0463, 0.0507, 0.0593, 0.1589 (2), 0.1974 mg/kg.

Residues for livestock dietary burden

[IN-F4106+1.068×IN-A5760]: 0.0139, 0.0140 (2), 0.0141, 0.0143, 0.0497 (2) mg/kg.

IN-QZY47: 0.0050 (3) 0.0051, 0.0053, 0.0178 (2) mg/kg.

IN-TMQ01: 0.0050 (4), 0.0051 0.0178 (2) mg/kg.

Residues for the TTC approach were.

IN-UJV12: 0.0050, 0.0050, 0.0050, 0.0050, 0.0051, 0.0178, 0.0178 mg/kg.

IN-TQD54: 0.0050, 0.0050, 0.0050, 0.0050, 0.0051, 0.0178, 0.0178 mg/kg.

Highest residues in soya bean, max of analytes for others

The Meeting estimated a maximum residue level of 0.04 mg/kg (based on soya beans) and an STMR of 0.0656 mg/kg (based on rape seed) for Oilseeds

For livestock dietary burden, the Meeting estimated, based on rape seed data, median residues of 0.0205 mg/kg for [IN-F4106+1.068×IN-A5760], of 0.0073 mg/kg for IN-QZY47 and of 0.0073 mg/kg for IN-TMQ01

For the TTC approach, the Meeting estimated, also based on rape seed data, median residues of 0.0073 mg/kg for IN-UJV12 and IN-TQD54.

Residues in animal feeds

All residues in forages and fodders discussed below were reported on an as received basis.

Legume forages

Bean vines (Canada PBI 14 days, n=2)

Fluazaindolizine: 0.0179, 0.0179 mg/kg.

[IN-F4106+1.068×IN-A5760]: 0.0501 (2) mg/kg.

IN-QZY47: 0.3136, 2.150 mg/kg.

IN-TMQ01: 0.0179 (2) mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0179, 0.0466 mg/kg.

IN-TQD54: 0.0179 (2) mg/kg.

Soya bean forage (Canada PBI 14 days, n=7)

Fluazaindolizine: 0.0050 (2), 0.0073, 0.0107, 0.0110, 0.0178 (2) mg/kg.

[IN-F4106+1.068×IN-A5760]: 0.0140, 0.0141, 0.0150, 0.0214, 0.0362, 0.0497 (2) mg/kg.

IN-QZY47: 0.0505, 0.0629, 0.0904, 0.1062, 0.1536, 0.1950, 0.3391 mg/kg.

IN-TMQ01: 0.0050 (4), 0.0051, 0.0178 (2) mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0078, 0.0114, 0.0146, 0.0178, 0.0265, 0.0455, 0.0651 mg/kg.

IN-TQD54: 0.0050 (4), 0.0051, 0.0178, 0.0178 mg/kg.

Pea forage (Canada PBI 14 days, n=5)

Fluazaindolizine: 0.0067, 0.0071, 0.0072, 0.0073 (2) mg/kg.

IN-F4106+1.068×IN-A5760: 0.0198, 0.0202, 0.0205, 0.0205, 0.0509 mg/kg.

IN-QZY47: 0.0133, 0.0155, 0.0241, 0.0264, 0.0464 mg/kg.

IN-TMQ01: 0.0067, 0.0071, 0.0072, 0.0073 (2) mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0071, 0.0072, 0.0073, 0.0073, 0.0094 mg/kg.

IN-TQD54: 0.0067, 0.0071, 0.0072, 0.0073, 0.0073 mg/kg.

Pea vines (Canada PBI 14 days, n=11)

Fluazaindolizine: 0.0049 (2), 0.0050, 0.0051, 0.0067, 0.0071, 0.0072, 0.0073 (2), 0.0076, 0.0105 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0198, 0.0202, 0.0205 (2), 0.0217, 0.0228, 0.0263, 0.0616, 0.0777, 0.1140, 0.1156 mg/kg.

IN-QZY47: 0.0071, 0.0072, 0.0110, 0.0175, 0.0191, 0.0597, 0.0717, 0.1280, 0.1337, 0.2663, 0.3034 mg/kg.

IN-TMQ01: 0.0049 (2), 0.0050 (2), 0.0051, 0.0053, 0.0067, 0.0071, 0.0072, 0.0073 (2) mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0067, 0.0071, 0.0072 (2), 0.0073 (2), 0.0092, 0.0262, 0.0318, 0.0488, 0.0641 mg/kg.

IN-TQD54: 0.0049 (2), 0.0050 (3), 0.0051, 0.0067, 0.0071, 0.0072, 0.0073 (2) mg/kg.

Based on soya bean forage data, the Meeting estimated a median and highest residue of 0.0107 and 0.0178 mg/kg respectively, for fluazaindolizine in Legume forages (bean, cowpea, crown vetch, Lespedeza, pea, peanut, soya bean, trefoil and vetch).

The Meeting also estimated based on soya bean or pea data:

[IN-F4106+IN-A5760]: median of 0.0214 mg/kg (soya bean) and highest residue of 0.0509 mg/kg (pea)

IN-QZY47: median and highest residues of 0.1062 and 0.3391 mg/kg, respectively, based on soya bean

IN-TMQ01: median of 0.0072 mg/kg (pea) and highest residue of 0.0178 mg/kg (soya bean)

For TTC approach

IN-UJV12: median of 0.0178 mg/kg and highest residues of 0.0651 mg/kg, based on soya bean

IN-TQD54: median of 0.0072 mg/kg (pea) and highest residues of 0.0178 mg/kg (soya bean)

Legume fodder

Bean hay (Canada PBI 14 days, n=2)

Fluazaindolizine residues were: 0.0179, 0.0305 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0501, 0.1073 mg/kg.

IN-TMQ01: 0.0179, 0.0179 mg/kg.

Residue for the TTC approach were:

IN-UJV12: 0.0323, 0.1971 mg/kg.

IN-TQD54: 0.0179, 0.0179 mg/kg.

Soya bean hay (Canada PBI 14 days, n=7)

Fluazaindolizine residues were: 0.0050, 0.0123, 0.0178, 0.0274, 0.0283, 0.0355, 0.0619 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0156, 0.0539, 0.0571, 0.0660, 0.0733, 0.1047, 0.1889 mg/kg.

IN-QZY47: 0.1867, 0.2064, 0.2950, 0.3296, 0.3415, 0.4206, 1.0709 mg/kg.

IN-TMQ01: 0.0050, 0.0051, 0.0063, 0.0109, 0.0178, 0.0178, 0.1150 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0259, 0.0467, 0.0497, 0.0669, 0.0700, 0.0748, 0.1383 mg/kg.

IN-TQD54: 0.0050, 0.0050, 0.0051, 0.0052, 0.0178, 0.0178, 0.0200 mg/kg.

Pea hay (Canada PBI 14 days, n=11)

Fluazaindolizine residues were: 0.0049, 0.0051, 0.0073, 0.0118, 0.0182, 0.0188, 0.0194, 0.0318, 0.0491, 0.0548, 0.0848 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0202, 0.0205, 0.0229, 0.0612, 0.1678, 0.1687, 0.1922, 0.1968, 0.3336, 0.4945, 0.8342 mg/kg.

IN-QZY47: 0.0072, 0.0078, 0.0279, 0.0296, 0.1144, 0.3012, 0.4005, 0.5121, 0.5662, 1.4933, 1.6035 mg/kg.

IN-TMQ01: 0.0049, 0.0051, 0.0071, 0.0072, 0.0073, 0.0073, 0.0110, 0.0114, 0.0116, 0.0118, 0.0305 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0071, 0.0072, 0.0073, 0.0073, 0.0303, 0.0462, 0.0710, 0.1438, 0.1452, 0.3478, 0.3559 mg/kg.

IN-TQD54: 0.0049, 0.0051, 0.0057, 0.0059, 0.0061, 0.0067, 0.0070, 0.0071, 0.0072, 0.0073, 0.0073 mg/kg.

Residues were highest in pea hay and soya bean hay.

Based on pea hay data, the Meeting estimated a maximum residue level of 0.17 mg/kg (dry weight basis) (assumed 88 percent dry matter) for fluazaindolizine in Legume fodders (bean, cowpea, crown vetch, Lespedeza, pea, peanut, soya bean, trefoil, vetch).

The Meeting also estimated, on a fresh weight basis:

Fluazaindolizine: median and highest residue of 0.0274 (soya bean) and 0.0848 (pea) mg/kg, respectively.

[IN-F4106+1.068×IN-A5760]: median and highest residues of 0.1687 and 0.8342 mg/kg, based on pea.

IN-QZY47: median and highest residues of 0.3296 (soya bean) and 1.6035 (pea) mg/kg,

IN-TMQ01: median and highest residues for of 0.0109 and 0.1150 mg/kg, based on soya bean

For the TTC approach

IN-UJV12: median and highest residues of 0.0669 (soya bean) and 0.3559 (pea) mg/kg

IN-TQD54: median and highest residues for of 0.0067 (pea) and 0.0200 (soya bean) mg/kg.

Cereal forages

Field corn forage (Canada PBI 14 days, n=10)

Fluazaindolizine residues were: 0.0050, 0.0050, 0.0050, 0.0050, 0.0050, 0.0067, 0.0067, 0.0067, 0.0067, 0.0068 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0141, 0.0165, 0.0190, 0.0193, 0.0201, 0.0361, 0.0441, 0.0768, 0.0881, 0.0974 mg/kg.

IN-QZY47: 0.0050, 0.0060, 0.0065, 0.0068, 0.0086, 0.0095, 0.0127, 0.0148, 0.0166, 0.0254 mg/kg.

IN-TMQ01: 0.0081, 0.0287, 0.0300, 0.0359, 0.0574, 0.0738, 0.0773, 0.0778, 0.1003, 0.1144 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050, 0.0050, 0.0050, 0.0057, 0.0067, 0.0067, 0.0067, 0.0067, 0.0068, 0.0107 mg/kg.

IN-TQD54: 0.0109, 0.0131, 0.0165, 0.0219, 0.0292, 0.0417, 0.0728, 0.1345, 0.1538, 0.1878 mg/kg.

Sorghum forage (Canada PBI 14 days, n=2)

Fluazaindolizine residues were: 0.0089, 0.0178 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0445, 0.0497 mg/kg.

IN-QZY47: 0.0222, 0.0509 mg/kg.

IN-TMQ01: 0.1671, 0.4507 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0089, 0.0178 mg/kg.

IN-TQD54: 0.0187, 0.0402 mg/kg.

Wheat forage (Canada PBI 14 days, n=13)

Fluazaindolizine residues were: 0.0050, 0.0050, 0.0050, 0.0051, 0.0067, 0.0070, 0.0072, 0.0073, 0.0073, 0.0076, 0.0178, 0.0179, 0.0179 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0139, 0.0161, 0.0194, 0.0201, 0.0214, 0.0251, 0.0270, 0.0358, 0.0470, 0.0479, 0.0525, 0.0633, 0.0654 mg/kg.

IN-QZY47: 0.0050, 0.0050, 0.0054, 0.0070, 0.0072, 0.0073, 0.0073, 0.0076, 0.0076, 0.0149, 0.0178, 0.0179, 0.0179 mg/kg.

IN-TMQ01: 0.0070, 0.0072, 0.0159, 0.0188, 0.0233, 0.0298, 0.0309, 0.0330, 0.0349, 0.0534, 0.0747, 0.0931, 0.1611 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050, 0.0050, 0.0050, 0.0050, 0.0051, 0.0070, 0.0072, 0.0073, 0.0073, 0.0076, 0.0178, 0.0179, 0.0179 mg/kg.

IN-TQD54: 0.0264, 0.0316, 0.0398, 0.0525, 0.0673, 0.0698, 0.1120, 0.1167, 0.1434, 0.1435, 0.1720, 0.2153, 0.4361 mg/kg.

Based on wheat data, the Meeting estimated a median and highest residue of 0.0072 and 0.0179 mg/kg, respectively for fluazaindolizine in cereal forages,

The Meeting also estimated (all on a fresh weight basis):

[IN-F4106+1.068×IN-A5760]: median and highest residues 0.0281 and 0.0974 mg/kg, respectively, based on maize

IN-QZY47: median and highest residues of 0.00905 and 0.0254 mg/kg, respectively, based on maize.

IN-TMQ01: median and highest residues of 0.0656 (maize) and 0.1611 (wheat) mg/kg, respectively.

Residues for the TTC approach were:

IN-UJV12: median and highest residues of 0.0072 and 0.0179 mg/kg, respectively, based on wheat

IN-TQD54: median and highest residues of 0.1120 and 0.4361 mg/kg, based on wheat.

Cereal fodder

Field corn stover (Canada PBI 14 days, n=10)

Fluazaindolizine residues were: 0.0050 (5), 0.0067 (4), 0.0068 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0219, 0.0418, 0.0544, 0.0669, 0.0676, 0.0744, 0.0751, 0.0815, 0.1422, 0.2650 mg/kg.

IN-QZY47: 0.0050, 0.0064, 0.0067, 0.0067, 0.0067, 0.0068, 0.0093, 0.0102, 0.0102, 0.0113 mg/kg.

IN-TMQ01: 0.0112, 0.0629, 0.0738, 0.0933, 0.1023, 0.1110, 0.1204, 0.1607, 0.1646, 0.2067 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0055, 0.0067, 0.0067, 0.0067, 0.0067, 0.0068, 0.0087, 0.0108, 0.0158, 0.0384 mg/kg.

IN-TQD54: 0.0165, 0.0307, 0.0649, 0.0698, 0.0765, 0.0800, 0.0804, 0.0869, 0.1073, 0.2200 mg/kg.

Sorghum straw (Canada PBI 14 days, n=2)

Fluazaindolizine residues were: 0.0089, 0.0178 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0462, 0.0592 mg/kg.

IN-QZY47: 0.0347, 0.0571 mg/kg.

IN-TMQ01: 0.3289, 0.6336 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0089, 0.0178 mg/kg.

IN-TQD54: 0.0453, 0.0754 mg/kg.

Wheat hay (Canada PBI 14 days, n=13)

Fluazaindolizine residues were: 0.0050 (3), 0.0051, 0.0055, 0.0070, 0.0072, 0.0073 (2), 0.0076, 0.0178, 0.0179 (2) mg/kg.

IN-F4106+1.068×IN-A5760: 0.0236, 0.0253, 0.0254, 0.0272, 0.0276, 0.0443, 0.0521, 0.0547, 0.0564, 0.0776, 0.0962, 0.1628, 0.2905 mg/kg.

IN-QZY47: 0.0050, 0.0050, 0.0056, 0.0070, 0.0072, 0.0073, 0.0073, 0.0076, 0.0178, 0.0179, 0.0179, 0.0201, 0.0315 mg/kg.

IN-TMQ01: 0.0070, 0.0094, 0.0172, 0.0222, 0.0357, 0.0428, 0.0485, 0.0735, 0.1447, 0.2044, 0.2150, 0.3196, 0.3469 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050, 0.0050, 0.0051, 0.0055, 0.0067, 0.0070, 0.0072, 0.0073, 0.0074, 0.0076, 0.0178, 0.0179, 0.0179 mg/kg.

IN-TQD54: 0.0577, 0.0613, 0.0796, 0.0850, 0.1031, 0.1171, 0.1459, 0.1996, 0.2995, 0.4659, 0.6117, 0.6899, 0.8673mg/kg.

Wheat straw (Canada PBI 14 days, n=13)

Fluazaindolizine residues found were: 0.0050, 0.0050, 0.0050, 0.0051, 0.0072, 0.0073, 0.0073, 0.0076, 0.0179, 0.0179, 0.0258, 0.0313, 0.0553 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0203, 0.0289, 0.0399, 0.0611, 0.0831, 0.0851, 0.1328, 0.1556, 0.1565, 0.1601, 0.2557, 0.4667, 0.5487 mg/kg.

IN-QZY47: 0.0050, 0.0050, 0.0070, 0.0072, 0.0073, 0.0073, 0.0076, 0.0144, 0.0159, 0.0178, 0.0179, 0.0179, 0.0224 mg/kg.

IN-TMQ01: 0.0165, 0.0356, 0.0495, 0.0500, 0.0682, 0.0949, 0.0986, 0.1025, 0.1820, 0.2061, 0.2467, 0.4956, 0.6133 mg/kg.

Residues for the TTC approach were:

IN-UJV12: 0.0050, 0.0050, 0.0050, 0.0050, 0.0051, 0.0070, 0.0073, 0.0073, 0.0076, 0.0136, 0.0178, 0.0179, 0.0179 mg/kg.

IN-TQD54: 0.0488, 0.0650, 0.0672, 0.1211, 0.1233, 0.1735, 0.2204, 0.2269, 0.2688, 0.2991, 0.3027, 0.4622, 0.7526 mg/kg.

The number of residue trials on sorghum was too few to allow estimation of median and highest residues for sorghum fodder. Of the crops with sufficient trials, residues were highest in maize and wheat fodders.

Based on wheat data, the Meeting estimated a maximum residue level of 0.09 mg/kg (dry weight basis) (assumed 88 percent dry matter) for fluazaindolizine in cereal straw and fodder dry,

The Meeting also estimated, based on a fresh weight basis:

Fluazaindolizine: median and highest residues of 0.0073 and 0.0553 mg/kg respectively, based on wheat.

[IN-F4106+1.068×IN-A5760]: median and highest residues of 0.1328 and 0.5487 mg/kg, respectively, based on wheat.

IN-QZY47: median and highest residues of 0.0076 and 0.0315 mg/kg, respectively, based on wheat.

IN-TMQ01: median and highest residues of 0.1067 (maize) and 0.6133 (wheat) mg/kg, respectively.

Residues for the TTC approach were:

IN-UJV12: median and highest residues of 0.0073 (wheat) and 0.0384 (maize) mg/kg, respectively.

IN-TQD54: median and highest residues of 0.2204 and 0.8673 mg/kg, respectively, based on wheat.

Group 052A Miscellaneous Feed Products with high water (≥20 percent) content

Rape seed, forage (Canada PBI 14 days, n=5)

Fluazaindolizine residues found were: 0.0071, 0.0072, 0.0073, 0.0073, 0.0075 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0198, 0.0201, 0.0205, 0.0205, 0.0209 mg/kg.

IN-QZY47: 0.0071, 0.0072, 0.0110, 0.0231, 0.0462 mg/kg.

IN-TMQ01: 0.0071, 0.0072, 0.0073, 0.0073, 0.0079 mg/kg.

Residues for the TTC approach:

IN-UJV12: 0.0071, 0.0072, 0.0073, 0.0073, 0.0075 mg/kg.

IN-TQD54: 0.0071, 0.0072, 0.0081, 0.0130, 0.0319 mg/kg.

Based on rape forage data, the Meeting estimated for oilseed forages, all on a fresh weight basis:

Fluazaindolizine: median and highest residue of 0.0073 (and 0.0075 mg/kg respectively.

[IN-F4106+1.068×IN-A5760]: median and highest residues of 0.0205 and 0.0209 mg/kg, respectively.

IN-QZY47: median and highest residues of 0.0110 and 0.0462 mg/kg, respectively.

IN-TMQ01: median and highest residues of 0.0073 and 0.0079 mg/kg, respectively.

Residues for the TTC approach:

IN-UJV12: median and highest residues of 0.0073 and 0.0075 mg/kg, respectively.

IN-TQD54 and median and highest residues of 0.0081 and 0.0319 mg/kg, respectively.

Group 052B Miscellaneous Feed Products with low water (<20 percent) content (hay, straw)

Rape seed, straw (Canada PBI 14 days, n=5)

Fluazaindolizine residues found were: 0.0073, 0.0073, 0.0075, 0.0170, 0.0251 mg/kg.

IN-F4106+1.068×IN-A5760: 0.0245, 0.0270, 0.0442, 0.0455, 0.2330 mg/kg.

IN-QZY47: 0.0073, 0.0129, 0.0181, 0.0219, 0.0777 mg/kg.

IN-TMQ01: 0.0072, 0.0073, 0.0073, 0.0190, 0.0210 mg/kg.

Residues for the TTC approach:

IN-UJV12: 0.0072, 0.0073, 0.0088, 0.0094, 0.0608 mg/kg.

IN-TQD54: 0.0266, 0.0731, 0.1018, 0.1084, 0.2968 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg (dry weight basis) for Oilseed fodders.

The Meeting also estimated, on fresh weight basis, for: Fluazaindolizine: median and highest residue of 0.0075 and 0.0251 mg/kg, respectively.

[IN-F4106+1.068×IN-A5760]: median and highest residues of 0.0442 and 0.2330) mg/kg, respectively.

IN-QZY47: median and highest residues of 0.0181 and 0.0777) mg/kg, respectively.

IN-TMQ01: median and highest residues of 0.0073 and 0.0210 mg/kg, respectively.

Residues for the TTC approach:

IN-UJV12: median and highest residues of 0.0088 and 0.0608) mg/kg , respectively.

IN-TQD54: median and highest residues of 0.1018 and 0.2968 mg/kg, respectively.

Fate of residues during processing

The Meeting received information on the fate of fluazaindolizine residues during conditions simulating commercial processing.

The hydrolytic stability of fluazaindolizine, was studied in sterile aqueous buffers at pH 4 at 90 °C for 20 minutes, pH 5 at 100 °C for 60 minutes and pH 6 at 120 °C for 20 minutes. Fluazaindolizine was not degraded and is hydrolytically stable under conditions simulating pasteurization (pH 4, 90 °C), baking/brewing/boiling (pH 5, 100 °C) and sterilization (pH 6, 120 °C).

Information on the fate of residues during processing was made available to the Meeting for strawberries, tomatoes, potatoes, soya beans, maize and wheat. In some cases residues for individual compounds were <LOQ and processing factors could not be calculated. In these cases residues are assumed to be zero.

Processing factors (PF) are estimated by dividing the residues of analyte in processed commodity by the sum of potential source residues in the raw commodity, expressed in terms of analyte.

The Table 1 summarises the processing factors for fluazaindolizine. Residues concentrated (PF> 1) on processing for dried tomato, tomato wet pomace, tomato paste and soya bean meal.

Table 192 Processing factors and median and highest residue values for fluazaindolizine used for estimation of maximum residue levels including livestock dietary burdens

Processed commodity	Raw commodity [median residue]	Raw commodity [highest residue]	Individual processing factors	Median or best estimate processing factor	Median residue-P (mg/kg)	Highest residue-P (mg/kg)
Tomato dried	0.01	0.0665	1.8 4.9	3.35	0.0335	0.2228
Tomato canned			0 0.22	0.11	0.0011	
Tomato juice			0 0.53	0.265	0.0026	
Tomato wet pomace			1 2.4	1.7	0.017	
Tomato paste			1.2 1.3	1.25	0.0125	
Tomato purée			0.53 0.8	0.665	0.0066	
Potato , flakes/granules	0.028	0.16	0 0.07	0.035	0.001	
Potato crisps			0.03 0.18	0.105	0.0029	
French fries peeled			0 0.04	0.02	0.0006	

Processed commodity	Raw commodity [median residue]	Raw commodity [highest residue]	Individual processing factors	Median or best estimate processing factor	Median residue-P (mg/kg)	Highest residue-P (mg/kg)
French fries unpeeled			0.08 0.91	0.495	0.01386	
Potato boiled unpeeled			0.07 0.15	0.11	0.00308	0.0176
Potato boiled peeled			0 0.02	0.01	0.00028	0.0016
Potato microwaved unpeeled			0.4 1.3	0.85	0.0238	0.1615
Soya bean meal (mechanically extracted)	0.0051		1.3 1.3 1.6	1.3	0.0066	
Soya bean meal (solvent extracted)			1.3 1.4 1.4	1.4	0.0071	
Soya bean hulls			0.9 0.9 1.7	0.9	0.0046	
Soya bean refined oil			0 0 0	0	0	

Using the estimated maximum residue level of 0.15 mg/kg for tomatoes and applying the processing factor of 3.35 for dried tomato, the Meeting estimated a maximum residue level of 0.5 mg/kg for tomato dried.

Inputs for estimation of dietary exposure are required for processed commodities. Table 3 summarises estimated STMR-P and HR-P values, calculated using information on the concentration of the relevant compounds on processing also summarised in the series of tables on processing that follow. The STMR-P and HR-P values were calculated as 2.26×IN-A5760+2.11×IN-F4106+1.52×IN-QZY47+1.51×IN-TMQ01.

Table 193 STMR-P and HR-P values

Processed commodity	STMR-P (mg/kg)	HR-P (mg/kg)
Strawberry juice	0.0142	0.0734
Strawberry canned	0.0081	0.0419
Strawberry jam	0.0040	0.0210
Strawberry frozen fruit	0.0121	0.0629
Strawberry, dried	0.0830	0.4297
Tomato dried	0.4624	6.6960
Tomato canned	0.0711	0.9389
Tomato juice	0.0590	0.8314
Tomato wet pomace	0.0708	1.0165
Tomato paste	0.2476	3.5309
Tomato purée	0.1268	1.8056
Potato culls	0.1022	0.8433
Potato, flakes/granules	0.0956	1.0275
Potato crisps	0.0673	0.6757
French fries peeled	0.0319	0.3213
French fries unpeeled	0.1215	1.0607

Processed commodity	STMR-P (mg/kg)	HR-P (mg/kg)
Potatoes boiled unpeeled	0.0560	0.6538
Potatoes boiled peeled	0.0343	0.3695
Potatoes baked microwaved unpeeled	0.1661	1.3600
Soya bean meal (mechanically extracted)	0.1876	
Soya bean meal (solvent extracted)	0.2118	
Soya bean hulls	0.1266	
Soya bean refined oil	0	
Maize starch	0	
Maize grits	0.0144	
Maize flour	0.0366	
Maize meal	0.0606	
Maize refined oil	0	
Wheat bran	0	
Wheat flour	0	
Wheat germ	0	

Table 194 Processing factors and median and highest residue values for [IN-F4106+1.068×IN-A5760], used as inputs to calculate total residues and for estimation of livestock dietary

Processed commodity	Raw commodity [median residue]	Raw commodity [highest residue]	Individual processing factors	Median or best estimate processing factor	Median residue-P (mg/kg)	Highest residue-P (mg/kg)
Tomato dried	0.0207	0.3385	4.6 7.5 14	7.5	0.1552	2.539
Tomato canned			0.7 0.9 1.1	0.9	0.0186	
Tomato juice			0.6 0.9 1.9	0.9	0.0186	
Tomato wet pomace			1.1 1.1 1.2	1.1	0.0228	
Tomato paste			2.6 3.9 5.7	3.9	0.0807	
Tomato purée			1.2 2.0 2.4	2	0.0414	
Potato , flakes/granules	0.0337	0.1127	0 0.1 0.6	0.1	0.0034	
Potato crisps			0.08 0.3 0.5	0.3	0.0101	
French fries peeled			0 0.04 0.2	0.04	0.0013	
French fries unpeeled			0.2 0.7 1.0	0.7	0.0236	
Potato boiled unpeeled			0.06 0.07 0.2	0.07	0.0024	0.0079
Potato boiled peeled			0 0.03 0.08	0.03	0.0010	0.0034
Potato microwaved unpeeled			0.4 1.2 1.4	1.2	0.0404	0.135
Soya bean meal (mechanically extracted)	0.0593		1.2 1.4 1.5	1.4	0.083	
Soya bean meal (solvent extracted)			1.5 1.6 1.6	1.6	0.095	

Processed commodity	Raw commodity [median residue]	Raw commodity [highest residue]	Individual processing factors	Median or best estimate processing factor	Median residue-P (mg/kg)	Highest residue-P (mg/kg)
Soya bean hulls			0.8 0.9 1.5	0.9	0.053	
Soya bean refined oil			0 0 0	0	0	
Maize starch	0.0164		0 0	0	0	
Maize grits			0 0	0	0	
Maize flour			0 2	0.1	0.00164	
Maize meal			0 1.7	0.85	0.0139	
Maize refined oil			0 0	0	0	

Table 195 Processing factors and median and highest residue values for IN-QZY47, used as inputs to calculate total residues and for estimation of livestock dietary burdens

Processed commodity	Raw commodity [median residue]	Raw commodity [highest residue]	Individual processing factors	Median or best estimate processing factor	Median residue-P (mg/kg)	Highest residue-P (mg/kg)
Tomato dried	0.01	0.058	4.4	4.4	0.044	0.2552
Tomato canned			1.2	1.2	0.012	
Tomato juice			0.7	0.7	0.007	
Tomato wet pomace			0.7	0.7	0.007	
Tomato paste			2.7	2.7	0.027	
Tomato purée			1.4	1.4	0.014	
Potato , flakes/granules	0.011	0.072	1.7 3.7	2.7	0.0297	
Potato crisps			0.6 1.2	0.9	0.0099	
French fries peeled			0.8 1.2	1	0.011	
French fries unpeeled			1.2 2.1	1.7	0.0187	
Potato boiled unpeeled			1.1 1.2	1.2	0.0132	0.0864
Potato boiled peeled			1.0 1.1	1	0.011	0.072
Potato microwaved unpeeled			1.4 1.6	1.5	0.0165	0.108
Soya bean meal (mechanically extracted)	0.0051		1.5 1.6	1.6	0.00816	
Soya bean meal (solvent extracted)			1.3 1.7	1.5	0.00765	
Soya bean hulls			1.8 1.9	1.8	0.00918	
Soya bean refined oil			0 0 0	0	0	
Maize starch	0.00675		0 0	0	0	
Maize grits			0.7 1.0	0.85	0.0057375	
Maize flour			1.3 1.6	1.45	0.0097875	

Processed commodity	Raw commodity [median residue]	Raw commodity [highest residue]	Individual processing factors	Median or best estimate processing factor	Median residue-P (mg/kg)	Highest residue-P (mg/kg)
Maize meal			1.5 1.6	1.55	0.0104625	
Maize refined oil			0 0	0	0	

Table 196 Processing factors and median and highest residue values for IN-TMQ01, used as inputs to calculate total residues and for estimation of livestock dietary burdens

Processed commodity	Raw commodity [median residue]	Raw commodity [highest residue]	Individual processing factors	Median or best estimate processing factor	Median residue-P (mg/kg)	Highest residue-P (mg/kg)
Strawberry juice	0.0123	0.0694	0.5 0.7 0.8	0.7	0.00861	
Strawberry canned			0.3 0.4 0.4	0.4	0.00492	
Strawberry jam			0.2 0.2 0.2	0.2	0.00246	
Strawberry frozen fruit			0.6 0.6 0.7	0.6	0.00738	0.0416
Strawberry dried			3.8 4.1 5.4	4.1	0.05043	0.2845
Tomato dried	0.01	0.14	4.5	4.5	0.045	0.63
Tomato canned			0.9	0.9	0.009	
Tomato juice			0.6	0.6	0.006	
Tomato wet pomace			0.8	0.8	0.008	
Tomato paste			2.4	2.4	0.024	
Tomato purée			1.2	1.2	0.012	
Potato , flakes/granules	0.0205	0.335	1.0 1.7	1.4	0.0287	
Potato crisps			0.6 1.3	1	0.0205	
French fries peeled			0.3 0.6	0.4	0.0082	
French fries unpeeled			0.8 2.0	1.4	0.0287	
Potato boiled unpeeled			0.8 1.2	1	0.0205	0.335
Potato boiled peeled			0.4 0.6	0.5	0.0102	0.168
Potato microwaved unpeeled			1.7 1.8	1.8	0.0369	0.603
Maize starch	0.00675		0 0 0	0	0	
Maize grits			0.4 0.5 0.7	0.5	0.0034	
Maize flour			0.8 1.4 1.7	1.4	0.0094	
Maize meal			1.1 1.4 1.7	1.4	0.00943	
Maize refined oil			0 0 0	0	0	

Median and highest residue values have been estimated for two compounds for which the Meeting decided to utilise the TTC approach.

Table 197 Processing factors and median and highest residue of IN-UJV12

Processed commodity	Raw commodity [median residue]	Raw commodity [highest residue]	Individual processing factors	Median or best estimate processing factor	Median residue-P (mg/kg)	Highest residue-P (mg/kg)
Tomato dried	0.01	0.01	4.7	4.7	0.047	0.047
Tomato canned			1.2	1.2	0.012	
Tomato juice			0.7	0.7	0.007	
Tomato wet pomace			0.8	0.8	0.008	
Tomato paste			2.8	2.8	0.028	
Tomato purée			1.3	1.3	0.013	
Potato , flakes/granules	0.01	0.016	1.5 3.1	2.3	0.023	
Potato crisps			0.5 1.3	0.9	0.009	
French fries peeled			0.6 1.1	0.8	0.008	
French fries unpeeled			0.8 2.0	1.4	0.014	
Potato boiled unpeeled			1.3 1.4	1.4	0.014	0.0224
Potato boiled peeled			0.7 1.1	0.9	0.014	0.0224
Potato microwaved unpeeled			1.5 1.8	1.6	0.016	0.0256
Maize starch	0.00585		0 0	0	0	
Maize grits			0 0.5	0.2	0.0012	
Maize flour			0 1.3	0.6	0.0035	
Maize meal			1 1.5	1.2	0.0070	
Maize refined oil			0 0	0	0	

Table 198 Processing factors and median and highest residues of IN-TQD54

Processed commodity	Raw commodity [median residue]	Raw commodity [highest residue]	Individual processing factors	Median or best estimate processing factor	Median residue-P (mg/kg)	Highest residue-P (mg/kg)
Strawberry juice	0.0051	0.0084	0.8 0.9	0.8	0.0041	
Strawberry canned			0 0.5	0.2	0.0010	
Strawberry jam			0 0	0	0	
Strawberry frozen fruit			0 0.5	0.2	0.0010	0.0017
Strawberry, dried			3.3 3.8	3.6	0.0184	0.0302
Tomato dried	0.01	0.061	3.5	3.5	0.035	0.2135
Tomato canned			1.1	1.1	0.011	
Tomato juice			0.6	0.6	0.006	
Tomato wet pomace			0.8	0.8	0.008	
Tomato paste			2.3	2.3	0.023	
Tomato purée			1.2	1.2	0.012	

Processed commodity	Raw commodity [median residue]	Raw commodity [highest residue]	Individual processing factors	Median or best estimate processing factor	Median residue-P (mg/kg)	Highest residue-P (mg/kg)
Potato , flakes/granules	0.01	0.0865	1.2 2.2	1.7	0.017	
Potato crisps			0.6 1.3	1	0.01	
French fries peeled			0.4 0.7	0.6	0.006	
French fries unpeeled			0.8 2.1	1.5	0.015	
Potato boiled unpeeled			1.0 1.2	1.1	0.011	0.0952
Potato boiled peeled			0.7 0.8	0.8	0.008	0.0692
Potato microwaved unpeeled			1.5 1.8	1.6	0.016	0.1384
Soya bean meal (mechanically extracted)	0.0050		0 0 0	0	0	
Soya bean meal (solvent extracted)			0 0 0	0	0	
Soya bean hulls			0 0 0.09	0	0	
Soya bean refined oil			0 0 0	0	0	
Maize starch	0.0067		0 0 0	0	0	
Maize grits			0.15 0.2 0.2	0.2	0.00134	
Maize flour			0.3 0.6 1.0	0.6	0.00402	
Maize meal			0 0.4 0.9	0.4	0.00268	
Maize refined oil			0 0 0	0	0	

Residues in animal commodities

The dietary risk assessment definition for animal commodities includes fluazaindolizine, IN-A5760, IN-F4106 and IN-TMQ01.

Farm animal feeding studies

The Meeting received a study on the transfer of fluazaindolizine to cow tissues and milk. Dairy cows were oral dosed once daily with fluazaindolizine at the equivalent of 2.3, 6.7 and 20.3 ppm in the feed for 28 days, with sacrifice 22–24 hours after the last dose.

Residues of fluazaindolizine in milk were <LOQ for the 2.3 ppm dose group, with the exception of one day-24 sample with residues of 0.01 mg/kg; however, fluazaindolizine in milk was quantifiable (≥ 0.01 mg/kg) in milk from the 6.7 and 20.3 ppm dose groups.

Fluazaindolizine residues in milk plateaued by day three, with average residues of 0.020 and 0.066 mg/kg, respectively, for the 6.7- and 20.3-ppm dose groups (milk samples from day 3–28). Fluazaindolizine residues in skim milk and cream were generally similar to or slightly lower than residue levels in whole milk. Mean fluazaindolizine residues were 0.020 mg/kg in milk, < 0.01 mg/kg in muscle, 0.020 mg/kg in fat, 0.021 mg/kg in liver and 0.091 mg/kg in kidney for the 6.7 ppm dose group. Maximum fluazaindolizine residues were <0.01 mg/kg in muscle, 0.022 mg/kg in fat, 0.023 mg/kg in liver and

0.096 mg/kg in kidney for the 6.7 ppm dose group. Mean residues in tissues showed a linear relationship with dose. Once dosing stopped, residues declined with a DT_{50} of < 0.5 days.

Residues of IN-REG72, IN-F4106, IN-A5760, IN-RYC33, and IN-R2W56 were not detected (< 0.003 mg/kg) in whole milk, skim milk, cream, and tissue samples, with the exception of IN-F4106 in cows dosed with fluazaindolizine – 6.7 ppm dose group < 0.01 mg/kg kidney, 20.3 ppm dose group < 0.01 mg/kg liver; 0.01 mg/kg kidney. IN-QEK31 was ≤ 0.01 mg/kg in all samples of milk, skim milk and cream.

In another study, lactating cows were dosed orally once daily with IN-QEK31 for 28 days at the equivalent of 19.5 ppm in the feed. IN-QEK31 was detected at above 0.01 mg/kg (LOQ) in milk and tissues. Residues in milk appeared to reach a plateau at 3 days of dosing. Mean IN-QEK31 residues were 0.203 mg/kg in milk, < 0.01 mg/kg in muscle, < 0.01 mg/kg in fat, 0.013 mg/kg in liver and 0.13 mg/kg in kidney. Maximum IN-QEK31 residues were < 0.01 mg/kg in muscle, < 0.01 mg/kg in fat, 0.016 mg/kg in liver and 0.19 mg/kg in kidney. Once dosing stopped, residues declined with a DT_{50} of <1.2 days.

IN-A5760 and IN-F4106 residues in tissues and milk may arise from feeding IN-A5760 and IN-F4106, from feeding IN-QZY47 or from feeding IN-TMQ01. Metabolism studies are available to allow estimation of the contribution from the various sources. IN-TMQ01 residues arise from the feeding of IN-TMQ01.

Farm animal dietary burden

Inputs for the livestock dietary burdens were obtained from primary treated crops (carrot culls, tomato pomace, potato culls) and from residues in rotational crops (cereal and legume forages, cereal and legume fodders, cereal grain and pulse seeds, oilseed fodder, kale, cabbage leaves, turnip roots and by-products from processing).

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the JMPR by the current Meeting. The dietary burdens, estimated using the most recent version of the OECD livestock dietary burden calculator, are presented in Annex 6 and summarised below.

Separate feeding studies were carried out in lactating cattle with fluazaindolizine and IN-QEK31 to determine residues. Metabolism studies in lactating goats were available for IN-QEK31, IN-QZY47, IN-TMQ01. For laying hens, metabolism studies were available for fluazaindolizine and IN-QEK31. The livestock dietary burdens were calculated separately for components required for dietary risk assessment:

- Fluazaindolizine;
- the sum of IN-F4106+1.068×IN-A5760
- IN-QZY47;
- IN-TMQ01;

In addition, livestock burdens were calculated for compounds being assessing using the TTC approach:

- IN-UJV12;
- IN-TQD54.

Inputs for the livestock dietary burdens were obtained from primary treated crops (carrot culls, tomato pomace, potato culls) and from residues in rotational crops (cereal and legume forages, cereal and legume fodders, cereal grain and pulse seeds, oilseed fodder, kale, cabbage leaves, turnip roots).

Residues in animal commodities from the fluazaindolizine transfer studies with lactating cows and laying hens were used in estimating maximum residue levels.

Table 199 Estimated maximum and mean dietary burdens of farm animals (fluazaindolizine)

	Animal dietary burden: fluazaindolizine, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.27	0.05	0.57	0.11	0.38	0.10	0.04	0.01
Dairy cattle	0.32	0.06	0.57 ❶ ❷	0.10	0.35	0.11 ❸ ❹	0.07	0.02
Broilers	0.01	0.002	0.23	0.02	0.01	0.01	0.01	-
Layers	0.01	0.002	0.28 ❺	0.04❻	0.01	0.01	0.01	-

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for HR estimates for mammalian tissues.

❷ Highest maximum dairy cattle dietary burden suitable for HR estimates for mammalian milk.

❸ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

❺ Highest maximum poultry dietary burden suitable for HR estimates for poultry tissues and eggs.

❻ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Additionally, metabolism studies with the various compounds were used to estimate the source of these compounds to the residues for dietary risk assessment.

Table 200 Estimated maximum and mean dietary burdens of farm animals (sum of IN-F4106+1.068×IN-A5760)

	Animal dietary burden: sum of IN-F4106+1.068×IN-A5760, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.42	0.12	0.85	0.25	0.99 ❶	0.31	0.36	0.09
Dairy cattle	0.83	0.20	0.86	0.26	0.98 ❷	0.32 ❸ ❹	0.69	0.16
Broilers	0.02	0.02	0.15	0.04	0.02	0.02	0.02	0.02
Layers	0.02	0.02	0.32❺	0.10❻	0.02	0.02	0.02	0.02

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for HR estimates for mammalian tissues.

❷Highest maximum dairy cattle dietary burden suitable for HR estimates for mammalian milk.

❸ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

❺ Highest maximum poultry dietary burden suitable for HR estimates for poultry tissues and eggs.

❻ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Table 201 Estimated maximum and mean dietary burdens of farm animals (IN-QZY47)

	Animal dietary burden: IN-QEK31, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.40	0.08	2.8	0.57	4.7❶	0.95❸	0.2	0.04
Dairy cattle	1.9	0.39	2.9	0.51	3.8❷	0.84❹	0.50	0.11
Broilers	0.01	0.01	0.42	0.03	0.01	0.01	0.01	-
Layers	0.01	0.01	1.16❺	0.19❻	0.01	0.01	0.01	-

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for HR estimates for mammalian tissues.

❷ Highest maximum dairy cattle dietary burden suitable for HR estimates for mammalian milk.

❸ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

❺ Highest maximum poultry dietary burden suitable for HR estimates for poultry tissues and eggs.

❻ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Table 202 Estimated maximum and mean dietary burdens of farm animals (IN-TMQ01)

	Animal dietary burden: IN-TMQ01, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.69	0.54	2.2❶	0.88❸	1.7	0.59	0.30	0.06
Dairy cattle	1.40	0.44	2.0❷	0.83❹	2.00	0.69	0.63	0.16
Broilers	0.01	0.01	0.50	0.17	0.01	0.01	0.01	0.01
Layers	0.01	0.01	0.78❺	0.25❻	0.01	0.01	0.01	0.01

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for HR estimates for mammalian tissues.

❷ Highest maximum dairy cattle dietary burden suitable for HR estimates for mammalian milk.

❸ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

❺ Highest maximum poultry dietary burden suitable for HR estimates for poultry tissues and eggs.

❻ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Livestock dietary burdens have been estimated for two compounds for which the Meeting decided to utilise the TTC approach.

Table 203 Estimated maximum and mean dietary burdens of farm animals (IN-UJV12)

	Animal dietary burden: IN-UJV12, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.11	0.05	0.51	0.13	1.02❶	0.19❸	0.06	0.01
Dairy cattle	0.35	0.08	0.48	0.13	0.82❷	0.17❹	0.14	0.03
Broilers	0.01	-	0.02	0.01	0.01	-	0.01	-
Layers	0.01	-	0.19❺	0.05❻	0.01	-	-	-

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for HR estimates for mammalian tissues

❷ Highest maximum dairy cattle dietary burden suitable for HR estimates for mammalian milk

❸ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

❺ Highest maximum poultry dietary burden suitable for HR estimates for poultry tissues and eggs.

❻ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Table 204 Estimated maximum and mean dietary burdens of farm animals (IN-TQD54)

	Animal dietary burden: IN-TQD54, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.21	0.07	2.34 ❶	0.50 ❸	2.16	0.45	0.44	0.11
Dairy cattle	1.52	0.33	2.02 ❷	0.44 ❹	2.85	0.58	1.15	0.29
Broilers	0.01	-	0.05	0.01	0.005	0.004	0.006	0.004
Layers	0.007	0.001	0.46❺	0.10❻	0.005	0.004	0.007	0.004

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for HR estimates for mammalian tissues and milk

❷ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues and milk.

❸ Highest maximum poultry dietary burden suitable for HR estimates for poultry tissues and eggs.

❹ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

❺ Highest maximum poultry dietary burden suitable for HR estimates for poultry tissues and eggs.

❻ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Animal commodity maximum residue levels

Cattle

The calculations used to estimate highest residues for use in estimating maximum residue levels, STMR and HR values are shown below.

Table 205 Animal commodity maximum residue levels for cattle

	Feed Level (ppm) for milk residues	Fluazaindolizine residues (mg/kg) in milk	Feed Level (ppm) for tissue residues	Fluazaindolizine residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest residue for maximum residue level estimation (beef or dairy cattle)							
Feeding Study	6.7	0.0199	6.7	< 0.01	0.0233	0.0957	0.0217
Dietary burden and estimate of highest residue	0.57	0.0017	0.57	0.00085	0.0020	0.0081	0.0018
Median Determination (beef or dairy cattle)							
Feeding Study	6.7	0.0199	6.7	< 0.01	0.0223	0.0912	0.0195
Dietary burden and estimate of median residue	0.11	0.00033	0.11	0.00016	0.00037	0.0015	0.00032

The Meeting estimated the following maximum residue levels: milk 0.01(*) mg/kg; meat (mammalian except marine mammals) 0.01(*) mg/kg, mammalian fat (except milk fat) 0.01(*) mg/kg and edible offal 0.01 mg/kg.

To estimate HR and STMR values for animal commodities, the residues in the following tables are combined according to the residue definition for risk assessment (total = fluazaindolizine + 2.11×(IN-F4106+1.068×IN-A5760) + 1.51×IN-TMQ01).

The Meeting estimated STMRs for fluazaindolizine of 0.0096 mg/kg in mammalian muscle, of 0.0098 mg/kg in mammalian fat, of 0.2217 in mammalian offal (based on liver) and of 0.0033 mg/kg in milk

The Meeting estimated HRs of 0.0415 mg/kg in mammalian muscle, 0.0431 mg/kg in mammalian fat and 0.7592 mg/kg in mammalian offal (based on kidney).

Residues of the fluazaindolizine were similar in milk fat compared to whole milk. Using this information, the Meeting estimated a maximum residue level of 0.01(*) mg/kg for milk fat and an STMR value of 0.0033 mg/kg.

Extrapolation of residues in the IN-QZY47 metabolism study with a lactating goat were used to estimate residues of IN-F4106 and IN-A5760.

Table 206 Residues of IN-F4106+1.068×IN-A5760 from feeding IN-F4106+1.068×IN-A5760

	Feed Level (ppm) for milk residues	IN-F4106+ 1.068×IN-A5760 (mg eq/kg) in milk	Feed Level (ppm) for tissue residues	IN-F4106+1.068×IN-A5760 residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest Determination (beef or dairy cattle)							
Metabolism Study	7.61*	0.00794	7.61*	0.03319	0.2054	0.5764	0.0336
Dietary burden and estimate of highest residue	0.98	0.0010 (+0.00090 +0.0028)	0.99	0.0043 (+0.00013 +0.0147)	0.0267 (+0.00029 +0.0911)	0.0750 (+0.00032 +0.2556)	0.0044 (+0.00005 +0.0149)
Median Determination (beef or dairy cattle)							
Metabolism Study	7.61*	0.00794	7.61*	0.03319	0.2054	0.5764	0.0336
Dietary burden and estimate of median residue	0.32	0.00033 (+0.00037 +0.00063)	0.32	0.0014 (+0.00005 +0.0030)	0.0086 (+0.00012 +0.0184)	0.0242 (+0.00013 +0.0517)	0.0014 (+0.00002 +0.0030)

*= IN-QZY47 metabolism dose in terms of IN-F4106.

*Additional IN-F4106 is produced from IN-TMQ01 and IN-QZY47, the figures in brackets are the contribution from livestock exposure to IN-TMQ01 and to IN-QZY47. Residues of IN-F4106+1.068×IN-A5760 are the sum of the three sources.

Table 207 Residues of IN-F4106+1.068×IN-A5760 from feeding TMQ01

	Feed Level (ppm) for milk residues	IN-F4106+1.068×IN-A5760 (mg/kg) in milk	Feed Level (ppm) for tissue residues	IN-F4106+1.068×IN-A5760 (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest Determination (beef or dairy cattle)							
Metabolism Study	10.9	0.004885	10.9	0.000624	0.001443	0.001574	0.00023
Dietary burden and estimate of highest residue	2.0	0.00090	2.2	0.00013	0.00029	0.00032	0.00005
Median Determination (beef or dairy cattle)							
Metabolism Study	10.9	0.004885	10.9	0.000624	0.001443	0.001574	0.00023
Dietary burden and estimate of median residue	0.83	0.00037	0.88	0.00005	0.00012	0.00013	0.00002

Table 208 Residues of IN-F4106+1.068×IN-A5760 from feeding IN-QZY47

	Feed Level (ppm) for milk residues	IN-F4106+1.068×IN-A5760 (mg/kg) in milk	Feed Level (ppm) for tissue residues	IN-F4106+1.068×IN-A5760 (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest Determination (beef or dairy cattle)							
Metabolism Study	10.6	0.0079	10.6	0.0332	0.2054	0.5764	0.0336
Dietary burden and estimate of highest residue	3.8	0.0028	4.7	0.0147	0.0911	0.2556	0.0149
Median Determination (beef or dairy cattle)							
Metabolism Study	10.6	0.0079	10.6	0.0332	0.2054	0.5764	0.0336
Dietary burden and estimate of median residue	0.84	0.00063	0.95	0.0030	0.0184	0.0517	0.0030

A metabolism study with IN-TMQ01 in a lactating goat was used to estimate residues of IN-TMQ01.

Table 209 Residues of IN-TMQ01

	Feed Level (ppm) for milk residues	IN-TMQ01 (mg/kg) in milk	Feed Level (ppm) for tissue residues	IN-TMQ01 (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest Determination (beef or dairy cattle)							
Metabolism Study	10.9	< 0.001	10.9	0.000854	0.009996	0.19074	0.001485
Dietary burden and estimate of highest residue	2.0	0.00018	2.0	0.00016	0.0018	0.0350	0.00027
Median Determination (beef or dairy cattle)							

	Feed Level (ppm) for milk residues	IN-TMQ01 (mg/kg) in milk	Feed Level (ppm) for tissue residues	IN-TMQ01 (mg/kg)			
				Muscle	Liver	Kidney	Fat
Metabolism Study	10.9	< 0.001	10.9	0.000854	0.009996	0.19074	0.001485
Dietary burden and estimate of median residue	0.57	0.00008	0.57	0.00004	0.00052	0.0100	0.00008

Median and highest residue values have been estimated for for IN-UJV12 and IN-TQD54, for which the Meeting decided to apply the TTC approach.

Extrapolation of residues in the IN-QZY47 metabolism study with a lactating goat were used to estimate residues of IN-UJV12.

Table 210 Residues of IN-UJV12

	Feed Level (ppm) for milk residues	IN-UJV12 (mg/kg) in milk	Feed Level (ppm) for tissue residues	IN-UJV12 (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest Determination (beef or dairy cattle)							
Metabolism Study	10.1*	0.000488	10.1*	< 0.001	< 0.001	< 0.001	< 0.001
Dietary burden and estimate of highest residue	0.82	0.00004	1.02	0.0001	0.0001	0.0001	0.0001
Median Determination (beef or dairy cattle)							
Metabolism Study	10.1*	0.000488	10.1*	< 0.001	< 0.001	< 0.001	< 0.001
Dietary burden and estimate of median residue	0.17	0.000008	0.19	0.00002	0.00002	0.00002	0.00002

Note:

*=Expressed IN-QZY47 metabolism dose in terms of IN-UJV12.

Extrapolation of residues in the IN-QZY47 metabolism study with a lactating goat were used to estimate residues of IN-TQD54.

Table 211 Residues of IN-TQD54

	Feed Level (ppm) for milk residues	IN-TQD54 (mg/kg) in milk	Feed Level (ppm) for tissue residues	IN-TQD54 (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest Determination (beef or dairy cattle)							
Metabolism Study	10.15*	0.000488	10.15*	< 0.001	< 0.001	< 0.001	< 0.001
Dietary burden and estimate of highest residue	2.85	0.00014	2.85	0.00028	0.00028	0.00028	0.00028
Median Determination (beef or dairy cattle)							
Metabolism Study	10.15*	0.000488	10.15*	< 0.001	< 0.001	< 0.001	< 0.001

	Feed Level (ppm) for milk residues	IN-TQD54 (mg/kg) in milk	Feed Level (ppm) for tissue residues	IN-TQD54 (mg/kg)			
				Muscle	Liver	Kidney	Fat
Dietary burden and estimate of median residue	0.58	0.00003	0.58	0.00006	0.00006	0.00006	0.00006

Note:

*=Expressed IN-QZY47 metabolism dose in terms of IN-TQD54.

Poultry

The calculations used to estimate maximum residue levels, as well as total residues for use in estimating STMR and HR values are shown below.

Table 212 Residues of fluazaindolizine

	Feed Level (ppm) for egg residues	Fluazaindolizine residues (mg/kg) in eggs	Feed Level (ppm) for tissue residues	Fluazaindolizine residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest residue for maximum residue level estimation (broilers or layers)							
Metabolism Study	13.1	0.013056	13.1	0.041495	0.032		0.0135
Dietary burden and estimate of highest residue	0.28	0.000279	0.28	0.000887	0.0146		0.00029
Median Determination (broilers or layers)							
Feeding Study	13.1	0.013056	13.1	0.041495	0.032		0.0135
Dietary burden and estimate of median residue	0.04	0.00004	0.04	0.00013	0.0021		0.00004

The Meeting estimated maximum residue levels of 0.01(*) mg/kg for eggs poultry meat and poultry fat 0.01(*) mg/kg and of 0.02 mg/kg for poultry edible offal (liver.

To estimate HR and STMR values, the residues in the following tables are combined in fluazaindolizine equivalents according to the residue definition for risk assessment (total = fluazaindolizine + 2.11×(IN-F4106+1.068×IN-A5760) + 1.51×IN-TMQ01).

The Meeting estimated STMRs of 0.0021 mg/kg for poultry muscle, of 0.00093 mg/kg for poultry fat, of 0.035 mg/kg poultry edible offal (liver) and of 0.0008 mg/kg in eggs.

The Meeting estimated HRs of 0.00708 mg/kg for poultry muscle, of 0.0032 mg/kg for poultry fat, of 0.1182 mg/kg for poultry edible offal (liver) and of 0.00263 mg/kg for eggs.

Extrapolation of TRRs in the fluazaindolizine metabolism study with laying hens was used to estimate residues of IN-F4106 + 1.068×IN-A5760. The dose level and TRR in the fluazaindolizine metabolism study were converted to IN-F4106 equivalents.

Table 213 Residues of IN-F4106+1.068×IN-A5760

	Feed Level (ppm) for egg residues	IN-F4106+1.068×IN-A5760 (mg/kg) in eggs	Feed Level (ppm) for tissue residues	IN-F4106+1.068×IN-A5760 residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest Determination (broilers or layers)							
Metabolism Study	6.2*	0.008048	6.2*	0.020356	0.346529		0.009468
Dietary burden and estimate of highest residue	0.32	0.0004	0.32	0.0011	0.0179		0.0005
Median Determination (broilers or layers)							
Metabolism Study	6.2*	0.008048	6.2*	0.020356	0.346529		0.009468
Dietary burden and estimate of median residue	0.1	0.00013	0.1	0.00033	0.00559		0.00015

Note:

* Dose in the fluazaindolizine metabolism study expressed in IN-F4106 equivalents.

Extrapolation of TRRs in the fluazaindolizine metabolism study with laying hens was used to estimate residues of IN-TMQ01. The dose level and TRR were converted to IN-TMQ01 equivalents.

Table 214 Residues of IN-TMQ01

	Feed Level (ppm) for egg residues	IN-TMQ01 (mg/kg) in eggs	Feed Level (ppm) for tissue residues	IN-TMQ01 (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest Determination (broilers or layers)							
Metabolism Study	8.67	0.0112	8.67	0.0284	0.484		0.0132
Dietary burden and estimate of highest residue	0.78	0.001	0.78	0.00256	0.0436		0.0012
Median Determination (broilers or layers)							
Metabolism Study	8.67	0.0112	8.67	0.0284	0.484		0.0132
Dietary burden and estimate of median residue	0.25	0.00032	0.25	0.00082	0.0140		0.00038

Median and highest residue values have been estimated for for IN-UJV12 and IN-TQD54, for which the Meeting decided to apply the TTC approach.

Extrapolation of TRRs in the fluazaindolizine metabolism study with laying hens was used to estimate residues of IN-UJV12. The dose level and TRR were converted to IN-UJV12 equivalents.

Table 215 Residues of IN-UJV12

	Feed Level (ppm) for egg residues	IN-UJV12 (mg/kg) in eggs	Feed Level (ppm) for tissue residues	IN-UJV12 (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest Determination (broilers or layers)							
Metabolism Study	8.24	0.0107	8.25	0.0271	0.4607		0.0126
Dietary burden and estimate of highest residue	0.19	0.00025	0.19	0.00062	0.0106		0.00029
Median Determination (broilers or layers)							
Metabolism Study	8.24	0.0107	8.25	0.0271	0.4607		0.0126
Dietary burden and estimate of median residue	0.05	0.00006	0.05	0.00016	0.0028		0.00007

Extrapolation of TRRs in the fluazaindolizine metabolism study with laying hens was used to estimate residues of IN-TQD54. The dose level and TRR were converted to IN-TQD54 equivalents.

Table 26 Residues of IN-TQD54

	Feed Level (ppm) for egg residues	IN-TQD54 (mg/kg) in eggs	Feed Level (ppm) for tissue residues	IN-TQD54 (mg/kg)			
				Muscle	Liver	Kidney	Fat
Highest Determination (broilers or layers)							
Metabolism Study	8.27	0.0107	8.27	0.0272	0.4623		0.0126
Dietary burden and estimate of highest residue	0.46	0.00060	0.46	0.0015	0.026		0.00070
Median Determination (broilers or layers)							
Metabolism Study	8.27	0.0107	8.27	0.0272	0.4623		0.0126
Dietary burden and estimate of median residue	0.10	0.00013	0.10	0.00033	0.0056		0.00015

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL for plant and animal commodities: *fluazaindolizine*.

Definition of the residue for dietary risk assessment for plant commodities:

fluazaindolizine, and free and conjugated forms of the following compounds: 2-chloro-5-hydroxybenzenesulfonamide (IN-A5760), 2-chloro-5-methoxybenzenesulfonamide (IN-F4106), 8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylic acid (IN-QEK31), 3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-L-alanine (IN-QZY47), 8-chloro-N-[(2-chloro-5-hydroxyphenyl)sulfonyl]-6-

(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide (IN-REG72), 8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxamide (IN-RYC33) and 3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-(2R)-hydroxypropanoic acid (IN-TMQ01) (expressed as fluazaindolizine). *This can be implemented by taking the maximum of the sum of compounds containing the imidazopyridine ring and hydrolysed using acid to IN-A5760, IN-F4106, IN-QZY47 and IN-TMQ01 (expressed as fluazaindolizine)* ***OR*** *compounds containing the phenyl ring and hydrolysed to 8-chloro-6-(trifluoromethyl)imidazo[1,2-a]pyridine-2-carboxylic acid (IN-QEK31) (expressed as fluazaindolizine).*

Definition of the residue for dietary risk assessment for animal commodities: the sum of fluazaindolizine, 2-chloro-5-hydroxybenzenesulfonamide (IN-A5760), 2-chloro-5-methoxybenzenesulfonamide (IN-F4106), and 3-[[(2-chloro-5-methoxyphenyl)sulfonyl]amino]-(2R)-hydroxypropanoic acid (IN-TMQ01) (expressed as fluazaindolizine).

The residue is not fat-soluble.

Table 217 Residue levels suitable for establishing maximum residue limits and for IEDI and IESTI assessments

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VC2039	Cucumbers and summer squashes, Subgroup of	0.15		0.1092	0.3674
VC2040	Melons, pumpkins and winter squashes, Subgroup of	0.1		0.1253	0.3395
VO2045	Tomato, Subgroup of	0.15		0.0748	0.963
VO2046	Subgroup of Eggplant	0.15		0.1348	0.3937
VO0051	Peppers, Subgroup of (except martynia, okra, roselle)	0.03		0.074	0.3102
HS0444	Peppers, Chili, dried	0.3		0.74	3.102
VR 0577	Carrot	0.4		0.1485	1.973
VR2071	Tuberous and corm vegetables, Subgroup of	0.2		0.1231	0.7356
FB 0275	Strawberry	0.015		0.0530	0.1416
VB 0040	Brassica vegetables (except Brassica leafy vegetables), Group of	0.02		0.04335	0.0705
VL0053	Leafy vegetables (including Brassica leafy vegetables, Group of	0.04		0.3880	1.388
VP0060	Legume vegetables, Group of [immature seeds with pods]	0.04		0.0709	0.1589
VD 0070	Pulses, Group of	0.09		0.0656	
VR 0075	Root vegetables, Group of [except carrot]	0.04		0.1935	0.9322
VS 0078	Stalk and stem vegetables, Group of	0.04		0.0674	0.8281
VA 0035	Bulb vegetables, Group of	0.04		0.0674	0.8281
GC 0080	Cereal grains, Group of	0.03		0.0676	
SO 0088	Oilseeds and oilfruits, Group of	0.04		0.0656	
MO 0105	Edible offal (Mammalian)	0.01		0.2217 (liver)	0.7592 (kidney)
MF 0100	Mammalian fats (except milk fats)	0.01*		0.0098	0.0431
MM 0095	Meat (from mammals other than marine mammals)	0.01*		0.0096	0.0415
ML 0106	Milks	0.01*		0.0033	0.0119
FM0183	Milk fats	0.01*		0.0033	0.0119
PE0112	Eggs	0.01*		0.0008	0.00263
PO0111	Poultry, Edible offal of	0.02		0.035 (liver)	0.1182 (liver)
PF0111	Poultry fats	0.01*		0.00093	0.0032
PM 0110	Poultry meat	0.01*		0.0021	0.0071
AS 0081	Straw and fodder (dry) of cereal grains	0.09 (dw)			

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
AL0157	Legume animal feeds, Group of	0.17 (dw)			
AM 3583	Rape seed, hay and/or straw	0.05 (dw)			
	Strawberry juice			0.0142	0.0734
	Strawberry canned			0.0081	0.0419
	Strawberry jam			0.0040	0.0210
DF 0275	Strawberry, dried			0.0830	0.4297
DV 0448	Tomato, dried	0.5		0.4624	6.6960
	Tomato canned			0.0711	0.9389
JF 0448	Tomato, juice			0.0590	0.8314
DM 3525	Tomato, pomace			0.0708	1.0165
	Tomato, paste			0.2476	3.5309
DM 3525	Tomato, purée			0.1268	1.8056
	Potato culls			0.1022	0.8433
DV 0589	Potato, flakes/granules			0.0956	1.0275
	Potato crisps			0.0673	0.6757
	French fries peeled			0.0319	0.3213
	French fries unpeeled			0.1215	1.0607
	Potatoes boiled unpeeled			0.0560	0.6538
	Potatoes boiled peeled			0.0343	0.3695
	Potatoes baked microwaved unpeeled			0.1661	1.3600
	Soya bean meal (mechanically extracted)			0.1876	
	Soya bean meal (solvent extracted)			0.2118	
AL 3538	Soya bean hulls			0.1266	
OR 0541	Soya bean oil, refined			0	
	Maize starch			0	
	Maize grits			0.0144	
CF 1255	Maize flour			0.0366	
	Maize meal			0.0606	
	Maize refined oil			0	
	Wheat bran (unprocessed)			0	
CF 1211	Wheat, flour			0	
CF 1210	Wheat, germ			0	

Note:

(as) – As received; (dw) – dry weight.

[a] residues resulting from rotational cropping.

Table 218 Residues of fluazaindolizine in livestock feeds (parent only)

CCN	Commodity	Median mg/kg	Highest mg/kg
VR 0577	Carrot	0.01	0.265
VR2071	Potato	0.028	0.16
VL0053	Leafy vegetables (including Brassica leafy vegetables, Group of	0.0178	0.0179
VD 0070	Pulses, Group of		
	Turnip roots	0.0077	0.0106
GC 0080	Cereal grains, Group of	0.0072	
AS 3303	Cereal grains (including pseudocereals) feed products with high water (≥20 percent) content (forage and silage), Subgroup of	0.0072	0.0179
AS 0081	Straw and hay of cereal grains	0.0073	0.0553
AL 3300	Products of legume feeds with high water (≥20 percent) content (forage and silage)	0.0107	0.0178
AL 3301	Products of legume feeds with low water (<20 percent) content (hay	0.0274	0.0848

CCN	Commodity	Median mg/kg	Highest mg/kg
VD 0070	Pulses, Group of	0.0094	
AM 0495	Rape seed, forage	0.0073	0.0075
	Tomato wet pomace	0.0125	
AL 3539	Soya bean meal	0.00714	
AL 3539	Soya bean hulls	0.00459	

Note:

(as) – As received; (dw) – dry weight; [a] residues resulting from rotational cropping.

Table 219 Residues of IN-F4106+1.068×IN-A5760 in livestock feeds (as IN-F4106)

CCN	Commodity	Median mg/kg	Highest mg/kg
VR 0577	Carrot	0.0207	0.1489
VR2071	Potato	0.0337	0.1127
VL0053	Leafy vegetables (including Brassica leafy vegetables, Group of	0.0501	0.0954
VD 0070	Pulses, Group of	0.0202	
	Turnip roots	0.0142	0.0143
GC 0080	Cereal grains, Group of	0.0204	
AS 3303	Cereal grains (including pseudocereals) feed products with high water (≥20 percent) content (forage and silage), Subgroup of	0.0281	0.0974
AS 0081	Straw and hay of cereal grains	0.1328	0.5487
AL 3300	Products of legume feeds with high water (≥20 percent) content (forage and silage)	0.0214	0.0509
AL 3301	Products of legume feeds with low water (<20 percent) content (hay	0.1687	0.8342
AM 0495	Rape seed, forage	0.0205	0.0209
	Tomato wet pomace	0.0228	
CF 0645	Maize, meal	0.0139	
AL 3539	Soya bean meal	0.0226	
AL 3539	Soya bean hulls	0.0127	

Note:

(as) – As received; (dw) – dry weight; [a] residues resulting from rotational cropping.

Table 220 Residues of IN-QZY47 in livestock feeds (as IN-QZY47)

CCN	Commodity	Median mg/kg	Highest mg/kg
VR 0577	Carrot	0.031	0.5
VR2071	Potato	0.011	0.072
VL0053	Leafy vegetables (including Brassica leafy vegetables, Group of	0.07955	0.2276
VD 0070	Pulses, Group of	0.0084	
	Turnip roots	0.0179	0.047
GC 0080	Cereal grains, Group of	0.0076	
AS 3303	Cereal grains (including pseudocereals) feed products with high water (≥20 percent) content (forage and silage), Subgroup of	0.00905	0.0254
AS 0081	Straw and hay of cereal grains	0.0072	0.0315
AL 3300	Products of legume feeds with high water (≥20 percent) content (forage and silage)	0.1062	0.3391
AL 3301	Products of legume feeds with low water (<20 percent) content (hay	0.3296	1.6035
AM 0495	Rape seed, forage	0.011	0.0462

Note:

(as) – As received; (dw) – dry weight; [a] residues resulting from rotational cropping.

Table 221 Residues of IN-TMQ01 in livestock feeds (as IN-TMQ01)

CCN	Commodity	Median mg/kg	Highest mg/kg
VR 0577	Carrot	0.045	0.595
VR2071	Potato	0.335	0.3685
VL0053	Leafy vegetables (including Brassica leafy vegetables, Group of	0.1472	0.4819
VD 0070	Pulses, Group of	0.0051	
	Turnip roots	0.0051	0.0051
GC 0080	Cereal grains, Group of	0.0073	
AS 3303	Cereal grains (including pseudocereals) feed products with high water (≥20 percent) content (forage and silage), Subgroup of	0.0656	0.1611
AS 0081	Straw and hay of cereal grains	0.1067	0.6133
AL 3300	Products of legume feeds with high water (≥20 percent) content (forage and silage)	0.0072	0.0178
AL 3301	Products of legume feeds with low water (<20 percent) content (hay	0.0109	0.115
AM 0495	Rape seed, forage	0.0073	0.0079
	Tomato wet pomace	0.008	
CF 0645	Maize, meal	0.00945	

Note:

(as) – As received; (dw) – dry weight; [a] residues resulting from rotational cropping.

Table 222 Residues of IN-UJV12 in foods (as IN-UJV12)

CCN	Commodity	Median mg/kg	Highest mg/kg
VC2039	Cucumbers and summer squashes, Subgroup of	0.01	0.021
VC2040	Melons, pumpkins and winter squashes, Subgroup of	0.01	0.0125
VO2045	Tomato, Subgroup of	0.01	0.01
VO2046	Subgroup of Eggplant	0.01	0.01
VO0051	Peppers, Subgroup of (except martynia, okra, roselle)	0.01	0.01
HS0444	Peppers, Chili, dried	0.1	0.1
VR 0577	Carrot	0.01	0.01
VR2071	Tuberous and corm vegetables, Subgroup of	0.01	0.016
FB 0275	Strawberry	0.0051	0.0051
VB 0040	Brassica vegetables (except Brassica leafy vegetables), Group of	0.005	0.0051
VL0053	Leafy vegetables (including Brassica leafy vegetables, Group of	0.0182	0.0602
VP0060	Legume vegetables, Group of	0.00565	0.0178
VD 0070	Pulses, Group of	0.0073	0.074
VR 0075	Root vegetables, Group of [except carrot]	0.0178	0.0245
VS 0078	Stalk and stem vegetables, Group of	0.0051	0.0088
VA 0035	Bulb vegetables, Group of	0.0051	0.0088
GC 0080	Cereal grains, Group of	0.0072	
SO 0088	Oilseeds and oilfruits, Group of	0.0073	
MO 0105	Edible offal (Mammalian)	0.00002	0.0001
MF 0100	Mammalian fats (except milk fats)	0.00002	0.0001
MM 0095	Meat (from mammals other than marine mammals)	0.00002	0.0001
ML 0106	Milks	0.000008	0.00004
FM0183	Milk fats	0.000008	0.00004
PE0112	Eggs	0.00006	0.00025
PO0111	Poultry, Edible offal of	0.0028	0.0106
PF0111	Poultry fats	0.00007	0.00029
PM 0110	Poultry meat	0.00016	0.00062

CCN	Commodity	Median mg/kg	Highest mg/kg
DV 0448	Tomato, dried	0.047	0.047
	Tomato canned	0.012	
JF 0448	Tomato, juice	0.007	
DM 3525	Tomato, pomace	0.008	
	Tomato, paste	0.028	
DM 3525	Tomato, purée	0.013	
DV 0589	Potato , flakes/granules	0.023	
	Potato crisps	0.009	
	French fries peeled	0.008	
	French fries unpeeled	0.014	
	Potato boiled unpeeled	0.014	0.0224
	Potato boiled peeled	0.014	0.0224
	Potato microwaved unpeeled	0.016	0.0256
	Maize starch	0	
	Maize grits	0.0012	
CF 1255	Maize flour	0.0035	
CF 0645	Maize meal	0.0070	
OR 0645	Maize oil, edible	0	

Note:

(as) – As received; (dw) – dry weight; [a] residues resulting from rotational cropping.

Table 223 Residues of IN-UJV12 in livestock feeds (as IN-UJV12)

CCN	Commodity	Median mg/kg	Highest mg/kg
VR 0577	Carrot	0.01	0.01
VR2071	Potato	0.016	0.0205
VL0053	Leafy vegetables (including Brassica leafy vegetables, Group of	0.0182	0.602
VD 0070	Pulses, Group of	0.0073	
	Turnip roots	0.005	0.0051
GC 0080	Cereal grains, Group of	0.0072	
AS 3303	Cereal grains (including pseudocereals) feed products with high water (≥20 percent) content (forage and silage), Subgroup of	0.0072	0.0179
AS 0081	Straw and hay of cereal grains	0.0073	0.0384
AL 3300	Products of legume feeds with high water (≥20 percent) content (forage and silage)	0.0178	0.0651
AL 3301	Products of legume feeds with low water (<20 percent) content (ha	0.0669	0.3559
AM 0495	Rape seed, forage	0.0072	0.0073
	Tomato wet pomace	0.008	
CF 0645	Maize meal	0.00702	

Note:

(as) – As received; (dw) – dry weight; [a] residues resulting from rotational cropping.

Table 224 Residues of IN-TQD54 foods (as IN-TQD54)

CCN	Commodity	Median mg/kg	Highest mg/kg
VC2039	Cucumbers and summer squashes, Subgroup of	0.01	0.01
VC2040	Melons, pumpkins and winter squashes, Subgroup of	0.01	0.0125
VO2045	Tomato, Subgroup of	0.01	0.061
VO2046	Subgroup of Eggplant	0.01	0.061

CCN	Commodity	Median mg/kg	Highest mg/kg
VO0051	Peppers, Subgroup of (except martynia, okra, roselle)	0.01	0.0375
HS0444	Peppers, Chili, dried	0.1	0.375
VR 0577	Carrot	0.01	0.049
VR2071	Tuberous and corm vegetables, Subgroup of	0.01	0.0865
FB 0275	Strawberry	0.0050	0.0084
VB 0040	Brassica vegetables (except Brassica leafy vegetables), Group of	0.0050	0.0051
VL0053	Leafy vegetables (including Brassica leafy vegetables, Group of	0.1175	0.6782
VP0060	Legume vegetables, Group of [immature seeds and pods]	0.0050	0.0178
VD 0070	Pulses, Group of	0.0051	0.0178
VR 0075	Root vegetables, Group of [except carrot]	0.0178	0.2008
VS 0078	Stalk and stem vegetables, Group of	0.0051	0.0121
VA 0035	Bulb vegetables, Group of	0.0051	0.0121
GC 0080	Cereal grains, Group of	0.0073	
SO 0088	Oilseeds and oilfruits, Group of	0.0073	
MO 0105	Edible offal (Mammalian)	0.00006	0.00028
MF 0100	Mammalian fats (except milk fats)	0.00006	0.00028
MM 0095	Meat (from mammals other than marine mammals)	0.00006	0.00028
ML 0106	Milks	0.00003	0.00014
FM0183	Milk fats	0.00003	0.00014
PE0112	Eggs	0.00013	0.0006
PO0111	Poultry, Edible offal of	0.0056	0.026
PF0111	Poultry fats	0.00015	0.00070
PM 0110	Poultry meat	0.00033	0.0015
	Strawberry juice	0.0041	
	Strawberry canned	0.0010	
	Strawberry jam	0	
DF 0275	Strawberry, dried	0.0184	0.0302
DV 0448	Tomato dried	0.035	0.2135
	Tomato canned	0.011	
JF 0448	Tomato juice	0.006	
DM 3525	Tomato wet pomace	0.008	
	Tomato paste	0.023	
DM 3525	Tomato purée	0.012	
DV 0589	Potato , flakes/granules	0.017	
	Potato crisps	0.01	
	French fries peeled	0.006	
	French fries unpeeled	0.015	
	Potato boiled unpeeled	0.011	0.0952
	Potato boiled peeled	0.008	0.0692
	Potato microwaved unpeeled	0.016	0.1384
	Soya bean meal (mechanically extracted)	0	
	Soya bean meal (solvent extracted)	0	
AL 3538	Soya bean hulls	0	
	Soya bean refined oil	0	
	Maize starch	0	
	Maize grits	0.00134	
CF 1255	Maize flour	0.00402	
CF 0645	Maize meal	0.00268	
OR 0645	Maize oil, edible	0	

Note:

(as) – As received; (dw) – dry weight; [a] residues resulting from rotational cropping.

Table 225 Residues of IN-TQD54 in livestock feeds (as IN-TQD54)

CCN	Commodity	Median mg/kg	Highest mg/kg
VR 0577	Carrot	0.01	0.049
VR2071	Potato	0.01	0.01
VL0053	Leafy vegetables (including Brassica leafy vegetables, Group of	0.1175	0.6782
VD 0070	Pulses, Group of	0.0051	
	Turnip roots	0.005	0.0051
GC 0080	Cereal grains, Group of	0.0073	
AS 3303	Cereal grains (including pseudocereals) feed products with high water (≥20 percent) content (forage and silage), Subgroup of	0.112	0.4361
AS 0081	Straw and fodder (dry) of cereal grains	0.2204	0.8673
AL 3300	Products of legume feeds with high water (≥20 percent) content (forage and silage)	0.0072	0.0178
AL 3301	Products of legume feeds with low water (<20 percent) content (hay)	0.0067	0.02
AM 0495	Rape seed, forage	0.0102	0.0319
	Tomato wet pomace	0.008	
CF 0645	Maize meal	0.00268	
AL 3539	Soya bean meal	0	
AL 3538	Soya bean hulls	0	

Note:

(as) – As received; (dw) – dry weight; [a] residues resulting from rotational cropping.

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for fluazaindolizine is 0–0.3 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for fluazaindolizine were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 0–1 percent of the maximum ADI.

Acute dietary exposure

The ARfD for fluazaindolizine is 1 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for fluazaindolizine were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs varied from 0–9 percent of the ARfD for children and 0–5 percent of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of fluazaindolizine from uses considered by the present Meeting is unlikely to present a public health concern.

Threshold of toxicological concern (TTC) consideration for metabolites

The Meeting agreed that metabolites IN-UJV12 and IN-TDQ54 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day).

The current Meeting estimated dietary exposures of 0.14–0.30 µg/kg bw per day for IN-UJV12 and of 0.15–0.44 µg/kg bw per day for IN-TQD54.

The Meeting concluded that the estimated dietary exposures to residues of IN-UJV12 and IN-TQD54 from uses considered by the JMPR are below the TTC for Cramer Class III compounds and are unlikely to present a public health concern. Should further uses be considered in the future, these conclusions may need to be re-evaluated.

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DuPont-42585	Manikandan, K.N.	2015	14C-IN-F4106: Laboratory study of hydrolysis as a function of pH. GLP, unpublished
DuPont-42610	Ballard, T.D.	2018	FDA PAM multiresidue method (MRM) testing for DPX-Q8U80 and four metabolites. GLP, unpublished
DuPont-42611	Čermák, J., Kwiecien, J.	2017	DFG-S19 multiresidue method trials for DPX-Q8U80 and its metabolites in tomato, soya bean, grapefruit and wheat straw using LC/MS/MS. GLP, unpublished
DuPont-42614	Cochrane, J.	2017	Metabolism of [14C]IN-TMQ01 in lactating goats. GLP, unpublished
DuPont-43188, Revision No. 1	Shepard, E.	2020	Magnitude of residues of DPX-Q8U80 and metabolites in potato and potato processed commodities from potatoes planted after treatment with fluazaindolizine 500 g/L SC - United States 2015. GLP, unpublished
DuPont-43191, Revision No. 1	Shepard, E.	2020	Magnitude of residues of DPX-Q8U80 and metabolites in soya bean and soya bean processed commodities from soya beans planted after simulated treatment of two previous crops with fluazaindolizine 500 g/L SC - 2015. GLP, unpublished
DuPont-43192, Revision No. 1	Shepard, E.	2020	Residues of DPX-Q8U80 and metabolites after treatment of carrots with fluazaindolizine 500 g/L SC in samples taken over multiple time intervals - United States and Canada 2015-2016. GLP, unpublished
DuPont-43193, Revision No. 1	Shepard, E.	2020	Residues of DPX-Q8U80 and metabolites after treatment of potatoes with fluazaindolizine 500 g/L SC in samples taken over multiple time intervals - United States and Canada 2015-2016. GLP, unpublished
DuPont-43220	Hobbs, G.	2017	The metabolism of [14C]DPX-Q8U80 in potatoes. GLP, unpublished
DuPont-43221, Revision No. 1	Cochrane, J.	2018	Metabolism of [IP-2-14C]IN-QEK31 in laying hens. GLP, unpublished
DuPont-43224, Revision No. 1	Shepard, E.	2020	Magnitude of residues of DPX-Q8U80 and metabolites in wheat grain and wheat grain processed commodities from wheat grain planted after simulated treatment of two previous crops with fluazaindolizine 500 g/L SC - United States 2015-2016. GLP, unpublished
DuPont-43225, Revision No. 1	Shepard, E.	2020	Magnitude of residues of DPX-Q8U80 and metabolites in field corn grain and field corn grain processed commodities from field corn grain planted after simulated treatment of two previous crops with fluazaindolizine 500 g/L SC - United States 2015-2016. GLP, unpublished
DuPont-43226, Revision No. 1	Shepard, E.	2020	Magnitude of residues of DPX-Q8U80 and metabolites in strawberry and strawberry processed commodities from strawberries planted after simulated treatment of two previous crops with fluazaindolizine 500 g/L SC - United States 2015 - 2016. GLP, unpublished
DuPont-44344	Indrani, B.K.	2016	DPX-Q8U80: Ready biodegradability - CO2 evolution test. GLP, unpublished
DuPont-44351	Xu, A.	2017	Independent laboratory validation of analytical method for the determination of DPX-Q8U80 and metabolites in liver, eggs and

Reference Number	Author(s)	Year	Study Title
			muscle using LC/MS/MS. GLP, unpublished
DuPont-44365	Čermák, J., Kwiecien, J.	2017	DFG-S19 multiresidue method trials for fluazaindolizine and its metabolites in animal tissues using multi-residue method DFG S 19 (LC-MS/MS module). GLP, unpublished
DuPont-44398	Cochrane, J.	2017	Metabolism of [14C]IN-QZY47 in lactating goats. GLP, unpublished
DuPont-45659	Rutt, D.	2017	Independent laboratory validation of DuPont-47054, revision no. 1, "Analytical method for the enforcement of fluazaindolizine and metabolite residue limits in crops using LC/MS/MS". GLP, unpublished
DuPont-46269	Anand, H.S.	2016	DPX-Q8U80: Laboratory study of bulk density. GLP, unpublished
DuPont-47054, Revision No. 2	Gesell, J.T.	2020	Analytical method for the enforcement of fluazaindolizine and metabolite residue limits in crops using LC/MS/MS. unpublished
DuPont-48155	MacDonald, R.	2018	DPX-Q8U80: Radiovalidation of the crop enforcement method for DPX-Q8U80. GLP, unpublished
DuPont-48540	Pushpalatha, K.G.	2017	IN-VM862: Laboratory study of vapour pressure. GLP, unpublished
DuPont-49059	Pushpalatha, K.G.	2017	DPX-Q8U80: Laboratory study of appearance (color, odor and physical state) and pH. GLP, unpublished
DuPont-49359	Schernikau, N., Colorado, C.S.	2017	ILV of the multiresidue method DFG S 19 for the determination of metabolites of fluazaindolizine in animal tissues (LC-MS/MS module). GLP, unpublished
DuPont-50715	Sharma, A.K.	2018	Calculated degradation rate of DPX-Q8U80 in top layer of water at multiple latitudes, GC solar based calculations. unpublished

FLUDIOXONIL (211)

First draft prepared by M. Le, Pest Management Regulatory Agency, Canada

EXPLANATION

Fludioxonil is a phenylpyrrole fungicide that was reviewed by the JMPR in 2004 (T, R), 2010 (R), 2012 (R), 2013 (R), and 2018 (R). The 2004 Meeting established and ADI of 0–0.4 mg/kg bw and decided that an ARfD was not necessary.

The residue definition for compliance with the MRLs and estimation of dietary intake in plant commodities is *fludioxonil*. The residue definition for compliance with the MRLs and estimation of dietary intake in animal commodities is the *sum of fludioxonil and its benzopyrrole metabolites, determined as 2,2-difluorobeno[1,1]dioxole-4-carboxylic acid and expressed as fludioxonil*. The residue is fat-soluble.

Fludioxonil was listed by the Fifty-second Session of CCPR for the evaluation of additional uses by the 2022 JMPR. Residue data for analytical methods, supervised residue trials, and processing studies for banana, mango, papaya, beans with pods, dry beans, dry peas, sugar beets, and tree nuts were submitted to the present Meeting.

RESIDUE ANALYSIS

Analytical methods

Method REM 133.06

Method REM 133.06 was reviewed by the 2012 JMPR and determined to be suitable for the analysis of fludioxonil in orange, kiwi, lettuce, wheat (grain, straw), grape, wine, sunflower seed, and mango, with an LOQ of 0.01 mg/kg in all crop matrices tested. In the current evaluation, Method REM 133.06 was used for the analysis of fludioxonil residues in banana, mango, papaya, fresh beans with pods, dry pea, and sugar beet matrices from the submitted supervised residue trials.

Method validation and concurrent recoveries of fludioxonil submitted to the current Meeting, from banana, mango, papaya, fresh beans with pods, dry pea, and sugar beet matrices, were determined at fortifications ranging from 0.01 mg/kg up to 40 mg/kg. Adequate recoveries (mean 76–108 percent) were demonstrated at all fortification levels in all matrices. Relative standard deviations (RSDs) were ≤ 15 percent in all matrices indicating that repeatability was also acceptable.

The method was adequately validated at an LOQ of 0.01 mg/kg for residues of fludioxonil in all matrices. Although there were a low number of samples at each fortification level in fresh beans with pods, at the highest fortification level in dry peas, and in numerous sugar beet matrices, Method REM 133.06 has been adequately validated in matrices from each of the five OECD commodity categories.

Method AG-597B

Method AG-597B was reviewed by the 2004 and 2006 JMPR and determined to be suitable for the analysis of fludioxonil in plant materials, with LOQs ranging from 0.008–0.05 mg/kg. In the current evaluation, Method AG-597B was used for the analysis of fludioxonil residues in dry edible beans and tree nuts from the submitted supervised residue trials.

Method validation and concurrent recoveries of fludioxonil submitted to the current Meeting, from dry edible beans were determined at fortifications ranging from 0.0033 mg/kg up to 0.4 mg/kg, in almond and pecan nutmeat at fortifications ranging from 0.01 mg/kg up to 0.50 mg/kg, and in almond

hulls at fortifications ranging from 0.01 mg/kg up to 10 mg/kg. Adequate recoveries (mean 96–117 percent) were demonstrated at all fortification levels in all matrices. Relative standard deviations (RSDs) were within the acceptable range for each fortification level indicating that repeatability was also acceptable.

The method was adequately validated at an LOQ of 0.01 mg/kg for residues in fludioxonil in dry edible beans, almond nutmeat, pecan nutmeat, and almond hulls. Although method validation data was conducted in dry edible beans at a fortification level of 0.003 mg/kg, given the limited number of samples validated at this level (i.e. n=3), the Meeting has determined that the LOQ for in dry edible beans using Method AG-597B is 0.01 mg/kg for residues of fludioxonil (n = 6). Although there were a low number of samples at most of the fortification levels in dry edible beans, at the higher fortification levels in almond nutmeat and hulls, and in almond processed commodities, Method AG-597B has been adequately validated in numerous matrices from each of the five OECD commodity categories.

Recovery data for all of the analytical methodsused to analyse samples from the supervised residue trials for fludioxonil reviewed by the current Meeting are summarized below.

Table 1 Summary of method validation (MV) and concurrent recovery (CR) data for fludioxonil from plant matrices.

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
Method REM 133.06							
Banana pulp	Fludioxonil	0.01	3	CR: 108, 107, 110	108	1.4	TK0167731
		40	3	CR: 90, 100, 93	94	5.4	
Banana whole fruit	Fludioxonil	0.01	3	CR: 103, 98, 99	100	2.6	
		40	3	CR: 94, 104, 103	100	5.5	
Mango peel	Fludioxonil (*m/z* 247/180)	0.01	10	MV: 90, 98, 96, 87, 90, 89, 90, 94, 87, 88	91	4.2	VR-024/20[A]
		1.0	10	MV: 95, 105, 103, 101, 97, 94, 97, 99, 93, 98	98	4.0	
		20	5	MV: 104, 101, 100, 107, 101	103	2.8	
	Fludioxonil (*m/z* 247/126)	0.01	10	MV: 89, 99, 96, 87, 89, 89, 99, 96, 87, 89	92	5.3	
		1.00	10	MV: 95, 104, 101, 100, 96, 95, 104, 101, 100, 96	99	3.5	
		20	5	MV: 102, 99, 104, 103, 104	102	2.0	
Mango pulp	Fludioxonil (*m/z* 247/180)	0.01	5	MV: 79, 88, 79, 76, 76	80	6.2	
		1.0	5	MV: 94, 101, 97, 97, 100	98	2.8	
	Fludioxonil (*m/z* 247/126)	0.01	5	MV: 78, 82, 75, 76, 76	77	3.6	
		1.0	5	MV: 98, 99, 102, 97, 101	99	2.1	
Mango peel	Fludioxonil	0.01	2	CR: 95, 106	101	-	LBS19053
		1.0	2	CR: 113, 99	106	-	
Mango pulp	Fludioxonil	0.01	2	CR: 97, 103	100	-	
		1.0	2	CR: 83, 103	93	-	
Papaya peel	Fludioxonil (*m/z* 247/180)	0.01	5	MV: 89, 90, 94, 87, 88	90	3.0	VR-030/20
		1.0	5	MV: 84, 97, 99, 93, 98	94	6.5	
		20	5	MV: 78, 84, 80, 89, 85	83	5.2	
	Fludioxonil (*m/z* 247/126)	0.01	10	MV: 88, 89, 94, 87, 88	89	3.1	
		1.0	10	MV: 93, 98, 100, 94, 96	96	3.0	
		20	5	MV: 76, 85, 79, 89, 87	83	6.6	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
Papaya pulp	Fludioxonil (*m/z* 247/180)	0.01	5	MV: 91, 100, 95, 95, 101	96	4.3	
		1.0	5	MV: 98, 99, 99, 96, 104	99	3.0	
	Fludioxonil (*m/z* 247/126)	0.01	5	MV: 92, 100, 96, 96, 103	97	4.3	
		1.0	5	MV: 98, 100, 99, 97, 103	99	2.3	
Papaya peel	Fludioxonil	0.01	2	CR: 91, 78	85	-	LBS19052
		1.0	2	CR: 97, 88	93	-	
Papaya pulp	Fludioxonil	0.01	2	CR: 88, 97	93	-	
		1.0	2	CR: 105, 98	102	-	
Beans green with green pods	Fludioxonil	0.01	3	CR: 108, 94, 106	103	7.4	S17-03822
		0.1	2	CR: 97, 89	93	-	
		16	1	CR: 104	-	-	
Beans remaining plant	Fludioxonil	0.01	5	CR: 98, 96, 103, 94, 106	99	5.0	
		0.1	3	CR: 92, 90, 99	94	5.1	
		8.0	1	CR: 100	-	-	
		20	1	CR: 79	-	-	
Dry peas	Fludioxonil	0.01	6	MV: 97, 95, 100 CR: 92, 91, 94	95	3.5	TK0256751
		0.1	6	MV: 94, 91, 91 CR: 87, 89, 90	90	2.6	
		0.4	3	MV: 95, 91, 93	93	2.2	
Sugar beet roots	Fludioxonil	0.01	5	MV: 102, 103 CR: 118, 102, 106	106	6.4	TK0044248
		0.1	4	MV: 83, 82 CR: 108, 102	94	14	
		3.0	2	CR: 77, 77	77	-	
		5.0	1	CR: 105	98	3.7	
Sugar beet raw juice	Fludioxonil	0.01	1	CR: 85	-	-	
		5.0	1	CR: 90	-	-	
Sugar beet thick juice	Fludioxonil	0.01	1	CR: 105	-	-	
		5.0	1	CR: 94	-	-	
Sugar beet raw sugar	Fludioxonil	0.01	1	CR: 89	-	-	
		5.0	1	CR: 77	-	-	
Sugar beet refined sugar	Fludioxonil	0.01	4	MV: 94, 84, 89 CR: 89	89	4.6	
		0.1	2	MV: 85, 81	83	-	
		5.0	1	CR: 87	-	-	
Sugar beet molasses	Fludioxonil	0.01	3	MV: 88, 87 CR: 90	88	1.7	
		0.1	2	MV: 76, 75	76	-	
		5.0	1	CR: 101	-	-	
Sugar beet wet pulp	Fludioxonil	0.01	1	CR: 72	-	-	
		5.0	1	CR: 83	-	-	
Sugar beet ensiled pulp	Fludioxonil	0.01	1	CR: 79	-	-	
		5.0	1	CR: 89	-	-	
Sugar beet dried pulp	Fludioxonil	0.01	3	MV: 108, 90 CR: 81	93	15	
		0.1	2	MV: 100, 83	92	-	
		5.0	1	CR: 101	-	-	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
Sugar beet pressed pulp	Fludioxonil	0.01	1	CR: 81	-	-	
		5.0	1	CR: 101	-	-	
Sugar beet press water	Fludioxonil	0.01	1	CR: 83	-	-	
		5.0	1	CR: 87	-	-	
Method AG-597B							
Dry edible beans	Fludioxonil	0.0033	3	MV: 116, 120, 115	117	2.3	CER04164/06
		0.01	6	MV: 114, 120, 119 CR: 70, 100, 82	101	20	
		0.05	3	MV: 114, 113 CR: 114	114	0.5	
		0.1	4	MV: 113, 110 CR: 111, 110	111	1.3	
Almond nutmeat	Fludioxonil	0.01	7	MV: 104, 106, 107 CR; 114, 106, 100	106	4.3	TK0351660
		0.10	7	MV: 99, 103, 103 CR: 112, 103, 92	102	6.4	
		0.50	1	CR: 76	-	-	
Pecan nutmeat	Fludioxonil	0.01	1	CR: 105	-	-	
		0.10	1	CR: 94	-	-	
Almond hulls	Fludioxonil	0.01	5	MV: 91, 94, 88 CR: 99, 106	96	7.4	
		0.10	5	MV: 107, 109, 100 CR: 91, 97	101	7.3	
		1.0	1	CR: 91	-	-	
		5.0	1	CR: 89	-	-	
		10	1	CR: 94	-	-	
Almond oil	Fludioxonil	0.01	4	MV: 107, 102, 105 CR: 112	107	4.0	
		0.10	4	MV: 99, 103, 103 CR: 105	103	2.5	
Roasted almonds	Fludioxonil	0.01	1	CR: 102	-	-	
		0.10	1	CR: 97	-	-	

Notes:

n = Number of replicates; RSD = relative standard deviation.

[A] In this study two MRM transitions were monitored for fludioxonil in each matrix tested. The 1st MRM is for quantitation while the second MRM was confirmatory.

STABILITY OF RESIDUES IN STORED ANALYTICAL SAMPLES

The stability of residues in samples during frozen storage was evaluated by the 2004 and 2010 JMPR in a range of commodities. Residues of fludioxonil are stable under freezer storage conditions for at least:

- 24 months in cereal (grain and straw), apples, tomatoes, fresh peas, rapeseed, maize (forage, grain, ears), sorghum hay, and potato tubers.

- 27 months in maize meal and potato flakes; 29 months in grapes.
- 427 days (~14 months) in whole grapefruit, 289 days (~9.5 months) in canned lemon juice, 303 days (~9.9 months) in lemon pulp, 312 days (~10.2 months) in sweet potato, and 159 days (approximately 5.2 months) in yam.

The stability data covered commodities that are representative of high water content (apples, tomatoes, fresh peas, maize forage, maize ears), high oil content (rapeseed), high starch content (cereal grain, maize grain, potato tubers, potato flakes, sweet potato, yam), and high acid content (grapes, whole grapefruit, canned lemon juice, lemon pulp) and can be extrapolated to the commodities considered at the current Meeting. Storage stability data are not available for any high protein content commodity, however it is noted that stability of fludioxonil was observed in cereal straw and sorghum hay (i.e. commodities that do not fall into any of the 5 OECD commodity categories) for at least 24 months.

Maximum storage to analysis intervals for samples from the submitted supervised field trials were: 5.3 months for bananas, 2.7 months for mangoes, 2.9 months for papayas, 13 months for beans with pods, 1.5 months for dry beans, 8.2 months for dry peas, 8.7 months for sugar beet roots and processed commodities, 3.0 months for pecan nutmeat, 10.4 months for almond nutmeat, 10.8 months for almonds hulls, and 1.7 months for almond processed commodities (i.e. roasted nutmeats and oil). The periods of demonstrated stability cover the frozen storage intervals used in the residue studies on crops.

USE PATTERN

The registered uses of fludioxonil relevant to the supervised residue studies made available to the current Meeting are summarized in Table 2.

Table 2 Registered uses of fludioxonil

Crop	Country	Form.	Application						PHI (days)
			Method, GS/Timing	Max. No.	Rate/ application	Interval (days)	Water (L/ha)	Max. Rate/ season	
006B Assorted tropical and sub-tropical Inedible Smooth Peel - Large									
Banana	Columbia	SC	Spray, Post-harvest	1	15–20 g ai/hL[A]	-	-	20 g ai/hL	-
Mango, papaya	Brazil	SC	Dip[B] or spray[C], Post-harvest	1	60–120 g ai/hL	-	-	120 g ai/hL	-
014 Legume Vegetables									
Peas, beans, fresh with pods	Latvia	WG	Foliar. Spray as a preventative measure in conditions favourable to the spread of disease.	3	250 g ai/ha	10–14	400–800	750 g ai/ha	14
Succulent beans[D]	Canada	WG	Foliar. Begin applications prior to or at the onset of disease.	3	194–244 g ai/ha	7	175–225	730 g ai/ha	7
015 Pulses									
Peas, beans, for dry consumption	Latvia	WG	Foliar. Spray as a preventative measure in conditions favourable to the spread of disease.	2	250 g ai/ha	10	400–800	500 g ai/ha	28
Dried shelled pea and bean (except soybean)[E]	Canada	WG	Foliar. Begin applications prior to or at the onset of disease.	3	194–244 g ai/ha	7	175–225	730 g ai/ha	7
016A Root Vegetables									
Sugar beets	United States	SC	In-line aqueous spray. Post-harvest	1	4.5 g ai/1000 kg of roots	-	0.2 L/kg	4.5 g ai/1000 kg of roots	-

Crop	Country	Form.	Application						PHI (days)
			Method, GS/Timing	Max. No.	Rate/ application	Interval (days)	Water (L/ha)	Max. Rate/ season	
02 Tree Nuts									
Tree Nuts[F]	United States	WG	Foliar. Make first application during early bloom. Repeat if conditions remain favourable for disease development.	4[G]	247 g ai/ha	14	≥ 187 (air); ≥ 94 (ground)	1009 g ai/ha	14

Notes:

Form = Formulation, NS = not specified.

[A] Use the lower rate when transport of fruit in shipments for a duration of < 15 days and the higher rate when duration is > 15 days.

[B] Use an equipment that allows complete immersion of the fruits in a water solution containing fludioxonil for 2 minutes. Then, move the fruits to a drying chamber.

[C] Ensure a homogeneous and uniform coverage of the fruits with an application system located within the process line, containing a closed chamber, through which the fruits pass, using controlled flow nozzles, avoiding leaks from the application chamber. Then, move the fruits to a drying chamber.

[D] Bean (*Phaseolus* spp.) (includes lima bean, snap bean and wax bean), Bean (*Vigna* spp.) (includes blackeyed pea, asparagus bean), Broad bean (fava bean) (*Vicia faba*).

[E] Chickpea (garbanzo bean) (*Cicer arietinum*), beans (*Lupinus* spp. including grain lupin, sweet lupin, white lupin, white sweet lupin), beans (*Phaseolus* spp. including field bean, kidney bean, lima bean (dry), navy bean, pinto bean, tepary bean), broad bean (fava bean) (*Vicia faba*), beans (*Vigna* spp. adzuki bean, black-eyed pea, catjang, cowpea, Crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean); Guar (*Cyamopsis tetragonoloba*); Lablab bean or Hyacinth bean (*Lablab purpureus*); Lentil (Lens esculenta); Pea (*Pisum* spp.) (includes field pea); Pigeon pea (*Cajanus cajan*)

[F] African nut-tree; almond; beechnut; Brazil nut; Brazilian pine; bunya; bur oak; butternut; Cajou nut; candlenut; cashew; chestnut; chinquapin; coconut; coquito nut; dika nut; ginkgo; Guiana chestnut; hazelnut (filbert); heartnut; hickory nut; Japanese horse-chestnut; macadamia nut; mongongo nut; monkey-pot; monkey puzzle nut; Okari nut; Pachira nut; peach palm nut; pecan; pequi; Pili nut; pine nut; pistachio; Sapucaia nut; tropical almond; walnut, black; walnut, English; yellowhorn; cultivars, varieties, and/or hybrids of these

[G] Make no more than 2 applications by air, the rest by ground.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials for fludioxonil on the following crops or crop groups:

Crop	Table No.
Assorted tropical and sub-tropical Inedible Smooth Peel - Large	
Banana	3
Mango	4
Papaya	5
Legume Vegetables	
Beans with pods	6
Pulses	
Dry beans	7
Dry peas	8

Crop	Table No.
Root Vegetables	
Sugar beets	9
Tree nuts	
Almonds and Pecans	10
Animal feeds	
Bean forage	11
Almond hulls	12

Residue values from the trials conducted according to the critical GAP (or a suitable alternative GAP) have been used for the estimation of maximum residue levels, STMR, and HR (where applicable). Those results included in the evaluation as adequately supporting the GAP are underlined. Non quantifiable residues are shown as below the reported LOQ (e.g. < 0.02 mg/kg). Where multiple analyses were conducted on a single sample, the average value is reported. Where multiple samples were taken from a single plot, the individual and average values are reported. For all trials except citrus fruits (which had two treated samples), only a single composite treated sample was analysed.

Banana

Table 3 Residues of fludioxonil (mg/kg) in banana following post-harvest application of fludioxonil in an SC-formulation (Lenz, 2017, TK0167731)

Location, year, variety BANANA	Timing/GS	Rate (g ai/hL)	Sample	Fludioxonil (mg/kg) [average]
Columbia GAP	Post-harvest spray	20		
Spray Application [A]				
Esmeraldas, La Union, Ecuador, 2016, Williams	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	1.57, 0.788 [1.2]
			Pulp	0.0102, <0.01 [<0.010]
La Villegas, La Concordia, Ecuador, 2016, Valery	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	0.747, 0.775 [0.76]
			Pulp	0.0155, 0.0148 [0.015]
La Independencia, Sto. Domingo, Ecuador, 2016, Grand Naine	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	0.823, 0.815 [0.82]
			Pulp	<0.01, <0.01 [<0.01]
Esmeraldas, La Parroquia, Ecuador, 2016, Cavendish	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	1.04, 0.818 [0.93]
			Pulp	0.0146, 0.0138 [0.014]
Sto. Domingo, Monterrey, Ecuador, 2016, Cavendish	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	0.758, 0.402 [0.58]
			Pulp	0.0276, 0.0134 [0.021]
Los Rios, Valencia 2016, Ecuador, Valery	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	1.02, 1.27 [1.1]
			Pulp	0.0112, <0.01 [<0.011]
Aqueous Dip Application[B]				
Esmeraldas, La Union, Ecuador, 2016, Williams	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	0.594, 0.756 [0.68]
			Pulp	<0.01, 0.0146 [<0.012]
La Villegas, La Concordia, Ecuador, 2016, Valery	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	1.21, 0.913 [1.1]
			Pulp	0.0247, 0.0277 [0.026]
La Independencia, Sto. Domingo, Ecuador, 2016, Grand Naine	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	0.623, 0.780 [0.70]
			Pulp	0.0178, 0.0137 [0.016]
Esmeraldas, La Parroquia, Ecuador, 2016, Cavendish	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	0.561, 0.740 [0.65]
			Pulp	0.0166, 0.0199 [0.018]
Sto. Domingo, Monterrey, Ecuador, 2016, Cavendish	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	0.660, 1.01 [0.84]
			Pulp	0.0129, 0.0180 [0.015]
Los Rios, Valencia 2016, Ecuador, Valery	Post-harvest/ Mature Commercial Harvest	20	Whole fruit	1.29, 1.02 [1.2]
			Pulp	0.0186, <0.01 [<0.014]

Notes:

[A] A backpack sprayer was used to spray the hands of fruit for approximately 2 to 4 minutes to ensure thorough coverage of the fruit (simulating commercial processes).

[B] Hands of bananas were dipped into the treatment solution for approximately 30 seconds. Fruit were removed from the solution, placed on a tray, and allowed to air dry for several minutes up to one hour.

Mango

Table 4 Residues of fludioxonil (mg/kg) in mango following post-harvest application of fludioxonil in an SC-formulation (Pereira, 2020, LBS19053)

Location, year, variety MANGO	Timing/GS	Rate (g ai/hL)	Storage Period (days)[C]	Fludioxonil (mg/kg) [average]		
				Peel	Pulp	Whole Fruit [D]
Brazil GAP	Post-harvest	120	NS	Dip or spray application		
Dip Application [A, B]						
Orocó, Pernambuco, Brazil, 2020, Palmer	Post-harvest/ BBCH 79-82	120	0	16.20, 15.52 [16]	0.01, 0.02 [0.02]	4.38, 4.14 [4.3]
			21	12.41, 15.07 [14]	0.01, <0.01 [<0.01]	3.44, 3.73 [3.6]
			42	12.55, 13.52 [13]	<0.01, 0.01 [<0.01]	3.18, 6.84 [5.0]
Juazeiro, Bahia, Brazil, 2020, Keit[E]	Post-harvest/ BBCH 79-81	120	0	13.40, 15.91 [15]	0.01, 0.01 [0.01]	3.59, 4.22 [3.9]
			21	15.00, 12.92 [14]	0.01, <0.01 [<0.01]	3.21, 2.69 [3.0]
			42	12.24, 13.63 [13]	0.01, <0.01 [<0.01]	2.93, 3.53 [3.2]
Juazeiro, Bahia, Brazil, 2020, Kent[E]	Post-harvest/ BBCH 79-81	120	0	15.08, 15.70 [15]	0.05, 0.05 [0.05]	3.30, 4.02 [3.7]
			21	13.69, 14.02 [14]	0.02, 0.04 [0.03]	3.49, 3.44 [3.5]
			42	12.05, 14.81 [13]	<0.01, 0.02 [<0.02]	2.88, 3.51 [3.2]
Petrolina, Pernambuco, Brazil, 2020, Tommy	Post-harvest/ BBCH 79-81	120	0	13.84, 15.75 [15]	0.05, 0.03 [0.04]	3.02, 4.39 [3.7]
			21	13.97, 14.69 [14]	0.02, 0.01 [0.02]	2.93, 3.20 [3.1]
			42	11.27, 10.28 [11]	0.01, 0.05 [0.03]	2.57, 2.45 [2.5]
Spray Application [B]						
Orocó, Pernambuco, Brazil, 2020, Palmer	Post-harvest/ BBCH 79-82	120	0	14.16, 20.61 [17]	<0.01, 0.01 [<0.01]	3.73, 5.19 [4.5]
			21	15.00, 16.70 [16]	0.01, <0.01 [<0.01]	3.89, 4.19 [4.0]
			42	13.72, 15.11 [14]	<0.01, <0.01 [<0.01]	3.69, 3.92 [3.8]
Juazeiro, Bahia, Brazil, 2020, Keit [E]	Post-harvest/ BBCH 79-81	120	0	17.05, 17.39 [17]	0.01, 0.01 [0.01]	4.72, 4.31 [4.5]
			21	17.46, 20.36 [19]	0.01, 0.06 [0.04]	3.63, 4.38 [4.0]
			42	15.89, 17.31 [17]	0.02, 0.03 [0.03]	3.94, 4.15 [4.1]
Juazeiro, Bahia, Brazil, 2020, Kent [E]	Post-harvest/ BBCH 79-81	120	0	20.46, 16.72 [19]	0.02, 0.02 [0.02]	5.35, 4.56 [5.0]
			21	22.56, 16.91 [20]	0.02, 0.04 [0.03]	5.47, 4.02 [4.8]
			42	16.52, 18.15 [17]	0.09, 0.03 [0.06]	4.36, 4.20 [4.3]
Petrolina, Pernambuco, Brazil, 2020, Tommy	Post-harvest/ BBCH 79-81	120	0	22.11, 16.74 [19]	0.05, 0.01 [0.03]	5.08, 3.71 [4.4]
			21	20.69, 14.06 [17]	0.01, 0.01 [0.01]	4.45, 3.28 [3.9]
			42	16.04, 21.00 [19]	0.02, 0.02 [0.02]	3.76, 4.97 [4.4]

Notes:

[A] Dipped for about 120 seconds.

[B] After the post-harvest treatments (dip or spray), the fruits were sprayed with carnauba wax.

[C] Storage period = Days after post-harvest treatment. Samples were stored in a cold room prior to being sub-sampled into peel and pulp samples and frozen.

[D] The amount of residues in whole fruit was calculated as follows: [(weight of the pulp sample × residue found in the pulp sample) + (weight of peel sample × residue found in the peel sample)] / weight of the whole fruit sample. The weight of the whole fruit sample was calculated as follows: weight of the pulp sample + weight of peel sample + weight of seeds.

[E] The trials conducted in Juazeiro, Bahia, Brazil were treated in different facilities on different days and separate treatment mixtures were prepared at each site. As such the trials are considered independent for the purposes of estimating maximum residue levels.

Papaya

Table 5 Residues of fludioxonil (mg/kg) in papaya following post-harvest application of fludioxonil in an SC-formulation (Pereira, 2020, LBS19052)

Location, year, variety PAPAYA	Timing/GS	Rate (g ai/hL)	Storage Period (days)[C]	Fludioxonil (mg/kg) [average]		
				Peel	Pulp	Whole Fruit[D]
Brazil GAP	Post-harvest	120	NS	Dip or spray application		
Dip Application[A, B]						
Vila Valério, Espíriro Santo, Brazil, 2020, Aliança	Post-harvest/ BBCH 79-81	120	0	8.43, 4.66 [6.5]	0.02, 0.01 [0.02]	1.57, 0.88 [1.2]
			6	6.53, 5.65 [6.1]	0.10, 0.10 [0.10]	1.21, 1.10 [1.2]
			13	1.09, 1.01 [1.1]	0.08, 0.09 [0.09]	0.98, 1.02 [1.0]
Mucuri, Bahia, Brazil, 2020, Golden/THB	Post-harvest/ BBCH 79-81	120	0	6.01, 9.76 [7.9]	0.02, 0.02 [0.02]	1.43, 4.91 [3.2]
			6	7.72, 8.40 [8.1]	0.11, 0.16 [0.14]	1.90, 2.11 [2.0]
			13	1.76, 1.79 [1.8]	0.17, 0.12 [0.15]	0.64, 0.47 [0.55]
Ibirapuã, Bahia, Brazil, 2020, Sunrise/BS	Post-harvest/ BBCH 79-81	120	0	5.37, 3.70 [4.5]	0.02, 0.02 [0.02]	1.19, 0.97 [1.1]
			6	6.57, 6.23 [6.4]	0.04, 0.08 [0.06]	1.57, 1.43 [1.5]
			13	1.16, 0.97 [1.1]	0.06, 0.06 [0.06]	0.32, 0.28 [0.30]
Baraúna, Rio Grande do Norte, Brazil, 2020, Tainung	Post-harvest/ BBCH 79-83	120	0	9.95, 15.01 [12]	0.01, <0.01 [<0.01]	1.83, 2.75 [2.3]
			6	1.62, 1.38 [1.5]	0.01, 0.01 [0.01]	0.49, 0.37 [0.43]
			13	0.91, 1.20 [1.1]	0.01, 0.04 [0.03]	0.18, 0.24 [0.21]
Spray Application[B]						
Vila Valério, Espíriro Santo, Brazil, 2020, Aliança	Post-harvest/ BBCH 79-82	120	0	5.93, 8.79 [7.4]	0.02, 0.01 [0.02]	1.28, 1.72 [1.5]
			6	8.27, 6.69 [7.5]	0.14, 0.17 [0.16]	1.36, 1.30 [1.3]
			13	1.22, 1.61 [1.4]	0.15, 0.17 [0.16]	1.24, 1.54 [1.4]]
Mucuri, Bahia, Brazil, 2020, Golden/THB	Post-harvest/ BBCH 79-81	120	0	6.18, 7.64 [6.9]	0.01, 0.02 [0.02]	1.34, 1.70 [1.5]
			6	9.34, 8.49 [8.9]	0.10, 0.15 [0.13]	2.19, 1.99 [2.1]
			13	2.14, 1.59 [1.9]	0.14, 0.16 [0.15]	0.55, 0.46 [0.51]
Ibirapuã, Bahia, Brazil, 2020, Sunrise/BS	Post-harvest/ BBCH 79-81	120	0	6.70, 5.45 [6.1]	0.02, 0.02 [0.02]	1.64, 1.40 [1.5]
			6	6.50, 8.22 [7.4]	0.16, 0.15 [0.16]	1.53, 1.85 [1.7]
			13	0.88, 1.27 [1.1]	0.07, 0.12 [0.10]	0.24, 0.39 [0.31]
Baraúna, Rio Grande do Norte, Brazil, 2020, Tainung	Post-harvest/ BBCH 79-83	120	0	14.96, 12.46 [14]	<0.01, <0.01 [<0.01]	3.06, 2.28 [2.7]
			6	3.48, 3.91 [3.7]	0.10, 0.12 [0.11]	0.93, 0.99 [0.96]
			13	4.63, 4.13 [4.4]	0.14, 0.15 [0.15]	1.09, 0.83 [0.96]

Notes:

[A] Dipped for about 120 seconds.

[B] After the post-harvest treatments (dip or spray), the fruits were sprayed with carnauba wax.

[C] Storage period = Days after post-harvest treatment. Samples were stored in a cold room prior to being sub-sampled into peel and pulp samples and frozen.

[D] The amount of residues in whole fruit was calculated in the study report as follows: [(weight of the pulp sample × residue found in the pulp sample) + (weight of peel sample × residue found in the peel sample)] / weight of the whole fruit sample. The weight of the whole fruit sample was calculated as follows: weight of the pulp sample + weight of peel sample. Since the weight of the whole fruit sample did not take into account the weight of the seed, the residue values reported in the study were recalculated to include the weight of the seed (i.e. weight of the whole fruit sample = weight of the pulp sample + weight of peel sample + weight of seeds).

Fresh beans with pods

Table 6 Residues of fludioxonil (mg/kg) in fresh beans with pods following foliar applications of fludioxonil in a WG-formulation (Yozgatli and Breyer, 2018, S17-03822)

Location, year, variety FRESH BEANS W/ PODS	N (interval)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Fludioxonil (mg/kg) [average]
Latvia GAP	3 (10)	250	400 -800		14		

Location, year, variety FRESH BEANS W/ PODS	N (interval)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Fludioxonil (mg/kg) [average]
Latvia GAP	3 (10)	250	400 -800		14		
Ravenna, Emilia Romagna, Italy, 2017, Shubert	3 (10, 11)	257 265 239	412 423 382	71 73 75	-0 3 7 11 14	Beans green with pods	0.19 0.26 0.23 0.16 0.13
Soria, Castilla y León, Spain, 2017, Kylie	3 (10, 11)	234 244 232	374 391 371	51 51-63 61-67	11 14	Beans green with pods	0.08 0.05
Tarn et Garonne, Midi Pyrenees, France, 2017, Manosi	3 (9, 10)	245 245 250	392 393 401	51 61-65 65-79	-0 3 7 11 14	Beans green with pods	0.05 0.33 0.16 0.05 0.07
Lovech, Severozapaden, Bulgaria, 2017, Plaja	3 (9, 11)	253 255 252	405 408 403	63 67 71	-0 3 7 10 14	Beans green with pods	<0.01 0.23 0.07 0.11 0.01
Pella, Kentriki Makedonia, Greece, 2017, SV1286GW	3 (10, 11)	261 256 231	627 616 553	51 55 61	14	Beans green with pods	0.03
Cadiz, Andalucia, Spain, 2017, Primel	3 (10, 11)	233 243 249	560 778 796	61 65-67 67-69	14	Beans green with pods	0.06
Thessaloniki, Kentriki Makedonia, Greece, 2017, Koala	3 (10, 10)	256 259 258	613 622 620	51 59 63	14	Beans green with pods	<0.01
Pazardzhik, Yuzhen tsentralen, Bulgaria, 2017, Gina	3 (9, 10)	266 261 256	426 417 409	69 73-74 75-77	14	Beans green with pods	0.48

Notes:

"-0" = harvested before the last application.

Dry beans

Table 7 Residues of fludioxonil (mg/kg) in dry edible beans following foliar applications of fludioxonil in a WG-formulation (Tout, 2006, CER 04164/06)

Location, year, variety DRY EDIBLE BEANS	N (interval)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Fludioxonil (mg/kg) [average]
Canada GAP	3 (7)	244	175–225		7		maximum 730 g ai/ha
Thorndale, Ontario, Canada, 2006, OAC Thunder	3 (6, 8)	246-249 237-243 244-245	200 200 200	74-76 77-79 97	7	Dry bean seed	<0.01, <0.01 [<0.01]
Elm Creek, Manitoba, Canada, 2006, Envoy[A]	3 (7, 7)	240-243 243-244 246-247	200 200 200	77-80 84-88 86-88	7	Dry bean seed	0.026[B], 0.031[B] [0.029]
Barnsley, Manitoba, Canada, 2006, Envoy	3 (7, 7)	240-242 243-247 244	200 200 200	78-79 84-86 87-88	7	Dry bean seed	0.024, 0.022 [0.023]

Location, year, variety DRY EDIBLE BEANS	N (interval)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Fludioxonil (mg/kg) [average]
Canada GAP	3 (7)	244	175–225		7		maximum 730 g ai/ha
Elm Creek, Manitoba, Canada, 2006, Pinto[A]	3 (7, 7)	246 244-250 245-246	200 200 200	78-80 82-83 87-88	7	Dry bean seed	<0.01, 0.011 [<0.011]
Taber, Alberta, Canada, 2006, Pinto	3 (7, 7)	241-242 233-237 235-240	200 200 200	79-81 79-83 79-86	7	Dry bean seed	0.020[B], 0.015[B] [0.018]

Notes:

[A] These trials were conducted at the same location however are considered independent for the purposes of estimating maximum residue levels on the basis of the different varieties (i.e. navy bean versus pinto bean) and last applications being made 15 days apart.

[B] These samples were re-extracted and re-analysed in duplicates to verify the original results. The original result and the duplicate re-analysed values were averaged to yield a single residue value.

Table 8 Residues of fludioxonil (mg/kg) in dry peas following foliar applications of fludioxonil in a WG-formulation (Sagan, 2017, TK0256751)

Location, year, variety DRY PEAS	N (interval)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Fludioxonil (mg/kg) [average]
Canada GAP	3 (7)	244	175–225		7		maximum 730 g ai/ha
Zealandia, Saskatchewan, Canada, 2015, CDC Amarillo	3 (7, 7)	237 242 243	200 200 200	76-77 79 88-89	3 6 9 13	Dry pea seed	0.19 0.082, 0.17 [0.13] 0.075 0.082
Delisle, Saskatchewan, Canada, 2015, CDC Amarillo	3 (7, 7)	234 249 254	200 200 200	75-77 79-82 79-83	4 6 10 13	Dry pea seed	0.087 0.041, 0.050 [0.046] 0.038 0.030
Dundurn, Saskatchewan, Canada, 2015, CDC Meadow	3 (6, 7)	247 254 246	200 200 200	79-82 82-83 83-84	7	Dry pea seed	0.018, 0.017 [0.018]
Moon Lake, Saskatchewan, Canada, 2015, CDC Amarillo	3 (6, 8)	251 242 243	200 200 200	79-80 79-81 79-82	6	Dry pea seed	0.097, 0.12 [0.11]
Blaine Lake, Saskatchewan, Canada, 2015, CDC Amarillo	3 (7, 7)	240 239 241	200 200 200	77-79 79-81 84-85	7	Dry pea seed	0.090, 0.13 [0.11]
Hague, Saskatchewan, Canada, 2015, CDC Amarillo	3 (8, 7)	247 253 241	200 200 200	76-77 78-79 81-84	7	Dry pea seed	0.048, 0.043 [0.046]
Glenboro, Manitoba, Canada, 2015, CDC Meadow	3 (7, 5)	245 247 244	200 200 200	79-81 81-82 82-83	6	Dry pea seed	0.23, 0.11 [0.17]

Table 9 Residues of fludioxonil (mg/kg) in sugar beets following post-harvest application of fludioxonil in a SC-formulation (Shepard, 2017, TK0044248)

Location, year, variety SUGAR BEET	Timing/Method/GS	Rate (g ai/1000 kg roots)	Sample	Fludioxonil (mg/kg) [average]
US GAP	Post-harvest/in-line aqueous spray	4.5		

Location, year, variety SUGAR BEET	Timing/Method/GS	Rate (g ai/1000 kg roots)	Sample	Fludioxonil (mg/kg) [average]
US GAP	Post-harvest/in-line aqueous spray	4.5		
Verona, Wisconsin, United States, 2015, BTS 60RR27 MP	Post-harvest/spray/ maturity (BBCH 49)	4.2	Roots[A]	0.71, 1.2 [0.96]
Ephrata, Washington, United States, 2015, 3574X0853	Post-harvest/spray/ maturity (BBCH 49)	4.7	Roots[A]	1.9, 1.9 [1.9]
Geneva, Minnesota, United States, 2015, 9425RR4M	Post-harvest/spray/ maturity (BBCH 49)	4.7	Roots[A]	0.69, 0.59 [0.64]
St. Cloud, Minnesota, United States, 2015, SX1521N	Post-harvest/spray/ maturity (BBCH 49)	4.7	Roots[A]	1.7, 1.6 [1.7]
Wyoming, Illinois, United States, 2015, Select Harvest, SUGB14151J	Post-harvest/spray/ maturity (BBCH 49)	4.7	Roots[A]	0.85, 0.95 [0.90]
Richland, Iowa, United States, 2015, Green Valley Lot# 160210	Post-harvest/spray/ maturity (BBCH 49)	4.6	Roots[A]	1.3, 1.2 [1.2]

Notes:

[A] After treatment, the test substance was allowed to dry and the sugar beet roots were halved or quartered prior to placing all pieces of the cut samples in bags for freezing. Samples were frozen within ~3.3 hours after collection.

Table 10 Residues of fludioxonil (mg/kg) in almonds and pecans following foliar applications of fludioxonil in a WG-formulation with adjuvant [A] (Baillargeon, 2020, TK0351660)

Location, year, variety TREE NUTS	N (interval)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Fludioxonil (mg/kg) [average]
US GAP	4 (14)	247	≥ 187 (air); ≥ 94 (ground)		14		
Almonds							
Porterville, California, United States, 2018, Butte	4 (14, 14, 14)	243 242 245 243	2759 2769 2759 2769		7 10 15 17 21	Nutmeat	0.011, <0.01 [<0.011] 0.016, 0.010 [0.013] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01]
Lost Hills, California, United States, 2018, Monterey	4 (14, 14, 14)	244 244 246 246	748 748 758 748		14	Nutmeat	0.017, 0.019 [0.018]
Terra Bella, California, United States, 2018, Nonpareil	4 (14, 14, 14)	243 249 243 247	243 249 243 247		14	Nutmeat	<0.01, <0.01 [<0.01]
Dinuba, California, United States, 2018, Carmel	4 (14, 14, 14)	245 245 245 245	245 245 245 245		14	Nutmeat	<0.01, <0.01 [<0.01]
Yuba City, California, United States, 2018, Mission	4 (14, 14, 14)	247 249 248 247	247 249 248 247		14	Nutmeat	0.142[B], 0.151[B] [0.15]
Pecans							
Bailey, North Carolina, United States, 2018, Stuart	4 (14, 12, 14)	71 429 243 244	2469 2516 2404 2441		13	Nutmeat	<0.01, <0.01 [<0.01]

Location, year, variety TREE NUTS	N (interval)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Fludioxonil (mg/kg) [average]
US GAP	4 (14)	247	≥ 187 (air); ≥ 94 (ground)		14		
Tifton, Georgia, United States, 2018, Sumner	4 (14, 13, 14)	245 245 246 249	608 589 617 458		7 10 14 16 21	Nutmeat	0.016, <0.01 [<0.013] <0.01, <0.01 [<0.01] <0.01, <0.01 [<u><0.01</u>] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01]
Port Barre, Louisiana, United States, 2018, Oconee	4 (14, 14, 14)	252 245 249 252	1787 1637 1749 1852		14	Nutmeat	<0.01, <0.01 [<u><0.01</u>]
Pearsall, Texas, United States, 2018, Cheyenne	4 (15, 13, 13)	244 249 248 250	1815 1983 1431 1441		14	Nutmeat	<0.01, <0.01 [<u><0.01</u>]
Lubbock, Texas, United States, 2018, Western Schley	4 (14, 13, 15)	247 248 243 244	374 374 374 374		13	Nutmeat	<0.01, <0.01 [<u><0.01</u>]

Notes:

[A] A non-ionic surfactant (NIS), a crop oil concentrate (COC), or a methylated seed oil (MSO) blend was added to each spray mixture at rates typical of the agricultural use.

[B] Average of duplicate or triplicate analyses.

Animal Feeds

Bean forage

Table 11 Residues of fludioxonil (mg/kg) in bean forage following foliar applications of fludioxonil in a WG-formulation (Yozgatli and Breyer, 2018, S17-03822)

Location, year, variety BEAN FORAGE	N (interval)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Fludioxonil (mg/kg) [average]
Latvia GAP	3 (10)	250	400 -800		14		
Canada GAP	3 (7)	244	175–225		7		maximum 730 g ai/ha
Ravenna, Emilia Romagna, Italy, 2017, Shubert	3 (10, 11)	257 265 239	412 423 382	71 73 75	14	Remaining plant	<u>5.1</u>
Soria, Castilla y León, Spain, 2017, Kylie	3 (10, 11)	234 244 232	374 391 371	51 51-63 61-67	0 3 7	Whole plant[A]	21 6.9 12
					14	Remaining plant	<u>11</u>[B]
Tarn et Garonne, Midi Pyrenees, France, 2017, Manosi	3 (9, 10)	245 245 250	392 393 401	51 61-65 65-79	14	Remaining plant	<u>2.3</u>
Lovech, Severozapaden, Bulgaria, 2017, Plaja	3 (9, 11)	253 255 252	405 408 403	63 67 71	14	Remaining plant	<u>0.50</u>
Pella, Kentriki Makedonia, Greece, 2017, SV1286GW	3 (10, 11)	261 256 231	627 616 553	51 55 61	14	Remaining plant	<u>3.3</u>

Location, year, variety BEAN FORAGE	N (interval)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Fludioxonil (mg/kg) [average]
Latvia GAP	3 (10)	250	400 -800		14		
Canada GAP	3 (7)	244	175–225		7		maximum 730 g ai/ha
Cadiz, Andalucia, Spain, 2017, Primel	3 (10, 11)	233 243 249	560 778 796	61 65-67 67-69	14	Remaining plant	7.8
Thessaloniki, Kentriki Makedonia, Greece, 2017, Koala	3 (10, 10)	256 259 258	613 622 620	51 59 63	14	Remaining plant	0.40
Pazardzhik, Yuzhen tsentralen, Bulgaria, 2017, Gina	3 (9, 10)	266 261 256	426 417 409	69 73-74 75-77	14	Remaining plant	4.2

Notes:

"-0" = harvested before the last application.

[A] the crop was immature, therefore whole plant without roots were sampled instead of beans green with pods. For whole plant without roots no control sample is available.

[B] Average of two determinations.

Almond hulls

Table 12 Residues of fludioxonil (mg/kg) in almond hulls following foliar applications of fludioxonil in a WG-formulation

Location, year, variety ALMOND HULLS	N (interval)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Fludioxonil (mg/kg) [average]
US GAP	4 (14)	247	≥ 187 (air); ≥ 94 (ground)		14		
Porterville, California, United States, 2018, Butte	4 (14, 14, 14)	243 242 245 243	2759 2769 2759 2769		7 10 15 17 21	Hulls	3.75, 3.48 [3.6] 4.81, 4.16 [4.5] 3.58, 2.94 [3.3] 1.27, 1.21 [1.2] 2.65, 2.82 [2.7]
Lost Hills, California, United States, 2018, Monterey	4 (14, 14, 14)	244 244 246 246	748 748 758 748		14	Hulls	6.86, 8.36 [7.6]
Terra Bella, California, United States, 2018, Nonpareil	4 (14, 14, 14)	243 249 243 247	243 249 243 247		14	Hulls	1.03, 1.11 [1.1]
Dinuba, California, United States, 2018, Carmel	4 (14, 14, 14)	245 245 245 245	245 245 245 245		14	Hulls	1.63, 1.76 [1.7]
Yuba City, California, United States, 2018, Mission	4 (14, 14, 14)	247 249 248 247	247 249 248 247		14	Hulls	1.99, 1.52 [1.8]

FATE OF RESIDUES IN STORAGE AND PROCESSING

Residues after processing

Sugar Beets

A processing trial on sugar beets was conducted in the United States in 2015 (Shepard, 2017, TK0044248). Fludioxonil, formulated as a SC (containing 235.9 g ai/L) was applied as a post-harvest spray to sugar beet roots at a rate of 4.7 g ai/1000 kg roots. The sugar beet roots were allowed to dry for about 2–3 hours prior to collection. The roots were quartered prior to placing samples in the bag to reduce the size of the beets for grinding purposes. The sugar beet samples were processed to generate raw juice, thick juice, raw sugar, refined sugar, molasses, wet pulp, ensiled pulp, dried pulp, pressed pulp, and press water using methods which are representative of commercial practice.

Sugar beets (~39 kg) were cleaned prior to processing and then sliced in a Hobart food chopper to form the cossettes. Cossettes were exposed to 88–92 °C water for 30–45 seconds and then diffused in 5 kettles in a 68–74 °C water bath for at least 9 minutes in each kettle. After diffusion, the raw juice was screened through a mesh sieve. Raw juice and wet pulp fractions were collected.

Diffused cossettes were then dewatered (pressed) in a filter bag and hydraulic press and pressed pulp fractions were collected. A portion of the pressed pulp was collected for ensiling. Dried beet pulp was produced by drying the remaining dewatered material in an oven at 54–71 °C until a final moisture content of 15 percent or less. Dried pump samples were collected.

In the first phosphatization step, raw juice from the dewatering process was screened through a mesh sieve and press water fractions were collected. Raw juice was mixed and heated to 79–85 °C and the pH was adjusted with calcium oxide solution to approximately 10.5. If the pH was above 11.2 it was lowered with 3M phosphoric acid. A precipitate/mud was formed and centrifugation was used to separate the mud and juice. In the second phosphatization step, the juice was again mixed and heated to 79–85 °C and the pH was reduced to 9.1–9.3 with 3M phosphoric acid and then centrifuged and vacuum filtered to separate the mud and clear/thin juice. The thin juice was mixed and heated to 79–85 °C, the pH was reduced to 8.8–9.0 with sodium bisulfite, and was evaporated under vacuum until the juice was 50–60 percent solids (thick juice). Temperature was maintained below 85 °C and after evaporation the thick juice was filtered using cotton and fractions of thick juice were collected.

The thick juice was evaporated under vacuum until a 70–80 percent solids juice (syrup) was achieved. A solution of white sugar was added to the juice to begin crystallization. The solution was allowed to cool and then raw sugar fractions were collected. Sugar and molasses were separated by centrifugation to remove all residual molasses from the crystallized sugar. After removing molasses, white sugar was dried (if necessary) in an oven at 54–71 °C until a final moisture content of approximately 1.0 percent. Sugar and molasses samples were collected.

Ensiled beet pulp was produced by placing vacuum sealed pressed pulp in a temperature controlled chamber set at 41–46 °C for 2 days. After, the pulp was removed and stored at ambient temperature for 12 days after which the ensiled beet pulp was collected.

All samples were stored frozen for a maximum of 131 days and then analysed for residues of fludioxonil using the validated method REM 133.06.

Table 19 Residues of fludioxonil (mg/kg) in sugar beet processed commodities (Shepard, 2017, TK0044248)

Location, year, variety SUGAR BEET ROOTS	N (int)	Rate (g ai/1000 kg roots)	Sample	Fludioxonil, mg/kg [median]	PF
St. Cloud, Minnesota, United States, 2015, SX1521N	1 (-)	4.7	Pre-processing sugar beet root	1.93, 1.23, 1.66 [1.7]	-
			Raw juice	0.24	0.15
			Thick juice	0.41	0.26
			Raw sugar	0.64	0.40
			Refined sugar	0.16	0.10
			Molasses	0.90	0.56
			Wet pulp	0.14	0.09
			Ensiled pulp	0.99	0.62
			Dried pulp	2.0	1.3
			Pressed pulp	1.3	0.81
			Press water	0.27	0.17

Almonds

A processing trial on almonds was conducted in the United States during the 2018 growing season (Baillargeon, 2020, TK0351660). Fludioxonil, formulated as a WG formulation (containing 25 percent fludioxonil) was applied as 4 foliar airblast applications at 1221–1230 g fludioxonil/ha, with an application interval of 14 days, totalling 4910 g fludioxonil/ha. Crop oil was added to the application mixtures. Samples of almond nutmeats were collected 15 days after the last application (DALA). Nutmeat was processed into roasted almonds and almond oil using methods which are representative of commercial practice.

Almond nutmeat samples (~22.7 kg) were ground to a homogenous consistency. The moisture content was determined for all samples using an electronic moisture balance. If the moisture was greater than 9.0 percent, samples were dried in an oven at 54–71 °C until a final moisture content of 3.0 to 9.0 percent was achieved. Nutmeats were cracked/broken into smaller pieces for feeding into the expeller.

Moisture content of the nutmeat material was adjusted to 12.0 percent by adding water and allowed to equilibrate for a minimum of 12 hours. Moisture adjusted nutmeat material was fed through an expeller to mechanically remove a majority of oil. Cold pressing produced crude oil and presscake (meal with residual oil). Crude oil was filtered and collected. Presscake (with residual oil) was discarded. For light roasting, whole nutmeats were dry roasted at a temperature of 129–149 °C for approximately 45 minutes in a roaster. After cooling, roasted almond fractions were collected.

All samples were stored frozen for a maximum of 10.4 months and then analysed for residues of fludioxonil using the validated method AG-597B.

Table 20 Residues of fludioxonil (mg/kg) in almond nutmeat processed commodities (Baillargeon, 2020, TK0351660)

Location, year, variety ALMOND NUTMEAT	N (int)	Rate (g ai/ha)	DALA	Sample	Fludioxonil, mg/kg [median]	PF
Lost Hills, California, United States, 2018, Monterey	4 (14, 14, 14)	1221 1230	15	Almond nutmeat (pre-processing)	0.082, 0.054, 0.062 [0.062]	-
		1230		Roasted almonds	0.053	0.80
		1230		Almond oil	0.096	1.5

APPRAISAL

Fludioxonil is a phenylpyrrole fungicide that was first evaluated for toxicology and residues by the JMPR in 2004. The Meeting derived an ADI of 0-0.4 mg/kg bw, decided that an ARfD is unnecessary and concluded that the residue definition for compliance with the MRL and for dietary risk assessment in plant commodities is *fludioxonil* and the residue definition for compliance with the MRL and for dietary risk assessment in animal commodities is the *sum of fludioxonil and its benzopyrrole metabolites, determined as 2,2-difluorobenzo[1,1]dioxole-4-carboxylic acid and expressed as fludioxonil*. The residue is fat-soluble.

Fludioxonil was listed at the Fifty-second Session of the CCPR for the evaluation of additional MRLs by the 2022 JMPR for banana, mango, papaya, beans and peas with pods, pulses, sugar beets, and tree nuts.

Additionally, new toxicology data (metabolism and toxicokinetics, genotoxicity, neurotoxicity, immunotoxicity and phototoxicity) for fludioxonil as well as genotoxicity studies for several of its metabolites were submitted to the current Meeting for follow up evaluation to the 2004 JMPR. As a result of the evaluation of these new data, the current Meeting agreed that exposure risks from the metabolites CGA 192155, SYN 551031, and CGA 339833 would be covered by the health-based guidance values (HBGVs) of the parent; while the exposure risks from the metabolites CGA 227731, CGA 308565/SYN 518579, CGA 265378, and CGA 308103 should be assessed using the Threshold of Toxicological Concern (TTC) approach.

Methods of analysis

Methods REM 133.06 and AG-597B, which were previously evaluated by the 2004, 2006, and 2012 JMPR, were used for the analysis of fludioxonil in banana, mango, papaya, beans with pods, pulses, sugar beets, and tree nuts. The Meeting received additional method validation and concurrent recovery data and both methods were demonstrated to have adequate performance for recovery of fludioxonil with an LOQ of 0.01 mg/kg in all matrices. Mean recoveries were within the acceptable range of 70–120 percent with RSDs of ≤ 20 percent.

The Meeting concluded that for the commodities considered by the Meeting, the methods used in the new residue trials were sufficiently validated and suitable to measure fludioxonil in plant commodities.

Stability of residues in stored analytical samples

The stability of fludioxonil residues in samples on frozen storage was evaluated by the 2004 and 2010 JMPR for a range of commodities. Although storage stability data are not available for any high protein content commodity, collectively the existing stability data for fludioxonil are acceptable to support the storage duration of samples in the trials considered by the current Meeting. Samples in the trials were stored frozen for periods less than the period of stability demonstrated in studies supplied to the 2004 and 2010 JMPR and were satisfactory.

Definition of the residue

The current Meeting considered the toxicological properties of the metabolites CGA 192155, SYN 551031, and CGA 339833 and concluded that they are covered by the HBGV for fludioxonil. When establishing the residues definition for dietary risk assessment, the Meeting considered their potential contribution to the dietary risk under the assumption them being covered by the parent HBGV and decided that CGA 192155, SYN 551031, and CGA 339833 would not be included.

The Meeting confirms its previous recommendation on the residue definition for fludioxonil.

Results of supervised residue trials on crops

The Meeting received information on supervised field trials on banana, mango, papaya, fresh beans with pods, dry edible beans, dry peas, sugar beets, almonds, and pecans.

Banana

The critical GAP for bananas is from the Republic of Colombia and is comprised of a single post-harvest spray application at 20 g ai/hL.

The Meeting received supervised residue trials conducted in Ecuador matching the critical GAP.

For estimation of maximum residue levels, residue levels of fludioxonil in bananas (whole fruit) ranked order were (n = 6): 0.58, 0.76, 0.82, 0.93, 1.1, and 1.2 mg/kg.

Residues in the edible portion (banana pulp) for dietary risk assessment in ranked order were (n = 6): <0.01 (2), <u><0.011</u>, <u>0.014</u>, 0.015, and 0.021 mg/kg.

The Meeting estimated an STMR value of 0.013 mg/kg (based on the pulp) and a maximum residue level (based on the mean + 4×SD, whole fruit) of 2 mg/kg (Po) for banana.

Mango

Mangoes were previously evaluated by the 2012 JMPR where a maximum residue level of 2 mg/kg and a STMR of 0.02 mg/kg were estimated based on the GAP from the Republic of South Africa, comprising a single post-harvest hot dip application at 52 °C at a maximum rate of 34.5 g ai/hL.

The Meeting received a new critical GAP for mangoes from Brazil comprising a single post-harvest spray or dip application at 120 g ai/hL.

Supervised residue trials were submitted to the Meeting that were conducted in Brazil matching the new critical GAP.

For estimation of maximum residue levels, residue levels of fludioxonil in mangoes (whole fruit, dip application) ranked order were (n = 4): 3.7 (2), 3.9, and 5.0 mg/kg.

Residues in the edible portion (mango pulp, dip application) for dietary risk assessment in ranked order were (n = 4): 0.01, 0.02, 0.04, and 0.05 mg/kg.

For estimation of maximum residue levels, residue levels of fludioxonil in mangoes (whole fruit, spray application) ranked order were (n = 4): 4.4, 4.5 (2), and 5.0 mg/kg.

Residues in the edible portion (mango pulp, spray application) for dietary risk assessment in ranked order were (n = 4): <0.01, 0.03, 0.04, and 0.06 mg/kg.

The Meeting noted that both treatments were applied at the same concentration (i.e., application rate) and considered the trials to be independent according to GAP.

Fludioxonil residues in mangoes (whole fruit, dip and spray applications) in ranked order were (n = 8): 3.7 (2), 3.9, 4.4, 4.5 (2), and 5.0 (2) mg/kg.

Fludioxonil residues in mango pulp (dip and spray applications) in ranked order were (n = 8): <0.01, 0.01, 0.02, 0.03, 0.04 (2), 0.05, and 0.06 mg/kg.

The Meeting estimated an STMR value of 0.04 mg/kg (based on the pulp) and a maximum residue level (based on the mean + 4×SD, whole fruit) of 7 mg/kg (Po) for mango. The latter replaces its previous recommended maximum residue level of 2 mg/kg for mango.

Papaya

The critical GAP for papayas is from Brazil and is comprised of a single post-harvest spray or dip application at 120 g ai/hL.

The Meeting received supervised residue trials conducted in Brazil matching the critical GAP.

For estimation of maximum residue levels, residue levels of fludioxonil in papayas (whole fruit, dip application) ranked order were (n = 4): 1.2, 1.5, 2.3, and 3.2 mg/kg.

Residues in the edible portion (papaya pulp, dip application) for dietary risk assessment in ranked order were (n = 4): 0.03, 0.06, 0.10, and 0.15 mg/kg.

For estimation of maximum residue levels, residue levels of fludioxonil in papayas (whole fruit, spray application) ranked order were (n = 4): 1.5, 1.7, 2.1, and 2.7 mg/kg.

Residues in the edible portion (papaya pulp, spray application) for dietary risk assessment in ranked order were (n = 4): 0.15 (2) and 0.16 (2) mg/kg.

The Meeting noted that both treatments were applied at the same concentration (i.e. application rate) which resulted in comparable residue levels in/on the fruit and decided to combine both datasets.

Fludioxonil residues in papayas (whole fruit, dip and spray applications) in ranked order were (n = 8): 1.2, 1.5 (2), 1.7, 2.1, 2.3, 2.7, and 3.2 mg/kg.

Fludioxonil residues in papaya pulp (dip and spray applications) in ranked order were (n = 8): 0.03, 0.06, 0.10, 0.15 (3), and 0.16 mg/kg.

The Meeting estimated an STMR value of 0.15 mg/kg (based on the pulp) and a maximum residue level (based on the mean + 4×SD, whole fruit) of 5 mg/kg (Po) for papaya.

Legume Vegetables

Subgroup of beans with pods and Subgroup of peas with pods

Beans with pods were previously evaluated by the 2013 JMPR where a maximum residue level of 0.6 mg/kg and a STMR of 0.02 mg/kg were estimated in Snap bean (young pods) and Beans, except broad bean and soya bean based on the GAP from the United States for snap beans (common beans) of 4 foliar applications × 250 g ai/ha, 7-day RTI, and 7-day PHI.

Peas with pods were previously evaluated by the 2004 JMPR where a maximum residue level of 0.3 mg/kg and a STMR of 0.04 mg/kg were estimated in Peas (pods and succulent=immature seeds) based residue data for beans with pods and the GAP from France for legume (pod and seed) of 250 g ai/ha, number of applications not specified, and a 14-day PHI.

Labels were provided for registrations in Canada on succulent beans (3 foliar applications × 250 g ai/ha, 7-day RTI, and 7-day PHI) and in Latvia on fresh beans and peas with pods (3 foliar applications ×

250 g ai/ha, 10-day RTI, and 14-day PHI). Based on the shorter RTI and PHI the Meeting decided that the GAP from Canada is the critical GAP.

The Meeting received three supervised residue trials conducted in Bulgaria, France and Italy approximating the Canadian GAP which is insufficient to support a recommendation. The Meeting agreed to consider the GAP from Latvia. The Meeting received eight supervised residue trials conducted in Bulgaria, Greece, Italy, Southern France and Spain matching the Latvian GAP.

Fludioxonil residues in beans green with pods in ranked order were (n = 8): <0.01, 0.01, 0.03, 0.05, 0.06, 0.07, 0.13, and 0.48 mg/kg.

The Meeting estimated a maximum residue level of 0.8 mg/kg and an STMR value of 0.055 mg/kg for the subgroup of beans with pods, except soya bean (succulent seeds in pods). The Meeting withdrew its previous recommendations of a maximum residue level 0.6 mg/kg for beans (*Phaseolus* spp.) immature pods and succulent seeds) and a maximum residue level of 0.6 m/kg for snap beans (young pods).

Residue trials for peas with pods were not available to the Meeting. Noting that beans with pods (*Phaseolus* spp.) is a representative crop for the subgroup of peas with pods, the Meeting decided to extrapolate its recommendation for the subgroup of beans with pods and estimated a maximum residue level of 0.8 mg/kg and an STMR value of 0.055 mg/kg for the subgroup of peas with pods. The Meeting withdrew its previous recommendation of a maximum residue level 0.3 mg/kg for peas (pods and succulent=immature seeds).

Pulses

Subgroup of dry beans (except soya beans)

Dry beans were previously evaluated by the 2013 JMPR where a maximum residue level of 0.5 mg/kg and a STMR of 0.04 mg/kg were estimated based on the GAP from the United States for dried beans (except cowpeas) of 4 foliar applications × 245 g ai/ha, 7-day RTI, and 7-day PHI.

The Meeting received a new GAP for dried shelled bean (except soya bean) from Canada consisting of 3 foliar applications × 244 g ai/ha, 7-day RTI, and 7-day PHI.

The Meeting received supervised residue trials for dry beans conducted in Canada matching the Canadian GAP. The Meeting also reassessed American dry bean residue data from the 2013 JMPR where 4 foliar applications were made at a rate of 245 g ai/ha, 6 to 8-day RTI and 5 to 8-day PHI. Residue decline data for dry peas indicate a half-life of fludioxonil of approximately 7.2 days. Based on the half-life, the Meeting decided that a first application (28 days before harvest) would not contribute significantly to residues at harvest. Therefore the Meeting determined that the dry bean trials from the 2013 JMPR sufficiently approximate the GAP from Canada and are suitable for making a recommendation.

Fludioxonil residues in dry beans in ranked order were (n = 13): <0.01, <0.011, 0.018, *0.02 (2)*, 0.023, 0.029, *0.04 (2)*, *0.06 (2)*, *0.12*, and *0.23* mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR value of 0.029 mg/kg for the subgroup of dry beans (except soya beans). The Meeting withdrew its previous recommendation of a maximum residue level 0.5 mg/kg for beans (dry).

Subgroup of dry peas

Dry peas were previously evaluated by the 2004 JMPR where a maximum residue level of 0.07 mg/kg and a STMR of 0.02 mg/kg were estimated based on the GAP from Austria and Spain for pulse, dry seed of 2

foliar applications × 250 g ai/ha and a 14-day PHI. Lentils and chick-peas were previously evaluated by the 2018 JMPR where a maximum residue level of 0.3 mg/kg and an STMR value of 0.11 mg/kg were estimated based on the GAP from Canada of 3 foliar applications 244 g ai/ha, 7-day RTI, and 7-day PHI.

The Meeting received a new critical GAP for dried peas from Canada consisting of 3 foliar applications at 244 g ai/ha, 7-day RTI, and 7-day PHI.

The Meeting received the same supervised residue trials for dry peas conducted in Canada that were assessed by the 2018 JMPR for lentils and chick-peas, matching the Canadian GAP for dry peas.

Fludioxonil residues in dry peas in ranked order were (n = 7): 0.018, 0.046 (2), 0.11 (2), 0.13, and 0.17 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR value of 0.11 mg/kg for the subgroup of dry peas. The Meeting withdrew its previous recommendations of maximum residue levels of 0.07 mg/kg for peas (dry), 0.3 mg/kg for chick-pea (dry), and 0.3 mg/kg for lentil (dry).

Sugar beet

The critical GAP for sugar beets is from the United States and is comprised of a single post-harvest spray application at 4.5 g ai/1000 kg of roots.

The Meeting received supervised residue trials conducted in the United States matching the critical GAP.

Fludioxonil residues in sugar beet roots in ranked order were (n = 6): 0.64, 0.90, 0.96, 1.2, 1.7, and 1.9 mg/kg.

The Meeting estimated a maximum residue level (based on the mean + 4×SD) of 4 mg/kg (Po) and an STMR value of 1.1 mg/kg for sugar beet.

Tree nuts

Pistachios were previously evaluated by the 2004 JMPR where a maximum residue level of 0.2 mg/kg and a STMR of 0.05 mg/kg were estimated based on the GAP from the United States of 4 foliar applications × 250 g ai/ha and a 7-day PHI.

The Meeting received a GAP from the United States for numerous tree nuts consisting of 4 foliar applications at a rate of 247 g ai/ha/application, with a 14-day RTI, and a 14-day PHI. For pistachios, the GAP assessed by the 2004 JMPR remains the critical GAP for this commodity.

The Meeting received supervised residue trials for almonds and pecans conducted in the United States matching the United States GAP for tree nuts.

Fludioxonil residues in almond nutmeat in ranked order were (n = 5): ≤0.01 (3), 0.018, and 0.15 mg/kg.

Fludioxonil residues in pecan nutmeat in ranked order were (n = 5): <0.01 (5) mg/kg.

Since residues of fludioxonil were higher in almonds than in pecans, the Meeting agreed to use the almond dataset for the estimation of maximum residue levels and dietary risk assessment for tree nut commodities.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR value of 0.01 mg/kg for the group of tree nuts (except Canarium nut, Chilean hazelnut, and Pistachios).

Residues in animal feeds

Bean forage

Labels were provided for registrations in Canada on succulent beans (3 foliar applications × 250 g ai/ha, 7-day RTI, and 7-day PHI) and in Latvia on fresh beans with pods (3 foliar applications × 250 g ai/ha, 10-day RTI, and 14-day PHI). Based on the shorter RTI and PHI the Meeting decided that the GAP from Canada is the critical GAP.

The Meeting received three supervised residue trials conducted in Italy, France, and Bulgaria approximating the Canadian GAP which is insufficient to support a recommendation. The Meeting agreed to consider the GAP from Latvia.

The Meeting received eight supervised residue trials conducted in Italy, Spain, France, Bulgaria, and Greece matching the Latvian GAP.

Fludioxonil residues in beans, remaining plant, in ranked order were (n = 8): 0.40, 0.50, 2.3, 3.3, 4.2, 5.1, 7.8, and 11 mg/kg.

The Meeting estimated a highest residue of 11 mg/kg and a median value of 3.75 mg/kg for bean forage (as received).

Almond hulls

The critical GAP for almonds is from the United States consisting of 4 foliar applications at a rate of 247 g ai/ha, with a 14-day RTI, and 14-day PHI.

The Meeting received supervised residue trials for almonds conducted in the United States matching the United States GAP.

Fludioxonil residues in almond hulls in ranked order were (n = 5): 1.1, 1.7, 1.8, 3.3, and 7.6 mg/kg.

The Meeting estimated a maximum residue level of 20 mg/kg (dw) and a median value of 1.8 mg/kg for almond hulls (as received).

Fate of residues in processing

Processing data on sugar beet roots and almond nutmeat were provided. All data relevant for an estimation of maximum residue levels in processed commodities or for dietary exposure calculations are summarized in the following table.

Table 21 Processing factors and residue estimates for fludioxonil

Raw commodity	Residue in RAC, mg/kg		Processed commodity	Processing Factors	Residue in processed commodity, mg/kg	
	Max	STMR		Fludioxonil [best estimate]	MRL	STMR-P
Sugar beet roots	4	1.1	Refined sugar	0.10	--	0.11
			Molasses	0.56	--	0.62
			Ensiled pulp	0.62	--	0.68
			Dried pulp	1.3	--	1.4
Almond nutmeat	0.2	0.01	Roasted almonds	0.80	--	0.008
			Almond oil	1.5	0.3	0.015

Residues in animal commodities

Farm animal feeding studies

Farm animal feeding studies are reported in the evaluations of the 2004 JMPR (lactating dairy cow), 2013 JMPR (laying hen), and 2018 JMPR (lactating dairy cow).

Farm animal dietary burden

The Meeting has added feed items (bean forage and almond hulls) and their associated residues to the dietary burden calculations used by the 2018 Meeting. Dietary burden calculations are provided in Annex 6; the dietary burden estimates are summarized below.

Table 22 Estimated maximum dietary burdens of farm animals

Animal	Dietary burden estimates, ppm							
	Canada-United States		European Union		Australia		Japan	
	Maximum	Mean	Maximum	Mean	Maximum	Mean	Maximum	Mean
Beef cattle	11	8.7	22	12	23	10	0.10	0.10
Dairy cattle	3.9	3.2	26	12❷	38❶	9.9	0.65	0.65
Broiler poultry	0.04	0.04	1.5	0.79	0.03	0.03	0.02	0.02
Laying hen	0.04	0.04	1.9❸	0.87❹	0.03	0.03	0.02	0.02

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues and milk.

❷ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues and milk.

❸ Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues and eggs.

❹ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Animal commodity maximum residue levels

Cattle

For beef and dairy cattle, the Meeting estimated a maximum dietary burden of 38 ppm and a mean dietary burden of 12 ppm. The burdens calculated by the current meeting are substantially greater than those used by the 2018 meeting (23 ppm and 6.4 ppm, respectively). Based on the new dietary burden and the results of the dairy cattle feeding studies evaluated by the 2018 JMPR, the calculations used to estimate highest total residues for use in estimating maximum residue levels and STMR values in mammalian commodities are shown below.

Table 23 Maximum residue level and STMR in mammalian commodities

Fludioxonil feeding study	Feed level (ppm) for milk residues	Total Residues (mg/kg) in milk a	Feed level (ppm) for tissue residues	Total Residues a (mg/kg)			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study b	20	0.030	20	<0.01	0.079	0.082	0.011
	100	0.15	100	0.012	0.35	0.29	0.033
Dietary burden and high residue	38	0.06	38	0.011	0.14	0.129	0.016
STMR beef or dairy cattle							
Feeding study b	20b	0.026	20	<0.01	0.055	0.062	0.01
Dietary burden and residue estimate	12	0.016	12	0.006	0.033	0.037	0.006

Notes:

[a] Total residues = fludioxonil and its benzopyrrole metabolites, determined as 2,2-difluorobenzo[1,1]dioxole-4-carboxylic acid and expressed as fludioxonil.

[b] Although the dairy cattle feeding study evaluated by the 2004 JMPR was conducted at a feeding level of 5.5 ppm, no quantifiable residues were observed in any matrices, as such it was decided to extrapolate from the feeding study evaluated by the 2018 JMPR where quantifiable residues were observed at a 20 ppm feeding level.

The Meeting confirmed its previous recommendation of a maximum residue level of 0.02 mg/kg for meat, based on fat (from mammals other than marine mammals) and mammalian fats (except milk fats).

The Meeting recommended a maximum residue level for milks at 0.07 mg/kg and edible offal (mammalian) at 0.15 mg/kg. The Meeting estimated STMRs of 0.006 mg/kg for muscle, 0.006 mg/kg for mammalian fat, 0.037 mg/kg for edible offal (mammalian), and 0.016 mg/kg in milks. These recommendations are intended to replace previous recommendations for these ruminant matrices.

Poultry

For poultry, the Meeting estimated a maximum dietary burden of 1.9 ppm and a mean dietary burden of 0.87 ppm which are the same as the dietary burdens estimated by the 2018 JMPR.

The Meeting therefore confirmed its previous recommendations.

RECOMMENDATIONS

On the basis of the data obtained from supervised residue trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue for compliance with the MRL and for dietary risk assessment for plant commodities: *fludioxonil*.

Definition of the residue for compliance with the MRL and for dietary risk assessment for animal commodities: *sum of fludioxonil and its benzopyrrole metabolites, determined as 2,2-difluoro-benzo[1,3]dioxole-4-carboxylic acid and expressed as fludioxonil*.

The residue is fat-soluble.

Table 24 Recommendations for residues of fludioxonil from the 2022 JMPR

CCN	Crop/Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
AM 0660	Almond hulls	20		1.8	
OR 0660	Almond oil	0.3		0.015	
FI 0327	Banana	2 (Po)		0.013	
VD 0071	Beans (dry)	W	0.5		
VP 0061	Beans (*Phaseolus* spp.) immature pods and succulent seeds)	W	0.6		
VP 2060	Beans with pods, subgroup of (except soya beans (succulent seeds in pods))	0.8		0.055	
VD 0524	Chick-pea (dry)	W	0.3		
VD 2065	Dry beans, subgroup of (except soya beans)	0.3		0.029	
VD 2066	Dry peas, subgroup of	0.3		0.11	
MO 0105	Edible offal (mammalian)	0.15	0.1	0.037	
VD 0533	Lentil (dry)	W	0.3		
MF 0100	Mammalian fats (except milk fats)	0.02	0.02	0.006	
FI 0345	Mango	7 (Po)	2	0.04	

CCN	Crop/Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
MM 0095	Meat (from mammals other than marine mammals)	0.02	0.02	fat 0.006 muscle 0.006	
ML 0106	Milks	0.07	0.04	0.016	
FI 0350	Papaya	5 (Po)		0.15	
VD 0072	Peas (dry)	W	0.07		
VP 0063	Peas (pods and succulent=immature seeds)	W	0.3		
VP 2061	Peas with pods, subgroup of	0.8		0.055	
VP 4453	Snap beans (young pods)	W	0.6		
VR 0596	Sugar beet	4 (Po)		1.1	
TN 0085	Tree nuts (except Canarium nut, Chilean hazelnut, and pistachios)	0.3		0.01	
For dietary risk assessment and/or dietary burden calculations					
-	Almonds, roasted			0.008	
AL 1030	Bean forage			3.75	11
DM 0596	Sugar beet molasses			0.62	
-	Sugar beet ensiled pulp			0.68	
AM 3599	Sugar beet, pulp, dry			1.4	
DM 3523	Sugar beet, sugar refined			0.11	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The International Estimated Daily Intakes (IEDIs) of fludioxonil were calculated for the 17 GEMS/Food cluster diets using STMRs and STMR-Ps estimated by the JMPR in 2004, 2006, 2010, 2012, 2013, 2018 and current Meeting. The results are shown in Annex 3.

The ADI is 0–0.4 mg/kg bw and the calculated IEDIs were 1–6 percent of the maximum ADI. The Meeting concluded that the long-term intake of residues of fludioxonil from the uses considered by the JMPR is unlikely to present a public health concern

Acute dietary exposure

The 2004 JMPR decided that an ARfD for fludioxonil was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of fludioxonil resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

Metabolites covered by the health-based guidance value (HBGV)

The Meeting concluded that metabolites CGA 192155, SYN 551031, and CGA 339833 are covered by the HBGV.

Residues of CGA 192155, SYN 551031, and CGA 339833 were not measured in any of the supervised field trials assessed by any of the previous Meetings or by the current Meeting. The Meeting estimated the dietary exposure to combined residues of fludioxonil, CGA 192155, SYN 551031, and CGA 339833 by applying conversion factors (based on the ratio of combined residues of fludioxonil, CGA 192155, SYN 551031, and CGA 339833 against residues of the parent) to the fludioxonil STMRs for those crops in which combined residue levels of these metabolites were significant when compared against the parent (i.e. ≥ 10 percent of the parent). The Meeting concluded that metabolites CGA 192155, SYN

551031, and CGA 339833 do not contribute significantly to the dietary exposure from fludioxonil (i.e. calculated IEDIs remained in the range of 1–6 percent of the HGBV) and are unlikely to present a public health concern.

Threshold of toxicological concern (TTC) consideration for metabolites

CGA 227731

The metabolite CGA 227731 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day).

Residues of CGA 227731 were not measured in any of the supervised field trials assessed by any of the previous Meetings or by the current Meeting but were observed in the soya bean seed treatment metabolism study and accounted for 1.9 percent of the TRR in forage (0.002 mg/kg) and 1.5 percent of the TRR in hay (<0.001 mg/kg). Residues of CGA 227731 were also observed in rotational wheat matrices accounting for 10.7 percent of the TRR in forage (0.006 mg/kg) and up to 22 percent of the TRR in straw (0.016 mg/kg). Residues of CGA 227731 were not found in any of the foliar metabolism studies conducted in plants (grapes, tomatoes, peaches, green onions, or head lettuce); seed treatment metabolism studies in potatoes, rice, wheat, or cotton; or in rotational lettuce, sugar beets, mustard greens, or turnips (2004 JMPR).

The Meeting concluded that no quantifiable residues of CGA 227731 are expected in soya bean forage and hay or rotational wheat forage, but that low concentrations of CGA 227731 might be expected in wheat straw grown in rotation with fludioxonil-treated crops.

Given that metabolite CGA 227731 was only present at insignificant levels in animal feed commodities, the Meeting agreed that dietary exposure to residues of CGA 227731 is expected to be below the TTC for Cramer Class III compounds of 1.5 µg/kg bw per day and is unlikely to present a public health concern. Should further uses be considered in the future, these conclusions may need to be re-evaluated.

CGA 308565/SYN 518579 tautomeric pair, CGA 265378 and CGA 308103

The CGA 308565/SYN 518579 tautomeric pair, CGA 265378 and CGA 308103 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day).

Residues of all components were not measured in any of the supervised field trials assessed by any of the previous Meetings or by the current Meeting. Based on metabolism studies previously evaluated by the 2004 JMPR, conversion factors to fludioxonil STMRs based on the ratio of each metabolite to parent were estimated. For post harvest uses and seed treatment uses, generally no residues of the metabolites were expected.

The Meeting estimated a dietary exposures to:

- CGA 308565/SYN 518579 metabolites of 0.0956 µg/kg bw/day;
- CGA 265378 of 0.236 µg/kg bw/day; and,
- CGA 308103 of 0.198 µg/kg bw/day.

The Meeting concluded that the estimated dietary exposure to residues of CGA 308565/SYN 518579 tautomeric pair, CGA 265378 and CGA 308103 from uses considered by the JMPR is below the TTC for Cramer Class III compounds and is unlikely to present a public health concern. Should further uses be considered in the future, these conclusions may need to be re-evaluated.

REFERENCES

Report No.	Author	Year	Title, Institution, Report reference
TK0167731	Lenz, C.	2017	A15696C - Magnitude of the Residues of Azoxystrobin and Fludioxonil in Post-Harvest Application to Bananas in Ecuador 2016 Final Report, Syngenta Crop Protection, Brazil, GLP, Unpublished
VR-024/20, amendment nº 01	Bento Magagnato, M.B.	2020	Validation study of analytical methodology for residue analysis of active ingredient Fludioxonil (CGA 173506) in mango fruits (peel and pulp) - REM 133.06, L.B.S.–Pesquisas Agrícolas, Brazil, GLP, Unpublished
LBS19053	Mastrotti Pereira, F. C.	2020	A13703 and A15696 - Magnitude of residues of Fludioxonil, Azoxystrobin and its isomer R230310 in mango fruits–Brazil, 2019-2020. Final Report, Syngenta Proteção de Cultivos Ltda., Brazil, GLP Unpublished
VR-030/20, amendment nº 01	Bento Magagnato, M.B.	2020	Validation study of analytical methodology for residue analysis of active ingredient Fludioxonil (CGA173506) in papaya fruits (peel and pulp) REM 133.06, L.B.S.–Pesquisas Agrícolas, Brazil, GLP, Brazil
VR-030/20, amendment nº 02	Bento Magagnato, M.B.	2022	Validation study of analytical methodology for residue analysis of active ingredient Fludioxonil (CGA173506) in papaya fruits (peel and pulp) REM 133.06, L.B.S.–Pesquisas Agrícolas, Brazil, GLP, Unpublished
LBS19052	Mastrotti Pereira, F. C.	2020	A13703 and A15696 - Magnitude of residues of Fludioxonil, Azoxystrobin and its isomer R230310 in papaya fruits–Brazil, 2019-2020. Final Report, Syngenta Proteção de Cultivos Ltda., Brazil, GLP Unpublished
S17-03822	Yozgatli, H.K. and Breyer, N.	2018	Cyprodinil and Fludioxonil - Residue Study on Fresh Beans with Pods in Italy, Spain, Southern France, Bulgaria and Greece in 2017 Final Report, Syngenta Ltd., United Kingdom, GLP, Unpublished
TK0256751	Sagan, K.	2017	Fludioxonil/Cyprodinil WG (A9219B) & Diquat SL (A1412H) Magnitude of the Residues in or on Dry Pea (Representative Commodities for Crop Group 6C) Canada 2015 Final Report, Syngenta Canada Inc., Canada, GLP, Unpublished
TK0044248	Shepard, E.	2017	Magnitude of the Residues on Sugarbeet and in Sugarbeet Processed Commodities Azoxystrobin + Fludioxonil Residues on Sugarbeet Following Post-Harvest Treatment in 2015 Final Report, Study Number 82545, Syngenta Crop Protection LLC, United States, GLP, Unpublished
CER04164/06	Tout, N.	2006	Fludioxonil and Cyprodinil–Residue Levels on Dry Edible Beans from Trials Conducted with SWITCH 62.5WG in Canada During 2006, Syngenta Crop Protection Canada Inc., Canada, GLP, Unpublished
TK0351660	Baillargeon, M.M.	2020	Fludioxonil WG (A9219B) - Magnitude of the Residues in or on Pecan and Almond Raw Agricultural and Processed Commodities as Representative Crops of the Tree Nuts Crop Group (14-12), United States 2018 Final Report, Syngenta Crop Protection LLC, United States, GLP, Unpublished

FLUINDAPYR (328)

First draft prepared by C.M. Mahieu, Centre for Nutrition, Prevention and Health Services (VPZ), National Institute for Public Health and the Environment (RIVM), The Netherlands

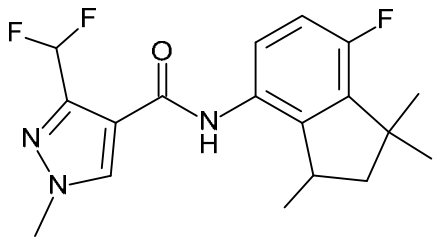
EXPLANATION

Fluindapyr (ISO name) is a new fungicide. The IUPAC name for fluindapyr is 3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-2,3- dihydro-1H-inden-4-yl)-1-methyl-1H-pyrazole-4-carboxamide. Fluindapyr is a pyrazole carboxamide fungicide that is a Complex II succinate dehydrogenase inhibitor (SDHI) that inhibits production of succinate dehydrogenase, which is a functional part of the tricarboxylic acid cycle and is linked to the mitochondrial electron transport chain.

Fluindapyr was scheduled at the Fifty-second Session of the CCPR (2020) for evaluation as a new compound by the 2021 JMPR, which was postponed to the 2022 JMPR for toxicology and for residues.

The Meeting received information on identity, physical chemical properties, plant and animal metabolism, soil degradation, residue analysis, storage stability, use patterns, residues resulting from supervised trials on cereal grains (wheat, sorghum, maize, sweet corn) and tree nuts (pecan and almond), fate of residues during processing (wheat, sorghum, maize) and livestock feeding studies.

IDENTITY

Chemical name	Fluindapyr
IUPAC:	3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-2,3-dihydro-1H-inden-4-yl)-1-methyl-1H-pyrazole-4-carboxamide
CAS:	3-(difluoromethyl)-N-(7-fluoro-2,3-dihydro-1,1,3-trimethyl-1H-inden-4-yl)-1-methyl-1H-Pyrazole-4-carboxamide
CAS Registry No:	1383809-87-7
CIPAC No:	not yet assigned
Synonyms and trade names	F9990, IR9792, F9944
Structural formula:	
	Structure is confirmed by UV-VIS, IR, ^{1}H-NMR, ^{13}C-NMR and HPLC-MS [Martinez, 2014p, 2013PCP-IFP0793]. Fluindapyr is a racemic mixture.
Molecular formula:	$C_{18}H_{20}F_3N_3O$
Molecular weight:	351.37 g/mole

Physical and chemical properties

Pure active ingredient (98.56 percent)

Parameter	Result	References	Guidelines/method
Appearance:	Purity: 98.56 percent Physical state: powdery solid Colour: light brown (but whiter than the technical active ingredient) Odour: odourless (20 °C)	[Martinez, 2014b, 2013PCP-IFP0782]	OPPTS 830.6302/ visual Munsell colour system, OPPTS 830.6303/ visual and OPPTS 830.6304/ olfactory determination
Vapour pressure:	Purity: 98.56 percent 2.85×10^{-8} Pa at 20 °C	[Martinez, 2014h, 2013PCP-IFP0795]	OPPTS 830.7950, EC Method A.4, and OECD 104/ gas saturation method
Melting point:	Purity: 98.56 percent 169.1 to 172.3 °C, without decomposition	[Martinez, 2014d, 2013PCP-IFP0787]	OECD 102, EC Method A.1, and OPPTS 830.7200/ Capillary/liquid bath
Henry's Law constant	Purity: 98.56 percent $6.13 \times 10^{-6} \times m^3 \times mol^{-1}$ at 20 °C	[Martinez, 2014h, 2013PCP-IFP0795]	OPPTS 830.7950 and OECD 104/ gas saturation method
Octanol/water partition coefficient:	Purity: 98.56 percent 4.12 at pH 7.7 and at 20°C	[Martinez, 2014i, 2013PCP-IFP0796]	OECD 107, EC Method A8, and OPPTS 830.7550 Shake flask method
Solubility in water at 20°C:	Purity: 98.56 percent 1.63 µg/mL in water at 20 ± 0.5 °C The solubility is not affected by the pH (tested at pH 4, 7 and 10).	[Martinez, 2014j, 2013PCP-IFP0798]	OECD 105, EC Method A6, CIPAC MT 157, and OPPTS 830.7840/ Shake flask method followed by column elution method
Solubility in organic solvents at 20 °C:	Purity: 98.56 percent 300-325 g/L in acetone 114-133 g/L in ethyl acetate 160-200 g/L in dichloromethane 0.31 g/L in n-heptane 80-100 g/L in methanol 20-25 g/L in toluene	[Gazzotti, 2015, 2015PCP-IFP1971]	CIPAC method MT 181 (solubility > 10 g/L), OECD 105, EC method A.6 (flask method for solubility <10 g/L), as well as OPPTS 830.7840 and OPPTS 830.7860
Density/Specific gravity/Relative density	Purity: 98.56 percent Density: 1.2735 g/mL Specific gravity: 1.2758 g/mL Relative density: 1.2735 at 20.09 °C (n=2) Was determined solvent mixture (1:5 of 2-propanol: water) and then converted to density in water.	[Martinez, 2014f, 2013PCP-IFP0780]	OECD 109, EC method A.3, CIPAC method MT 3.2, and OPPTS 830.7300, Pycnometer
Hydrolysis:	[^{14}C-5-Pyrazole]- and [^{14}C-U-Phenyl]-fluindapyr Radiochemical purity >95 percent Stable (no degradation) at pH 4, 7 and 9 over 5 days at 50 ± 0.5 ºC in the dark under sterile conditions at 0.45 mg ai/L. No degradation products were detected and the enantiomeric ratio remained unchanged.	[Russo, 2013, 2013EFT-IFP0692]	OECD 111, OPPTS 835.2110

Parameter	Result	References	Guidelines/method
	Hydrolytically stable at environmental conditions.		
Photolysis:	[^{14}C-5-Pyrazole] fluindapyr with radiochemical purity: >98 percent [^{14}C-U-Phenyl]-fluindapyr with radiochemical purity: >98 percent Photodegradation of fluindapyr was studied under simulated sunlight in sterile non-buffered water at 25± 1.0 ºC with 0.369 and 0.383 mg ai/L, for the [^{14}C-U-phenyl]-fluindapyr and the [^{14}C-pyrazole]-fluindapyr, respectively. Samples were taken after 1, 3, 6, 8, 10, 12 and 14 days and analysed by radio-TLC and LSC. Mass balances ranged from 91.7 to 100.7 percent. Degradation for fluindapyr equivalent to summer sunlight (55° North in June): U-Ph-^{14}C Pyr-^{14}C DT_{50} (years) 4.3 2.9 (arithmetic mean 3.5 years) Some minor degradation products were identified, all below 10 percent AR and the majority even below 5 percent AR. Two of the minor product were confirmed as 3-OH-fluindapyr and the pyrazole-amide. Photolysis of fluindapyr in water is water is insignificant. The distribution of the two enantiomers remained 1:1 throughout the entire irradiation duration.	[Hüben, 2017, 2015EFT-IFP2139]	OECD 316 and OPPTS 835.224, First tier: UV/VIS spectrum
Dissociation constant:	Could not be determined as fluindapyr is not capable of ionization.	[Martinez, 2014l, 2013PCP IFP0794]	OECD 112 and OPPTS 830.7370

Technical material (purity: 96.31 percent)

Parameter	Result	References	Guidelines
Appearance:	Purity: 96.31 percent Physical state: powdery solid Colour: light brown Odour: odourless (20 °C)	[Martinez, 2014a, 2013PCP-IFP0781]	OPPTS 830.6302/ visual Munsell colour system, OPPTS 830.6303/ visual and OPPTS 830.6304/ olfactory determination
Density/Specific gravity/Relative density	Purity: 96.31 percent Density: 1.2719 g/mL Specific gravity: 1.2742 g/mL Relative density: 1.2719 at 20.03 °C (n=2) Was determined solvent mixture (1:5 of 2-propanol: water) and then converted to density in water.	[Martinez 2014e, 2013PCP-IFP0779]	OECD 109, EC method A.3, CIPAC method MT 3.2, and OPPTS 830.7300, Pycnometer
pH (1 percent w/v	Purity: 96.31 percent	[Martinez, 2014g,	CIPAC, MT 75.3 and

Parameter	Result	References	Guidelines
aqueous dispersion)	5.5 at 20 °C (mean of 2 measurements)	2013PCP-IFP0784]	OPPTS 830.7000
Solubility in organic solvents at 20 °C:	Purity: 96.31 percent 300-325 g/L in acetone 114-133 g/L in ethyl acetate 160-200 g/L in dichloromethane 0.3 g/L in n-heptane 80-100 g/L in methanol 20-25 g/L in toluene	[Martinez, 2014k, 2013PCP-IFP0797]	CIPAC method MT 181 (solubility > 10 g/L), OECD 105, EC method A.6 (flask method for solubility <10 g/L).
Melting range:	Purity: 96.31 percent 160.9 to 170.5°C, without decomposition	[Martinez, 2014c, 2013PCP-IFP0786]	OECD 102, EC Method A.1, and OPPTS 830.7200/ Capillary/liquid bath
Stability:	Stable under:- ambient temperature warehouse conditions for 24 months, accelerated storage conditions for 14 days at 54 °C, and in the presence of metals/metal ions for 14 days at 54 °C.	[Martinez, 2016, 2013PCP-IFP0792] [Crane, 2014, 2013SST-IFP0846] [Martinez, 2014m, 2013PCP-IFP0783]	GIFAP Monograph No. 17, CIPAC MT 46, and OPPTS 830.6313

Formulations

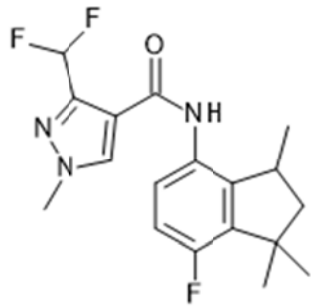
Fluindapyr has not been evaluated by JMPS and therefore no FAO specifications for technical and formulated fluindapyr have been published.

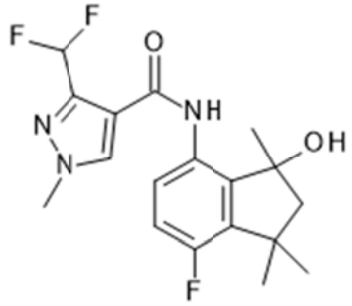
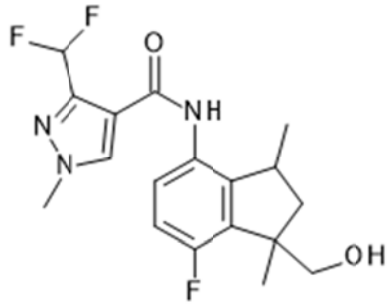
A suspension concentrate (SC) formulation, F9944-74, containing 42.4 percent (w/w) fluindapyr is commercially available in the United States of America.

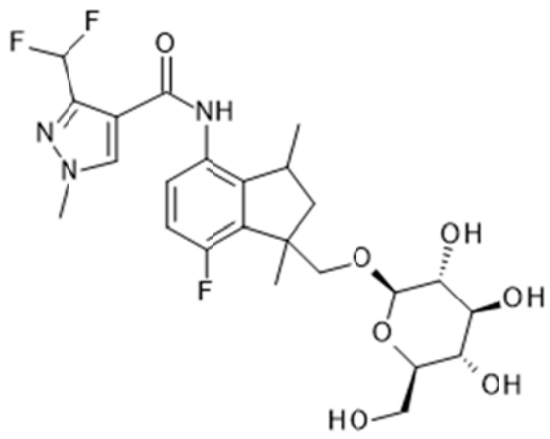
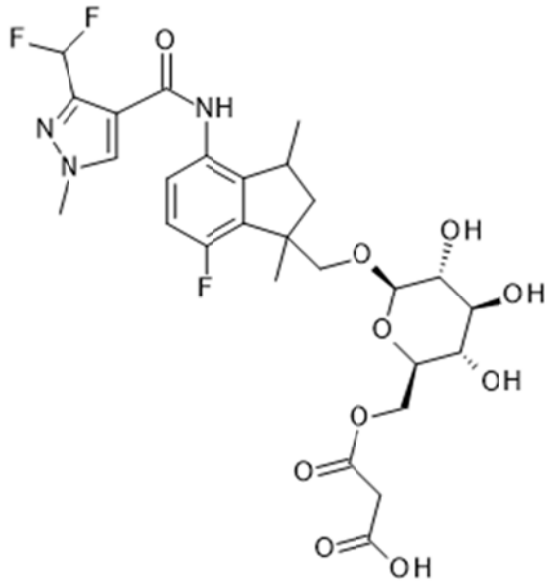
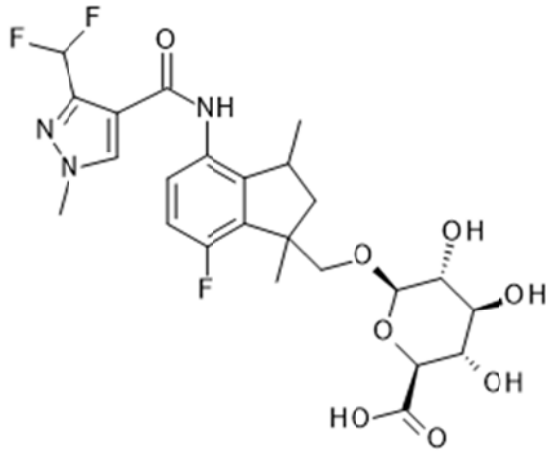
List of reference compounds used in various study reports

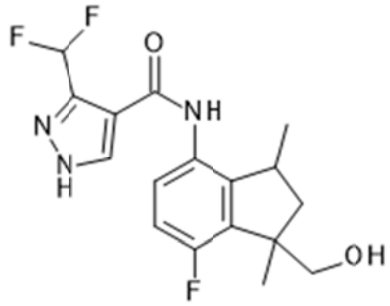
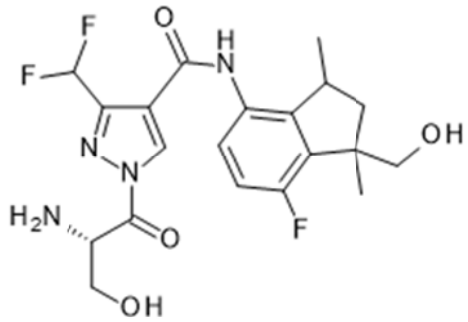
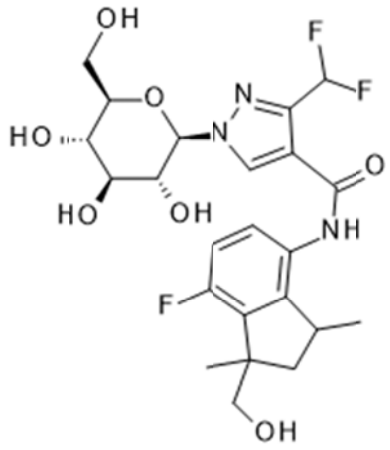
The reference compounds used in the various study reports are listed in Table 1.

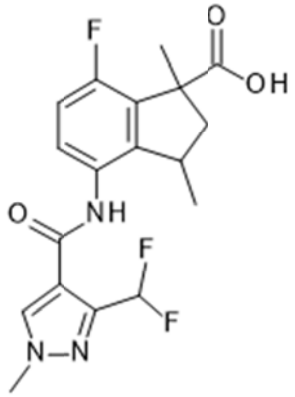
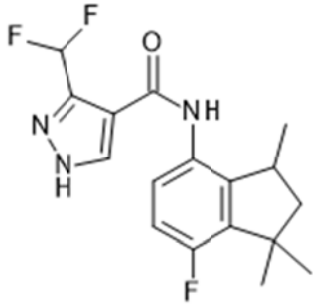
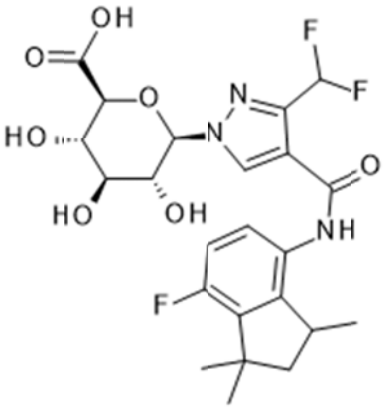
Table 1 List of reference compounds used in various study reports

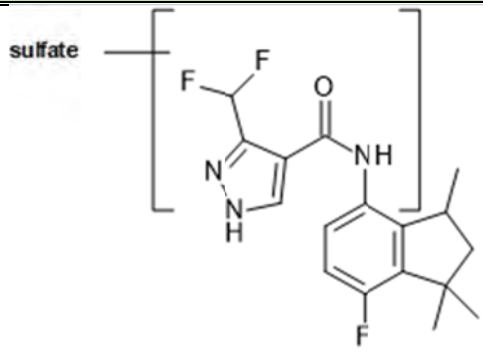
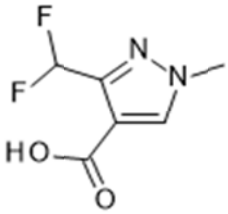
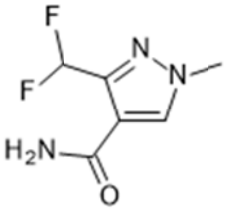
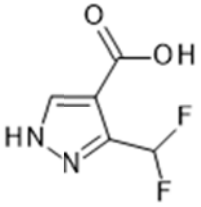
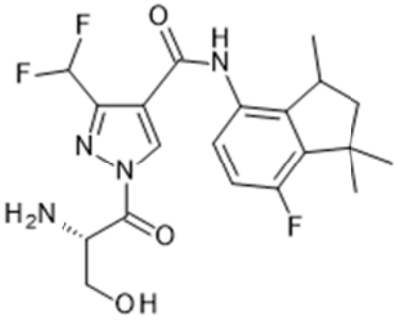
Name & abbreviation	Chemical structure	Found in
Fluindapyr *(Code: 510142)* *IR9792/F9990* 3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-indan-4-yl)-1-methyl-pyrazole-4-carboxamide *Parent*	 MW = 351 g/mol	Present in all primary (wheat, soybean, sugar beet, rice and grape) & rotational crops (lettuce, carrot and wheat) Present in all goat and hen matrices with highest residues in fat

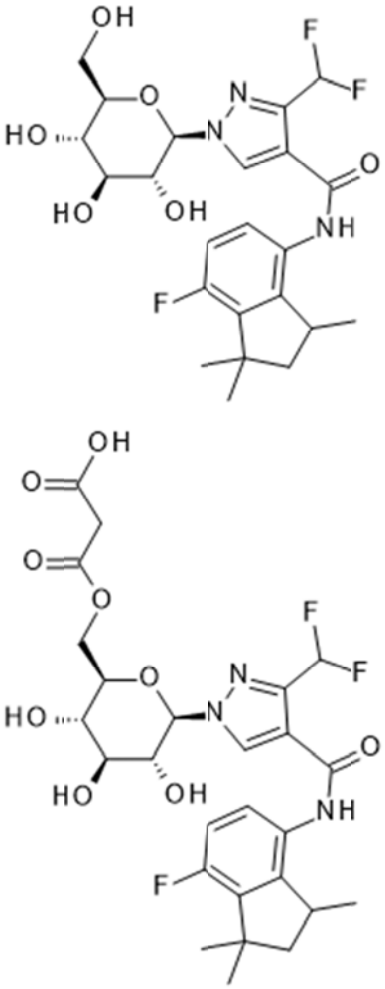
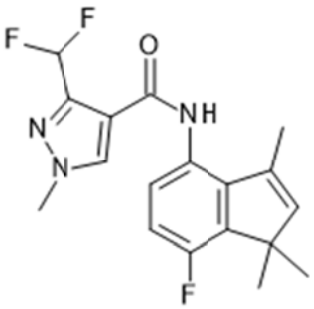
Name & abbreviation	Chemical structure	Found in
3-OH-fluindapyr *(Code:510152)* *3-Hydroxy-fluindapyr* *3-Hydroxy-IR9792/F9990* 3-(difluoromethyl)-N-(7-fluoro-3-hydroxy-1,1,3-trimethyl-2,3-dihydro-1*H*-inden-4-yl)-1-methyl-1*H*-pyrazole-4-carboxamide *M34 in animal studies*	 MW = 367 g/mol	Grape: fruit and leaves (9.3-15 percent TRR) Sugar beet: foliage and mature root (1.3-2.0 percent TRR) Wheat: forage, hay, straw (4.5-14 percent TRR), grain (20-22 percent TRR) Rice: grain and straw (8.2-11 percent TRR) Soya bean: forage and hay (2.4-4.4 percent TRR) Confined rotational crops: carrot root, leaves, lettuce, wheat hay, straw, grain Goat, fat, muscle, milk cream: 1.5-7.4 percent TRR Hen, all matrices: 0.1-1.0 percent TRR, <0.001-0.001 mg/kg Rat metabolite, faeces: 0.7-3.0 percent TRR
1-OH-Met-fluindapyr *(Code: 510153)* *1-Hydroxymethyl-fluindapyr* *1-Hydroxymethyl-IR9792/F9990* 3-(difluoromethyl)-*N*-[7-fluoro-1-(hydroxymethyl)-1,3-dimethyl- 2,3-dihydro-1*H*-inden-4-yl]-1-methyl-1*H*-pyrazole-4-carboxamide *M24, M26 in animal studies*	 MW = 367 g/mol	Grape: fruit and leaves (3.6-4.1 percent TRR) Sugar beet: foliage (62-66 percent TRR) and mature root (8.4-25 percent TRR) Wheat: forage, hay, straw (35-60 percent TRR) Rice: grain and straw (17-23 percent TRR) Confined rotational crops: carrot root, leaves, lettuce, wheat hay, straw, grain Major in: Human, rat hepatocytes Goat: 2.1-52 percent TRR in various matrices Hen, all matrices: 1.3-31.8 percent TRR, 0.001-0.019 mg/kg Rat metabolite, urine + bile: 14.4 percent of AD (applied dose)

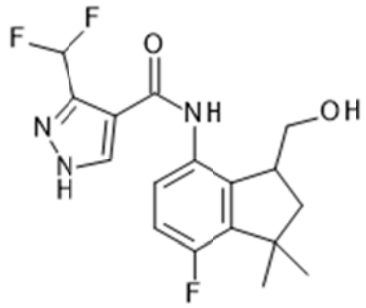
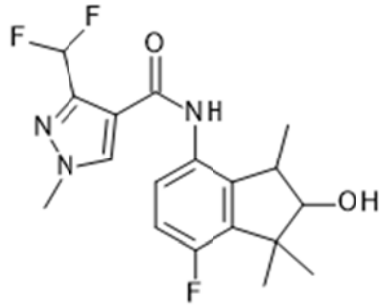
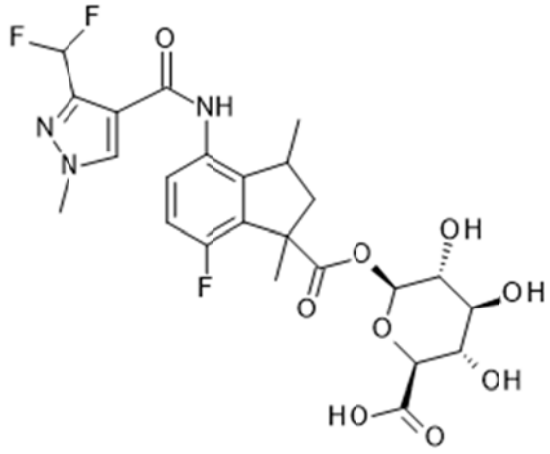
Name & abbreviation	Chemical structure	Found in
1-OH-Met-fluindapyr-Glu *(Code: N/A)* *1-Hydroxymethyl-fluindapyr glucoside (plant)* *1-Hydroxymethyl-fluindapyr gluc-mal (plant)* *1-Hydroxymethyl-fluindapyr glucuronide (animals)* 3-(difluoromethyl)-N-(7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1H-inden-4-yl)-1-methyl-1H-pyrazole-4-carboxamide gluc *M13, M15 in animal studies*	   MW-Glu= 529 g/mol MW-Glu-Mal = 615 g/mol MW-Glucuronide = 544 g/mol	As Glu: Grape: fruit (9.5-9.8 percent TRR) and leaves (20-33 percent TRR) As Glu-Mal: Grape: fruit and leaves (4.7-7.1 percent TRR) Wheat forage, hay, straw (as glucosyl conjugate and glucosyl sulphate conjugate) → see total 1-OH-Met-fluindapyr above Soya bean: forage and hay (also as Glu- or Glu-mal conjugate) → see total 1-OH-Met-fluindapyr above Goat liver: 2.9-27.7 percent TRR (max M13&M15 combined 29.6 percent TRR) Not in hen

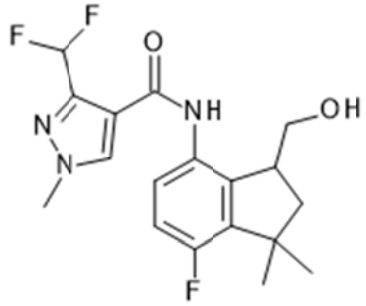
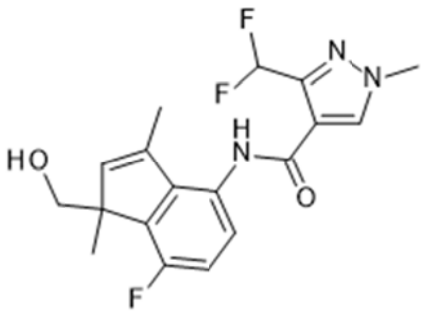
Name & abbreviation	Chemical structure	Found in
1-OH-Met-N-DesMet-fluindapyr *(Code: 510215)* *1-Hydroxymethyl-N-Desmethyl-fluindapyr* *1-Hydroxymethyl-N-Desmethyl-IR9792/F9990* 3-(difluoromethyl)-*N*-[7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1*H*-inden-4-yl]-1*H*-pyrazole-4-carboxamide *M18, M19 in animal studies*	Diastereoisomer ratio = 2.2:1  MW = 353 g/mol	Sugar beet: roots and leaves (0.4-1.8 percent TRR) Wheat: forage, hay and straw only as glucosyl conjugate, see below Soya bean: forage and hay (after acid hydrolysis) Major in: Human, rat, mouse hepatocytes Goat muscle and skimmed milk: n.d.-5.2 percent TRR, <0.001-0.001 mg/kg (M18 only) Not in hen Rat metabolite, urine + bile: 13.2 percent AD
1-OH-Met-N-DesMet-fluindapyr-gluc *(Code: N/A)* *1-hydroxymethyl-N-desmethyl-fluindapyr glucoside (plant)* *1-hydroxymethyl N-desmethyl-fluindapyr-glucuronide (animal)* 3-(difluoromethyl)-N-(7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1H-inden-4-yl)-1H-pyrazole-4-carboxamide gluc *M10 and M11 in animal metabolism studies*	  MW-N-Ser-conjugate = 440 g/mol MW-N-Glu-conjugate = 515 g/mol	Wheat: forage, hay, straw (as glucosyl conjugate) (0.38-5.1 percent TRR) Soya bean: forage and hay (as N-ser or N-glu conjugate) (9.9-12 percent TRR) Goat: liver (4.3-8.9 percent TRR) and kidney (12-24 percent TRR) Not in hen tissues and eggs.
1-COOH-fluindapyr *(Code: 510216)* *1-Carboxy-fluindapyr*	Diastereoisomer ratio = 1.72:1	Major in: Human, rat hepatocytes Sugar beet: mature root (2.1-4.1 percent TRR) Rice: grain and straw (3.7-4.4

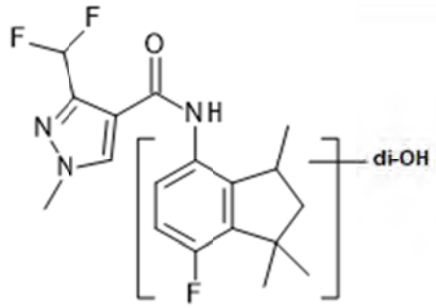
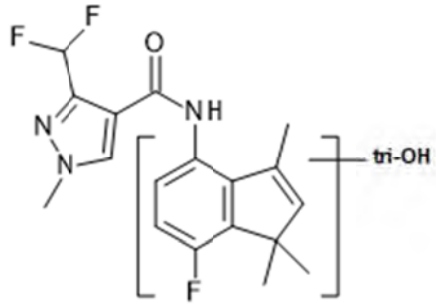
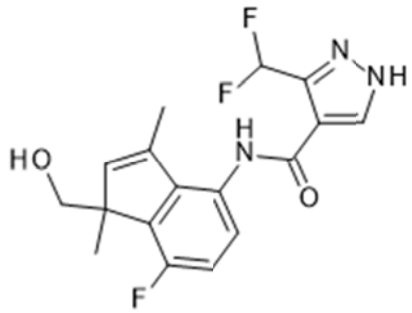
Name & abbreviation	Chemical structure	Found in
1-Carboxy-IR9792/F9990 4-(3-(difluoromethyl)-1-methyl-1*H*-pyrazole-4-carboxamido)-7-fluoro-1,3-dimethyl-2,3-dihydro-1*H*-indene-1-carboxylic acid *M23, M25 in animal studies*	 MW = 381 g/mol	percent TRR) Confined rotational crops: carrot root, leaves, lettuce, wheat hay, straw, grain Goat: liver 20-27 percent TRR) and kidney (5.3-11 percent TRR) Hen: liver and muscle: 4.7-12 percent TRR Rat metabolite, urine + bile: 10.8 percent AD
N-DesMet-fluindapyr *(Code: 510220)* *N-Desmethyl-fluindapyr* *N-Desmethyl-IR9792/F9990* 3-(difluoromethyl)-*N*-(7-fluoro-1,1,3-trimethyl-2,3-dihydro-1*H*-inden-4-yl)-1*H*-pyrazole-4-carboxamide *M33 in animal studies*	 MW=337 g/mol	Major in: human, rat, dog, mouse hepatocytes Soya bean: forage and hay (1-4.6 percent TRR) Sugar beet: foliage and mature root (0.39-2.5 percent TRR) Grape: fruit and leaves (0.1-0.2 percent TRR) Rice: grain and straw (0.4-1.0 percent TRR) Confined rotational crops: carrot leaves, mature lettuce, wheat forage, straw, and grain Goat fat, muscle and cream: 2.0-4.7 percent TRR Hen all matrices: 1.2-61.1 (liver) percent TRR, <0.001-0.066 mg/kg Rat metabolite, faeces: 2 percent AD
N-DesMet-fluindapyr-glu *(Code: N/A)* *N-Desmethyl-fluindapyr-glucuronide* *N-Desmethyl-IR9792/F9990-glucuronide* 3-(difluoromethyl)-*N*-(7-fluoro-1,1,3-trimethyl-2,3-dihydro-1*H*-inden-4-yl)-1*H*-pyrazole-4-carboxamide glucuronide *M6 and M7 in animal studies*	 MW = 513 g/mol	Goat kidney: n.d.-6.3 percent TRR, 0.003-0.005 mg/kg Not in hen tissues and eggs.

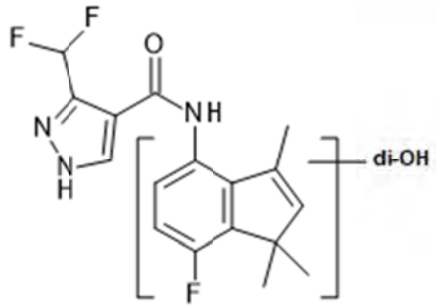
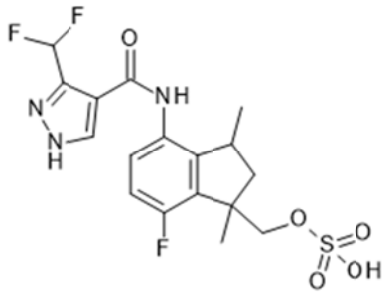
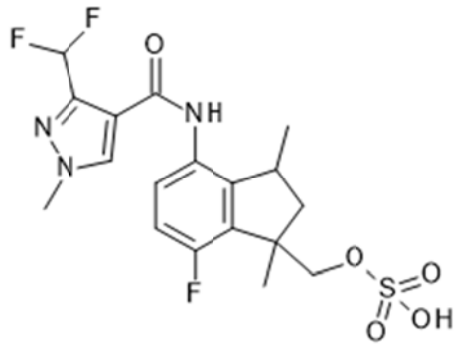
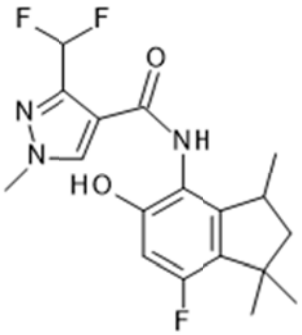
Name & abbreviation	Chemical structure	Found in
N-DesMet-fluindapyr-sulfate *Code: N/A* *M38 and M43 in animal studies* *Chemical Name: N/A*	 MW = 433 g/mol	Hen liver (0.2-1.3 percent TRR)
Pyrazole carboxylic acid *(Code: 510147)* 3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxylic acid	 MW = 176 g/mol	Wheat: forage, hay, straw (2.9-3.8 percent TRR) Sugar beet: foliage and mature root (2.4-4.6 percent TRR) Grape: fruit and leaves (0.5-1.4 percent TRR) Rice: grain and straw (3.2-3.5 percent TRR)
Pyrazole carboxamide *(Code: 510151)* 3- (difluoromethyl)- 1-methyl-1H-pyrazole-4-carboxamide	 MW = 175 g/mol	Wheat: forage, hay, straw (0.4-2.9 percent TRR) Soya bean: forage (1.1 percent TRR) Sugar beet foliage (2.6 percent TRR) Grape: fruit and leaves (1.1-2.0 percent TRR)
N-DesMet-pyrazole carboxylic acid *(Code: 510219)* 3- (difluoromethyl)- 1H-pyrazole-4- carboxylic acid	 MW = 162 g/mol	Sugar beet mature root (1.9 percent TRR, 0.002 mg/kg)
N-DesMet-fluindapyr-N-ser *(Code: N/A)* 1-(2-amino-3-hydroxy-propanoyl)-3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-indan-4-yl)pyrazole-4-carboxamide	 MW = 424 g/mol	Soya bean: forage and hay (14-18 percent TRR)
N-DesMet-fluindapyr-N1-Glu *(Code: 510171)* *and*		Soya bean: forage and hay (3.6-4.3 percent TRR)

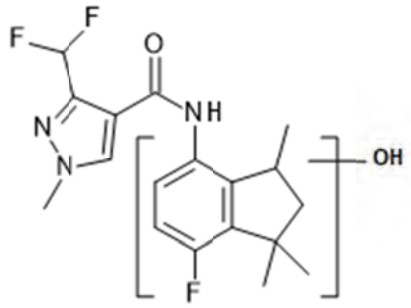
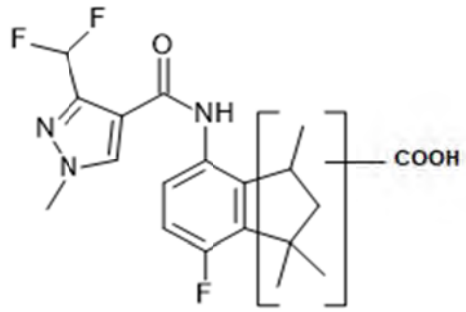
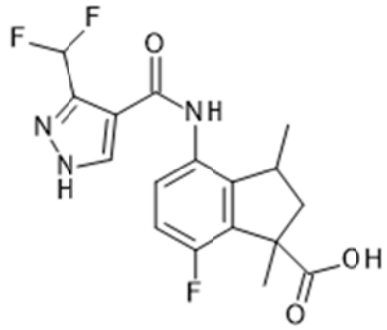
Name & abbreviation	Chemical structure	Found in
DesMet-fluindapyr-N1-Glu-Mal *(Code: N/A)* 3-(difluoromethyl)-*N*-(7-fluoro-1,1,3-trimethyl-2,3-dihydro-1*H*-inden-4-yl)-1*H*-pyrazole-4-carboxamide N1-Glu and Glu-Mal	 MW = 499 g/mol (glu) MW = 585 g/mol (glu-mal)	
Dehydro-fluindapyr *(Code: 510143)* *Dehydro-fluindapyr* *Dehydro-IR9792/F9990* 3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-1*H*-inden-4-yl)-1-methyl-1*H*-pyrazole-4-carboxamide	 MW = 349 g/mol	Grape: fruit and leaves (0.1-0.3 percent TRR) Rice: grain and straw (0.8-1.1 percent TRR) Soya bean: forage and hay (0.046-0.13 percent TRR) Not in goat tissues. Not in hen tissues and eggs (used as reference standard)

Name & abbreviation	Chemical structure	Found in
3-OH-Met-N-DesMet-fluindapyr *(Code:510329)* *3-Hydroxymethyl-N-desmethyl-fluindapyr* *3-Hydroxymethyl-N-desmethyl- IR9792/F9990* 3-(difluoromethyl)-N-[7-fluoro-3-(hydroxymethyl)-1,1-dimethyl-indan-4-yl]-1H-pyrazole-4-carboxamide *M37 in animal studies*	 MW = 353 g/mol	Sugar beet: foliage (0.24-0.64 percent TRR) Goat liver (0.1-0.9 percent percent TRR)
2-OH-fluindapyr *(Code: 510321)* *2-Hydroxy-fluindapyr* *2-Hydroxy-IR9792/F9990* 3-(difluoromethyl)-N-(7-fluoro-2-hydroxy-1,1,3-trimethyl-2,3-dihydro-1*H*-inden-4-yl)-1-methyl-1*H*-pyrazole-4-carboxamide *M27 in animal metabolism studies*	 MW = 367 g/mol	Trace in: human, rat, dog, mouse hepatocytes Goat all tissues: 0.2-8.5 percent TRR Hen all tissues: 0.1-5.7 percent TRR
1-COOH-fluindapyr-glu *(Code: N/A)* *1-Carboxy-fluindapyr-glucuronide* *1-Carboxy-IR9792/F9990-glucuronide* 4-(3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamido)-7-fluoro-1,3-dimethyl-2,3-dihydro-1H-indene-1-carboxylic acid glucuronide *M14 in animal metabolism studies*	 MW = 557 g/mol	Goat: liver (5.8-8.3 percent TRR) and trace in kidney (0.7 percent TRR) Not in hen tissues or eggs.
3-OH-Met-fluindapyr *(Code: 510218)*		Not in goat tissues. Trace in hen liver and egg: 0.1

Name & abbreviation	Chemical structure	Found in
3-Hydroxymethyl- fluindapyr *3-Hydroxymethyl-IR9792/F9990* 3-(difluoromethyl)-N-[7-fluoro-3-(hydroxymethyl)-1,1-dimethyl-2,3-dihydro-1*H*-inden-4-yl]-1-methyl-1*H*-pyrazole-4-carboxamide *M36 in hen metabolism study*	 MW = 367 g/mol	percent TRR, <0.001 mg/kg
N-DesMet-OH-fluindapyr-gluc *(Code: N/A)* *N-desmethyl-hydroxy-fluindapyr-glucuronide* *N-desmethyl-hydroxy-IR9792/F9990-glucuronide* *Structure and subsequently chemical name not confirmed* *M17 in goat metabolism studies and M5 in hen metabolism study?*	**OH** O-glucuronide Precise location of OH- not confirmed MW = 529 g/mol	Traces in goat kidney: 0.6-0.9 percent TRR, <0.001 mg eq/kg. Hen muscle: 1.8-4.0 percent TRR, <0.001 mg/kg.
1-OH-Met-dehydro-fluindapyr *(Code: N/A)* *1-hydroxymethyl- dehydro-fluindapyr* *1-hydroxymethyl- dehydro-IR9792/F9990* 3-(difluoromethyl)-N-(7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-1H-inden-4-yl)-1-methyl-1H-pyrazole-4-carboxamide *M20 in animal metabolism studies*	 MW = 365 g/mol	Goat skimmed milk: 15 percent TRR Not in hen tissues and eggs.
Di-OH-fluindapyr *(Code: N/A)* *Dihydroxy-fluindapyr*		Skimmed milk goat: 28-43 percent TRR and goat muscle: 8.6-9.4 percent TRR Not in hen tissues or eggs.

Name & abbreviation	Chemical structure	Found in
Dihydroxy-IR9792/F9990 *Chemical name: N/A* *M4 and M8 in animal metabolism studies*	 MW = 383 g/mol	Rat metabolite, urine and faeces up to 1 percent AD
Tri-OH-dehydro-fluindapyr *(Code: N/A)* *Trihydroxy-dehydro fluindapyr* *Trihydroxy-dehydro IR9792/F9990* *Chemical Name: N/A* *M2 and M3 in animal metabolism studies*	 MW = 397 g/mol	Skimmed milk goat: 5-5.6 percent TRR
1-OH-Met-N-DesMet-dehydro-fluindapyr *(Code: N/A)* *1-hydroxymethyl-N-desmethyl-dehydro-fluindapyr* *1-hydroxymethyl-N-desmethyl-dehydro-IR9792/F9990* 3-(difluoromethyl)-N-(7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-1H-inden-4-yl)-1H-pyrazole-4-carboxamide *M12 in animal metabolism studies*	 MW = 351 g/mol	Skimmed milk goat: 2.3-11 percent TRR and goat muscle: 0.8-2.9 percent TRR) Not in hen tissues and eggs. Rat metabolite, urine and faeces up to 3.3 percent AD

Name & abbreviation	Chemical structure	Found in
Di-OH-N-DesMet-fluindapyr *(Code: N/A)* *Dihydroxy-N-desmethyl-fluindapyr* *Dihydroxy-N-desmethyl-IR9792/F9990* Chemical name: N/A *M31 in animal studies*	 MW = 369 g/mol	Trace in goat kidney: 0.3-1.2 percent TRR Not in hen tissues or eggs. Rat metabolite; present in urine up to 2.2 percent AD
1-SO_4-Met-N-DesMet-fluindapyr *(Code: N/A)* *[4-[[3-(difluoromethyl)-1H-pyrazole-4-carbonyl]amino]-7-fluoro-1,3-dimethyl-indan-1-yl]methyl hydrogen sulfate* *M39 and M40 in hen metabolism study*	 MW = 433 g/mol	Hen liver 4.0-8.5 percent TRR
1-SO_4-Met-fluindapyr *(Code: N/A)* [4-[[3-(difluoromethyl)-1-methyl-pyrazole-4-carbonyl]amino]-7-fluoro-1,3-dimethyl-indan-1-yl]methyl hydrogen sulfate M41 and M42 in hen metabolism study	 MW = 447 g/mol	Hen liver 16.9-18.3 percent TRR
5'-OH-fluindapyr *(Code: 510217)* *5'-Hydroxy-fluindapyr* *5'-Hydroxy-IR9792/F9990* 3-(difluoromethyl)-N-(7-fluoro-5-hydroxy-1,1,3-trimethyl-2,3-dihydro-1*H*-inden-4-yl)-1-methyl-1*H*-pyrazole-4-carboxamide *M35 in hen metabolism study*		Not in goat tissues In various hen tissues and eggs: 1.3-6.4 percent TRR

Name & abbreviation	Chemical structure	Found in
	MW = 367 g/mol	
OH-fluindapyr *(Code: N/A)* *Hydroxy-fluindapyr* *Hydroxy- IR9792/F9990* Chemical name: N/A *M21, M22, M28, M30 in hen metabolism study*	 MW = 367 g/mol	Not in goat tissues (traces of) M21 and M22 were only observed in hen excreta. M28 was found at trace level of 1.5 percent TRR in eggs only (one label only). M30 was found at trace level of 1.2 percent TRR in hen liver.
COOH-fluindapyr *(Code: N/A)* *Carboxy-fluindapyr* *Carboxy-IR9792/F9990* Chemical name: N/A *M29 in hen metabolism study*	 MW = 381 g/mol	Not in goat tissues Trace level in hen fat 2.0 percent TRR (one label only)
1-COOH-N-DesMet-fluindapyr *(Code: 510323)* *1-Carboxy-N-Desmethyl-fluindapyr* *1-Carboxy-N-Desmethyl-IR9792/F9990* 4-[3-(difluoromethyl)-1H-pyrazole-4-amido]-7-fluoro-1,3-dimethyl-2,3-dihydro-1H-indene-1-carboxylic acid	Diastereoisomer ratio = 2.02:1  MW = 367 g/mol	Found in rat Not in goat Not in hen (used as reference standard) Major rat metabolite, urine + bile up to 24 percent AD

METABOLISM AND ENVIRONMENTAL FATE

Plant metabolism

The meeting received metabolism studies for fluindapyr after foliar applications, conducted with crops representative of four different crop groups; fruit (grape), root crops (sugar beet), cereal/grass (wheat and rice), pulses and oilseeds (soya bean). Fluindapyr was applied using [phenyl-^{14}C] and [pyrazole-^{14}C] labelled fluindapyr. The structural formula for both radiolabels is given in Figure 1.

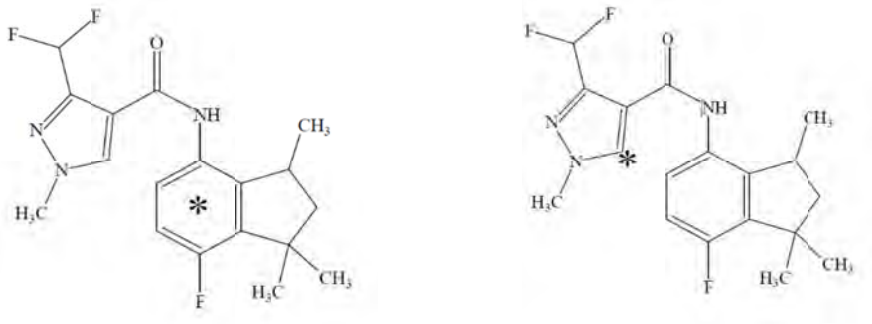


Figure 1 Position of radiolabels in fluindapyr used in plant metabolism studies with on the left the phenyl label and on the right the pyrazole label.

Foliar applications to fruits

A metabolism study using [phenyl-^{14}C] and [pyrazole-^{14}C] labelled fluindapyr was conducted outdoor in grapes (variety: Thompson seedless) after two applications of the test substance [Desai, 2017a, 2016MET-IFP2538] in Fresno CA, United States in 2016. Three established grape vines planted in 1960 were selected for the study: one untreated plot, one plot treated with phenyl-labelled fluindapyr, and one plot treated with pyrazole-labelled fluindapyr. Each plot contained one plant. A total of two applications of 237–313 g ai/ha each were conducted with [^{14}C]-labelled fluindapyr on grape vines. The first application was performed at growth stage BBCH 55 and the second application was performed at BBCH 85, resulting in an RTI of 123 days.

Fluindapyr was administered as an emulsifiable concentrate (EC) formulation by foliar spraying. Samples of mature crop (leaves and grapes) were harvested 14 days after the 2nd application. Samples were refrigerated until shipment. Grapes were kept refrigerated and leaves were frozen during shipment for analysis. All samples were processed within two weeks. At the time of processing, grape berries were rinsed with methanol, the rinse was collected, and the leaves and rinsed grapes were cryogenically homogenised, and the processed samples were kept frozen at -20 °C. All the processed samples were extracted within one month of processing. Initial sample analysis and metabolite profiling along with LC-MS characterisation/identification was performed within 6 months from harvest. Therefore, no additional extraction was conducted for storage stability evaluation.

Aliquots were taken for TRR determination by combustion. The TRR of grapes was obtained by summing radioactivity content in the grape rinse and combusted homogenized grapes.

Leaves and grapes were first extracted with acetonitrile/water (1:1, v/v). The extraction procedure was then repeated using methanol/water (1:1, v/v). All extracts were combined, assigned Ext-1 fraction and assayed by liquid scintillation counting (LSC). The remaining post extraction solid (PES) samples were combusted, followed by LSC to determine any residual ^{14}C-bound material. A further fraction of Ext-1 was evaporated to dryness and reconstituted in an aqueous solution. The concentrated Ext-1 fraction was then subjected to partitioning with dichloromethane. Subsamples of aqueous fractions remaining after dichloromethane separation of Phase I metabolites were concentrated and individually subjected to acid hydrolysis (1N HCl) under reflux at 100 °C for approximately 1 hour. All the fractions, solvent extract, concentrated extracts, and dichloromethane fractions were analysed by HPLC. Selected extracts and isolated metabolites were analysed by LC/RAM/ESI-MS and LC-MS/MS to obtain structural information of fluindapyr and metabolites. The available reference standards assayed were fluindapyr, pyrazole carboxamide, pyrazole carboxylic acid, N-DesMet-fluindapyr, dehydro-fluindapyr, 1-OH-Met-

fluindapyr (2 diastereomers), and 3-OH-Met-fluindapyr. In addition, 1-OH-Met-fluindapyr-Glu and 1-OH-Met-fluindapyr-Glu-sugar (M661/1-4) were identified by LC-MS/MS.

In leaves, the PES was greater than 0.05 mg/kg, and therefore, these fractions were further subjected to sequential enzyme (cellulase, alpha amylase, pectinase, protease), acid and base hydrolysis (both at 1N and 6N). All hydrolysis steps were conducted at 37 °C for 24 hours, except for the 6N HCl and 6N NaOH, which occurred at 100 °C for 1 hour. These fractions were quantified, but not further subjected to identification.

The enantiomeric composition of fluindapyr was determined by collection of the fluindapyr HPLC peak and further analysis by a chiral HPLC method.

The TRR, distribution of radioactivity and the identified metabolites are shown in Table 2 and Table 3. The TRR in grapes was low, while higher TRRs were detected in leaves. Rinsing of treated bunches dislodged 79 to 81 percent of TRR from the grapes. The extractable radioactivity of grapes and leaves for both labels (phenyl and pyrazole) was 99 percent TRR in grapes and 90–91 percent of the TRR in leaves. TRR in untreated control samples were ≤ 0.001 mg/kg.

The identification results of the organic/aqueous extracts (Ext-1) are shown in the table below. These extracts did not undergo any hydrolysis. The identification results of the extracts which were subjected to dichloromethane partitioning, after which the aqueous fractions were subjected to acid hydrolysis, are not shown, since these were parallel extractions and the identifications results are similar to the results in Ext-1. In the 'aqueous fraction after acid hydrolysis', some residues were retrieved, which would have originated from their conjugated counterparts, however, in the organic/aqueous extracts these metabolites were already present in their unconjugated form. The identification results of the organic/aqueous extracts are considered to better represent the original residue situation, since they did not undergo additional sample processing steps (i.e. partitioning and hydrolysis). Furthermore, high identification levels are already achieved with these organic/aqueous extracts.

Parent was the main component of the residue in grapes (63–65 percent TRR, 0.024–0.056 mg eq/kg) and leaves (38–54 percent TRR, 5.9–14 mg eq/kg). Further identified metabolites were 1-OH-Met-fluindapyr and its different conjugates (20 percent TRR, 0.017–0.07 mg eq/kg in grapes and 25–39 percent TRR, 6.0–6.4 mg eq/kg in leaves), and 3-OH-fluindapyr (12–15 percent TRR, 0.013–0.043 mg eq/kg in grapes and 9.3–12 percent TRR, 1.8–2.4 mg eq/kg in leaves), and in minor amounts N-DesMet-fluindapyr and dehydro-fluindapyr (0.1–0.3 percent TRR, 0.000–0.001 mg eq/kg in grapes and 0.019–0.064 mg eq/kg in leaves). In addition, in the extracts from the pyrazole-labelled leaves and grapes, pyrazole carboxylic acid and pyrazole carboxamide were detected at low levels (0.5–1.1 percent TRR, 0.002–0.003 mg eq/kg in grapes and 1.4–2.0 percent TRR, 0.35–0.53 mg eq/kg in leaves. Metabolite profiles from the phenyl and pyrazole labelled grape samples were similar with only minor differences in the magnitudes of their distributions.

The hydrolysis processes of the PES fractions of the leaves released small percentages of the TRR. Only the HPLC chromatograms are available as raw data in the study report (no quantitative information), showing the presence of 1-OH-Met-fluindapyr (2 diastereomers), 1-OH-Met-fluindapyr-Glu, fluindapyr, pyrazole carboxylic acid, and pyrazole carboxamide in these fractions.

Chiral analysis of fluindapyr in leaves showed no significant change in the enantiomeric composition, indicating non-selective metabolic biotransformation. However, in grapes, the parent dislodged by rinsing (79–81 percent of TRR found in grapes) showed an enantiomeric ratio approximately 50:50 while the parent found in grape extract has an *R/S* ratio of approximately 70:30 (see Table 4). No analysis of R/S isomerization for metabolites was performed.

Table 2 Total Radioactive Residues (TRRs) and distribution of radioactivity from grape samples, foliar treated with fluindapyr (2 × 237-313 g ai/ha, PHI 14d)

	Grapes				Leaves			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.089	100	0.36	100	16	100	26
Grape rinse (methanol)	79	0.070	81	0.29	-	-	-	-
Grapes after rinsing	21	0.019	19	0.069	-	-	-	-
Organic/aqueous extract	20	0.018	18	0.065	90	14	91	24
Dichloromethane extract	*7.2*	*0.006*	*5.8*	*0.021*	*62*	*9.7*	*68*	*18*
Aqueous fraction	*13*	*0.012*	*12*	*0.044*	*28*	*4.3*	*23*	*6.0*
PES (unextracted after solvent extraction)	1.2	0.001	1.0	0.04	9.7	1.5	8.9	2.3
Unextracted residue after exhaustive extraction	-	-	-	-	2.7	0.41	3.8	0.98

Table 3 Identification and characterisation of radioactivity from grape samples (organic/aqueous extract), foliar treated with fluindapyr (2 × 237-313 g ai/ha, PHI 14d)

	Grapes				Leaves			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Fluindapyr (M351/1)	63	0.056	65	0.24	38	5.9	54	14
Pyrazole carboxamide (M175/1)	-	-	1.1	0.003	-	-	2.0	0.53
Pyrazole carboxylic acid (M176/1)	-	-	0.5	0.002	-	-	1.4	0.35
Total 1-OH-Met-fluindapyr metabolites	20	0.017	20	0.07	39	6.0	25	6.4
1-OH-Met-fluindapyr-Glu-sugar (M661/1-4)	*7.1*	*0.006*	*5.8*	*0.021*	*6.0*	*0.94*	*4.7*	*1.2*
1-OH-Met-fluindapyr-Glu (M529/1)[a]	*9.0*	*0.008*	*9.3*	*0.032*	*27*	*4.2*	*18*	*4.6*
1-OH-Met-fluindapyr-Glu (M529/2)[a]	*0.5*	*0.000*	*0.5*	*0.002*	*5.7*	*0.89*	*2.4*	*0.61*
1-OH-Met-fluindapyr (M367/1)[b]	*3.3*	*0.003*	*3.8*	*0.014*	-	-	-	-
1-OH-Met-fluindapyr (M367/2)[b]	*0.3*	*0.000*	*0.3*	*0.001*	-	-	-	-
3-OH-fluindapyr (M367/3)[c]	15	0.013	12	0.043	12	1.8	9.3	2.4
N-DesMet-fluindapyr (M337/1)[c]	0.1	<0.000	0.2	0.001	0.2	0.031	0.1	0.037
Dehydro-fluindapyr (M349/1)[c]	0.1	<0.000	0.3	0.001	0.1	0.019	0.2	0.064
Unknown	-	-	-	-	2.1	0.33	-	-
Total identified	99	0.086	99	0.36	88.2	14	92	24
PES (unextracted after solvent extraction)	1.2	0.001	1.0	0.04	9.7	1.5	8.9	2.3
Cellulase hydrolysis	-	-	-	-	0.52	0.080	0.61	0.16
Alpha amylase hydrolysis	-	-	-	-	0.61	0.095	0.50	0.13
Pectinase hydrolysis	-	-	-	-	0.25	0.040	0.24	0.062

	Grapes				Leaves			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Protease hydrolysis	-	-	-	-	0.40	0.062	0.35	0.091
Acid (1N) hydrolysis	-	-	-	-	0.33	0.052	0.29	0.076
Base (1N) hydrolysis	-	-	-	-	4.0	0.63	2.1	0.55
Acid (6N) hydrolysis	-	-	-	-	0.06	0.010	0.18	0.046
Base (6N) hydrolysis	-	-	-	-	0.82	0.13	0.83	0.21
Unextracted residue after exhaustive extraction	-	-	-	-	2.7	0.41	3.8	0.98

Notes:

[a] A pair of diastereomers.

[b] A pair of diastereomers.

[c] Co-eluting peaks, separated using a secondary HPLC method in the 'dichloromethane extract + aqueous extract after hydrolysis'.

Table 4 Enantiomeric ratios of fluindapyr in grapes

Sample	Label	S (%)	R (%)
Grape leaves	Phenyl	50	50
	Pyrazole	50	50
Grape rinse	Phenyl	43	57
	Pyrazole	47	53
Grapes	Phenyl	28	72
	Pyrazole	28	72

Foliar application to root crops

The metabolic fate of fluindapyr following foliar application was studied in sugar beets [Tuffnail, 2017, 2013MET-IFP0758]. Sugar beet plants (variety: Pasteur) were grown outdoors in pots filled with sandy loam soil, in Essex, United Kingdom. A total of three applications of 113–149 g ai/ha each of either phenyl or pyrazole labelled fluindapyr were performed.

The first application was performed at BBCH 35-38, the second at BBCH 39–49 and third at BBCH 49, resulting in RTI's of 33 and 28 days, respectively. In addition, plants in two separate test plots were also treated at an exaggerated rate of once 646–648 g ai/ha at BBCH 39–49 for generating metabolites for identification purposes, if needed. Fluindapyr was administered as an emulsifiable concentrate (EC) formulation by foliar spraying. Samples of immature foliage were harvested at BBCH 39 (32 days after application 1 and 1 day prior to the second application), and mature samples (foliage and root) were harvested 30 days after the 3rd application. At maturity, leaves were separated from the roots, and roots were cleaned by gentle washing. Samples were stored frozen.

The samples were homogenised over dry ice, and the TRR was determined by combustion. The remaining samples were stored frozen. All samples were processed within 1 month, and subsequently extracted within 1 month. Initial analysis and metabolic profiling occurred within approximately 6 months after harvest. Further additional extractions were conducted *ca* 20 months (616 days) after harvest, and the metabolite profiles, generated during subsequent analysis, were compared with the initial analysis to

confirm that no significant degradation of the metabolites had taken place. The metabolic profiles of the major components were considered to be qualitatively similar. TRR levels in the experiment with the nominal use rate of 125 g ai/ha were deemed sufficient for qualitative and quantitative metabolite identification, therefore the samples from the exaggerated use rate were not analysed.

The homogenized samples were sequentially extracted with acetonitrile and acetonitrile/water (1:1 v/v). Aliquots of the resulting extracts were taken for radioactivity measurements (LSC) and for profiling using HPLC. The remaining radioactivity in the unextracted residue (PES) was determined by combustion and LSC. The concentrated acetonitrile/water extracts of mature foliage and root samples were partitioned twice with dichloromethane to separate parent and Phase I metabolites from the Phase II conjugated metabolites. Aliquots of each fraction were taken for LSC analysis. Following partitioning, a portion of the dichloromethane fraction was analysed by HPLC. Subsamples of aqueous fractions remaining after dichloromethane separation were subjected to acid hydrolysis (1N HCl) under reflux at 100 °C for approximately 1 hour, and then also analysed by LSC and HPLC. After the dichloromethane partitioning step, the aqueous root fractions contained high levels of sugars, therefore, they were subjected to a further clean-up. Aliquots were taken for LSC analysis, and chromatographic metabolite profiles generated using HPLC.

Aliquots of the concentrated acetonitrile/water extracts were treated with concentrated HCl to give solutions of approximately 1 M [H^+] concentration. Samples were incubated at ambient temperature for approximately 16 hours, and then incubated further at 60°C for 16 hours before being neutralised with NaOH (5 M). Aliquots were analysed by HPLC. Subsamples of phenyl and pyrazole concentrated foliage acetonitrile/water extracts were mixed with 5M NaOH to obtain an approximately 1 M basic solution. The samples were incubated at ambient temperature overnight before being neutralised by addition of concentrated HCl. Sample clean-up was performed, and aliquots of the supernatants were analysed by HPLC. The acetonitrile/water extract of phenyl and pyrazole foliage samples was reconstituted in 0.1 M acetate buffer, β-glucosidase enzyme was added and incubated at 37 °C for approximately 16 hours. Samples were then analysed by HPLC.

Selected leaf and root PES subsamples after extraction were subjected to enzyme hydrolysis. A PES subsample was suspended in 100 mM sodium acetate buffer (pH 5.0). Cellulase was added and incubated at 37 °C for approximately 24 hours. The enzyme hydrolysis procedure was repeated sequentially using amylase in 100 mM potassium phosphate buffer, pH 6.9 at approximately 22 °C, pectinase in 100 mM sodium acetate buffer, pH 4.0 at approximately 25 °C, and pronase in 100 mM phosphate buffer, pH 7.5 at approximately 37 °C. These fractions were quantified by LSC, but not further subjected to identification. After enzyme treatment further hydrolysis was performed on the remaining PES after the pronase treatment with EGTA in 50 mM sodium acetate buffer, pH 4.5 at approximately 80 °C for approximately 4 hours. The solid residue from this treatment (PES-2) was retained for further hydrolysis. The entire remaining PES-2 fraction was suspended in 1N HCl and agitated at 37 °C for approximately 24 hours. Aliquots of the acid hydrolysed aqueous fraction were taken for LSC evaluation of released radioactivity. The solid fraction after acid hydrolysis was individually suspended in 1N NaOH, and agitated at 37 °C for approximately 24 hours. of the base-hydrolysed aqueous fraction were taken for LSC.

Metabolite identities were confirmed by direct LC-MS/MS comparison with certified synthetic reference standards (fluindapyr and potential metabolites: N-DesMet-pyrazole carboxylic acid, pyrazole carboxamide, pyrazole carboxylic acid, 1-OH-Met-N-DesMet-fluindapyr, 1-COOH-fluindapyr, 1-OH-Met-fluindapyr (2 diastereomers), 2-Dehydro-fluindapyr, DesMet-fluindapyr-N-1-Gluc, 3-OH-fluindapyr, N-DesMet-fluindapyr, 2-OH-fluindapyr, 3-OH-fluindapyr, N-DesMet-3-COOH-fluindapyr, 3-COOH-fluindapyr, 3-

OH-Met-N-DesMet-fluindapyr). The enantiomeric ratio of fluindapyr was determined in the foliage samples.

The TRR, distribution of radioactivity and the identified metabolites in mature root and foliage are shown in Tables 5 to 7. The TRR in mature roots was relatively low (max. 0.122 mg eq/kg), with the TRR in immature foliage being higher, but with the highest TRR in mature foliage (maximum of 1.67 mg eq/kg). The TRR in control samples was below the LOQ of 0.01 mg/kg. The extractable radioactivity for all samples ranged from 90–93 percent TRR in both sugar beet roots and foliage. The remaining mature foliage and root PES samples contained residues > 0.05 mg eq/kg or >10 percent TRR, and were further investigated.

The identification results of the extracts which were subjected to dichloromethane partitioning, after which the aqueous fractions were subjected to acid hydrolysis are shown in Table 6 and Table 7. Immature foliage was not partitioned with dichloromethane, and therefore, no identification results are available for these type of extracts of immature foliage. The identification results of the organic/aqueous extracts are not shown here. These were parallel extractions and the identifications results of the organic/aqueous extracts are only shown in an appendix of the study report. This appendix has been checked and results are similar to the identification results shown here in Table 6 and Table 7. In the organic/aqueous extracts, in mature foliage, conjugated 1-OH-Met-fluindapyr has been specified as glucosyl conjugates and glucosylsulfate conjugates. In mature roots, no conjugated 1-OH-Met-fluindapyr has been detected in the acetonitrile extracts. The results of the acetonitrile extracts of the immature foliage are similar to the results of the extracts of mature foliage.

The β-glucosidase enzyme hydrolysis of extractable conjugated metabolites from the mature foliage samples from both labels was similar and showed minimal partial de-conjugation of the glucosylsulfate conjugate of 1-OH-Met-fluindapyr to liberate 1-OH-Met-fluindapyr. The results suggest a complex conjugation product. Therefore, the 1N HCl acid hydrolysis procedure was adopted for the hydrolysis of the conjugated residues remaining in the aqueous fractions after dichloromethane partitioning of the original extract. The acid hydrolysis of the conjugated metabolites in the concentrated acetonitrile/water extracts, at 60 °C gave a partial de-conjugation of the glucosylsulfate conjugate of 1-OH-Met-fluindapyr to the glucosyl conjugate and some liberation to 1-OH-Met-fluindapyr. After dichloromethane partitioning, the 1N HCl hydrolysis procedure of the conjugated metabolites performed at 100 °C gave full de-conjugation of the glucosylsulfate conjugate of 1-OH-Met-fluindapyr to the liberation of 1-OH-Met-fluindapyr.

Parent fluindapyr was a major component of the residue in sugar beet, both in mature roots (43–50 percent TRR, 0.036–0.062 mg eq/kg) and mature foliage (15–18 percent TRR, 0.25–0.30 mg eq/kg). The main component of the residue in mature foliage was 1-OH-Met-fluindapyr, both free and conjugated (62–66 percent TRR, 1.0–1.1 mg eq/kg). In sugar beet roots, this metabolite 1-OH-Met-fluindapyr was also retrieved (8.4–25 percent TRR, 0.007–0.031 mg eq/kg).

Levels of pyrazole carboxamide, pyrazole carboxylic acid and N-DesMet pyrazole carboxylic acid were low (each <5 percent TRR).

Further hydrolysis of the PES was performed on the phenyl-labelled root PES and on phenyl and pyrazole foliage PES samples, which showed very low amounts of radioactivity released. No further analysis was conducted.

Chiral analysis of fluindapyr showed no significant change in the enantiomeric composition, indicating non-selective metabolic biotransformation in sugar beet mature foliage. The chiral analysis results are presented in Table 8. No further analysis of R/S isomerization was performed.

Table 5 Total Radioactive Residues (TRRs) and distribution of radioactivity from sugar beet samples, foliar treated with fluindapyr (3 × 113-149 g ai/ha, PHI 30d)

	Mature foliage				Mature root			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	1.67	100	1.64	100	0.084	100	0.122
Organic/aqueous extract	92	1.5	93	1.5	90	0.075	92	0.11
Dichloromethane extract	*44*	*0.74*	*34*	*0.55*	*62*	*0.052*	*68*	*0.084*
Aqueous fraction	*51*	*0.85*	*60*	*0.97*	*33*	*0.027*	*23*	*0.029*
PES (unextracted after solvent extraction)	8.0	0.13	6.2	0.10	10	0.009	7.6	0.009
Unextracted residue after exhaustive extraction	2.3	0.039	1.2	0.019	6.3	0.006	-	-

Table 6 Identification and characterization of radioactivity in mature sugar beet roots, foliar treated with fluindapyr (3 × 113-149 g ai/ha, PHI 30d)

	Mature Root, phenyl label						Mature Root, pyrazole label					
	DCM fraction		Aqueous fraction after acid hydrolysis		Total		DCM fraction		Aqueous fraction after acid hydrolysis		Total	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Fluindapyr	43	0.036	-	-	43	0.036	50	0.062	-	-	50	0.062
N-DesMet-pyrazole carboxylic acid (M162)	-	-	-	-	-	-	-	-	1.9	0.002	1.9	0.002
Pyrazole carboxylic acid (M176)	-	-	-	-	-	-	-	-	2.4	0.003	2.4	0.003
1-OH-Met-N-DesMet-fluindapyr (M353)[a]	-	-	1.2	0.001	1.2	0.001	-	-	0.4	0.001	0.4	0.001
1-COOH-fluindapyr diastereoisomer 1 (M381)	-	-	4.1	0.003	4.1	0.003	-	-	2.1	0.003	2.1	0.003
1-OH-Met-fluindapyr (M367)[d]	4.4	0.004	4.0	0.003	8.4	0.007	8.6	0.011	16	0.02	25	0.031
	4.4	*0.004*	*4.0*	*0.003*	*8.4*	*0.007*	*3.0*	*0.004*	*3.0*	*0.004*	*6.0*	*0.008*
	-	-	-	-	-	-	*5.6*	*0.007*	*13*	*0.016*	*19*	*0.023*
1-OH-Met-fluindapyr diastereoisomer 2 (M367) and 1-COOH-fluindapyr diastereoisomer 2 (M381)	7.8	0.007	19	0.015	26	0.022	-	-	-	-	-	-
3-OH-fluindapyr (M367)	-	-	-	-	-	-	1.7	0.002	-	-	1.7	0.002

	Mature Root, phenyl label						Mature Root, pyrazole label					
	DCM fraction		Aqueous fraction after acid hydrolysis		Total		DCM fraction		Aqueous fraction after acid hydrolysis		Total	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
3-OH-fluindapyr (M367) and N-DesMet-fluindapyr (M337)	6.1	0.005	-	-	6.1	0.005	-	-	-	-	-	-
N-DesMet-fluindapyr (M337)	-	-	-	-	-	-	2.5	0.003	-	-	2.5	0.003
Unknowns	-	-	6.0	0.005	6.0[b]	0.005[b]	5.4	0.007	0.3	<0.001	5.5[c]	0.007[c]
Total identified:					89	0.074					86	0.11
Unextracted residue after acetonitrile extraction	-	-	-	-	10.3	0.009	-	-	-	-	7.6	0.009
Cellulase hydrolysis	-	-	-	-	1.6	0.001	-	-	-	-	-	-
Amylase hydrolysis	-	-	-	-	<LOQ	<LOQ	-	-	-	-	-	-
Pectinase hydrolysis	-	-	-	-	<LOQ	<LOQ	-	-	-	-	-	-
Pronase hydrolysis	-	-	-	-	<LOQ	<LOQ	-	-	-	-	-	-
EGTA	-	-	-	-	<LOQ	<LOQ	-	-	-	-	-	-
Acid (1N) hydrolysis	-	-	-	-	<LOQ	<LOQ	-	-	-	-	-	-
Base (1N) hydrolysis	-	-	-	-	2.4	0.002	-	-	-	-	-	-
Unextracted residue after exhaustive extraction	-	-	-	-	6.3	0.006	-	-	-	-	-	-

Notes:

[a] HPLC characterisation based on *Rt* and reference standard *Rt* value; in the raw data of the pyrazole-labelled mature foliage, this peak/region 29 was not assigned as 1-OH-Met-N-DesMe fluindapyr, which is contrary to the summary results.

[b] Includes 9 unidentified regions, the largest of which contained 1.7 percent TRR, 0.001 mg/kg.

[c] Includes 3 unidentified regions, the largest of which contained 4.6 percent TRR, 0.006 mg/kg.

[d] Pair of diastereomers; results of individual diastereomers are reported in italics.

Table 7 Identification and characterization of radioactivity in mature sugar beet foliage, foliar treated with fluindapyr (3 × 113-149 g ai/ha, PHI 30d)

	Mature foliage, phenyl label						Mature foliage, pyrazole label					
	DCM fraction		Aqueous fraction after acid hydrolysis		Total		DCM fraction		Aqueous fraction after acid hydrolysis		Total	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Fluindapyr	18	0.30	-	-	18	0.30	15	0.25	-	-	15	0.25
Pyrazole carboxamide	-	-	-	-	-	-	-	-	2.6	0.043	2.6	0.043

	Mature foliage, phenyl label						Mature foliage, pyrazole label					
	DCM fraction		Aqueous fraction after acid hydrolysis		Total		DCM fraction		Aqueous fraction after acid hydrolysis		Total	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
(M175)												
Pyrazole carboxylic acid (M176)	-	-	-	-	-	-	-	-	4.6	0.076	4.6	0.076
1-OH-Met-N-DesMet-fluindapyr (M353)[a]	0.89	0.015	0.015	0.87	1.8	0.029	1.2	0.02	0.47	0.008	1.7	0.028
1-OH-Met-fluindapyr (M367)[d]	19	0.32	47	0.8	66	1.1	13	0.22	49	0.80	62	1.0
	12	*0.20*	*24*	*0.41*	*36*	*0.60*	*9.7*	*0.16*	*26*	*0.42*	*35*	*0.58*
	7.1	*0.12*	*23*	*0.39*	*30*	*0.51*	*3.4*	*0.055*	*23*	*0.38*	*27*	*0.44*
3-OH-Me-N-DesMet-fluindapyr (M353)	-	-	0.24	0.004	0.24	0.004	-	-	0.64	0.010	0.64	0.010
3-OH-fluindapyr (M367)	2.0	0.034	-	-	2.0	0.034	1.3	0.022	-	-	1.3	0.022
N-DesMet-fluindapyr (M337)	0.6	0.009	-	-	0.56	0.009	0.39	0.006	-	-	0.39	0.006
Unknowns	3.8	0.062	1.9	0.03	5.6[b]	0.092[b]	3.6	0.059	2.7	0.045	6.4[c]	0.10[c]
Total identified:					**90**	**1.5**					**88**	**1.4**
Unextracted residue after acetonitrile extraction					8.0	0.13					6.3	0.10
Cellulase hydrolysis	-	-	-	-	1.4	0.023	-	-	-	-	1.2	0.019
Amylase hydrolysis	-	-	-	-	1.1	0.019	-	-	-	-	0.86	0.014
Pectinase hydrolysis	-	-	-	-	0.26	0.004	-	-	-	-	0.28	0.005
Pronase hydrolysis	-	-	-	-	0.79	0.013	-	-	-	-	0.94	0.015
EGTA	-	-	-	-	0.20	0.003	-	-	-	-	0.18	0.003
Acid (1N) hydrolysis	-	-	-	-	0.19	0.003	-	-	-	-	0.20	0.003
Base (1N) hydrolysis	-	-	-	-	1.7	0.029	-	-	-	-	1.4	0.024
Unextracted residue after exhaustive extraction	-	-	-	-	2.3	0.039	-	-	-	-	1.2	0.019

Notes:

[a] HPLC characterization based on *Rt* and reference standard *Rt* value; in the raw data of the pyrazole-labelled mature foliage, this peak/region 29 was not assigned as 1-OH-Met-N-DesMet fluindapyr, which is contrary to the summary results.

[b] Includes 11 unidentified regions, the largest of which contained 1.0 percent TRR, 0.016 mg/kg.

[c] Includes 11 unidentified regions, the largest of which contained 1.7 percent TRR, 0.028 mg/kg.

[d] Pair of diastereomers; results of individual diastereomers are reported in italics.

Table 8 Enantiomeric ratios of fluindapyr in mature sugar beet foliage

Label	S (%)	R(%)
Phenyl	51	49
Pyrazole	52	48

Foliar application to cereals

Metabolism of fluindapyr following foliar application was investigated in wheat [Mainolfi & Garau, 2016, 2013MET-IFP0694]. Wheat plants (variety: San Carlo) were grown outdoors in pots with sandy loam soil in Italy. Two applications of 124–130 g ai/ha each of either phenyl or pyrazole labelled fluindapyr were

performed. The first application was performed at BBCH 31–33, the second at BBCH 65. Separate treatment pots received two exaggerated applications at a rate of 601–624 g ai/ha to be used to assist in the identification of specific metabolites, in case necessary (this treatment was not further analysed). Fluindapyr was administered as an emulsifiable concentrate (EC) formulation by foliar spraying. Harvesting took place at the following growth stages; immature whole plants at forage stage (BBCH 47–49; only 1 application has taken place yet, 3–4 days after application), immature whole plants at hay stage (BBCH 83; 21–22 DALA), and mature grain and straw (41–42 DALA) separately. Samples were stored at -20 °C until analysis. Samples were stored for a maximum period of 99 days (grain) before extraction, after which TLC analysis took place within max. 21 days.

Each RAC was thoroughly and finely ground to obtain a homogeneous sample and the TRR was determined by combustion of ground subsamples. Ground samples from each RAC were sequentially extracted with acetone/water mixtures four times. A fifth extraction was conducted on straw and grain with acetone/HCl (0.1 N 50:50, v/v). The extracts from forage, hay and straw were combined by matrix and sequentially partitioned with n-heptane (extract A) and ethyl acetate (extract B; remaining aqueous phase = C). The extracts from grain were evaporated and the solid residues were solubilized in methanol for analysis. All the organic extracts and aqueous phases were analysed by LSC to determine their radioactive content.

Bound residues (after the acetone extractions) from grain and straw were processed further to promote the release of the unextracted radioactivity. The solid residues were suspended in 0.5 N $NaOH/CH_3OH$ (50:50 v/v) and incubated for 2 hours at ambient temperature. The solid residues were subsequently suspended into 0.1 percent Tween 20 and incubated for 2 hours at ambient temperature. The solid residues were subsequently incubated with enzymes capable of degrading macromolecules from the matrices. Grain residue was suspended in 50 mM ammonium acetate buffer, pH 5, and sonicated for 5 minutes. An aliquot of the suspension was taken, β-amylase was added and the sample was incubated at 37 °C for 24 hours. Then, the grain residue was suspended in the buffer and sonicated as described above, prior to incubation with cellulase. The sample was incubated at 37 °C for 24 hours. Bound residue in straw was subjected to hydrolysis with cellulase, following the same procedure described above.

Aliquots of concentrated ethyl acetate (extract B) and aqueous extracts (phase C) from pyrazole or phenyl straw samples were subjected to enzymatic hydrolysis with β-glucosidase. The aliquots were evaporated to dryness. Enzyme was added to the obtained residues and suspended with sodium acetate 0.1 M (pH 4.8). The suspensions were incubated at 37 °C for 48 hours. The aqueous extract from the pyrazole straw sample after enzymatic hydrolysis was further subjected to chemical hydrolysis. Three aliquots were incubated with HCl at different acid strengths, 1N, 2N and 6N, for 1 hour at 80 °C. The samples were then neutralised by adding ammonia solution. In addition, aliquots of concentrated ethyl acetate (extract B) and aqueous phase (phase C) from pyrazole or phenyl samples (all RAC's) were subjected to acid hydrolysis with 6N HCl for 1 hour at 80 °C.

The extractable radioactivity from each RAC was analysed by TLC. Additionally, representative extracts from straw were analysed also by HPLC. Representative RAC extracts (extract A) of different commodities were analysed by chiral HPLC in order to evaluate the enantiomeric ratio of the unchanged fluindapyr. Reference standards were fluindapyr, pyrazole carboxylic acid, pyrazole carboxamide, 3-OH-fluindapyr, 1-OH-Met-fluindapyr, and 1-OH-Met-N-DesMet-fluindapyr. The identity of each metabolite was confirmed by comparison of the LC-MS data with an authentic reference standard, or in the case of conjugates with the reference standard of the corresponding aglycone.

The TRR and distribution of radioactivity is shown in Table 9. The TRR of grain was lower than the TRR obtained from the other wheat RACs. The highest TRR was observed in straw. The extractability was

at least 93 percent in forage and hay, while it was higher than 84 percent for straw. In grain, extractability was 66 percent with the phenyl-label and 77 percent with the pyrazole-label. Radioactivity in post-extraction solid residues from forage and hay was not further investigated, but the residual radioactivity in post-extraction solid residues from straw and grain required further characterization. Partitioning of extracts from forage, hay and straw showed that the water-soluble fractions contained relevant amounts of extractable radioactivity for all samples.

Identification and characterization results are shown in Table 10 and Table 11. The majority of the detected metabolites were found in both the phenyl-labeled and pyrazole-labeled samples. Parent fluindapyr was a major metabolite in all RACs (46–56 percent TRR, 0.0093–0.021 mg eq/kg in grain and 28–37 percent TRR, 0.46–4.3 mg eq/kg in forage, hay and straw). Unconjugated 3-OH-fluindapyr was present at 20–22 percent TRR (0.0042–0.0084 mg eq/kg) in grain and 4.5–14 percent TRR (0.56–1.8 mg eq/kg) in forage, hay and straw.

HPLC radio chromatograms of the ethyl acetate extract (B) and the aqueous phase (C) from pyrazole-labelled straw, chosen as representative, before and after enzymatic hydrolysis show that the metabolic profile was simplified by the hydrolysis with β-glucosidase, demonstrating that several glucosyl conjugated compounds are present in the extracts. In addition, the profile obtained from enzymatic hydrolysis was further simplified after the subsequent chemical hydrolysis by HCl. The peak for O-glucosyl-sulfate conjugate of 1-OH-Met-fluindapyr gradually disappeared while increasing the concentration of HCl from 1N up to 6N. Meanwhile, the peak corresponding to 1-OH-Met-fluindapyr gradually increased. Acid hydrolysis (6N HCl) of the ethyl acetate extract (B) and aqueous phase (C) from forage, hay and straw produced the complete conversion of all the conjugates to the two diastereomers of 1-OH-Met-fluindapyr. The two diastereomers of 1-OH-Met-N-DesMet-fluindapyr were found in residual aqueous phases (phase C) from forage, hay and straw after acid and enzymatic hydrolysis. Therefore, after enzymatic and chemical hydrolysis, four free aglycones were detected: the main two were identified as the diastereomers of 1-OH-Met-fluindapyr, the minor two as diastereomers of 1-OH-Met-N-DesMet-fluindapyr. Free and conjugated levels of 1-OH-Met-fluindapyr ranged from 35 to 60 percent TRR (0.64–7.1 mg eq/kg) in forage, hay and straw, with the largest part being conjugated, since free 1-OH-Met-fluindapyr was <1 percent TRR in all samples. This metabolite was not detected in grain. Glucosyl conjugated 1-OH-Met-N-DesMet-fluindapyr was present at 0.38–5.1 percent TRR (0.037–0.067 mg eq/kg) in forage, hay and straw, while it was not detected in grain.

The radioactivity levels in grain extracts were too low to be analysed by chiral HPLC. The starting enantiomeric ratio S:R was approximately 50:50 in the test formulations, however, a mean ratio of 34:66 was found in the forage, hay and straw RAC samples (see Table 12).

Table 9 Total Radioactive Residues (TRRs) and distribution of radioactivity in wheat samples, foliar treated with fluindapyr (2 × 124-130 g ai/ha)

	Forage				Hay				Straw				Grain			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	1.2	100	2.2	100	5.5	100	7.4	100	15	100	13	100	0.020	100	0.038
Total extracted	99	1.2	97	2.1	103	5.7	93	6.9	90	14	84	11	66	0.014	77	0.030
n-heptane	*40*	*0.49*	*33*	*0.71*	*22*	*1.2*	*25*	*1.8*	*32*	*4.8*	*31*	*4.1*	-	-	-	-
Ethyl	*13*	*0.16*	*12*	*0.26*	*27*	*1.5*	*25*	*1.8*	*22*	*3.2*	*22*	*2.8*	-	-	-	-

	Forage				Hay				Straw				Grain			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
acetate																
Aqueous phase	*46*	*0.57*	*52*	*1.1*	*54*	*3.0*	*43*	*3.2*	*37*	*5.5*	*31*	*4.1*	-	-	-	-
PES (unextracted after solvent extraction)[a]	1.8	0.022	3.3	0.071	0.63	0.035	0.65	0.048	10	1.5	16	2.0	34	0.006	23	0.008
Unextracted residue after exhaustive extraction	-	-	-	-	-	-	-	-	6.8	1.0	7.4	0.96	27	0.005	18	0.007

Notes:

[a] For grain and straw: determined by calculation.

Table 10 Identification and characterization of radioactivity in wheat forage and hay samples, foliar treated with fluindapyr (2 × 124-130 g ai/ha)

Component[a]	Forage				Hay			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR
Fluindapyr	0.46	37	0.66	31	1.7	31	2.1	28
3-OH-fluindapyr	0.056	4.5	0.12	5.5	0.57	10	0.79	11
1-OH-Met-fluindapyr, free and conjugated (glucosyl and glucosyl sulfate conjugate)[b]	0.64	52	1.2	54	3.3	60	3.4	47
	0.45	*36*	*0.80*	*37*	*2.1*	*38*	*2.4*	*32*
	0.19	*15*	*0.36*	*17*	*1.2*	*21*	*1.0*	*14*
1-OH-Met-N-DesMet-fluindapyr as glucosyl conjugate[b]	0.063	5.1	0.057	2.6	0.037	0.66	0.066	0.89
	0.030	*2.4*	*0.029*	*1.3*	*0.021*	*0.38*	*0.022*	*0.29*
	0.033	*2.7*	*0.028*	*1.3*	*0.016*	*0.28*	*0.044*	*0.60*
Pyrazole carboxylic acid	-	-	0.082	3.8	-	-	0.22	2.9
Pyrazole carboxamide	-	-	0.0087	0.40	-	-	0.21	2.8
Sum characterised as organosoluble	0.0094	0.75	0.0059	0.27	0.031	0.56	0.040	0.54
Organic soluble compounds	*0.0076*	*0.61*	*0.0059*	*0.27*	*0.012*	*0.21*	*0.016*	*0.22*
	0.0018	*0.14*	-	-	*0.019*	*0.35*	*0.024*	*0.32*
Total characterised/identified	1.2	99	2.1	97	5.7	103	6.9	93

Notes:

[a] Results are shown for the total of the different extracts (n-heptane/extract A, ethyl acetate/extract B and residual water/phase C).

[b] A pair of diastereomers; results of individual diastereomers are reported in italics.

Table 11 Identification and characterization of radioactivity in wheat straw and grain samples, foliar treated with fluindapyr (2 × 124-130 g ai/ha)

Component[a]	Straw				Grain			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR
Fluindapyr	4.3	29	3.7	28	0.0093	46	0.021	56
3-OH-fluindapyr	1.8	12	1.8	14	0.0042	20	0.0084	22
1-OH-Met-fluindapyr, free and conjugated (glucosyl and glucosyl sulfate conjugate)[b]	7.1	48	4.6	35	-	-	-	-
	5.2	*35*	*3.1*	*24*	-	-	-	-
	2.0	*13*	*1.5*	*11*	-	-	-	-
1-OH-Met-N-DesMet-fluindapyr as glucosyl conjugate[b]	0.057	0.38	0.067	0.51	-	-	-	-
	0.027	*0.18*	*0.033*	*0.25*	-	-	-	-
	0.029	*0.20*	*0.034*	*0.26*	-	-	-	-
Pyrazole carboxylic acid	-	-	0.42	3.2	-	-	-	-
Pyrazole carboxamide	-	-	0.38	2.9	-	-	-	-
Sum characterised as organosoluble	0.11	0.74	0.074	0.57	-	-	-	-
Organic soluble compounds	*0.032*	*0.21*	*0.028*	*0.21*	-	-	-	-
	0.041	*0.27*	*0.016*	*0.12*	-	-	-	-
	0.040	*0.26*	*0.031*	*0.24*	-	-	-	-
Total characterised/identified	14	90	11	84	0.014	66	0.029	78
0.5N NaOH/MeOH (1:1)	0.20	1.3	0.18	1.4	0.0015	7.2	0.0025	6.6
1 percentTween 20 in water	0.044	0.29	0.047	0.36	<LOD	-	0.0003	0.79
B-amylase hydrolysate (starch fraction)	-	-	-	-	0.0011	5.4	0.0006	1.6
Cellulase hydrolysate (cellulose fraction)	0.71	4.8	0.72	5.5	0.0018	8.8	0.0014	3.7
Sum characterised from bound	0.96	6.4	0.95	7.3	0.0044	22	0.005	13

Notes:

[a] Results are shown for the total of the different extracts (n-heptane/extract A, ethyl acetate/extract B and residual water/phase C).

[b] A pair of diastereomers; results of individual diastereomers are reported in italics.

Table 12 Enantiomeric ratios of fluindapyr in wheat RACs

Label	Enantiomer (%)	Forage	Hay	Straw	Mean
Phenyl	S	36	35	31	34
	R	64	65	69	66
Pyrazole	S	30	37	34	34
	R	70	63	66	66

Foliar application to rice

A metabolism study using phenyl-^{14}C and pyrazole-^{14}C labelled fluindapyr was conducted in rice (variety: M-205) [Desai *et al*. 2017, 2015MET-IFP1891]. Rice plants were grown outdoors in boxes filled with sandy loam, in California, United States of America. Rice was seeded in May 2015. Two foliar applications of 114–122 g ai/ha each were performed at crop stage BBCH 33 and BBCH 75, corresponding with an RTI of 70 days. Fluindapyr was administered as an emulsifiable concentrate (EC) formulation. Samples of mature crop (dehulled grain and straw) were harvested 58 days after the 2nd application and frozen

(approximately at -20 °C) prior to shipment for analysis. All samples received were processed within two weeks. All the processed samples were extracted within one month and initial sample analysis was performed within 6 months (41 days) from harvest.

Combustion analysis of plant samples was performed using finely homogenised/ground straw and grain. Subsamples of rice samples were extracted three times with a solution of acetonitrile/water (1:1, v/v). Further extraction was repeated three additional times using methanol/water (1:1, v/v). All extracts were combined, assigned Ext-1 fraction, and analysed by LSC. A fraction of Ext-1 was also concentrated to dryness, reconstituted in aqueous solution and subjected to partitioning with dichloromethane. After dichloromethane partitioning, the separated aqueous fraction was subjected to acid hydrolysis (1N HCl) under reflux at 100 °C for approximately 1 hour. All the fractions were then concentrated and analysed by HPLC analysis. The remaining PES samples were air-dried and combusted, followed by LSC to determine residual ^{14}C-bound. The initial solvent extracts (Ext-1) were concentrated to remove organic solvent. Representative sample extracts were further analysed by chiral HPLC. Selected extracts and isolated metabolites were analysed by LC/RAM/ESI-MS and MS/MS. Metabolite reference standards were used for qualitative HPLC chromatographic analyses only. Reference standards were fluindapyr, pyrazole carboxylic acid, pyrazole carboxamide, N-DesMet-fluindapyr, dehdyro-fluindapyr, 1-OH-Met-fluindapyr (2 diastereomers), 3-OH-fluindapyr, fluindapyr-1-COOH (2 diastereomers).

The TRR and distribution of radioactivity are shown in Table 13. The TRR in rice grain was moderate (0.65 mg eq/kg and 0.78 mg eq/kg). Straw contained higher TRR levels of 1.83 mg eq/kg and 2.25 mg eq/kg for phenyl and pyrazole label respectively. Residues in control samples were <0.001 mg eq/kg. The data show ≥92 percent of the TRR was extracted with acetonitrile/water solvent mixture. The PES fractions from both phenyl- and pyrazole treatment constituted <10 percent TRR and <0.05 mg/kg, respectively. Therefore, the PES fractions from these samples were not subjected to further analysis.

The identification results of the organic/aqueous extracts (Ext-1) are shown in Table 14. These extracts did not undergo any hydrolysis. The identification results of the extracts which were subjected to dichloromethane partitioning, after which the aqueous fractions were subjected to acid hydrolysis, are not shown, since these were parallel extractions and the sum of the identifications results in the dichloromethane extract' and the 'aqueous fraction after acid hydrolysis' are similar to the results in Ext-1. In the 'aqueous fraction after acid hydrolysis', some residues were retrieved (parent, the two diastereomers of 1-OH-Met-fluindapyr, and 1-COOH-fluindapyr), which would have originated from their conjugated counterparts, however, in the organic/aqueous extracts these metabolites were already present in their unconjugated form. The identification results of the organic/aqueous extracts are considered to better represent the original residue situation, since they did not undergo additional sample processing steps (i.e. partitioning and hydrolysis). Furthermore, high identification levels are already achieved with these organic/aqueous extracts.

Metabolite profiles from the straw and mature grain samples of the phenyl and pyrazole labels were essentially similar with minor differences in the magnitudes of their distributions. Parent was the major residue in all samples, ranging from 53 to 57 percent TRR (0.37–1.2 mg eq/kg) in grain and straw. The two diastereomers of 1-OH-Met-fluindapyr, and 3-OH-fluindapyr were also present in large quantities, ranging from 17 to 23 percent TRR (0.11–0.43 mg eq/kg) and from 8.2 to 11 percent TRR (0.053–0.25 mg eq/kg) for each compound, respectively in rice grain and straw. Metabolites 1-COOH-fluindapyr, N-DesMet-fluindapyr and dehydro-fluindapyr accounted for <5 percent TRR (<0.1 mg eq/kg) in both matrices. In addition, in the extracts from the pyrazole-labelled grain and straw, pyrazole carboxylic acid was detected, indicating that breakage of the carboxamide bond in fluindapyr was a minor metabolic pathway.

Chiral analysis of fluindapyr in rice grain (husked rice) and straw indicated that a slight change in enantiomeric ratio (R:S) took place and was determined to be approximately 60:40. This is presented in Table 15.

Table 13 Total Radioactive Residues (TRRs) and distribution of radioactivity from rice samples, foliar treated with fluindapyr (2 × 114-122 g ai/ha, PHI 58d)

	Grain (husked rice)				**Straw**			
	Phenyl-label		**Pyrazole-label**		**Phenyl-label**		**Pyrazole-label**	
	percent TRR	**mg eq/kg**	**percent TRR**	**mg eq/kg**	**percent TRR**	**mg eq/kg**	**percent TRR**	**mg eq/kg**
TRR	**100**	**0.78**	**100**	**0.65**	**100**	**1.8**	**100**	**2.2**
Organic/aqueous extract	94	0.74	93	0.60	98	1.8	98	2.2
Dichloromethane extract	*78*	*0.61*	*71*	*0.46*	*69*	*1.3*	*77*	*1.7*
Aqueous fraction	*16*	*0.13*	*22*	*0.14*	*29*	*0.52*	*21*	*0.48*
PES (unextracted after solvent extraction)	6.0	0.047	7.4	0.048	2.4	0.044	2.1	0.047

Table 14 Identification and characterisation of radioactivity from rice samples (organic/aqueous extract), foliar treated with fluindapyr (2 × 114-122 g ai/ha, PHI 58d)

	Grain (husked rice)				Straw			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Fluindapyr (M351/1)	53	0.41	57	0.37	55	1.0	56	1.2
Pyrazole carboxylic acid (M176/1)	-	-	3.2	0.021	-	-	3.5	0.079
1-OH-Met-fluindapyr (M367)[a]	22	0.17	17	0.11	23	0.43	19	0.21
	13	*0.1*	*9.7*	*0.063*	*14*	*0.26*	*11*	*0.026*
	9.5	*0.074*	*7.4*	*0.048*	*9.0*	*0.17*	*8.1*	*0.18*
1-COOH-fluindapyr (M381/1)	4.2	0.033	4.0	0.026	3.7	0.068	4.4	0.099
3-OH-fluindapyr (M367/3)[b]	9.1	0.072	8.2	0.053	11	0.20	11	0.25
N-DesMet-fluindapyr (M337/1)[b]	0.8	0.006	0.4	0.003	1.0	0.018	0.7	0.016
Dehydro-fluindapyr (M349/1)[b]	0.8	0.006	1.0	0.006	0.9	0.016	1.1	0.025
Unknown	4.5	0.035	-	-	-	-	-	-
Total identified	**94**	**0.74**	**91**	**0.59**	**95**	**1.7**	**96**	**1.9**

Notes:

[a] a pair of diastereomers; results of individual diastereomers are reported in italics.

[b] co-eluting peaks, separated using a secondary HPLC method in the 'dichloromethane extract'.

Table 15 Enantiomeric ratios of fluindapyr in rice RACs

Label	Enantiomer (%)	Grain	Straw

Phenyl	S	42	42
	R	58	58
Pyrazole	S	44	43
	R	56	57

Foliar application to soya bean

The metabolism of fluindapyr has been investigated in soya beans (variety: Mycogen 5N45122) after three foliar applications [Desai, 2016, 2013MET-IFP0730]. Soya bean seeds were sown in May 2013 in boxes and maintained outdoors in sandy loam soil in California, United States. Three applications of 117–129 g ai/ha each of either phenyl or pyrazole labelled fluindapyr were performed. The first application was performed at BBCH 15–16, the second at BBCH 55-60, and the third application at BBCH 79, corresponding with RTIs of 21 and 60 days. Separate treatment plots received once an exaggerated application at a rate of 667–676 g ai/ha at BBCH 55–60 to be used to assist in the identification of specific metabolites, in case necessary (this treatment was not further analysed). Fluindapyr was administered as an emulsifiable concentrate (EC) formulation by foliar spraying. Immature forage was sampled at 21 days after the first application (28 prior to the second application), hay was harvested after two applications, and mature seeds were collected 30 days after the third application. Harvested commodities were processed cryogenically. The TRR in each sample was determined via combustion. The remaining samples were stored frozen (approximately -20 °C). Samples were subsequently extracted within one month after processing, and initially analysed within 6 months from harvest.

Subsamples of soya bean samples were first extracted with a solution of 1:1 acetonitrile/water (v/w). The remaining solids were then re-blended with a solution of 1:1 acetonitrile/water (v/w). The extraction step was repeated once more with 1:1 acetonitrile/water. The extraction procedure was repeated three additional times using 1:1 methanol:water (v/w). All extracts were combined, assigned Ext-1 fraction, and assayed by LSC. Ext-1 fraction was concentrated and analysed by HPLC. A fraction of Ext-1 was also concentrated to dryness and reconstituted in aqueous solution. This was then subjected to partitioning with dichloromethane. After dichloromethane partitioning, the separated aqueous fraction was subjected to acid hydrolysis (1N HCl) under reflux at 100°C for approximately 1 hour. All the fractions were then concentrated separately for analysis by HPLC. The remaining PES samples were air-dried and combusted, followed by LSC to determine residual ^{14}C-bound. The initial solvent extracts (Ext-1) were concentrated to remove organic solvent.

The hay PES samples were subjected to sequential enzyme hydrolysis. A representative PES subsample was suspended in 100 mM sodium acetate buffer (pH ~5.0). Cellulase solution was prepared in 100 mM sodium acetate buffer. Fractions were then mixed with cellulase enzyme and incubated at 37 °C for about 24 hours. Aliquots were taken for LSC. The procedure was repeated for sequential enzyme hydrolysis with amylase, pectinase, and protease enzymes. The solid residues (PES-2) were transferred into a container for further hydrolysis. The bound residue samples remaining after sequential enzyme hydrolyses were subjected to further hydrolysis with 1N acid and 1N base. The entire remaining PES-2 fraction from each sample was suspended in 2× v/w of 1 N HCl. The mixture was sonicated for 15 minutes and stirred in a water bath at 37 °C for approximately 24 hours. A single aliquot of the aqueous fraction was taken for LSC evaluation of released radioactivity. The solid fraction after acid hydrolysis was individually suspended in 2 × v/w of 1 N NaOH and the mixture was vortex-mixed and stirred at ambient temperature for approximately 24 hours. A single aliquot of the aqueous fraction was taken for LSC.

Representative sample extracts were analysed by chiral HPL. Selected extracts and isolated metabolites were analysed by LC/MS with on-line UV and radio-detection and LC-MS/MS.

Reference standards were fluindapyr, pyrazole carboxylic acid, pyrazole carboxamide, N-DesMet-fluindapyr, dehydro-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, 1-OH-Met-fluindapyr (2 diastereomers), 3-OH-fluindapyr, and DesMet-fluindapyr-N1-glycoside.

The TRR and distribution of radioactivity are shown in Table 16. The TRR in seeds was clearly lower than the TRR in forage and hay. Residues in control samples were <0.001 mg eq/kg.

The data showed ≥90 percent of the TRR was extracted with acetonitrile/water. Less than 10 percent of the TRR remained as bound residues. Based on the values ≥ 0.050 mg/kg in the PES from the two hay samples, these samples were subjected to enzyme, acid, and base hydrolysis. Sequential treatment of the PES fractions from mature hay samples released relatively insignificant amounts of TRR (less than 0.01 mg/kg in each fraction) from the PES fractions (see Table 17), suggesting natural incorporation. Since the amount of released radioactivity from various PES fractionations was less than 0.01 mg eq/kg, no further characterisation of these fractions was performed.

The identification results of the organic/aqueous extracts (Ext-1) are shown in Table 17. These extracts did not undergo any hydrolysis. The identification results of the extracts which were subjected to dichloromethane partitioning, after which the aqueous fractions were subjected to acid hydrolysis, are not shown, since these were parallel extractions and the sum of the identifications results in the dichloromethane extract' and the 'aqueous fraction after acid hydrolysis' are similar to the results in Ext-1. There is one small exception: pyrazole carboxylic acid was detected in the 'aqueous fraction after acid hydrolysis', while not observed in the organic/aqueous extracts and the 'dichloromethane extract'. This would imply that conjugated pyrazole carboxylic acid is present in low amounts. Interestingly, in the 'aqueous fraction after acid hydrolysis', some residues were retrieved (the two diastereomers of 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and N-DesMet-fluindapyr-N-Ser), which probably have originated from their conjugated counterparts, and which in the organic/aqueous extracts were indeed present in their conjugated form. The identification results of the organic/aqueous extracts are considered to better represent the original residue situation, since they did not undergo additional sample processing steps (i.e. partitioning and hydrolysis). Furthermore, high identification levels are already achieved with these organic/aqueous extracts.

The identification results in Table 17 show that the metabolite profiles from the forage and hay samples for both labels were similar with only minor differences in the magnitudes of their distributions. Several metabolites have been observed, therefore, it can be concluded that fluindapyr has been rather intensively metabolised in soya bean plants. Parent was only present at 5.7–12 percent TRR (0.017–0.22 mg eq/kg) in forage and hay. Conjugated 1-OH-Met-fluindapyr, DesMet-fluindapyr, and 1-OH-Met-N-DesMet-fluindapyr were detected in larger quantities: 1-OH-Met-fluindapyr-conjugates ranged from 31–40 percent TRR (0.12–0.57 mg eq/kg), DesMet-fluindapyr-conjugates were present from 14–18 percent TRR (0.046–0.33 mg eq/kg) and 1-OH-Met-N-DesMet-fluindapyr-conjugates ranged from 9.5–12 percent TRR (0.034–0.17 mg eq/kg). In addition, N-DesMet-fluindapyr-N-Ser, 3-OH-Fluindapyr, N-DesMet-Fluindapyr and dehydro-fluindapyr were present in quantities < 5 percent TRR (< 0.1 mg eq/kg). Pyrazole carboxamide was only present in forage (1.1 percent TRR, 0.006 mg eq/kg).

No tabular results are shown for the soya bean seeds, since insufficient radioactivity was detected for characterisation and identification of metabolites in soya bean seed. In the phenyl-labelled soya bean seed samples, the profile contained 13 unknowns at levels of <0.001–0.001 mg eq/kg; and also in these samples after acid hydrolysis only unknowns were observed (maximum of 0.003 mg eq/kg). The pyrazole-labelled soya bean samples contained 13 unknowns at levels of 0.001–0.023 mg eq/kg, which

included 3 unknowns that were pyrazole carboxylic acid fragments. In addition, in the pyrazole-labelled soya bean sample, pyrazole carboxamide was identified at 6.7 percent TRR (0.006 mg eq/kg). In these samples after acid hydrolysis, similarly, only unknowns were retrieved (max. 0.013 mg eq/kg), in addition to pyrazole carboxamide at 0.032 mg eq/kg and pyrazole carboxylic acid at 0.003 mg eq/kg.

The chiral analysis results are presented in Table 18. The enantiomeric ratio R/S changed from approximately 50:50, as found in the test formulations, to 60:40 in hay and forage.

Table 16 Total Radioactive Residues (TRRs) and distribution of radioactivity in soya bean samples, foliar treated with fluindapyr (3 × 117-129 g ai/ha)

	Forage				Hay				Seed			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.30	100	0.51	100	1.8	100	1.6	100	0.013	100	0.090
Organic/aqueous extract	90	0.27	90	0.47	95	1.7	94	1.5	92	0.012	98	0.088
Dichloromethane extract	*30*	*0.090*	*22*	*0.11*	*18*	*0.33*	*18*	*0.29*	*5.6*	*0.001*	*1.3*	*0.001*
Aqueous fraction	*64*	*0.19*	*70*	*0.36*	*79*	*1.4*	*74*	*1.2*	*90*	*0.011*	*96*	*0.086*
PES (unextracted after solvent extraction)	9.7	0.029	8.9	0.046	5.0	0.090	5.9	0.095	8.2	0.001	1.8	0.002
Unextracted residue after exhaustive extraction	-	-	-	-	2.8	0.049	2.9	0.046	-	-	-	-

Table 17 Identification and characterisation of radioactivity from soya bean samples (organic/aqueous extract), foliar treated with fluindapyr (3 × 117-129 g ai/ha)

	Forage				Hay			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Fluindapyr (M351/1)	5.7	0.017	5.9	0.031	12	0.22	12	0.19
Pyrazole carboxamide (M175/1)	-	-	1.1	0.006	-	-	ND	NA
Sum of 1-OH-Met-N-DesMet-fluindapyr-N-conjugates	12	0.034	12	0.063	9.5	0.17	9.9	0.16
1-OH-Met-N-DesMet-fluindapyr-N-Ser (M440/1)	*5.5*	*0.016*	*5.5*	*0.028*	*3.7*	*0.066*	*4.6*	*0.072*
1-OH-Met-N-DesMet-fluindapyr-N-Glu (M515/1)	*6.1*	*0.018*	*6.8*	*0.035*	*5.8*	*0.10*	*5.3*	*0.085*
Sum of 1-OH-Met-fluindapyr conjugates	40	0.12	38	0.19	32	0.57	31	0.48
1-OH-Met-fluindapyr-Glu (M529/1)	*6.3*	*0.019*	*8.2*	*0.042*	*8.1*	*0.14*	*9.0*	*0.14*
1-OH-Met-fluindapyr-Glu-Mal (M615/1)	*27*	*0.082*	*26*	*0.14*	*21*	*0.37*	*18*	*0.29*
1-OH-Met-fluindapyr-Glu-Mal	*6.1*	*0.018*	*3.0*	*0.016*	*3.2*	*0.057*	*3.4*	*0.054*

	Forage				Hay			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
(M615/2)								
Unknown Ph-5	3.6	0.011	4.3	0.022	3.8	0.067	3.8	0.06
N-DesMet-fluindapyr-N-Ser (M424/1)	4.0	0.012	4.0	0.021	3.5	0.062	3.9	0.062
Sum of DesMet-fluindapyr-N1-conjugates	15	0.046	14	0.072	18	0.33	16	0.26
DesMet-fluindapyr-N1-Glu (M499/1)	*5.5*	*0.016*	*5.4*	*0.028*	*8.3*	*0.15*	*7.9*	*0.13*
DesMet-fluindapyr-N1-Glu-Malo (M585/1)	*9.8*	*0.030*	*8.6*	*0.044*	*10*	*0.18*	*8.2*	*0.13*
Unknown	1.7	0.005	1.7	0.009	2.8	0.05	2.5	0.039
3-OH-fluindapyr (M367/3)[a]	4.0	0.012	2.4	0.012	4.4	0.077	4.4	0.068
N-DesMet-fluindapyr (M337/1)[a]	4.6	0.013	4.2	0.021	1.0	0.017	1.6	0.025
Dehydro-fluindapyr (M349/1)[a]	0.082	0.00	0.13	0.001	0.046	0.001	0.059	0.001
Total identified	**90**	**0.27**	**88**	**0.45**	**87**	**1.6**	**85**	**1.4**
Cellulase hydrolysis	-	-	-	-	0.34	0.006	0.43	0.007
Alpha amylase hydrolysis	-	-	-	-	0.31	0.005	0.53	0.009
Pectinase hydrolysis	-	-	-	-	0.43	0.008	0.51	0.008
Protease hydrolysis	-	-	-	-	0.42	0.008	0.54	0.009
Acid (1N) hydrolysis	-	-	-	-	0.25	0.004	0.53	0.008
Base (1N) hydrolysis	-	-	-	-	0.48	0.009	0.54	0.009
Unextracted residue after exhaustive extraction	-	-	-	-	2.8	0.049	2.9	0.046

Notes:

[a] co-eluting peaks, separated using a secondary HPLC method in the 'dichloromethane extract'.

Table 18 Enantiomeric ratios of fluindapyr in soya bean RACs

Label	Enantiomer (%)	Forage	Hay	Dose solution
Phenyl	S	41	44	51
	R	59	56	49
Pyrazole	S	43	44	48
	R	57	56	52

Overview of the metabolic pathway of fluindapyr in primary crops

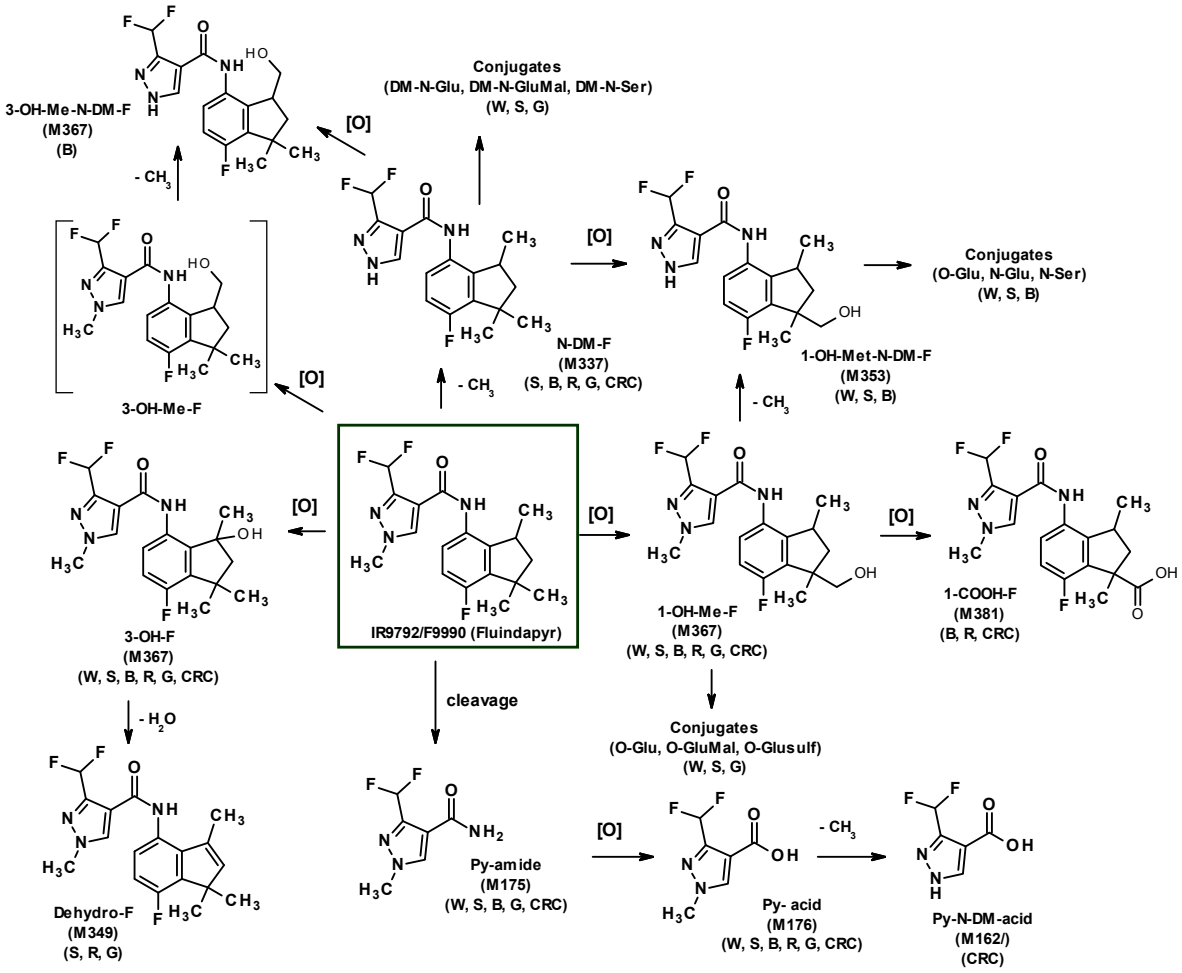
Plant metabolism studies have been presented covering foliar treatments to the crop categories of fruits (grape), root crops (sugar beet), cereal/grass (wheat and rice), pulses and oilseeds (soya bean).

The enantiomeric ratio R/S in some crops remained 50:50 (grape leaves and rinse, sugar beet foliage) however, in other crops a shift could be observed into a ratio of 60:40 or 70:30 (grape extract, wheat forage/hay/straw, rice grain/straw, soya bean hay/forage).

The major metabolic pathways of fluindapyr were hydroxylation, often followed by O-glycosylation, and oxidative-N-demethylation, both followed by conjugation, yielding the important

metabolites 1-OH-Met-fluindapyr (free and conjugated), 3-OH-fluindapyr, and 1-OH-Met-N-DesMet-fluindapyr (free and conjugated). Hydrolysis resulting in the parent breakage of the carboxamide bond constituted a relatively minor route.

In all crops most major pathways were observed, however, in some crops metabolism was more advanced (e.g. soya bean) than in other crops. See Figure 2, note that the figure below also includes the proposed metabolic pathways of the rotational crops, which are discussed in the section on the rotational crops after the section on environmental fate in soil. For better view an enlargement of the scheme can be found at the end of the evaluation.



F: fluidapyr; Me: methyl; DM: desmethyl; py: pyrazole; Glu: glucoside; GluMal: malonylglucoside; Ser: serine; Glusulf: glucosyl sulfate
M: Mol. Wt.
Primary crops: W: wheat, S: soybean; B: sugarbeet; R: rice, G: grape
CRC: confined rotational crops (carrot, lettuce and wheat)

Figure 2 Proposed metabolic pathways of fluindapyr (F9990/IR9792) in primary and rotational crops

Environmental fate in soil

The Meeting received information on the environmental fate in soils. Information included studies on the route and rate of degradation of fluindapyr and its metabolites in aerobic soils under laboratory conditions and dissipation of fluindapyr under field conditions. The fate and behaviour of fluindapyr was investigated using ^{14}C-phenyl and ^{14}C-pyrazole-labeled fluindapyr. Furthermore, soil photolysis, soil

dissipation and confined and field rotational crop studies were provided. The anaerobic soils studies as well as the mobility studies (adsorption and desorption of the active substance and its soil metabolites) that were submitted, are not required according to the JMPR manual on the Submission and Evaluation of Pesticide Residues Data (2016). These studies were not further considered (see references submitted but not used).

Photodegradation on the soil surface

The photodegradation of fluindapyr was studied in a soil from Europe [Vanini & Pizzella, 2016, 2014EFT-IFP1406]. Soil characteristics are reported in Table xx.

Table 19 Soil characteristics for study 2014EFT-IFP106

Soil name	Stir-2
Location	
Soil texture (USDA) [a]	Clay loam
-- Sand (%)	30
-- Silt (%)	35
-- Clay (%)	35
Organic Carbon (%) [b]	2.35
CEC (meq/100 g)	18.6
pH (H_2O)	7.5
Water Holding Capacity at pF 2.0 (0.1 bar) [percent]	29.0
Disturbed bulk density (g/cm^3)	1.1
Dry Mass (%)	90.52

Notes:

[a] Classification according to United States Department of Agriculture (USDA).

The [^{14}C-pyrazole]-labelled fluindapyr or the [^{14}C-phenyl]-labelled fluindapyr was applied to a thin layer of soil. The soil layer was prepared by applying an aqueous slurry of soil to a stainless steel plate in such a way to form a soil layer of about 2 mm thick and of about an area of 12.5 cm^2.

A set of samples was allowed to air-dry, about 24 hours, to reach a soil moisture of about 75 percent of field capacity. A set of samples was completely dried out. ^{14}C-fluindapyr was applied to each unit at the nominal rate of 125 g a.i./ha. Half of the samples of the first set (named *Wet*) and all samples of the second set (named *Dry*) were placed in a stainless steel tray covered with a quartz lid, exposed to the Xenon lamp at a irradiance mean value of 708.5 W/m2 and maintained at a temperature of 20±2 °C. The other half of the samples of the first set were incubated under identical conditions but not irradiated (named *Dark*).

Duplicate incubation units were collected and analysed at the following sampling times after treatment: 0, 1, 2, 3, 6, 10 and 15 days (15 days of continuous irradiation corresponded to about 46 days of natural summer sunlight at latitude 30°N).

Soil was extracted with two different solvent mixtures and the extractable radioactivity was determined by Liquid Scintillation Counting (LSC). The radioactivity distribution in soil extracts was determined by Thin Layer Chromatography (TLC) analyses and the enantiomeric ratio of fluindayr by High Pressure Liquid Chromatography (HPLC) analyses of representative samples. The non-extractable radioactivity was determined by LSC, after oxidation of aliquots of soil residue by means of a biological oxidizer.

The soil extractable radioactivity ranged from 96 percent to 99 percent of Applied Radioactivity (AR) in *Dark* samples, from 94 percent to 98 percent AR in *Dry* samples and from 93 percent to 98 percent AR in *Wet* samples.

Fluindapyr progressively degraded in Wet and Dry samples, while in Dark samples no a evident decline was observed in 15 days of incubation. DT_{50} values were calculated assuming single first order (SFO) kinetics and using KinGUI version 1.1 software. The fit of the SFO model is based on visual assessment of goodness of fit and on Chi-squared error. Results are shown in Table 20.

Table 20 Degradation rate in irradiated soils for fluindapyr

	Dark controls soils				Irradiated dry soil				Irradiated wet soil			
	DT_{50} (days)	DT_{90} (days)	χ^2 (%)	r^2	DT_{50} (days)	DT_{90} (days)	χ^2 (%)	r^2	DT_{50} (days)	DT_{90} (days)	χ^2 (%)	r^2
[^{14}C-pyrazole] fluindapyr	318	1058	0.8	0.448	56	186	1.3	0.923	56	187	2.2	0.810
[^{14}C-phenyl] fluindapyr	281	933	0.9	0.430	65	217	1.2	0.907	52	172	2.2	0.831

The endpoint obtained from SFO equation were converted in equivalent days of natural summer sunlight and the results are listed in Table 21.

Table 21 Degradation rate in equivalent of natural summer light

Sample	endpoint	experimental days (mean of both labels)	equivalent days natural sunlight		
			sunlight 30°N	sunlight 40°N	sunlight 50°N
Dry	DT_{50}	61	188	183	193
	DT_{90}	202	624	608	640
Wet	DT_{50}	54	167	163	172
	DT_{90}	180	556	542	570

The route of degradation on the soil surface is similar for both the wet and dry samples and showed that the extracts contained the same main photo transformation products. Five or six photodegradation products were found. See Table 22. Two main compounds were identified; Co-chromatography identified one of the degradation products as 3-OH-fluindapyr (code 510152). On the basis of TLC, HPLC and LC-MS analyses, the second degradation compound was identified as pyrazole-carboxamide (code 510151). In Dark samples no degradation products were found.

The non-extractable radioactivity (bound residue) ranged from 0.77 percent to 4.6 percent AR. The Mass Balance was always higher than 96 percent AR.

HPLC analyses of representative soil extracts showed that the enantiomeric ratio S/R of unchanged ^{14}C-fluindapyr remained constant during the study and was found to be about 50 : 50.

Table 22 Average (n=2) total ^{14}C distribution in irradiated dry and wet soils and dark dry and wet controls

Time point [days]	fluindapyr (percent AR)	3-OH-fluindapyr (percent AR)	pyrazole-carboxamide (percent AR)	S4 (percent AR)	S5 (percent AR)	S6 (percent AR)	S7 (percent AR)	PES (percent AR)	Total (percent AR)
	[^{14}C-pyrazole]-fluindapyr, dark soil								
0	95	2.4	ND	ND	ND	-	-	1.2	98
1	97	2.7	ND	ND	ND	-	-	1.4	101
2	95	2.5	ND	ND	ND	-	-	0.77	98
3	95	2.4	ND	ND	ND	-	-	1.9	100

Time point [days]	fluindapyr (percent AR)	3-OH-fluindapyr (percent AR)	pyrazole-carboxamide (percent AR)	S4 (percent AR)	S5 (percent AR)	S6 (percent AR)	S7 (percent AR)	PES (percent AR)	Total (percent AR)
6	93	3.4	ND	ND	ND	-	-	3.1	100
10	94	2.7	ND	ND	ND	-	-	1.5	99
15	94	2.8	ND	ND	ND	-	-	3.7	100
	[^{14}C-phenyl]-fluindapyr, dark soil								
0	95	2.9	ND	ND	ND	-	-	1.4	100
1	96	3.3	ND	ND	ND	-	-	1.1	100
2	94	2.8	ND	ND	ND	-	-	1.1	98
3	95	2.5	ND	ND	ND	-	-	2.2	99
6	94	3.4	ND	ND	ND	-	-	1.2	99
10	93	2.6	ND	ND	ND	-	-	3.9	99
15	93	3.3	ND	ND	ND	-	-	1.6	98
	[^{14}C-pyrazole]-fluindapyr, dry soil								
0	95	2.4	ND	ND	ND	ND	-	1.6	99
1	92	3.8	1.0	ND	ND	ND	-	1.2	98
2	93	3.5	1.4	ND	ND	ND	-	1.2	99
3	93	3.5	1.8	ND	ND	ND	-	1.5	100
6	87	4.0	2.8	1.2	1.0	ND	-	1.2	97
10	85	4.1	3.8	1.6	ND	ND	-	4.0	98
15	80	5.9	4.1	1.6	1.4	3.1	-	3.7	99
	[^{14}C-phenyl]-fluindapyr, dry soil								
0	95	2.7	NA	ND	ND	ND	-	1.0	99
1	94	3.6	NA	ND	ND	ND	-	1.7	99
2	91	3.7	NA	ND	ND	ND	-	2.1	97
3	92	4.1	NA	ND	ND	ND	-	2.3	99
6	91	5.6	NA	ND	ND	ND	-	2.2	99
10	85	6.6	NA	ND	2.2	1.6	-	3.4	99
15	82	7.9	NA	ND	3.2	3.4	-	3.9	100
	[^{14}C-pyrazole]-fluindapyr, wet soil								
0	94	2.6	ND	ND	ND	ND	-	1.1	97
1	89	4.1	1.6	ND	ND	ND	-	1.9	97
2	87	4.2	2.8	1.3	ND	ND	-	1.9	97
3	85	5.4	3.9	1.6	ND	ND	-	2.7	99
6	86	5.4	4.3	ND	ND	ND	-	2.7	98
10	81	6.2	6.7	2.5	1.4	ND	-	2.0	100
15	77	7.7	7.3	2.6	1.2	2.6	-	2.8	101
	[^{14}C-phenyl]-fluindapyr, wet soil								
0	93	2.6	NA	ND	ND	ND	ND	1.0	96
1	90	5.5	NA	ND	ND	ND	ND	3.5	99
2	88	6.0	NA	ND	ND	ND	ND	2.2	96
3	85	6.9	NA	ND	2.5	ND	ND	3.2	98
6	82	7.7	NA	ND	2.9	ND	ND	4.5	97
10	78	7.7	NA	ND	3.0	3.	4.3	4.0	100
15	78	9.7	NA	ND	2.9	2.9	3.5	4.6	101

Notes:

ND = not detected (detection limit = 0.88 percent).

NA = not applicable because of the labelling site.

- = Not analysed.

Overview of the metabolic pathway of fluindapyr after soil surface photolysis

Fluindapyr was only limited metabolised after soil surface photolysis with approximately 80 percent unchanged parent, two main degradation products could be identified, 4 minor, unidentified metabolites. The results indicates that some hydroxylation takes place resulting in 3-OH-fluindapyr (up to 9.7 percent AR) and that there is cleavage of the parent forming the pyrazole carboxamide (up to 7.3 percent AR) and finally some mineralization (formation of bound residues and CO_2), reaching maximums of 4.6 percent of the AR.

Aerobic degradation of fluindapyr in soil

Studies 1-4 In two studies (study 1 and 2) the route and the rate of degradation was studied in one European and oneUnited States soil [Mainolfi & Colombini, 2016a, 2013EFT-IFP0873; Mainolfi & Colombini, 2016b, 2013EFT-IFP0874] using both [^{14}C-phenyl]-fluindapyr and [^{14}C-pyrazole]-fluindapyr. In two additional studies (study 3 and 4) the route of degradation of fluindapyr was investigated in three European and three soils using only the [^{14}C-pyrazole]-labelled fluindapyr [Vanini, 2016a, 2013EFT-IFP0735; Vanini, 2016b, EFT-IFP0763]. Soil characteristics from the different soils are reported in Table 23.

In all four studies the soil samples (50 g dry weight) were maintained in the dark at a temperature of 20 ± 2 °C and the soil moisture was maintained at 60 percent of the Maximum Water Holding Capacity (MWHC) corresponding to a pF value of 2 (0.1 bar). After 7 days of acclimatization, [^{14}C-phenyl]-fluindapyr and [^{14}C-pyrazole]-fluindapyr were applied separately to the respective soil incubation units at 0.5 mg/kg, equivalent to a field application of 127–128 g ai/ha (1 × test solution) in all studies.

Each unit was then connected to 2N KOH and ethylene glycol traps to collect volatile radioactivity and incubated in the dark at 20 ± 2 °C for 123 days. Moistened CO_2-free air was passed through units to maintain aerobic conditions. The soil moisture content was adjusted by adding purified water during the study. For each soil, an untreated sample was set up and used as blank. Duplicate incubation units were collected and analysed at 0, 9, 19, 36, 61, 90 and 120 days in studies 1 and 2. In study 3 and 4 the European soil samples were taken at 0, 10, 24, 48, 91, 132, and 151 days for Stir-2 and Z-1 soils and at 0, 10, 34, 70, 98, and 125 days for Sp2.1 soil and for the three United States soils. Soil samples were stored at 4 °C for a maximum of three months after collection [Mainolfi & Colombini, 2016a, 2013EFT-IFP0873; Mainolfi & Colombini, 2016b, 2013EFT-IFP0874]. No storage data were reported in the other study reports [Vanini, 2016a, 2013EFT-IFP0735; Vanini, 2016b, EFT-IFP0763]. The experimental phase of both studies was performed within 9 months. The storage stability of fluindapyr and four of its metabolites was established by Skags, 2018 [Report X1509BK]. See section on storage stability.

Table 23 Soil characteristics for studies 2013EFT-IFP0873, 2013EFT-IFP0874, 2013EFT-IFP0735 and 2013EFT-IFP0763

Soil name	Sp-2.2	Stir-2	Z-1	Sp-2.1	Iowa	CA-SL	DU-PF	DU-L
Study	Mainolfi and Colombini, 2016a	Vanini, 2016a	Vanini, 2016a	Vanini, 2016a	Mainolfi and Colombini, 2016b	Vanini, 2016b	Vanini, 2016b	Vanini, 2016b
Report ID	2013EFT-IFP0873	2013EFT-IFP0735	2013EFT-IFP0735	2013EFT-IFP0735	2013EFT-IFP0874	2013EFT-IFP0763	2013EFT-IFP0763	2013EFT-IFP0763
Location	Germany	Italy	Italy	Germany	IA United States	CA, United States	ND, United States	ND, United States
Soil texture (USDA) [a]	Loamy sand	Clay loam	Loam	Sand	Sandy loam	Sandy loam	Sandy loam	Clay loam

Soil name	Sp-2.2	Stir-2	Z-1	Sp-2.1	Iowa	CA-SL	DU-PF	DU-L
Study	Mainolfi and Colombini, 2016a	Vanini, 2016a	Vanini, 2016a	Vanini, 2016a	Mainolfi and Colombini, 2016b	Vanini, 2016b	Vanini, 2016b	Vanini, 2016b
Report ID	2013EFT-IFP0873	2013EFT-IFP0735	2013EFT-IFP0735	2013EFT-IFP0735	2013EFT-IFP0874	2013EFT-IFP0763	2013EFT-IFP0763	2013EFT-IFP0763
--Sand (%)	76.5	30	43	87	20.0	72	53	37
-- Silt (%)	15.3	35	38	10	61.0	23	27	31
-- Clay (%)	8.2	35	19	3	19.0	5	20	32
Organic Carbon (%)	1.74	2.35	2.35	0.65	2.50	0.39	4.7	3.48
CEC (meq/100 g)	10.2	18.6	20.9	4.3	13.0	5.9	22.4	20.7
pH (in water)	5.5[b]	7.5	6.8	6.1	6.1[b]	7.5	6.7	5.5
Water Holding Capacity at pF 2.0 (0.1 bar) [percent]	42.5	29.0	23.0	n.d.	42.0	16.0	50.0	45.5
Bulk density (disturbed [g/cm^3]	n.d.	1.09	1.12	1.47	n.d.	1.38	0.90	1.02
Dry mass (%)	91.38	90.52	85.36	97.66	83.85	99.59	77.55	86.71
Microbial biomass initial [mg microbial C/100 g dry weight]	98.16	63.18	42.27	25.42	211.75	108.16	242.01	198.13
Microbial biomass initial (percent referred to total organic carbon)	5.64	2.69	1.80	3.91	8.47	27.73	5.15	5.69
Microbial biomass final [mg microbial C/10 g dry weight]	23.13	60.51	32.89	19.22	68.77	78.78	194.94	153.54
Microbial biomass final (percent referred to total organic carbon)	1.33	2.57	1.40	2.96	2.75	20.20	4.15	4.41

Notes:

n.d. = Not determined.

[a] Classification according to United States Department of Agriculture (USDA).

[b] pH in 0.01 M $CaCl_2$.

Soils were extracted with acetone:water (9:1 v/v and 1:1 v/v) for all sample points, and with acetone:0.1N HCl (1:1 v/v) for all sample points except time 0. The extractable radioactivity was determined by LSC. Suitable aliquots of soil extracts were pooled, concentrated and analysed by TLC to quantify parent degradation and metabolite formation. Representative extract samples were analysed by LC-MS and compared to relevant reference compounds in order to identify the main degradation compounds. The unextracted radioactivity was determined by LSC, after oxidation of aliquots of soil residue using a biological oxidizer. The radioactivity content in the traps (volatiles) was determined by LSC, except at 0 day in all studies. Chiral High Performance Liquid Chromatography (HPLC) was used to confirm the enantiomeric ratio (S/R) of fluindapyr.

In study 1 and 2 the soils treated with an application rate of 1587-1599 g ai/ha (15 × test solution) were extracted only after 123 days of incubation. Each sample was extracted with acetone:water (9:1 v/v and 1:1 v/v) and pooled together. These extract samples were used as a source of metabolites for

characterisation/identification purposes as they have the same profile of radioactivity as the soil samples treated with the application rate of 125 g/ha.

The extractable radioactivity was higher than 90 percent of Applied Radioactivity (AR) in all samples. The unextracted radioactivity (bound residue) slightly increased during the study but did not exceed 5 percent AR. Volatile radioactivity did not exceed 4.3 percent AR, in either the KOH or ethylene glycol traps.

The mass balance ranged from an average of 93 percent to 104 percent AR and the individual mass balance values were within 90-110 percent AR throughout the studies.

In Sp-2.2 soil [Mainolfi&Columbini, 2016a, 2013EFT-IFP0873], the concentration of fluindapyr gradually decreased to averages of 71 percent AR at the end of the study (120 days) for the ^{14}C-phenyl and the ^{14}C-pyrazole labelled samples, respectively. In Iowa soil [Mainolfi & Columbini, 20156b, 2013EFT-IFP0874], the concentration of fluindapyr gradually decreased to averages of 66 percent AR at the end of the study (120 days) for both the ^{14}C-phenyl and the ^{14}C-pyrazole labelled samples. The same metabolic profile was observed for the ^{14}C-phenyl and the ^{14}C-pyrazole labels, indicating that no cleavage of the carboxamide bond occurred in the fluindapyr structure. The three main degradation compounds found during the study corresponded to 3-hydroxy-fluindapyr (code 510152), and the diastereomers *cis*-1-carboxy-fluindapyr (code 510170) and *trans*-1-carboxy-fluindapyr (code 510169). No other degradation compounds exceeded 3 percent AR at any sampling time. A similar pattern was observed in the studies only using ^{14}C-pyrazole labels [Vanini, 2016a, 2013EFT-IFP0735; Vanini, 2016b, EFT-IFP0763].

The concentration of all the three metabolites was still increasing at the end of the studies. The occurrence of fluindapyr and its metabolites in the eight tested soils are presented in Table 24.

Table 24 Distribution of radioactivity (percent of applied radioactivity) in 4 European and 4 United States soils after application of ^{14}C-phenyl or ^{14}C-pyrazole labelled fluindapyr (means of duplicate samples)

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval						
Sp-2.2 (1) 2013EFT-IFP0873 (^{14}C-phenyl)	Interval in days	0	9	19	36	61	90	120
	Fluindapyr	96.93	93.51	90.43	85.27	79.39	74.30	70.67
	3-hydroxy-fluindapyr	1.36	2.86	4.47	7.56	10.44	11.89	14.16
	cis-1-carboxy-fluindapyr	ND	0.59	1.24	1.58	2.35	3.01	3.74
	trans-1-carboxy-fluindapyr	ND	0.64	1.38	1.62	2.15	2.55	3.29
	Extracted unknown 3	0.85	0.72	1.39	1.24	1.61	1.16	0.62
	Extracted unknown 6	ND	ND	ND	ND	ND	ND	ND
	Extracted unknown 7	ND	ND	ND	0.89	0.93	1.30	1.42
	Extracted unknowns 8	ND	ND	ND	ND	0.54	1.06	1.04
	Total extracted [a]	99.14	98.32	98.91	98.16	97.41	95.27	94.94
	Bound (after 3 extractions)	0.00	0.00	0.00	0.76	0.86	1.64	1.89
	Volatile - KOH	n.a.	ND	ND	ND	0.38	0.51	0.49
	Volatile - Ethylene glycol	n.a.	ND	ND	ND	0.29	ND	ND
	Mass balance	99.14	98.32	98.91	98.92	98.94	97.42	97.32
Sp-2.2 (1) 2013EFT-IFP0873 (^{14}C-pyrazole)	Interval in days	0	9	19	36	61	90	120
	Fluindapyr	99.54	95.61	92.95	89.89	82.66	76.89	70.90
	3-hydroxy-fluindapyr	1.35	2.66	4.72	6.51	9.48	11.84	13.40
	cis-1-carboxy-fluindapyr	ND	0.62	1.32	2.06	2.52	2.72	3.56
	trans-1-carboxy-fluindapyr	ND	0.62	1.40	2.05	2.52	2.55	3.35
	Extracted unknown 3	ND	ND	ND	ND	ND	ND	1.20
	Extracted unknown 6	ND	0.53	1.08	0.73	ND	ND	ND
	Extracted unknown 7	ND	ND	ND	0.76	0.84	1.22	1.13
	Extracted unknowns 8	ND	ND	ND	0.34	0.37	1.13	0.96

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval						
	Total extracted [a]	100.89	100.04	101.47	102.34	98.39	96.35	94.5
	Bound (after 3 extractions)	0.00	0.00	0.00	0.86	0.81	1.07	1.60
	Volatile - KOH	n.a.	ND	ND	ND	0.66	0.75	0.55
	Volatile - Ethylene glycol	n.a.	ND	ND	ND	ND	ND	ND
	Mass balance	100.89	100.04	101.47	103.2	99.86	98.17	96.65
Iowa (2) 2013EFT-IFP0874 (^{14}C-phenyl)	Interval in days	0	9	19	36	61	90	120
	Fluindapyr	95.56	93.25	90.96	89.13	80.87	70.91	66.15
	3-hydroxy-fluindapyr	1.09	2.22	3.32	4.88	8.27	11.31	13.16
	cis-1-carboxy-fluindapyr	ND	0.54	0.81	1.70	2.47	3.09	3.27
	trans-1-carboxy-fluindapyr	ND	0.68	1.02	2.64	3.81	5.24	5.88
	Extracted unknown 3	0.85	0.78	1.28	1.32	1.94	1.27	1.17
	Extracted unknown 6	ND	ND	ND	0.87	ND	ND	ND
	Extracted unknown 7	ND	ND	ND	ND	0.96	1.04	1.68
	Extracted unknowns 8	ND	ND	ND	ND	1.10	1.21	1.36
	Total extracted [a]	97.5	97.47	97.39	100.54	99.42	94.07	92.67
	Bound (after 3 extractions)	0.00	0.00	0.00	1.18	1.05	2.86	3.41
	Volatile - KOH	n.a.	ND	ND	ND	0.28	0.66	0.69
	Volatile - Ethylene glycol	n.a.	ND	ND	ND	0.29	0.56	0.59
	Mass balance	97.5	97.47	97.39	101.72	101.04	98.15	97.36
Iowa (2) 2013EFT-IFP0874 (^{14}C-pyrazole)	Interval in days	0	9	19	36	61	90	120
	Fluindapyr	101.34	99.39	96.66	98.08	80.62	71.83	65.91
	3-hydroxy-fluindapyr	1.24	2.32	3.85	6.57	7.69	11.70	14.06
	cis-1-carboxy-fluindapyr	ND	0.55	0.92	1.66	2.26	2.63	3.22
	trans-1-carboxy-fluindapyr	ND	0.72	1.27	2.60	3.55	4.68	6.16
	Extracted unknown 3	ND	ND	ND	ND	ND	ND	ND
	Extracted unknown 6	ND	0.64	1.18	1.34	2.38	2.49	2.93
	Extracted unknown 7	ND	ND	ND	0.65	1.08	1.03	1.39
	Extracted unknowns 8	ND	ND	ND	ND	1.22	1.30	1.47
	Total extracted [a]	102.58	103.62	103.88	110.9	98.8	95.66	95.14
	Bound (after 3 extractions)	0.00	0.00	0.00	1.19	1.24	1.71	2.2
	Volatile - KOH	n.a.	ND	ND	ND	0.31	0.64	0.82
	Volatile - Ethylene glycol	n.a.	ND	ND	ND	ND	0.57	0.48
	Mass balance	102.58	103.62	103.88	112.09	100.35	98.58	98.64
Stir-2 (3) 2013EFT-IFP0735 (^{14}C-pyrazole)	Interval (days)	0	10	24	48	91	132	151
	Fluindapyr	101.18	92.00	88.15	85.69	77.14	69.85	66.15
	3-hydroxy-fluindapyr	0.95	1.73	3.05	3.54	5.12	6.38	8.08
	cis-1-carboxy-fluindapyr	ND	2.07	4.44	5.48	8.21	10.46	12.52
	trans-1-carboxy-fluindapyr	ND	2.33	3.97	4.67	6.68	8.99	11.06
	Extracted unknown 5	ND	ND	ND	ND	ND	0.83	ND
	Extracted unknown 6	ND	ND	ND	ND	ND	0.97	1.68
	Total extracted [a]	102.13	98.13	99.61	99.38	97.15	97.48	99.49
	Bound (after 3 extractions)	0.80	0.34	0.33	0.32	2.68	2.77	2.81
	Volatile - KOH	ND	ND	ND	ND	ND	ND	ND
	Volatile - Ethylene glycol	ND	ND	ND	ND	ND	ND	ND
	Mass balance	102.93	98.47	99.94	99.7	99.83	100.25	102.3
Z-1 (3) 2013EFT-IFP0735 (^{14}C-pyrazole)	Interval (days)	0	10	24	48	91	132	151
	Fluindapyr	101.14	94.40	91.34	91.77	81.55	76.79	72.73
	3-hydroxy-fluindapyr	0.95	1.99	3.55	4.64	6.85	10.22	12.68
	cis-1-carboxy-fluindapyr	ND	0.48	2.07	2.26	4.26	6.52	7.58
	trans-1-carboxy-fluindapyr	ND	1.79	2.67	2.69	4.16	6.06	6.22
	Extracted unknown 5	ND	ND	ND	ND	ND	ND	ND
	Extracted unknown 6	ND	ND	ND	ND	1.47	ND	2.63

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval						
	Total extracted [a]	102.09	98.66	99.63	101.36	98.29	99.59	101.84
	Bound (after 3 extractions)	0.72	0.87	0.82	1.11	1.01	1.26	1.42
	Volatile - KOH	ND	ND	ND	ND	ND	ND	ND
	Volatile - Ethylene glycol	ND	ND	ND	ND	ND	ND	ND
	Mass balance	102.81	99.53	100.45	102.47	99.3	100.85	103.26
Sp-2.1 (3) 2013EFT-IFP0735 (^{14}C-pyrazole)	Interval (days)	0	10	34	70	98	125	
	Fluindapyr	100.47	93.92	90.06	77.28	70.13	65.93	
	3-hydroxy-fluindapyr	1.03	3.09	7.14	12.61	15.30	18.59	
	cis-1-carboxy-fluindapyr	ND	ND	1.03	1.80	2.13	4.09	
	trans-1-carboxy-fluindapyr	ND	NDP	1.44	2.18	2.69	3.01	
	Extracted unknown 5	ND	ND	ND	ND	ND	ND	
	Extracted unknown 6	ND	ND	ND	ND	ND	ND	
	Total extracted [a]	101.5	97.01	99.67	93.87	90.25	91.62	
	Bound (after 3 extractions)	ND	1.18	0.92	3.26	3.89	4.12	
	Volatile - KOH	ND	ND	ND	0.33	2.64	2.98	
	Volatile - Ethylene glycol	ND	ND	ND	ND	ND	ND	
	Mass balance	101.5	98.19	100.59	97.46	96.78	98.72	
CA-SL (4) 2013EFT-IFP0763 (^{14}C-pyrazole)	Interval (days)	0	10	34	70	98	125	
	Fluindapyr	100.61	98.86	85.50	71.97	64.11	55.66	
	3-hydroxy-fluindapyr	1.56	3.31	6.41	10.48	12.39	14.94	
	cis-1-carboxy-fluindapyr	ND	ND	1.67	2.56	2.55	3.02	
	trans-1-carboxy-fluindapyr	ND	0.85	2.75	4.31	3.69	4.14	
	Extracted unknown 5	ND	ND	2.221	4.12	3.48	3.54	
	Extracted unknown 6	ND	ND	1.51	2.98	4.12	3.08	
	Extracted unknown 7	ND	ND	ND	ND	0.92	1.15	
	Extracted unknown 8	ND	ND	ND	ND	0.68	1.30	
	Total extracted [a]	102.17	103.02	100.061	96.42	91.94	86.83	
	Bound (after 3 extractions)	ND	ND	ND	0.88	3.61	4.73	
	Volatile - KOH	n.a.	ND	ND	ND	1.76	3.33	
	Volatile - Ethylene glycol	n.a.	ND	ND	ND	ND	ND	
	Mass balance	102.17	103.02	100.061	97.3	97.31	94.89	
DU-PF (4) 2013EFT-IFP 0763 (^{14}C-pyrazole)	Interval (days)	0	10	34	70	98	125	
	Fluindapyr	98.78	95.71	91.16	81.61	71.62	63.10	
	3-hydroxy-fluindapyr	1.39	2.24	5.51	7.69	10.15	11.86	
	cis-1-carboxy-fluindapyr	ND	ND	1.40	1.55	2.26	2.07	
	trans-1-carboxy-fluindapyr	ND	ND	1.77	2.25	3.18	3.01	
	Extracted unknown 6	ND	ND	ND	2.08	2.07	2.32	
	Extracted unknown 7	ND	ND	ND	ND	1.42	1.81	
	Total extracted [a]	100.17	97.95	99.84	95.18	90.7	84.17	
	Bound (after 3 extractions)	ND	ND	ND	1.94	3.73	4.96	
	Volatile - KOH	n.a.	ND	ND	ND	2.74	4.13	
	Volatile - Ethylene glycol	n.a.	ND	ND	ND	ND	ND	
	Mass balance	100.17	97.95	99.84	97.12	97.17	93.26	
DU-L (4) 2013EFT-IFP0763 (^{14}C-pyrazole)	Interval (days)	0	10	34	70	98	125	
	Fluindapyr	100.03	98.25	86.82	80.15	73.92	67.65	
	3-hydroxy-fluindapyr	1.43	2.92	7.66	11.57	15.01	17.28	
	cis-1-carboxy-fluindapyr	ND	ND	1.87	2.61	2.95	3.47	
	trans-1-carboxy-fluindapyr	ND	ND	1.26	1.73	1.89	2.07	
	Total extracted [a]	101.46	101.17	97.61	96.06	93.77	90.47	
	Bound (after 3 extractions)	ND	0.16	2.00	1.11	3.74	4.19	
	Volatile - KOH	n.a.	ND	ND	0.57	2.63	3.20	
	Volatile - Ethylene glycol	n.a.	ND	ND	ND	ND	ND	

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval						
	Mass balance	101.46	101.33	99.61	97.74	100.14	97.86	

Notes:

n.a. = Not analysed.

ND = below detection limit: bound residue = 0.19-0.22 percent AR, KOH solution = 0.20 percent AR, ethylene glycol = 0.18 percent AR, compounds = 0.15 percent AR.

[a] Calculated by the reviewer by summing all extracted components.

The enantiomeric ratio remained constant during the studies (ca. 50:50). See Table 25 and Table 26.

Table 25 Enantiomeric ratio of fluindapyr in representative soil extracts for studies 2013EFT-IFP0873 and 2013EFT-IFP0874

Soil (study no)	Time point [days]	^{14}C-phenyl label			^{14}C-pyrazole label		
		S enantiomer	R enantiomer	Ratio S/R	S enantiomer R enantiomer Ratio S/R	R enantiomer	Ratio S/R
Sp-2.2 (1)	TS 1 X	49.39	49.75	0.99	49.67	49.92	1.00
	61 days	49.17	50.84	0.97	50.26	49.75	1.01
	120 days	49.72	50.29	0.99	49.59	50.42	0.98
Iowa (2)	TS 1 X	49.39	49.75	0.99	49.67	49.92	1.00
	61 days	49.99	50.02	1.00	50.03	49.98	1.00
	120 days	49.78	50.23	0.99	49.97	50.03	1.00

Table 26 Enantiomeric ratio of fluindapyr in representative soil extracts for studies 2013EFT-IFP0735 and 2013EFT-IFP0763

Soil (study no)	Time point [days]	^{14}C-pyrazole label			Soil (study no)	^{14}C-pyrazole label		
		S enantiomer	R enantiomer	Ratio S/R		S enantiomer R enantiomer Ratio S/R	R enantiomer	Ratio S/R
Stir-2 (3)	48	49.74	50.26	0.99	CA-SL (4)	49.52	50.48	0.98
	151	49.51	50.50	0.98		49.22	50.78	0.97
Z-1 (3)	48	49.33	50.68	0.97	DU-PF (4)	49.55	50.45	0.98
	151	49.44	50.57	0.98		49.25	50.75	0.97
Sp-2.1 (3)	70	49.17	50.83	0.97	DU-L (4)	49.31	50.69	0.97
	125	49.57	50.43	0.98		49.77	50.23	0.99

The rate of degradation of fluindapyr has been assessed using the FOCUS DEGKIN v2 Excel tool following FOCUS Kinetics guidance (2006). Single First-Order (SFO) kinetics adequately described the degradation in both radiolabels in the eight soils. The DT_{50} and DT_{90} values ranged from 141-353 and 469-1173 days, respectively. The individual results are summarized in Table 27.

Kinetic endpoints for aerobic degradation in soil

The rate of degradation of fluindapyr was calculated using kinetic modelling of the residue data for four soils in Europe [Mainolfi & Colombini, 2016a, 2013EFT-IFP0873; Vanini, 2016a, 2013EFT-IFP0735] and four soils in the United States [Mainolfi & Colombini, 2016b, 2013EFT-IFP0874; Vanini, 2016b, EFT-IFP0763]. The data were first fitted according to a single first order kinetic model (SFO) and secondly, if necessary, according to a bi-phasic kinetic model, First-Order Multi-Compartment kinetic model (FOMC).

The rate of degradation of fluindapyr has been assessed using the FOCUS DEGKIN v2 (June 2007) Excel tool following FOCUS Kinetics guidance (2006). The goodness of fit was assessed by visual inspection and an error criterion based on a chi-squared test. The findings are summarised in Table 27. Single First-Order (SFO) kinetics adequately described the degradation in both radiolabels in the eight soils.

The best fit DT_{50} and DT_{90} values ranged from 141-353 and 469-1173 days, respectively. When the data were combined, the geometric mean DT_{50} for fluindapyr is 265 days for the European soils, 187 days for the United States soils and 223 days for all soils together.

Table 27 Summary of fluindapyr DT_{50}'s in eight soils under aerobic conditions

Soil name	ref	pH (water)	C_{org} (%)	Clay (%)	Label position	Model	DT_{50} (days)	DT_{90} (days)	χ^2	r^2
Sp-2.2	2013EFT-IFP0873	5.5 [a]	1.74	8.2	[phenyl]	SFO	255	847	1.1	0.972
		5.5 [a]	1.74	8.2	[pyrazole]	SFO	249	828	0.5	0.986
					mean [b]		252	837		
Stir-2	2013EFT-IFP0735	7.5	2.35	35	[pyrazole]	SFO	273	908	2.1	0.964
Z-1	2013EFT-IFP0735	6.8	2.35	19	[pyrazole]	SFO	353	1173	1.7	0.949
Sp-2.1	2013EFT-IFP0735	6.1	0.65	3	[pyrazole]	SFO	204	676	1.3	0.983
Iowa	2013EFT-IFP0874	6.1 [a]	2.50	19	[phenyl]	SFO	220	731	1.4	0.980
		6.1 [a]	2.50	19	[pyrazole]	SFO	183	609	0.8	0.991
					mean [b]		202	670		
CA-SL	2013EFT-IFP0763	7.5	0.39	5	[pyrazole]	SFO	141	469	0.9	0.996
DU-PF	2013EFT-IFP0763	6.7	4.7	20	[pyrazole]	SFO	200	663	1.6	0.979
DU-L	2013EFT-IFP0763	5.5	3.48	32	[pyrazole]	SFO	216	716	1.4	0.980
Geometric mean European soils [b]							265	881		
Geometric mean United States soils [b]							187	621		
Geometric mean all soils [b]							223	740		

Notes:

[a] pH in 0.01 M $CaCl_2$

[b] Calculated by reviewer, representing mean of the results of the phenyl and pyrazole-label of one soil.

Overview of the metabolic pathway of fluindapyr in aerobic soil

Fluindapyr was metabolised in aerobic soil (depicted in Figure 3) either by hydroxylation resulting in 3-hydro-fluindapyr, or by carboxylation resulting in *cis*-1-carboxy-fluindapyr or *trans*-1-carboxy-fluindapyr. Mineralization (formation of bound residues and CO_2) was limited, reaching a maximum of almost 5 percent of the AR in one soil.

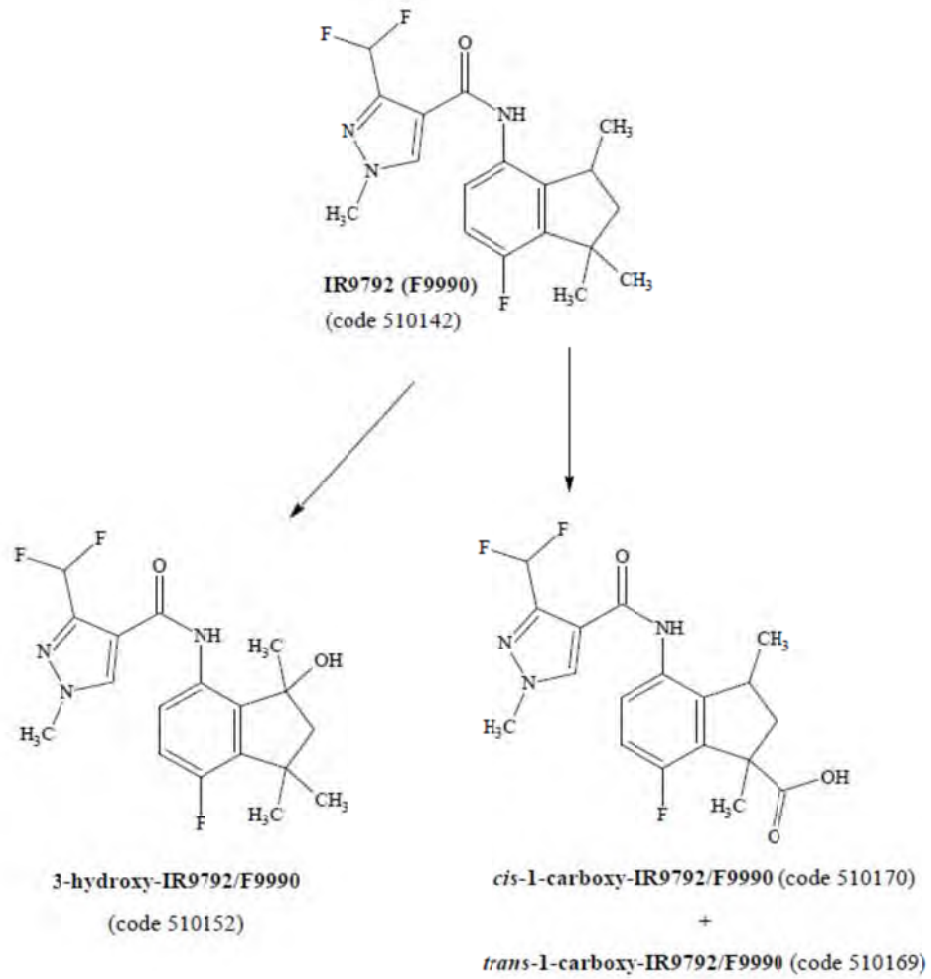


Figure 3 The proposed metabolic pathway for fluindapyr in aerobic soil

Aerobic degradation of metabolites in soil – laboratory studies with 3-OH-fluindapyr

The degradation of ^{14}C-3-hydroxy-fluindapyr under aerobic laboratory conditions was studied in three terrestrial soils from Europe [Vanini & Zerbinati, 2017, 2016EFT-IFP2696] and from the United States [Vanini, 2016c, 2015EFT-IFP2086]. Soil characteristics are reported in Table 28.

In both studies the soil samples (50 g dry weight) were maintained in the dark at a temperature of 20 ± 2 °C and the soil moisture was maintained at 60 percent of the Maximum Water Holding Capacity (MWHC) corresponding to a pF value of 2 (0.1 bar). After 7 days of acclimatization [^{14}C-pyrazole]-1-hydroxy fluindapyr was applied to the respective soil incubation units at 0.5 mg/kg, equivalent to a field application rate of 236–250 g ai/ha in all studies.

Each unit was then connected to 2N KOH traps to collect volatile radioactivity and incubated in the dark at 20 ± 2 °C for 123 days. Moistened CO_2-free air was passed through units to maintain aerobic conditions. The soil moisture content was adjusted by adding purified water during the study. For each soil, an untreated sample was set up and used as blank. Duplicate incubation units were collected and analysed at 0, 13, 31, 52, 81, and 117 days in the European soil studies and 0, 7, 25, 48, 81, and 123 day in the United States soils. No storage data were reported. The experimental phase of both studies was performed within 6 months. The storage stability of fluindapyr and four of its metabolites over a period of 2 years was established by Skags, 2018 [Report X1509BK]. See section on storage stability.

Table 28 Soil characteristics for studies 2016EFT-IFP2696 and 2015EFT-IFP2086

Soil name	Sp-2.1	Sp-2.2	Stir-2	CA-SL	DU-L	Iowa
Study	Vanini&Zerbatini, 2017	Vanini&Zerbatini, 2017	Vanini&Zerbatini, 2017	Vanini, 2017	Vanini, 2017	Vanini, 2017
Report ID	2016EFT-IFP2696	2016EFT-IFP2696	2016EFT-IFP2696	2015EFT-IFP2086	2015EFT-IFP2086	2015EFT-IFP2086
Location	Germany	Germany	Italy	CA, US	ND, US	IA, US
Soil texture (USDA) [a]	Sand	Sandy loam	Clay loam	Loamy sand	Sandy clay loam	Silt loam
--Sand (%)	86	76	32	82	46	16
-- Silt (%)	11	16	34	14	26	66
-- Clay (%)	3	8	34	4	28	18
Organic Carbon (%)	0.71	1.61	2.21	0.35	2.96	1.86
CEC (meq/100 g)	4.3	9.7	31.2	5.0	21.8	12.1
pH (in 0.01M $CaCl_2$)	4.9	5.4	7.3	6.7	4.9	5.9
Water Holding Capacity at pF 2.0 (0.1 bar) [percent]	19.3	26.9	25.6	15.1	41.8	44.7
Bulk density (disturbed [g/cm^3]	1.50	1.26	1.08	1.46	1.01	1.02
Dry mass (%)	98.77	90.50	93.60	99.42	81.96	84.90
Microbial biomass initial [mg microbial C/100 g dry weight]	29.41	36.93	73.84	54.46	79.68	68.77
Microbial biomass initial (percent referred to total organic carbon)	4.14	2.29	3.34	15.56	2.69	3.70
Microbial biomass final [mg microbial C/10 g dry weight]	27.35	32.82	71.78	78.49	85.64	91.20
Microbial biomass final (percent referred to total organic carbon)	3.85	2.04	3.25	22.43	2.89	5.06

Notes:

n.d. = Not determined

[a] Classification according to United States Department of Agriculture (USDA)

[b] pH in 0.01 M $CaCl_2$

Soils were extracted with acetone:water (7:3 v/v and 1:1 v/v)) for all sample points and with acetone:0.5N HCl (1:1 v/v) from 13 days [2016EFT-2696] and 7 days [2015EFT-IFP2086] onwards. The extractable radioactivity was determined by LSC. Suitable aliquots of soil extracts were pooled, concentrated and analysed by TLC to quantify parent degradation and metabolite formation. Representative extract samples were analysed by Liquid Chromatography - Mass Spectrometry (LC-MS) and compared to relevant reference compounds in order to identify the main degradation compounds. The unextracted radioactivity was determined by LSC, after oxidation of aliquots of soil residue using a biological oxidizer. The radioactivity content in the traps (volatiles) was determined by LSC, except at 0 day in all studies. Chiral High Performance Liquid Chromatography (HPLC) was used to confirm the enantiomeric ratio (S/R) of the metabolite in the United States soils [Vanini, 2016c, 2015EFT-IFP2086].

The extractable radioactivity ranged from 93-102 percent of Applied Radioactivity (AR) in all samples from both studies. The unextracted radioactivity (bound residue) slightly increased during the study but did not exceed 5.8 percent AR in both studies. Volatile radioactivity in the KOH solution trap was below detection limit in all samples from both studies.

The mass balance ranged from an average of 97 percent to 102 percent AR and the individual mass balance values were within 90–110 percent AR throughout the studies.

The chromatographic pattern of the European soil extracts showed a very slow degradation of 3-OH-fluindapyr. The unchanged 3-OH-fluindapyr decreased to 98 percent, 99 percent, and to 96 percent AR at the last sampling interval in SP-2.1, SP-2.2, and Stir-2 soils, respectively. No significant degradation compounds were detected in the extracts. The chromatographic pattern of the United States soil extracts also showed a very slow degradation of 3-hydroxy-fluindapyr and the formation of two or three minor degradation compounds depending on soil. None of them was increasing and the maximum amount reached was 4.2 percent AR. The unchanged 3-hydroxy-fluindapyr decreased to 89 percent, 87 percent, and to 92 percent AR at the last sampling interval in CA-SL, DU-L, and Iowa soils, respectively.

HPLC analyses of representative soil extracts showed that the enantiomeric ratio of unchanged ^{14}C-3-OH-fluindapyr remained constant during the study and was found to be about 50:50 in theUnited Statesstudy (ratios ranged from 0.97 to 1.01).

The occurrence of 3-OH-fluindapyr in the six tested soils are presented in Table 29.

Table 29 Distribution of radioactivity (percent of applied radioactivity) in 3 European and 3 United States soils after application of ^{14}C-phenyl or ^{14}C-pyrazole labelled 3-OH-fluindapyr (means of duplicate samples)

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval					
Sp-2.1	Interval in days	0	13	31	52	81	117
2016EFT-IFP2696	3-OH-fluindapyr	100.45	99.72	101	99.99	99.45	97.97
(^{14}C-pyrazole)	Bound (after 3 extractions)	0.43	ND	ND	ND	0.28	1.78
	Volatile - KOH	n.a.	ND	ND	ND	ND	ND
	Mass balance	100.88	99.72	101	99.99	99.74	99.75
Sp-2.2	Interval in days	0	13	31	52	81	117
2016EFT-IFP2696	3-OH-fluindapyr	99.98	100.31	101.35	100.16	98.05	99.1
(^{14}C-pyrazole)	Bound (after 3 extractions)	0.56	ND	ND	ND	1.67	0.33
	Volatile - KOH	n.a.	ND	ND	ND	ND	ND
	Mass balance	100.53	100.31	101.35	100.16	98.05	99.43
Stir-2	Interval in days	0	13	31	52	81	117
2016EFT-IFP2696	3-OH-fluindapyr	99.93	100.28	100.15	98.88	98.87	95.9
(^{14}C-pyrazole)	R_F = 0.12	ND	ND	ND	0.91	ND	1.87
	Bound (after 3 extractions)	0.55	ND	ND	ND	0.46	1.54
	Volatile - KOH	n.a.	ND	ND	ND	ND	ND
	Mass balance	100.48	100.28	100.15	99.79	99.32	99.3
CA-SL	Interval in days	0	7	25	48	81	123
2015EFT-IFP2086	3-OH-fluindapyr	98	95.81	96.33	97.73	91.75	89.23
(^{14}C-pyrazole)	Unknown S2	ND	ND	1.79	2.78	3.51	3.61
	Unknown S3	ND	ND	ND	ND	1.98	2.54
	Unknown S4	ND	ND	ND	ND	ND	3.03
	Bound (after 3	0.7	2.18	0.43	0.48	1.09	1.18

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval					
	extractions)						
	Volatile - KOH	n.a.	ND	ND	ND	ND	ND
	Mass balance	98.7	97.99	98.55	100.99	98.33	99.59
DU-L	Interval in days	0	7	25	48	81	123
2015EFT-IFP2086	3-OH-fluindapyr	98.36	95.63	96.86	95.19	92.51	86.94
(^{14}C-pyrazole)	Unknown S2	ND	ND	ND	ND	ND	ND
	Unknown S3	ND	ND	0.94	2.27	3.88	4.14
	Unknown S4	ND	ND	ND	ND	ND	2.47
	Bound (after 3 extractions)	0.94	2.01	1.32	ND	2.56	5.58
	Volatile - KOH	n.a.	ND	ND	ND	ND	ND
	Mass balance	99.3	97.64	99.12	97.46	98.95	99.13
Iowa	Interval in days	0	7	25	48	81	123
2015EFT-IFP2086	3-OH-fluindapyr	99.03	98.98	99.35	97.03	97.18	92.25
(^{14}C-pyrazole)	Unknown S2	ND	ND	ND	ND	ND	ND
	Unknown S3	ND	ND	ND	1.05	1.75	ND
	Unknown S4	ND	ND	ND	ND	ND	2.11
	Bound (after 3 extractions)	0.69	0.52	0.5	1.05	1.06	5.64
	Volatile - KOH	n.a.	ND	ND	ND	ND	ND
	Mass balance	99.72	99.5	99.85	99.13	99.99	100

Notes

n.a. = Not analysed.

ND = Not detected.

3-Hydroxy-fluindapyr very slowly degraded in the studies and the data fit well with the Single First-Order kinetic model (SFO) for each soil. The DT_{50} and DT_{90} values for 3-OH-fluindapyr were in higher than 1000 days in the European soils. The DT_{50} values were 970, 794, and 1302 days in the CA-SL, DU-L, and IOWA soils, respectively and the DT_{90} values 3222, 2639, and 4325 days, respectively. The results are summarized in Table 34.

Aerobic degradation of metabolites in soil – laboratory studies with cis-1-COOH-fluindapyr and trans-1-carboxy-fluindapy

The degradation of ^{14}C-*cis*-1-COOH-fluindapyr and ^{14}C-*trans*-1-COOH-fluindapyr under aerobic laboratory conditions was studied in three terrestrial soils from Europe [Mainolfi & Elmini, 2017, 2016EFT-IFP2510; Vanini, 2016d, 2016EFT-IFP2504]. Soil characteristics are reported in Table 30.

In both studies the soil samples (50 g dry weight) were maintained in the dark at a temperature of 20 ± 2 °C and the soil moisture was maintained at 60 percent of the Maximum Water Holding Capacity (MWHC) corresponding to a pF value of 2 (0.1 bar). After 7 days of acclimatization [^{14}C-pyrazole]-*cis*-1-COOH-fluindapyr or [^{14}C-pyrazole]-*trans*-1-COOH-fluindapyr was applied to the respective soil incubation units at 0.5 mg/kg, equivalent to a field application rate of 252 and 260 g ai/ha, respectively in both studies.

Each unit was then connected to 2N KOH traps to collect volatile radioactivity and incubated in the dark at 20 ± 2 °C for 117-120 days. Moistened CO_2-free air was passed through units to maintain

aerobic conditions. The soil moisture content was adjusted by adding purified water during the study. For each soil, an untreated sample was set up and used as blank. Duplicate incubation units were collected and analysed at 0, 10, 24, 48, 78, and 120 days in studies with ^{14}C-cis-1-COOH-fluindapyr and 0, 11, 24, 52, 81, and 117 day in the studies with ^{14}C-*trans*-1-COOH-fluindapyr. The experimental phase of both studies was performed within 6 months. The storage stability of fluindapyr and four of its metabolites over a period of 2 years was established by Skags, 2018 [Report X1509BK]. See section on storage stability.

Table 30 Soil characteristics for studies using ^{14}C-*cis*-1-COOH-fluindapyr or ^{14}C-*trans*-1-COOH-fluindapyr

Soil name	Sp-2.1	Sp-2.2	Stir-2	Sp-2.1	Sp-2.2	Stir-2
Study	Mainolfi& Elmini, 2017	Mainolfi& Elmini, 2017	Mainolfi& Elmini, 2017	Vanini, 2016d	Vanini, 2016d	Vanini, 2016d
Report ID	2016EFT-IFP2510	2016EFT-IFP2510	2016EFT-IFP2510	2016EFT-IFP2504	2016EFT-IFP2504	2016EFT-IFP2504
metabolite	[^{14}C-pyrazole]-*cis*-1-COOH-fluindapyr			[^{14}C-pyrazole]-*trans*-1-COOH-fluindapyr		
Location	Germany	Germany	Italy	Germany	Germany	Italy
Soil texture (USDA) [a]	Sand	Sandy loam	Clay loam	Sand	Sandy loam	Clay loam
--Sand (%)	86	76	32	86	76	32
-- Silt (%)	11.5	16	34	11.5	16	34
-- Clay (%)	2.5	7.7	34	2.5	7.7	34
Organic Carbon (%)	0.71	1.59	2.13	0.71	1.59	2.13
CEC (meq/100 g)	4.2	9.7	29.8	4.2	9.7	29.8
pH (in 0.01M $CaCl_2$)	4.9	5.4	7.8	4.9	5.4	7.8
Water Holding Capacity at pF 2.0 (0.1 bar) [percent]	19.5	26.1	28.8	19.5	26.1	28.8
Bulk density (disturbed [g/cm^3]	1.5	1.34	1.12	1.53	1.34	1.12
Dry mass (%)	99.61	99.14	97.35	99.67	98.95	94.53
Microbial biomass initial [mg microbial C/100 g dry weight]	29.28	37.46	69.46	30.10	23.28	73.84
Microbial biomass initial (percent referred to total organic carbon)	4.1	2.36	3.3	4.24	2.41	3.47
Microbial biomass final [mg microbial C/10 g dry weight]	28.5	34.95	69.2	29.46	34.37	72.94
Microbial biomass final (percent referred to total organic carbon)	4.0	2.2	3.2	4.15	2.16	3.42

Notes:

[a] Classification according to United States Department of Agriculture (USDA).

Soils were extracted with acetone:water (7:3 v/v and 1:1 v/v) for all sample points in both studies and with acetone:0.5N HCl (1:1 v/v) from 11 days onwards in study 2016EFT-IFP2504. In study 2016EFT-IFP2504 all samples were extracted twice with acetone:0.5N HCl (1:1 v/v). The extractable radioactivity was determined by LSC. Suitable aliquots of soil extracts were pooled, concentrated and analysed by TLC. The unextracted radioactivity was determined by LSC, after oxidation of aliquots of soil residue using a biological oxidizer. The radioactivity content in the traps (volatiles) was determined by LSC.

For the *cis*-metabolite, the extractable radioactivity ranged from 90.34 percent to 105.55 percent AR in SP-2.1 soil, from 90.38 percent to 101.58 percent AR in SP-2.2 soil and from 86.47 percent to 101.61 percent AR in Stir-2 soil. The extractable radioactivity ranged from 91.49 percent to 97.41 percent

AR in SP-2.1 soil, from 90.86 percent to 96.65 percent AR in SP-2.2 soil, and from 92.26 percent to 96.06 percent AR in Stir-2 soil with the *trans*-metabolite.

The non-extractable radioactivity (bound residue) slowly increased during the study to 2.21 percent, 4.00 percent and 9.03 percent AR in SP-2.1, SP-2.2 and Stir-2 soil respectively with the cis-metabolite. In the study with the trans-metabolite, the non-extractable radioactivity (bound residue) slowly increased to 4.09 percent, 4.44 percent, and 4.46 percent AR in SP-2.1, SP-2.2, and Stir-2 soil, respectively.

The radioactivity found in KOH traps reached 1.11 percent in SP-2.1 soil, 1.69 percent in SP-2.2 soil and 1.67 percent in Stir-2 soil with the *cis*-metabolite. The volatile radioactivity increased from 0.46 percent, 0.37 percent, and 0.23 percent AR at 52 days to 1.45 percent, 1.56 percent, and 0.92 percent AR in SP-2.1, SP-2.2, and Stir-2 soils, respectively with the *trans*-metabolite.

The ^{14}C-Mass Balance for each individual sample was always higher than 92 percent and 95 percent throughout the entire incubation duration for the cis and the trans metabolite, respectively in both studies and ranged from 93 percent to 106 percent AR and from 95 to 98 percent, respectively.

Twelve versus eight degradation products were found, always lower than 10 percent AR, in the studies with the *cis*- and *trans*-metabolite, respectively.

The occurrence of *cis*- and *trans*-1-COOH-fluindapyr in the three tested soils are presented in Table 31.

Table 31 Distribution of radioactivity (percent of applied radioactivity) in 3 European soils after application of ^{14}C-pyrazole labelled *cis*- or *trans*-1-COOH-fluindapyr (means of duplicate samples)

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval					
Sp-2.1	Interval in days	0	10	24	48	78	120
2016EFT-IFP2510 [^{14}C-pyrazole]-*cis*-1-COOH fluindapyr) [a]	Extract I + II + III	99.1	98.6	99.5	98.1	95	92.3
	cis-1-COOH	99.1	97.1	97.8	92.8	84	76.9
	Unknown S2	ND	ND	ND	ND	ND	0.54
	Unknown S3	ND	ND	ND	ND	ND	0.7
	Unknown S7	ND	ND	ND	0.78	3.3	3.78
	Unknown S8	ND	ND	ND	0.62	1.1	1.37
	Unknown S9	ND	ND	ND	0.98	2.18	2.6
	Unknown S10	ND	ND	ND	0.96	1.68	3
	Unknown S11	ND	ND	ND	ND	ND	0.31
	Unknown S12	ND	ND	ND	ND	0.72	1
	Unknown S13	ND	1.48	1.67	1.93	1.97	2.04
	Bound (after 3 extractions)	0.04	0.42	0.39	0.53	1.6	2.1
	Volatile - KOH	n.a.	ND	ND	0.07	0.58	1.1
	Mass balance	99.2	99.3	99.9	98.7	97.2	95.5
Sp-2.2	Interval in days	0	10	24	48	78	120
2016EFT-IFP2510 [^{14}C-pyrazole]-*cis*-	Extract I + II + III	100.4	100.6	99.6	98.2	92.7	90.7
	cis-1-COOH	100.2	99.6	98.5	88	72.6	64.7

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval					
1-COOH fluindapyr) [a]							
	Unknown S2	ND	ND	ND	ND	0.74	0.61
	Unknown S3	ND	ND	ND	ND	0.65	0.72
	Unknown S4	ND	ND	ND	0.74	1.32	1.96
	Unknown S5	ND	ND	ND	ND	1.38	1.1
	Unknown S6	ND	ND	ND	ND	ND	0.68
	Unknown S7	ND	ND	0.8	1.59	3.3	2.84
	Unknown S8	ND	ND	ND	1.19	2.02	1.86
	Unknown S9	ND	ND	ND	1.48	2.3	2.87
	Unknown S10	ND	ND	ND	1.41	2.32	2.46
	Unknown S11	ND	ND	ND	ND	ND	1.74
	Unknown S12	ND	ND	ND	2.87	5.36	8.38
	Unknown S13	ND	1.02	0.6	0.94	0.72	0.8
	Bound (after 3 extractions)	0.11	0.96	0.6	1.2	2.4	3.8
	Volatile - KOH	ND	ND	ND	0.32	0.84	1.5
	Mass balance	100.6	101.2	100.2	99.8	95.9	96
Stir-2	Interval in days	0	10	24	48	78	120
2016EFT-IFP2510 [^{14}C-pyrazole]-*cis*-1-COOH fluindapyr) [a]	Extract I + II + III	99.9	101.6	97.3	97.5	92.3	86.8
	cis-1-COOH	99.9	101.6	94.1	75.2	59.7	46.6
	Unknown S2	ND	ND	ND	ND	1.475	0.8
	Unknown S3	ND	ND	ND	0.82	1.52	0.72
	Unknown S4	ND	ND	ND	1.28	0.77	4.08
	Unknown S6	ND	ND	ND	0.75	ND	1.38
	Unknown S7	ND	ND	1.37	1.24	4.38	4.07
	Unknown S8	ND	ND	ND	1.99	2.32	2.72
	Unknown S9	ND	ND	ND	2.16	2.95	3.23
	Unknown S10	ND	ND	ND	1.32	2.96	2.63
	Unknown S11	ND	ND	ND	2.245	ND	3.6
	Unknown S12	ND	ND	1.84	10.8	16.2	16.9
	Bound (after 3 extractions)	0.22	1.49	4.4	3.1	4.7	9
	Volatile - KOH	ND	ND	ND	0.24	0.94	1.5
	Mass balance	100.1	103.1	101.7	100.8	98	97.3
Sp-2.1	Interval in days	0	11	24	52	81	117
2016EFT-IFP2510 [^{14}C-pyrazole]-*trans*-1-COOH fluindapyr)	Extract I + II + III	97.21	97.9	93.5	95.6	92.4	91.5
	trans-1-COOH	97.21	88.11	87.63	80.01	70.53	67.93
	Unknown S2	ND	ND	ND	1.54	1.54	1.46
	Unknown S3	ND	ND	1.04	2.79	3.02	3.55
	Unknown S4	ND	1.16	1.11	3.23	4.4	4.84
	Unknown S5	ND	0.69	1.83	2.19	3.39	4.62
	Unknown S6	ND	0.79	ND	ND	ND	ND

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval					
	Unknown S7	ND	ND	ND	ND	1.17	1.17
	Unknown S8	ND	4.13	4.85	56.9	6.43	6.68
	Unknown S9	ND	ND	ND	ND	1.92	1.29
	Bound (after 3 extractions)	0.46	1.7	1.4	1.4	4.1	4.1
	Volatile - KOH	ND	ND	ND	0.47	0.69	1.4
	Mass balance	97.7	96.6	97.9	97.6	97.2	97
Sp-2.2	Interval in days	0	11	24	52	81	117
2016EFT-IFP2510 [^{14}C-pyrazole]-*trans*-1-COOH fluindapyr)	Extract I + II + III	94.4	95.2	95.1	95.7	93.5	90.9
	trans-1-COOH	95.95	92.38	86.02	77.86	68.18	60.07
	Unknown S2	ND	ND	1.83	3.65	4.89	4.87
	Unknown S3	ND	ND	1.15	3.31	3.65	3.98
	Unknown S4	ND	0.64	1.26	3.49	3.22	4.39
	Unknown S5	ND	0.51	1.44	1.77	3.42	4.72
	Unknown S6	ND	0.39	ND	ND	ND	ND
	Unknown S7	ND	0.35	1.51	3.97	6.71	8.82
	Unknown S8	ND	0.95	1.89	1.67	1.8	1.91
	Unknown S9	ND	ND	ND	ND	1.59	2.16
	Bound (after 3 extractions)	0.6	1.5	1.3	2.1	3.7	4.3
	Volatile - KOH	ND	ND	ND	0.38	0.6	1.6
	Mass balance	95	96.7	96.4	98.2	97.8	96.8
Stir-2	Interval in days	0	11	24	52	81	117
2016EFT-IFP2510 [^{14}C-pyrazole]-*trans*-1-COOH fluindapyr)	Extract I + II + III	97	95.6	95.5	95.3	93.1	92.3
	trans-1-COOH	93.96	93.78	86.14	76.44	68.9	66.29
	Unknown S2	ND	ND	1.1	2.35	3.35	2.81
	Unknown S3	ND	ND	1.11	2.71	3.1	2.98
	Unknown S4	ND	0.39	2.22	3.76	3.24	4.68
	Unknown S5	ND	0.35	0.98	3.2	3.13	3.82
	Unknown S6	ND	0.35	0.41	0.74	ND	ND
	Unknown S7	ND	0.76	2.84	6.14	8.25	9.47
	Unknown S8	ND	ND	0.73	ND	ND	ND
	Unknown S9	ND	ND	ND	ND	3.1	2.32
	Bound (after 3 extractions)	1.6	0.92	1.4	2	4.1	4.4
	Volatile - KOH	ND	ND	ND	0.24	0.68	0.92
	Mass balance	95.5	96.5	96.9	97.6	97.9	97.7

Notes:

[a] The mean percent AR of the unknowns were calculated by the reviewer.

The concentration of *cis*-1-COOH fluindapyr gradually decreased during incubation, according to a Single First-Order (SFO) kinetic in all soil, reaching 77 percent of AR in SP-2.1 soil, 65 percent of AR in SP-2.2 soil and 47 percent of AR in Stir-2 soil by the end of the incubation. ^{14}C-*trans*-1-COOH-fluindapyr also degraded slowly according to a Single First-Order (SFO) kinetic in all soils. Its amounts remaining at the last sampling interval were 67.93 percent, 60.07 percent, and 66.29 percent AR in SP-2.1, SP-2.2, and Stir-2 soils, respectively. The DT_{50lab} values for *cis*-1-COOH-fluindapyr ranged from 102–320 days. The DT_{50lab} values for *trans*-1-COOH-fluindapyr ranged from 170–223 days. The individual results and geometric means are summarized in Table 34.

Aerobic degradation of metabolites in soil–laboratory studies with pyrazole carboxamide

The rate of degradation of the photolytic soil metabolite pyrazole carboxamide (CSCC210616) was studied under aerobic conditions in 3 European and one United States soils [Simmonds & MacKenzie, 2009, NC/08/027]. Soil characteristics are reported in Table 32.

The soil samples (50 g dry weight) were maintained in the dark at a temperature of 20 ± 2 °C and the soil moisture was maintained at a moisture content equivalent to pF value of 2 (0.1 bar) for up to 119 days. After acclimatization [^{14}C-pyrazole]-pyrazole carboxamide was applied to the respective soil incubation units at an actual field application rate of 22 g ai/ha.

At intervals of 0, 3, 7, 14, 30, 60, 91 and 119 days after application duplicate flasks of each

soil were removed from the incubation system. Soil samples were extracted on the day that they were collected for analysis. Extracts were then stored refrigerated for a maximum of 10 days until processed. Concentrated extracts were stored frozen and initially analysed within 21 days of being generated. The longest storage period for extracts before the final HPLC analysis was 116 days. Comparison of chromatograms of the zero time and 3 day extracts of the North Dakota soil profiled following 116 days and 113 days storage confirmed that the test item pyrazole carboxamide (CSCC210616) and the metabolite CSAA798670 were stable throughout the respective storage periods.

Each soil sample was extracted three times with acetonitrile / water (1:1, v/v) at room temperature, including dilute formic acid in the final extraction. Extracted soil samples were air-dried, ground to a fine powder and the residual radioactivity quantified by combustion. Extracts from each soil sample were concentrated and analysed by reverse phase high performance liquid chromatography (HPLC). In addition, selected soil extracts were analysed by thin layer chromatography (TLC) and by mass spectrometry (LC-MS/MS) to provide confirmation of structural identity. At each sampling interval, the radioactivity in the trap solutions associated with each sample was quantified by liquid scintillation counting (LSC). Traps were also changed between sampling intervals and the radioactivity quantified by LSC.

Table 32 Soil characteristics for studies using ^{14}C-pyrazole carboxamide

Soil name	Marsillargues	Gartenacker	18 Acres	North Dakota
Location	La Paulette Marsillargues France	Les Barges Vouvry Switzerland	Nupton Road Warfield, Bracknell, United Kingdom	Gardner, Cass County, NF, United States
Soil texture (USDA) [a]	Clay	Loam	Sandy clay loam	Sandy loam
--Sand (%)	16	39	54	60
-- Silt (%)	39	49	21	26
-- Clay (%)	45	12	25	14
Organic Carbon (%) [b]	1.0	1.9	2.3	3.9
CEC (meq/100 g)	19.2	8.8	17.2	19.7
pH (in 0.01M $CaCl_2$)	7.7	7.1	6.4	6.7

Soil name	Marsillargues	Gartenacker	18 Acres	North Dakota
Water Holding Capacity at pF 2.0 (0.1 bar) [percent]	22.7	39.0	29.8	34.7
Bulk density (disturbed [g/cm^3]	1.15	0.95	1.19	1.02
Dry mass (%)	n.r.	n.r.	n.r.	n.r.
Microbial biomass initial [mg microbial C/100 g dry weight]	239	386	699	455
Microbial biomass final [mg microbial C/10 g dry weight]	2001	296	623	453

Notes:

[a] Classification according to United States Department of Agriculture (USDA).

[b] Organic carbon percent calculated from organic matter percent assuming a conversion factor of 1.724.

The overall mean recovery values for each soil ranged from 95 percent to 97 percent AR. Recoveries for individual flasks ranged between 91 percent and 103 percent AR.

In all soils the extractable radioactivity decreased with time with a corresponding increase in

the unextracted radioactivity (maximum ca 35 percent in the 18 Acres sandy loam) and carbon dioxide (maximum ca 32 percent in the Gartenacker loam) after 119 days of incubation. The level of pyrazole carboxamide (CSCC210616) declined rapidly, reaching ≤ 6.0 percent AR in all soils by 14 days incubation. The results are shown in the Table 33.

Table 33 Distribution of radioactivity (percent of applied radioactivity) in three European and one United States soil after application of ^{14}C- pyrazole carboxamide (means of duplicate samples)

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval							
Marsillargues	Interval in days	0	3	7	14	30	60	91	119
NC/08/027	Carboxamide (CSCC210616)	98	67	30	5.1	0	0	0	0
(^{14}C-pyrazole)	CSCD465008	0	0.88	5.2	13	41	75	57	54
	CSAA798670	0	26	57	65	46	0	0	0
	Total extracted	98	94	91	93	87	75	57	54
	Unexctracted	1.24	3.4	5.54	8.5	7.5	14	24	25
	Volatiles	n.a.	0.31	0.67	0.73	2.1	5.8	12	16
	Mass balance	99	98	97	92	96	95	93	94
Gartenacker	Interval in days	0	3	7	14	30	60	91	119
NC/08/027	Carboxamide (CSCC210616)	98	49	9.2	0.84	0	0	0	0
(^{14}C-pyrazole)	CSCD465008	0	12	40	87	73	56	44	36
	CSAA798670	0	33	41	4.2	0	0	0	0
	Total extracted	98	93	89	83	73	56	44	36
	Unextracted	2.6	4.4	8.3	11	15	21	25	27
	Volatiles	n.a.	0.87	1.7	2.3	7.2	16	27	32
	Mass balance	100	99	99	96	95	93	96	96
18 Acres	Interval in days	0	3	7	14	30	60	91	119
NC/08/027	Carboxamide (CSCC210616)	100	31	0.79	0	0	0	0	0
(^{14}C-pyrazole)	CSCD465008	0	5	24	49	60	53	41	37
	CSAA798670	0	56	62	24	3.8	0	0	0
	Total extracted	100	95	86	73	64	53	41	37

Soil/Study (label)	Compounds	percent of applied radioactivity per time interval							
	Unextracted	1.4	3.8	10	18	22	30	32	35
	Volatiles	n.a.	0.73	2.1	3.2	7.2	13	22	26
	Mass balance	102	96	99	94	92	96	95	97
North Dakota	Interval in days	0 3 7 14 30 60 91 119	3	7	14	30	60	91	119
NC/08/027	Carboxamide (CSCC210616)	98	58	23	6	0.84	0	0	0
(^{14}C-pyrazole)	CSCD465008	0	0	9.6	21	48	61	52	45
	CSAA798670	0	34	56	57	26	0	0	0
	Total extracted	98	92	88	84	75	61	52	45
	Unextracted	2.1	3.2	7.4	6.2	11	18	21	27
	Volatiles	n.a.	61	1.1	2.4	6.34	13	21	23
	Mass balance	100	96	97	93	92	92	95	95

Notes:

n.a. = Not analysed CSCD465008 = N-DesMet-pyrazole carboxamide acid; CSAA798670 = pyrazole carboxamide acid

Two metabolites, identified as CSAA798670 and CSCD465008, were observed in all soils, reaching maximum values of 65 percent AR in the Marsillargues clay and 78 percent in the Gartenacker loam soils respectively, after 14 days of incubation.

In all soils, aerobic degradation of [^{14}C]-pyrazole carboxamide led to the formation of $^{14}CO_2$ and non-extractable residues via two intermediate metabolites. The amide group was hydrolysed to form the acid metabolite CSAA798670 (pyrazole carboxamide acid), which then underwent demethylation to form N-DesMet-pyrazole carboxamide acid (CSCD465008). A proposed degradation pathway is depicted in Figure 4

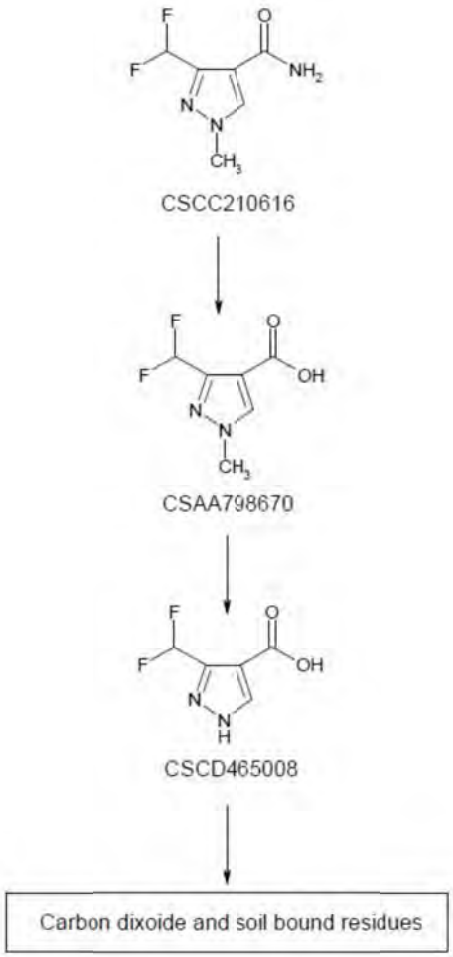


Figure 4 Aerobic degradation pathway of pyrazole carboxamide

DT_{50} and DT_{90} values for pyrazole carboxamide (CSCC210616) were determined following the recommendations of the FOCUS work group using a simple first order model (SFO). The results are shown in Table 34. Summary of DT_{50} and DT_{90} values of (photolytic) soil metabolites of fluindapyr in aerobic soils

Table 34 Summary of DT_{50} and DT_{90} values of (photolytic) soil metabolites of fluindapyr in aerobic soils

Soil name	Metabolite	Model	DT_{50} (days)	DT_{90} (days)	χ^2 error	r^2	Reference
Sp-2.1	3-OH-fluindapyr	SFO	>1000	>1000	0.4	0.486	Vanini & Zerbinati, 2017, 2016EFT-IFP2696
Sp-2.2	3-OH-fluindapyr	SFO	>1000	>1000	0.6	0.317	
Stir-2	3-OH-fluindapyr	SFO	>1000	>1000	0.5	0.683	
CA-SL	3-OH-fluindapyr	SFO	970	3222	0.5	0.932	Vanini, 2016c, 2015EFT-IFP2086
DU-L	3-OH-fluindapyr	SFO	794	2639	1.0	0.893	
Iowa	3-OH-fluindapyr	SFO	1302	4325	0.8	0.807	
	Geometric mean		n.c.	n.c.			
Sp-2.1	*cis*-1-COOH-fluindapyr	SFO	320	>1000	1.4	0.869	Mainolfi&Elmini, 2017, 2016EFT-IFP2510
Sp-2.2	*cis*-1-COOH-fluindapyr	SFO	175	583	2.6	0.956	
Stir-2	*cis*-1-COOH-fluindapyr	SFO	102	340	3.3	0.973	
	Geometric mean		179	n.c.			
Sp-2.1	*trans*-1-COOH-fluindapyr	SFO	223	741	2.21	0.949	Vanini, 2016d, 2016EFT-IFP2504
Sp-2.2	*trans*-1-COOH-fluindapyr	SFO	170	564	0.615	0.995	
Stir-2	*trans*-1-COOH-fluindapyr	SFO	202	672	2.27	0.955	
	Geometric mean		197	655			

Soil name	Metabolite	Model	DT_{50} (days)	DT_{90} (days)	χ^2 error	r^2	Reference
Marsillargues	pyrazole carboxamide [a]	SFO	4.1	14	7.22	n.r.	Simmonds&Mackenzie, 2009, NC/08/27
Gartenacker	pyrazole carboxamide [a]	SFO	2.5	8.3	8.12	n.r.	
18 Acres	pyrazole carboxamide [a]	SFO	1.7	5.5	5.85	n.r.	
North Dakota	pyrazole carboxamide [a]	SFO	3.5	12	3.36	n.r.	
	Geometric mean		2.8	9.4			

Notes:

n.r. = Not reported.

[a] metabolite found in soil photolysis study, but not in aerobic soil degradation study.

Field dissipation studies in Europe

Study 1+2: Four field dissipation trials were carried out in Germany, United Kingdom, France and Italy in July 2015-August 2017 to study the dissipation and mobility of residues of fluindapyr and its metabolites in soil, following two applications of the a formulated product to bare soil [Gemroth, 2018, 2015-IFP1999 and 2015-IFP1999 Amendment no.1]. In order to assess the behaviour of fluindapyr and its metabolites 3 years after the last application, a second study was performed [Gemroth, 2020a, 2018-IFP4409].

Fluindapyr was sprayed twice, with a seven day interval, to bare soil at actual rates of 229–271 g ai/ha. Soil characteristics for the four soils are summarised in Table 35. Soil cores were taken immediately prior to the first spraying (-1DAA1), after the first application (0DAA2), prior to the second application (-1DAA2), after the second application (0DAA2) and at 1, 4–7, -12, 19–30, 88–1, 114–120, 143–146, 208–218, 358–373, 452–462, and 713–727 days after application. All soil cores were frozen at -18 °C or lower on the day of collection. The maximum storage interval from sampling to extraction was less than 730 days [Gemroth, 2018, 2015-IFP1999], which was within the 24 month period of storage stability demonstrated in study [Skaggs, 2018, 2015EFT-IFP1940]. In the extended study soil cores were taken 1080–1105 and 1232–1261 days after the last application [Gemroth, 2020a, 2018-IFP4409].

Table 35 Soil characteristics for European soils in field dissipation studies

Soil name	8202031/1	8202031/2	820203/3	8202031/4
Location	Lower-Saxony, Germany	Warwickshire, United Kingdom	Aquitaine, Southern France	Lombardy, Italy
Soil texture (USDA) [a]	Loamy sand	Sandy loam	Sandy loam	Loam
-- Sand (%)	82	59	54	34
-- Silt (%)	9	24	31	45
-- Clay (%)	9	17	15	21
Organic Carbon (%) [b]	1.3	1.8	0.64	1.49
Organic Matter (%)	2.25	2.04	1.11	2.56
CEC (meq/100 g)	5.3	10.7	7.5	13.9
pH (H_2O)	5.2	5.9	6.0	6.3
Maximum Water Holding Capacity [percent w/w]	35.3	42.4	28.1	42.3
Water Holding Capacity at pF 2.0 (FC at 0.1 bar) [percent]	15.0	23.8	20.8	31.3
Water Holding Capacity at pF 2.5 (FC at 0.33 bar) [percent]	8.4	12.9	15.9	19.9
Disturbed density g/m^3	1.18	1.22	1.24	1.04
Microbial biomass (mg microbial carbon/kg soil DM) prior to application	188	116	92	141
Microbial biomass (mg microbial	106	n.d.	134	220

carbon/kg soil DM) at the end of the trial				

n.d. = not determined

[a] Classification according to United States Department of Agriculture (USDA)

For residue analysis, the soil cores were cut into soil horizons (0–5 cm, 5–10 cm and 10–25 cm). The segmented soils were homogenized with dry ice and passed through a sieve with 3 mm mesh. Soils were extracted twice with acetone (trial GE01 and IT04) or with a mixture of acetone/water 51 (v/v) (trails United Kingdom02 and FR03). After drying by evaporation, residues from trials GE01 and IT04 were redissolved in methanol/water 8:2 (v/v/). For United Kingdom02 and FR03 methanol was added to the water phase to achieve methanol/water 8:2 (v/v). Soil samples were analysed for fluindapyr, 3-OH-fluindapyr, pyrazole carboxamide and *cis*- and *trans*-COOH-fluindapyr using LC-MS/MS with a reported LOQ of 0.01 for fluindapyr, 0.005 mg/kg for 3-OH-fluindapyr and pyrazole carboxamide and 0.003 and 0.002 mg/kg for *cis*- and *trans*-COOH-fluindapyr, respectively. The results were not corrected for recoveries.

The report included individual analytical results in terms of mg/kg soil, but no summarizing tables (mean of analytical results/sample). The results in terms of g/ha for the 0–5 cm, 5–10 cm and 10–25 cm soil layers were available for the four soils and are shown in Table 36.

Residues in control samples were generally below 0.3LOQ. The procedural recovery values for each analytical batch were within the acceptance criteria of 70–120 percent. The results were not correct for recoveries.

Parent was predominantly found in the 0-5 cm soil layer and less in 5-10 cm soil layer. Maximum levels of parent were found in the 0–10 cm soil layers on day 0–9, except FR (89 days) and ranged from 351 g/ha (Italian trial, day 0) to 522 g/ha (German trial, day 6). Parent declined to about 10 percent after approximately 700 days (French and Italian trials) and after approximately 1100 days in the trials from Germany and the United Kingdom.

Metabolite pyrazole carboxamide incidentally appeared at trace levels (1-6 g/ha) from day 1, but was not observed after 636 days.

Metabolite *cis*-1-COOH-fluindapyr and *trans*-1-COOH-fluindapyr generally appeared in trace levels (1–9 g /ha) at day 1 in all soil levels. Only in the French trial higher levels of *cis*-1-COOH-fluindapyr and *trans*-1-COOH-fluindapyr were observed; 11–45 g/ha from day 28–143, mainly in the top 0-5 cm layer.

Metabolite 3-OH-fluindapyr was observed in all soil samples (0–5 cm) and increased over time with a distribution into the second soil layer (5–10 cm) after approximately 200 days or more. Maximum levels of metabolite 3-OH-fluindapyr were reached after approximately 210–373 days and ranged from 42 to 113 g/ha.

Table 36 Soil residues (expressed in g/ha) in field dissipation studies

DAT (days)	fluindapyr				pyrazole carboxamide			*cis*-1-COOH fluindapyr			*trans*-1-COOH fluindapyr			-3-OH-fluindapyr			
	0-5 cm	5-10 cm	10-25 cm	Total 0-25	0-5 cm	5-10 cm	10-25 cm	0-5 cm	5-10 cm	10-25 cm	0-5 cm	5-10 cm	10-25 cm	0-5 cm	5-10 cm	10-25 cm	Total 0-25
Lower-Saxony, Germany																	
-1 [a]	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1 [b]	372	10	0	382	0	0	0	0	0	0	0	0	0	4	3	0	7
-1	275	11	1	287	0	0	0	0	0	0	1	0	0	12	1	0	13
0	450	22	1	473	0	0	0	0	0	0	1	0	0	12	1	0	13

DAT (days)	fluindapyr				pyrazole carboxamide			*cis*-1-COOH fluindapyr			*trans*-1-COOH fluindapyr			-3-OH-fluindapyr			
	0-5 cm	5-10 cm	10-25 cm	Total 0-25	0-5 cm	5-10 cm	10-25 cm	0-5 cm	5-10 cm	10-25 cm	0-5 cm	5-10 cm	10-25 cm	0-5 cm	5-10 cm	10-25 cm	Total 0-25
1	479	10	0	489	0	0	0	1	0	0	1	0	0	15	0	0	15
6	522	11	0	533	0	0	1	1	1	0	1	1	0	22	0	0	22
9	506	11	0	517	1	0	0	1	1	0	1	1	0	26	1	0	27
22	490	7	1	498	1	1	0	2	2	0	2	1	0	25	2	1	28
30	434	12	0	446	0	0	0	1	1	0	1	1	0	40	1	0	41
89	352	11	0	363	0	0	0	1	2	3	1	2	3	56	2	0	58
120	248	11	3	262	0	0	0	1	2	3	1	1	3	54	4	0	58
146	246	8	2	256	1	0	0	1	1	2	1	1	3	56	4	0	60
208	198	28	6	232	0	0	1	0	1	2	0	1	2	45	14	1	60
358	182	19	8	209	0	0	0	2	1	1	2	2	5	88	24	1	113
432	132	13	1	146	0	0	0	2	1	1	3	1	2	87	17	1	105
636	151	18	1	170	0	0	1	1	1	2	1	1	2	80	28	2	110
713	85	17	1	103	0	0	0	1	1	2	1	1	2	53	28	1	82
1096	64	19	0	83	0	0	0	0	0	0	0	0	0	42	47	5	94
1131	44	13	0	57	0	0	0	0	0	0	0	0	0	36	39	0	75
Warwickshire, United Kingdom																	
-1 [a]	0	0	0	0	0	0	0	0	0	0	0	0	0	1	0	0	1
1 [b]	230	15	4	249	0	0	0	0	0	0	0	0	0	5	0	0	5
-1	131	9	0	140	0	0	0	0	0	0	1	0	0	6	1	0	7
0	538	38	1	577	1	0	0	1	0	0	2	1	0	20	1	0	21
1	455	9	31	495	1	0	0	2	0	0	3	1	0	18	0	1	19
7	422	51	24	497	2	1	0	1	1	0	2	1	0	30	5	3	38
12	442	47	1	490	3	0	0	1	1	0	3	1	0	41	4	2	47
22	265	22	10	297	1	0	0	3	1	0	3	1	0	22	1	0	23
30	344	18	7	369	0	0	0	5	1	2	8	2	1	33	2	0	35
91	252	28	7	287	0	0	0	6	7	3	7	9	3	36	5	0	41
114	215	27	4	246	3	0	0	5	6	4	6	7	5	47	6	4	57
145	192	21	4	217	1	0	0	5	6	7	6	6	7	43	6	1	50
218	225	20	4	249	1	0	0	4	4	6	4	4	5	36	6	2	44
373	91	52	7	150	1	0	1	4	3	3	5	4	4	46	34	4	84
462	62	28	7	97	1	0	1	5	3	1	5	3	3	39	24	6	69
646	55	24	3	82	0	0	0	2	1	1	2	2	1	49	23	3	75
727	37	23	1	61	0	0	0	1	1	1	2	2	2	34	26	2	62
1105	31	15	0	46	0	0	0	1	0	0	2	2	0	29	23	16	68
1261	22	8	0	30	0	0	0	0	0	0	0	0	0	25	16	7	48
						Aquitane, Southern France											
-1 [a]	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	2	2
1 [b]	198	2	0	200	0	0	0	1	0	0	1	0	0	3	0	2	5
-1	120	12	6	138	0	0	0	1	0	0	1	0	0	7	1	2	10
0	359	13	0	372	1	0	0	1	0	0	1	0	0	11	0	3	14
1	353	28	0	381	1	0	0	1	0	0	1	0	0	19	2	2	23
6	330	43	2	375	3	0	0	1	0	0	2	0	0	40	6	2	48
10	304	23	0	327	3	0	0	2	0	0	3	0	0	36	4	2	42
21	298	18	0	316	1	0	0	5	2	0	7	3	0	59	4	1	64
28	405	10	34	449	6	0	0	11	2	2	13	2	3	22	1	4	27
89	450	12	1	463	1	0	0	34	4	1	45	5	0	40	3	0	43
118	149	6	4	159	0	0	0	13	8	2	19	9	2	43	2	1	46

DAT (days)	fluindapyr				pyrazole carboxamide			*cis*-1-COOH fluindapyr			*trans*-1-COOH fluindapyr			-3-OH-fluindapyr			
	0-5 cm	5-10 cm	10-25 cm	Total 0-25	0-5 cm	5-10 cm	10-25 cm	0-5 cm	5-10 cm	10-25 cm	0-5 cm	5-10 cm	10-25 cm	0-5 cm	5-10 cm	10-25 cm	Total 0-25
143	126	10	7	143	1	0	0	15	4	2	18	11	4	43	3	1	47
216	98	13	6	117	0	0	0	2	2	5	3	2	5	40	7	3	50
372	39	11	7	57	1	0	0	2	1	2	2	1	2	39	14	5	58
461	31	10	2	43	0	0	0	2	1	2	2	1	2	29	14	3	46
647	23	6	2	31	0	0	0	1	0	0	1	1	1	34	14	9	57
721	22	13	1	36	0	0	0	1	0	0	1	1	0	33	28	12	73
1102	0	0	0	0	0	0	0	0	0	0	0	0	0	11	13	0	24
1232	0	0	0	0	0	0	0	0	0	0	0	0	0	12	7	0	19
						Lombardy, Italy											
-1 [a]	1	0	0	1	0	0	1	0	0	0	0	0	0	0	0	2	2
1 [b]	170	44	2	216	1	0	1	0	0	0	0	0	0	5	3	2	10
-1	166	15	0	181	1	0	1	0	0	0	0	0	0	9	1	1	11
0	351	35	8	394	1	0	1	0	0	0	1	0	0	7	1	1	9
1	300	26	1	327	1	0	0	0	0	0	1	0	0	14	1	1	16
6	331	36	4	371	1	0	0	1	0	0	1	0	0	16	2	1	19
9	330	32	73	435	1	0	0	1	0	0	1	0	1	12	2	1	15
22	187	10	3	200	0	0	0	4	0	0	3	0	0	12	1	1	14
30	152	8	6	166	0	0	0	4	0	0	2	0	0	11	1	0	12
89	181	2	8	191	0	0	0	7	1	2	6	1	1	23	3	1	27
120	104	10	26	140	0	0	0	7	2	2	5	2	1	18	2	2	22
140	134	24	14	172	0	0	0	8	2	4	9	2	3	25	5	1	31
210	153	24	39	216	0	0	0	3	1	1	2	0	2	29	5	8	42
366	39	16	5	60	0	0	0	0	1	0	1	1	0	13	7	3	23
460	39	8	5	52	0	0	1	0	1	2	1	1	2	15	6	3	24
646	35	5	0	40	0	0	0	0	0	0	0	0	0	18	5	0	23
721	20	5	0	25	0	0	0	1	0	0	0	0	0	14	5	0	19
1080	5	3	0	8	0	0	0	1	0	0	0	0	0	3	8	0	11
1237	4	0	0	4	0	0	0	0	0	0	0	0	0	7	5	0	12

Notes:

[a] Prior to first application.

[b] Immediately after second application.

Field dissipation studies in the United States

Study 3-6: Four field trials were carried out in the United States to study the dissipation and mobility of residues of fluindapyr and its metabolites in soil, following two applications of a formulated product to bare soil. The studies were performed in California [Schreier, 2017, 2014EFT-IFP1203], New York [Schreier, 2018a, EFT-IFP1205], Georgia [Schreier, 2018b, EFT-IFP1206], and Nebraska [Schreier, 2018c, EFT-IFP1331].

Fluindapyr was sprayed twice, with a seven day interval, to bare soil at actual rates of 215–359 g ai/ha, with one outlier at 535 g ai/ha in the Nebraska trial. The reported soil characteristics (ranges in the different soil levels 0–15 to 76–91 cm) are summarised in Table 37. Soil cores were taken prior to and immediately after each spraying as well as 7, 14–15, 21, 30–31, 56–60, 90–96, 120–122, 180–235, 240–301, 449–453, 538–551, 657–666, and 714–720 days after application. All soil core samples were frozen

at -18 °C or lower on the day of collection. The maximum storage interval from sampling to extraction was 760–873 days, which was within the 24 month period of storage stability demonstrated in study [Skaggs, 2018, 2015EFT-IFP1940].

Table 37 Soil characteristics (0–15 cm) for European soils in field dissipation studies

Location	California, United States	New York, United States	Georgia, United States	Brunswick, Nebraska, United States
Reference	2014EFT-IFP1203	2014EFT-IFP1205	2014EFT-IFP1206	2014EFT-IFP1331
Soil texture (USDA) [a]	Sandy loam	Silt loam	Loamy sand	Loamy sand
-- Sand (%)	57	25	86	83
-- Silt (%)	23	60	5	10
-- Clay (%)	20	15	9	7
Organic Carbon (%) [b]	.6	2.5	0.55	0.90
Organic Matter (%)	n.r.	n.r.	n.r.	1.5
CEC (meq/100 g)	14	8.2	4.6	8.8
pH (H_2O)	6.2	5.1	5.9	6.0
Water Holding Capacity at pF 2.5 (FC at 0.33 bar) [percent]	20	29	7.9	10.4
Bulk density g/m^3	1.1	0.97	1.2	1.34

Notes:

[a] Classification according to United States Department of Agriculture (USDA).

For residue analysis, the treated soil cores were segmented and composited by depth (0–15 cm), 15–30 cm, 30–46 cm, 46–61 cm, 61–76 cm, 76–91 cm) per subplot yielding 6 samples per subplot at each time interval except for Day 0. Residues of fluindapyr, *cis*-1-COOH-fluindapyr, *trans*-1-COOH-fluindapyr, 3-OH-fluindapyr, and pyrazole carboxamide were extracted from soil using acetone:water followed by acetone:0.5N HCl. The acetone was removed and the resulting sample diluted with methanol. The resulting solution was diluted as necessary and the residues were quantified by LC-MS/MS. The method had a limit of quantitation of 0.005 mg/kg for all analytes. The average concurrent method recoveries and relative standard deviation (n = 34/study report) for fluindapyr and its degradates in soil were within 70–110 percent with a mean RSD of 6.3–17, for fluindapyr, *cis*-1-COOH-fluindapyr, *trans*-1-COOH-fluindapyr, 3-OH-fluindapyr, and pyrazole carboxamide. All soil residues were corrected for percent moisture and presented on a dry weight basis. Residues in control samples were generally below 0.3LOQ. The results were not corrected for recoveries.

Residues of fluindapyr, *cis*-1-COOH-fluindapyr, *trans*-1-COOH-fluindapyr, 3-OH-fluindapyr, and pyrazole carboxamide were found mainly in the 0–15 cm soil layer. Trace residues of the parent and various degradates were occasionally found in the 15–31 cm layer. Residues below the 0–15 cm layer generally remained below the LOQ of 0.005 mg/kg, indicating minimal mobility in the soil. The results for the 0–15 cm soil layer are shown in Table 38.

Table 38 Distribution of the average total fluindapyr residues (mg/kg soil)

Depth (cm) Layer	Days after last application													
	0	7	14-15	21	30-31	56-60	90-96	120-122	180-235	240-301	449-455	538-551	657-666	714-720
California, United States, 2014EFT-IFP1203														
0-15	0.32	0.18	0.25	0.15	0.25	0.16	0.20	0.026	0.077	0.042	0.053	0.009	0.039	0.047
15-31	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
31-46	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.006	<LOQ	<LOQ
New York, United States, 2014EFT-IFP1205														

Depth (cm) Layer	Days after last application													
	0	7	14-15	21	30-31	56-60	90-96	120-122	180-235	240-301	449-455	538-551	657-666	714-720
0-15	0.35	0.35	0.29	0.20	0.16	0.098	0.15	0.12	0.16	0.17	0.10	0.050	0.064	0.052
15-31	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.005	<LOQ	0.008	<LOQ	0.019	0.008	0.013	0.010
31-46	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
46-61	n.a.	n.a.	n.a.	n.a.	n.a.	0.010	<LOQ	n.a.	n.a.	n.a.	n.a.	<LOQ	<LOQ	<LOQ
Georgia, United States, 2014EFT-IFP1206														
0-15	0.30	0.15	0.065	0.12	0.13	0.072	0.11	0.098	0.083	0.032	0.033	0.019	0.023	0.014
15-31	<LOQ	0.013	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
31-46	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Nebraska, United States, 2014EFT-IFP1331														
0-15	0.17	0.12	0.10	0.054	0.12	0.069	0.079	0.054	0.060	<LOQ	0.006	<LOQ	<LOQ	0.005
15-31	0.040	0.035	0.032	0.006	0.011	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
31-46	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

Notes:

n.a. = Not analysed.

The average total fluindapyr (fluindapyr plus *cis*-1-COOH-fluindapyr, *trans*-1-COOH-fluindapyr, 3-OH-fluindapyr, and pyrazole carboxamide) residue at day 0 (0 days after the last application) in the 0–15 cm depth was 0.167–0.348 mg/kg following the second application. This value dropped to 120–183 ppb on day 7, except in the trial from New York where it increased to 0.353. The total residue dropped to 60–124 ppb by day 180-182 and 5-52 ppb by day 714-720.

At day 0, in the 0-15 cm layer, residues consisted almost entirely of parent fluindapyr. The degradates *cis*-1-COOH- fluindapyr, *trans*-1-COOH- fluindapyr, 3-OH-fluindapyr and pyrazole carboxamide contributed only little (1-5 percent) to the total fliunapyr equivalent residues in the trails from Georgia and California. In the New York trial degradate pyrazole carboxamide did not contribute to the total fluindapyr equivalent residues in this layer at day 0. In the Nebraska trial, the degradates *cis*-1-COOH-F9990, *trans*-1-COOH-F9990, and pyrazole carboxamide did not contribute to the total fluindapyr equivalent residues at day 0, but 3-OH-F9990 was 0.013 mg eq/kg. In the New York trial, 3-OH-fluindapyr was 0.008 mg eq/kg, on day 0 and *cis*-1-COOH-fluindapyr and *trans*-1-COOH-fluindapyr levels were 0.002 mg eq/kg and 0.004 mg eq/kg, respectively. On day 0, 3-OH-fluindapyr was 0.008 and 0.013 mg/kg in, except for the trial in California, where it did not contribute to the residue. On subsequent sampling days the cis-1-COOH-fluindapyr, trans-1-COOH- fluindapyr, 3-OH-fluindapyr, and pyrazole carboxamide residues remained below 10 percent of the total residue until day 14–59 in all trials except the trial in Georgia, where it remained below 15 percent of the total residue until day 180. The relative parent percentage of the total residue continued to drop until reaching approximately 45–67 percent of the total residue by day 452–540.

Residues of the soil degradates *cis*-1-COOH-fluindapyr and *trans*-1-COOH-fluindapyr were low with the individual diastereomers remaining at or below 0.002-0.018 mg eq/kg, or even below LOQ (Georgia trial) throughout the study.

Residues of 3-OH-fluindapyr were detected in the 0-15 com horizon starting after the first application in the Nebraska trial to one week after the first application. In the Californian trial. Residues remained low at 0.015–0.024 mg/kg or less throughout the study.

Residues of pyrazole carboxamide were either not detected or occasionally detected throughout the study, reaching a maximum average of 0.011 mg eq/kg on day 31. The results are also presented in Table 39.

Table 39 Distribution of the average residues (mg/kg soil fluindapyr equivalents) in soil depth of 0 – 15 cm inUnited Statesterrestrial field dissipation studies

	Average Residue (mg/kg soil eq) in soil depth (0 - 15 cm)						
Days after last application	0	7	14-15	21	30-31	56-60	90-96
California, United States, 2014EFT-IFP1203							
Fluindapyr	0.316	0.170	0.230	0.141	0.227	0.144	0.165
cis-1-COOH-fluindapyr	ND	ND	ND	ND	ND	0.002	0.004
trans-1-COOH-fluindapyr	ND	ND	ND	ND	ND	ND	<LOQ
3-OH-fluindapyr	<LOQ	0.007	0.012	0.006	0.013	0.013	0.018
Pyrazole carboxamide	ND	0.006	0.007	0.006	0.011	<LOQ	0.005
Total fluindapyr residues	0.320	0.183	0.250	0.153	0.251	0.163	0.196
New York, United States, 2014EFT-IFP1205							
Fluindapyr	0.333	0.327	0.267	0.177	0.124	0.076	0.111
cis-1-COOH-fluindapyr	0.002	0.003	0.007	<LOQ	0.007	0.005	0.009
trans-1-COOH-fluindapyr	0.004	0.005	0.010	0.009	0.015	0.009	0.018
3-OH-fluindapyr	0.008	0.018	0.010	0.008	0.009	0.008	0.013
Pyrazole carboxamide	ND	ND	ND	ND	ND	ND	ND
Total fluindapyr residues	0.348	0.353	0.294	0.199	0.157	0.099	0.152
Georgia, United States, 2014EFT-IFP1206							
Fluindapyr	0.292	0.14	0.059	0.118	0.128	0.064	0.093
cis-1-COOH-fluindapyr	ND	ND	ND	<LOQ	<LOQ	<LOQ	<LOQ
trans-1-COOH-fluindapyr	ND	ND	ND	ND	ND	ND	ND
3-OH-fluindapyr	0.009	0.007	<LOQ	<LOQ	0.005	0.008	0.014
Pyrazole carboxamide	<LOQ	<LOQ	<LOQ	ND	ND	ND	ND
Total fluindapyr residues	0.304	0.154	0.065	0.122	0.134	0.072	0.108
Nebraska, United States, 2014EFT-IFP1331							
Fluindapyr	0.153	0.11	0.096	0.051	0.11	0.06	0.068
cis-1-COOH-fluindapyr	ND	<LOQ	ND	ND	ND	<LOQ	<LOQ
trans-1-COOH-fluindapyr	ND	ND	<LOQ	ND	<LOQ	<LOQ	<LOQ
3-OH-fluindapyr	0.013	0.01	0.009	<LOQ	0.01	<LOQ	ND
Pyrazole carboxamide	ND	ND	ND	ND	ND	ND	ND
Total fluindapyr residues	0.167	0.12	0.105	0.054	0.121	0.069	0.079
Days after last application	120-122	180-235	240-301	449-455	538-551	657-666	714-720
California, United States, 2014EFT-IFP1203							
Fluindapyr	0.022	0.059	0.030	0.030	<LOQ	0.021	0.023
cis-1-COOH-fluindapyr	0.002	0.003	0.003	0.004	0.003	0.002	0.003
trans-1-COOH-fluindapyr	<LOQ	<LOQ	<LOQ	0.003	0.003	<LOQ	<LOQ
3-OH-fluindapyr	ND	0.011	0.006	0.016	<LOQ	0.014	0.020
Pyrazole carboxamide	ND	<LOQ	ND	ND	ND	ND	ND
Total fluindapyr residues	0.026	0.077	0.042	0.053	0.009	0.039	0.047
New York, United States, 2014EFT-IFP1205							
Fluindapyr	0.093	0.129	0.136	0.067	0.029	0.039	0.028
cis-1-COOH-fluindapyr	0.009	0.009	0.008	0.005	0.005	0.004	0.004
trans-1-COOH-fluindapyr	0.011	0.012	0.009	0.007	0.006	0.005	0.005
3-OH-fluindapyr	0.012	0.010	0.017	0.024	0.010	0.016	0.015

	Average Residue (mg/kg soil eq) in soil depth (0 - 15 cm)						
Days after last application	0	7	14-15	21	30-31	56-60	90-96
Pyrazole carboxamide	ND	ND	ND	ND	ND	ND	ND
Total fluindapyr residues	0.125	0.162	0.171	0.104	0.05	0.065	0.052
Georgia, United States, 2014EFT-IFP1206							
Fluindapyr	0.092	0.067	0.032	0.017	0.01	0.012	0.007
cis-1-COOH-fluindapyr	ND	ND	ND	ND	ND	ND	ND
trans-1-COOH-fluindapyr	ND	ND	ND	ND	ND	ND	ND
3-OH-fluindapyr	0.007	0.016	ND	0.015	0.008	0.01	0.007
Pyrazole carboxamide	ND	ND	ND	ND	ND	ND	ND
Total fluindapyr residues	0.098	0.083	0.032	0.033	0.019	0.023	0.014
Nebraska, United States, 2014EFT-IFP1331							
Fluindapyr	0.049	0.047	<LOQ	<LOQ	<LOQ	ND	0.005
cis-1-COOH-fluindapyr	ND	ND	ND	ND	ND	ND	ND
trans-1-COOH-fluindapyr	<LOQ	<LOQ	ND	ND	ND	ND	ND
3-OH-fluindapyr	ND	<LOQ	ND	ND	ND	ND	ND
Pyrazole carboxamide	ND	ND	ND	ND	ND	ND	ND
Total fluindapyr residues	0.054	0.06	<LOQ	0.006	<LOQ	ND	0.005

Notes:

ND = Not detected; Note that all residues are expressed in fluindapyr equivalents: trans-1-COOH-fluindapyr ppb fluindapyr equivalents = (ppb trans-1-COOH-fluindapyr, dry weight) × (351.4/381.4), where 351.4 and 381.4 are the molecular weights of fluindapyr and trans-1-COOH-fluindapyr, respectively; cis-1-COOH-fluindapyr ppb fluindapyr equivalents = (ppb cis-1-COOH-fluindapyr, dry weight) × (351.4/381.4), where 351.4 and 381.4 are the molecular weights of fluindapyr and cis-1-COOH-fluindapyr, respectively; 3-OH-fluindapyr ppb fluindapyr equivalents = (ppb 3-OH-fluindapyr, dry weight) × (351.4/367.4), where 351.4 and 367.4 are the molecular weights of fluindapyr and 3-OH-fluindapyr, respectively; Pyrazole Carboxamide ppb fluindapyr equivalents = (ppb Pyrazole Carboxamide, dry weight) × (351.4/176.0), where 351.4 and 176.0 are the molecular weights of fluindapyr and Pyrazole Carboxamide, respectively; Total ppb fluindapyr equivalents = (ppb fluindapyr) + (trans-1-COOH-fluindapyr ppb fluindapyr equivalents) + (cis-1-COOH-fluindapyr ppb fluindapyr equivalent) + (3-OH-fluindapyr ppb fluindapyr equivalents) + (Pyrazole Carboxamide).

Kinetic endpoints for aerobic degradation in soil under field conditions

The dissipation and mobility of residues of fluindapyr and its metabolites under field conditions was determined in four European soils [Gemroth, 2018, 2015-IFP1999 and 2015-IFP1999 Amendment no.1, and the one year extension in Gemroth, 2020a, 2018-IFP4409]. The method and results are summarized above. The kinetic evaluation of these studies was described in Gemroth, 2020b [2018-EFT-IFP1999]. The rate of degradation of fluindapyr was assessed using the FOCUS DEGKIN v2 (June 2007) Excel tool and KinGUII (v2.1) following FOCUS Kinetics guidance (2006).

The data were first fitted according to a single first order kinetic model (SFO) and secondly, if necessary, according to a bi-phasic kinetic models, First-Order Multi-Compartment kinetic model (FOMC), Double-First-Order in Parallel (DFOP) or Hockey Stick (HS) kinetics models. The goodness of fit was assessed by visual inspection and an error criterion based on a chi-squared test. The findings are summarised in Table 40. The best fits for the European trials were obtained with the bi-phasic kinetic models.

The dissipation and mobility of residues of fluindapyr and its metabolites under field conditions was also determined in 4 United States soils in four different studies [Schreier, 2017, 2014EFT-IFP1203;

Schreier, 2018a, EFT-IFP1205; Schreier, 2018b, EFT-IFP1206; Schreier, 2018c, EFT-IFP131]. The residue values for the 0-15 cm depth at each sampling interval were used to calculate the total fluindapyr residue half-life (using first order rate kinetics), DT_{50}, DT_{75} and DT_{90}. For these calculations residues of *cis*-1-COOH-fluindapyr, *trans*-1-COOH-fluindapyr, 3-OH-fluindapyr, and pyrazole carboxamide were corrected for molecular weight to parent fluindapyr equivalents. All calculations were performed on a dry weight basis. The results of the kinetic evaluation are included in Table 40.

Table 40 Field DT_{50} and DT_{90} values of total fluindapyr residues from all the European and United States sites

Study location	Sand (%)	Silt (%)	Clay (%)	percent OC	pH (H_2O)	Soil [a]	Kinetic model	DT_{50} (days) [b]	DT_{90} (days) [b]	Chi^2	References
Lower-Saxony, GE	82	9	9	1.3	5.2	Loamy sand	DFOP	168	>1000	8	Gemrot, 2018a,b and 2020a,b
Warwickshire, United Kingdom	59	24	17	1.18	5.9	Sandy loam	DFOP	81	836	10	
Aquitaine, France	54	31	15	0.64	6.0	Sandy loam	HS	129	222	20	
Lombardy, Italy	34	45	21	1.49	6.3	loam	DFOP	55	687	24	
California, United States	57	23	20	1.51	6.2	Sandy loam	SFO	122	DT75 180	n.r.	Schreier, 2017
New York, United States	25	60	15	2.5	5.1	Silt loam	SFO	30	DT75 540	n.r.	Schreier, 2018a
Georgia, United States	86	5	9	0.55	5.9	Loamy sand	SFO	120	DT75 240	n.r.	Schreier, 2018b
Nebraska, United States	83	10	7	0.90	6.0	Loamy sand	SFO	120	DT75 182	n.r.	Schreier, 2018c

Notes:

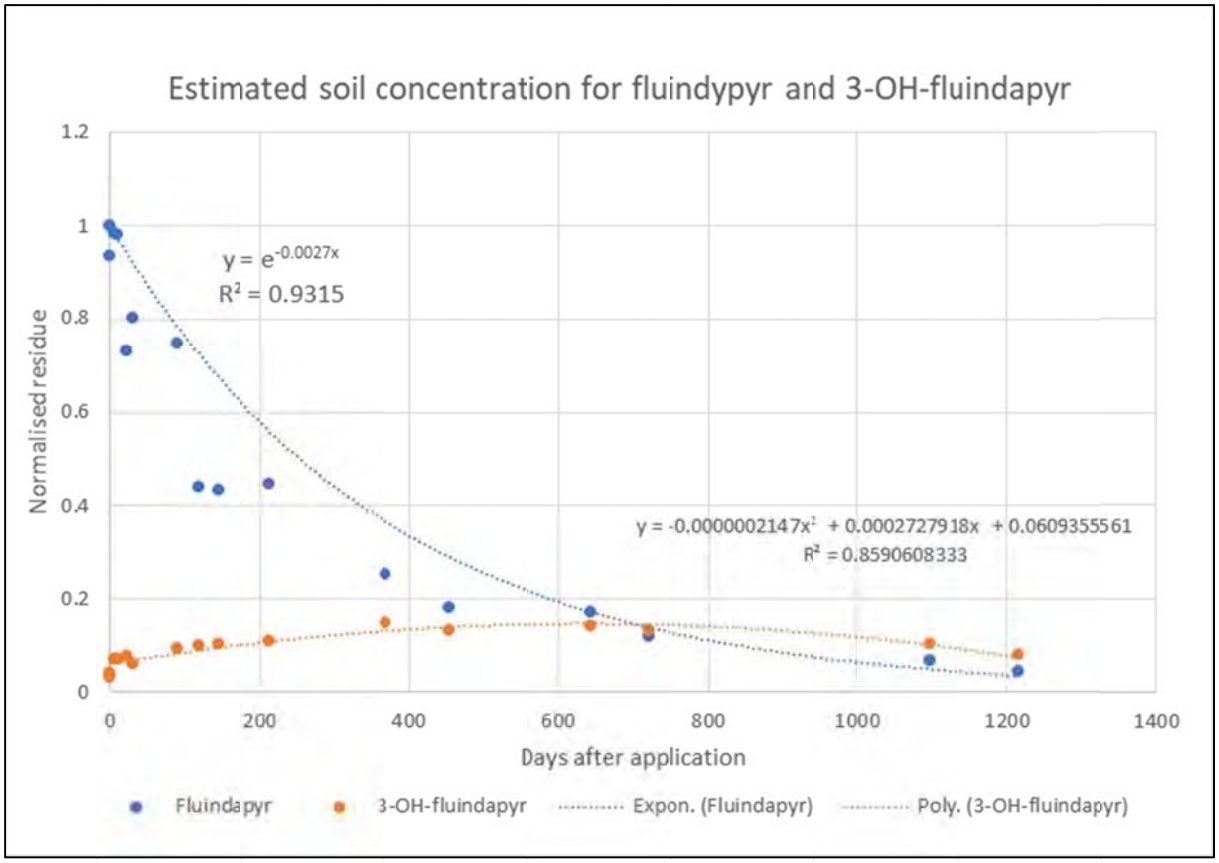
[a] Soil properties are for soils at depth of 0-10 and 0-15 cm for European and United States sites, respectively.

[b] Non-normalized field DT_{50}.

Note by the reviewer: The actual method description and calculation of the DT_{50}, DT_{75} and DT_{90} from the United States trials are not included in the reports (Schreier, 2017-2018c). The lack of this information does not hamper the assessment in the context of the JMPR.

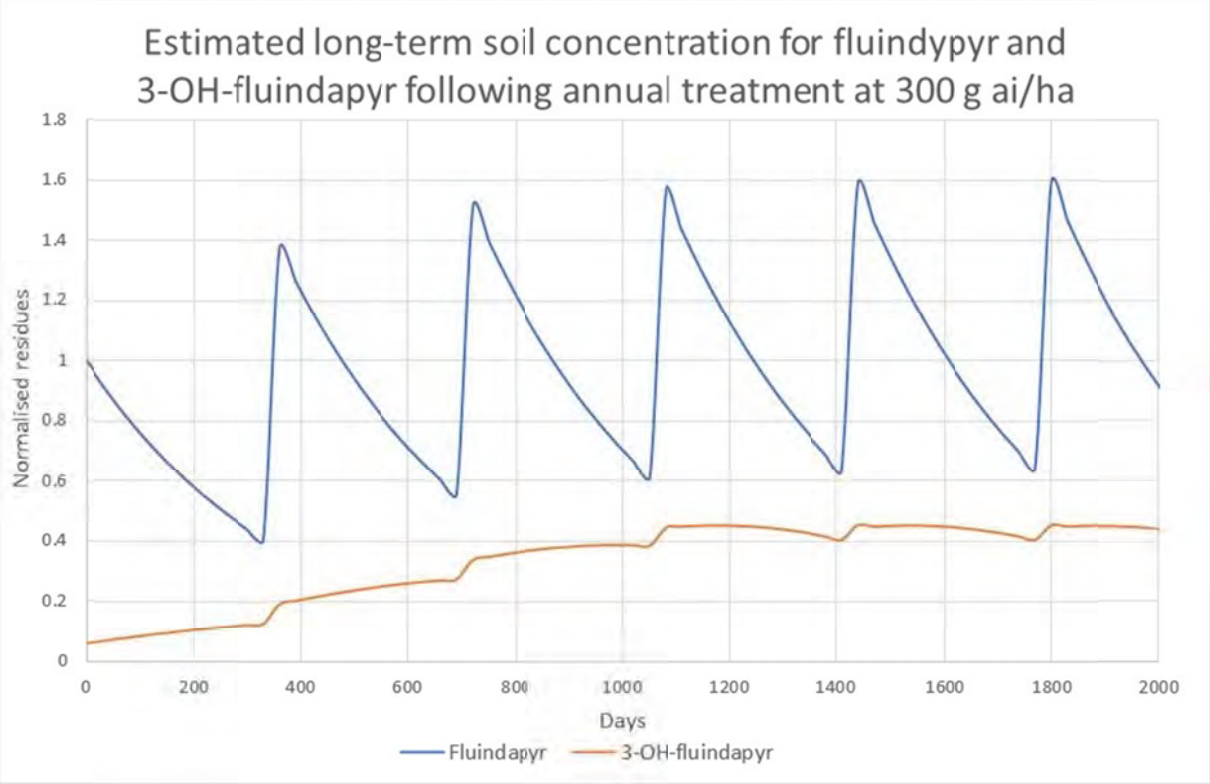
Estimated soil concentrations of fluindapyr and 3-OH-fluindapyr following single or subsequent annual application

In order to estimate potential soil residue plateau levels for fluindapyr and 3-OH-fluindapyr, the Meeting considered the field accumulation study results by Gemrot, 2018a,b and 2020a,b, since the interval investigated was sufficiently long. Residues for both analytes were initially expressed as kg ai/ha (0–25 cm soil layer) equivalents and have been normalised based on residues for fluindapyr at day zero of each plot. Regression considered the mean normalized concentration from each of the four plots in the study.



Based on the single application, a plateau of 15 percent of the dose rate for 3-OH-fluindapyr per ha was estimated, reached after approximately 700 days. Expressed in mg/kg soil, this plateau represents a concentration of 0.012 mg/kg soil (300 g/ha max. annual application rate, 25 cm soil layer, 1.5 g/cm³ soil density).

Due to the persistence of fluindapyr residues in soil, long term accumulation was taken into account by estimating plateau levels following annual application at the maximum seasonal rate:



Based on multiple annual applications, a plateau of 45 percent of the single maximum dose rate for fluindapyr per ha and year was estimated, reached approximately after 4 years. Expressed in mg/kg soil, this plateau represents a concentration of 0.036 mg/kg soil (300 g/ha max. annual application rate, 25cm soil layer, 1.5 g/cm³ soil density).

Environmental fate in water/sediment systems

The Meeting received information on the environmental fate in water/sediment systems. Studies on the environmental fate in water/sediments are not required for the envisaged uses, except for hydrolysis and photolysis in water. These studies were summarised in the physico-chemical properties section.

If the uses are to be extended to rice, the studies on the environmental fate in water/sediment systems will have to be evaluated (see references submitted but not used).

Rotational crops

Confined rotational crop studies

Study 1 and 2

Two confined rotational crop studies were conducted in Italy to provide information on the uptake and metabolism of fluindapyr in rotational crops [Mainolfi, 2017, 2013MET-IFP0693 and Vanini, 2017a, 2013MET-IFP0717]. Pots with bare sandy loam soil were treated dropwise with either phenyl-labelled fluindapyr or pyrazole-labelled fluindapyr at a dose rate of 360–387 g ai/ha. The treated soil was aged outdoors, for three time intervals prior to the sowing of each succeeding crop: 30, 120 and 300 days. Three different representative crops were used: lettuce (variety: Canasta), carrot (variety: Nantese F1) and wheat (variety: Bologna). From each plant-back interval (PBI), crop samples were harvested at maturity: lettuce, carrots (roots and leaves) and wheat (grain and straw). Samples of immature lettuce and wheat

forage and hay were also collected from each PBI. Carrot (roots and tops) and lettuce samples were gently rinsed with water in order to remove any soil adhering to the leaves. Hay samples were air dried to a moisture content of 10–20 percent. All plant materials were immediately frozen and stored at -20 °C prior to processing and analysis. Duplicate soil cores were removed from one of the treated pots at time of treatment and again at each crop harvest. The soil cores were immediately analysed or stored frozen at -20 °C. Sample preparation, extraction and analysis were carried out within 6 months of crop harvest, so further storage stability data were not required.

The crop samples were finely ground with dry ice. The ground crop material was analysed for total radioactivity by LSC after combustion. Extraction was carried out on the samples with TRR higher than 0.01 mg/kg. Each aliquot was homogenized with acetone-water (70:30 v/v), shaken and centrifuged [Extract I]. Shaking and centrifugation was repeated three times on each sample: twice with acetone-water (50:50 v/v) [Extract II and Extract III] and then once with acetone [Extract IV]. All extracts were analysed by LSC and stored at <7 °C. Suitable aliquots of extracts I to IV were pooled, concentrated and analysed by LSC and TLC. Suitable aliquots of the extracts were pooled, concentrated to remove the organic solvent, and the aqueous portion was extracted three times with n-heptane (1:2 v/v).

Reversed-phase HPLC was used to isolate the parent compound from aliquots of concentrated heptane phase for analysis by chiral HPLC. Further aliquots of the heptane phase were analysed by TLC, HPLC, and LC-MS for metabolite identification. For each RAC, a portion of the concentrated aqueous phase, after heptane extraction, was incubated with 6 N HCl for 1 hour at 80 °C. After hydrolysis, the samples were neutralized and analysed by HPLC and by LC-MS for metabolite profiling and identification. Reference compounds were 3-OH-fluindapyr, *cis*-1-COOH-fluindapyr, *trans*-1-COOH-fluindapyr, 1-OH-Met-fluindapyr (2 diastereomers), N-DesMet-fluindapyr, pyrazole carboxamide, pyrazole carboxylic acid and N-DesMet-pyrazole carboxylic acid.

Aliquots of selected samples of concentrated total extract and concentrated aqueous phase were subjected to enzymatic hydrolysis with β-glucosidase. The aliquots were dried and suspended in sodium acetate 0.1 M (pH 4.8) containing the enzyme and incubated at 37 °C for 48 hours under stirring. After incubation, samples were analysed by TLC.

After acetone extraction, the remaining solid residues from the RAC samples were air-dried at room temperature, and the radioactive content determined by LSC. The PES samples containing significant residues (≥0.05 mg/kg or 10 percent of TRR; i.e. grain and straw) were subjected to sequential base, some to surfactant, and enzymatic (β-amylase and cellulase) treatments to release and characterize the un-extractable radioactivity. The solid residues were suspended in 0.5 N NaOH - CH_3OH (1:1 v/v) and incubated for 2 hours at ambient temperature, under stirring (Extract Base). Subsequently, from some samples the solid residues were suspended in 0.1 percent Tween20 for 2 hours under stirring (Extract Surfactant). Grain residue was suspended in 50 mM ammonium acetate buffer, pH 5, and enzyme β-amylase was added. The sample was incubated at 37 °C for 24 hours under stirring (Extract Amylase). Subsequently, the solid residue was re-suspended in the buffer and incubated with cellulase at 37 °C for 24 hours, under stirring (Extract Cellulase). Straw residue was subjected to hydrolysis with cellulase, following the same procedure described above for grain residue (Extract Cellulase).

The soil samples were extracted by shaking with acetone. The soil residues were dried, then oxidized before determination of the radioactivity by LSC.

The TRR, distribution, and identification of radioactivity are shown in Tables 41 to Table 56. The TRR in carrot and lettuce samples showed a decline from the 30d PBI towards the 300d PBI for the phenyl-label, while no decline in TRR could be observed in the carrot and lettuce samples from the 30d PBI towards the 300d PBI for the pyrazole-label. Furthermore, for the phenyl-label, the TRR in wheat

forage and hay remained rather constant throughout the study, while a decline in TRR could be observed from the 120-day PBI to the 300-day PBI in wheat grain and straw. In the wheat RACs from the pyrazole-label, the TRR remained relatively constant throughout the study.

Extractable radioactivity from all different crop matrices was high and generally ranged from 83 to 99 percent TRR. Only the radioactivity in the PES of wheat straw and grain needed further investigation and was present mainly as cellulose ^{14}C-incorporated natural products and represented 6–10 percent TRR in straw, and 3–5 percent TRR in grain.

The residual water soluble fraction, remaining after the heptane extraction, was subjected to enzymatic hydrolysis using glucosidase, which resulted in partial hydrolysis of several glucoside conjugates. However, the chemical hydrolysis (6 N HCl) of the aqueous metabolite fraction (after heptane extraction) showed complete hydrolysis of the conjugated metabolites and further simplification of the chromatographic metabolite profile, thus allowing adequate quantification of individual aglycones.

The identified major residues (fluindapyr, 3-OH-fluindapyr, 1-COOH-fluindapyr, 1-OH-Met-fluindapyr, N-DesMet-fluindapyr, N-DesMet-pyrazole carboxylic acid, pyrazole carboxylic acid, pyrazole carboxamide) were common in all crops but their magnitude varied depending on the individual crop and matrix. Parent and 3-OH-fluindapyr were identified in the total acetone extracts and heptane extracts. On the other hand, pyrazole carboxamide, pyrazole carboxylic acid, N-DesMet-pyrazole carboxylic acid, 1-COOH-fluindapyr, 1-OH-Met-fluindapyr, and N-DesMet-fluindapyr were all identified as aglycones in the hydrolysed aqueous extracts. No quantitative results are available for the aqueous extracts before hydrolysis, therefore, it is not known how much of the respective metabolites were already present in their free form before the hydrolysis. A minor residual fraction in the TLC radio-profiles of almost all pooled acetone extracts of RACs was characterized as organo-soluble as it was completely extracted in the heptane phase after partitioning. Minor polar residues detected in the HPLC radio-profiles of the hydrolysed aqueous phases were characterized as water soluble as they remained in aqueous phase after partitioning with heptane. They could be present as sugar conjugates in the initial unhydrolysed extract.

Parent was the main component found in carrot roots ranging from 65 to 70 percent TRR (0.013–0.026 mg eq/kg) at the three different plant back intervals for the phenyl-label, while for the pyrazole-label parent was present at similar quantitative levels, but in a less pronounced percentage of the TRR (10–38 percent TRR, 0.011–0.031 mg eq/kg). Parent represented also less of the radioactive residue in immature and mature lettuce (2.8–20 percent TRR, 0.006–0.015 mg eq/kg) and wheat grain (6.1–15 percent TRR, 0.084–0.39 mg eq/kg).

In carrot root, the pyazole-label specific metabolite N-DesMet-pyrazole carboxylic acid (including its conjugates) was a major contributor to the TRR (28–65 percent TRR, 0.022–0.068 mg eq/kg), while 3-OH-fluindapyr, free and conjugated 1-COOH-fluindapyr, and free and conjugated 1-OH-Met-fluindapyr were present in quantities <0.01 mg eq/kg. The metabolite pyrazole carboxamide (including its conjugates) increased with longer PBIs in carrot root (from 6.6 percent TRR, 0.005 mg eq/kg at 30d PBI to 18 percent TRR, 0.018 mg eq/kg at 300d PBI).

For the phenyl-label, in mature and immature lettuce the free and conjugated metabolites 1-COOH-fluindapyr and 1-OH-Met-fluindapyr represented the majority of the radioactive residue, each ranging from 16 to 34 percent TRR (0.011–0.027 mg eq/kg), whereas these metabolites were clearly less present in the pyrazole-labeled studies. In the latter case, pyrazole-label specific free and conjugated N-DesMet-pyrazole carboxylic acid was present at high levels in lettuce (62–82 percent TRR, 0.12–0.21 mg eq/kg). Other detected metabolites in lettuce were 3-OH-fluindapyr (< 10 percent TRR, < 0.01 mg eq/kg), pyrazole carboxylic acid (≤ 12 percent TRR, ≤ 0.039 mg eq/kg), pyrazole carboxamide (≤ 13 percent TRR, ≤ 0.032 mg eq/kg), and N-DesMet-fluindapyr (< 5 percent TRR, < 0.01 mg eq/kg).

In wheat grain, 1-OH-Met-fluindapyr and its conjugates was the major contributor to the radioactive residue (12–52 percent TRR, 0.34–1.1 mg eq/kg), as well as free and conjugated pyrazole carboxylic acid (12–29 percent TRR, 0.34–0.83 mg eq/kg). 3-OH-fluindapyr was present at approximately 10 percent TRR, and free and conjugated 1-COOH-fluindapyr was detected at somewhat lower levels (2.7–7.0 percent TRR, 0.076–0.13 mg eq/kg). Also the label-specific metabolites N-DesMet-pyrazole carboxylic acid and pyrazole carboxamide were retrieved in wheat grain (1.7–16 percent TRR, 0.050–0.45 mg eq/kg; both increasing with longer PBIs; free and conjugated). N-DesMet-fluindapyr was observed at <5 percent TRR.

In feed commodities, the levels of parent varied from 0.24–11 percent TRR (0.004–0.013 mg eq/kg) in carrot tops to 13–32 percent TRR (0.050–0.14 mg eq/kg) in wheat forage. In feed crops, the (conjugated) metabolite 1-OH-Met-fluindapyr represented a major part of the radioactive residue in the phenyl-label, ranging from 11–36 percent TRR (0.061–0.13 mg eq/kg) in wheat forage to 17–46 percent TRR (0.49–1.6 mg eq/kg) in wheat straw. In the pyrazole-label, in particular free and conjugated pyrazole carboxylic acid was present in large quantities in cereal feed items (forage: 13–24 percent TRR, 0.050–0.12 mg eq/kg; straw: 9.1–28 percent TRR, 0.34–0.89 mg eq/kg), while in carrot tops free and conjugated N-DesMet-pyrazole carboxylic acid was more pronounced (52–58 percent TRR, 0.64–0.99 mg eq/kg). 3-OH-fluindapyr, pyrazole carboxamide (and its conjugates), N-DesMet-fluindapyr (and its conjugates), and 1-COOH-fluindapyr (and its conjugates) also contributed to the total radioactive residues, with levels depending on crop matrix and on PBI.

For the phenyl label, radioactivity found in soil after treatment ranged between 108-110 percent AR (Applied Radioactivity) and between 73 and 82 percent AR at crop harvest for the 30d PBI. Radioactivity recovered from soil at harvest from the 120d and 300d PBI gradually decreased, ranging between 61 and 71 percent AR and between 51 and 64 percent AR, respectively. For the pyrazole-label, radioactivity found in soil after treatment ranged between 81-86 percent AR (Applied Radioactivity), while radioactivity recovered from soil at crop harvest for the 30d PBI ranged between 69 and 91 percent AR. Radioactivity recovered from soil at harvest from the 120d and 300d PBI ranged between 67 and 90 percent AR and between 72 and 83 percent AR, respectively.

The chiral analysis results are presented in Table 57. A shift in the enantiomeric ratio *R/S* of 50/50 in the formulation to up to 23:77 after application can be observed in the rotational crop samples.

Table 41 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated carrot root

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.037	100	0.081	100	0.029	100	0.079
Extractable	99	0.037	99	0.080	98	0.029	100	0.079
Fluindapyr [a]	70	0.026	38	0.031	65	0.019	23	0.018
3-OH-fluindapyr [a]	9.8	0.004	6.7	0.005	15	0.005	5.6	0.004
N-DesMet-pyrazole carboxylic acid [b]	-	-	28	0.022	-	-	41	0.032
Pyrazole carboxamide [b]	-	-	6.6	0.005	-	-	14	0.011
1-COOH-fluindapyr [b]	9.5	0.004	5.3	0.004	9.6	0.003	6.0	0.005
trans-1-COOH-fluindapyr [b]	*4.6*	*0.002*	5.3	0.004	*5.1*	*0.001*	5.8	0.005
cis-1-COOH-fluindapyr [b]	*4.9*	*0.002*	-	-	*4.5*	*0.001*	*5.8*	*0.005*
1-OH-Met-fluindapyr [b,c]	9.4	0.004	8.8	0.007	8.2	0.002	-	-
	6.7	*0.003*	*4.8*	*0.004*	*8.2*	*0.002*	-	-
	2.7	*0.001*	*4.0*	*0.003*	-	-	-	-
Total identified	99	0.037	93	0.074	98	0.029	95	0.075

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Unknown (water soluble) 1-2 compounds [d,e]	-	-	5.8	0.005			5.4	0.004
PES	3.6	0.001	2.0	0.002	4.1	0.001	2.4	0.002

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] Each compound never exceeded 0.004 mg/kg or 5.4 percent TRR.

[e] Detected in hydrolysed aqueous extracts.

Table 42 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated carrot root

	300d PBI		300d PBI	
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.019	100	0.11
Extractable	95	0.018	102	0.11
Fluindapyr [a]	70	0.013	10	0.011
3-OH-fluindapyr [a]	25	0.005	2.9	0.003
N-DesMet-pyrazole carboxylic acid [b]	-	-	65	0.068
Pyrazole carboxamide [b]	-	-	18	0.018
1-COOH-fluindapyr [b]	-	-	7.2	0.008
trans-1-COOH-fluindapyr [b]	-	-	*7.2*	*0.008*
cis-1-COOH-fluindapyr [b]	-	-	-	-
Total identified	95	0.018	102	0.11
PES	5.8	0.001	2.7	0.003

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

Table 43 Total Radioactive Residues (TRRs), Identification and distribution of radioactivity in rotated carrot tops

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.18	100	1.1	100	0.16	100	1.4
Extractable	92	0.17	95	1.1	91	0.14	96	1.4
Fluindapyr [a]	7.3	0.013	0.85	0.010	6.5	0.010	0.92	0.013
3-OH-fluindapyr [a]	6.5	0.012	0.73	0.008	6.8	0.011	1.0	0.014
N-DesMet-pyrazole carboxylic acid [b]	-	-	56	0.64	-	-	52	0.74
Pyrazole carboxylic acid [b]	-	-	2.0	0.023	-	-	8.9	0.13
Pyrazole carboxamide [b]	-	-	10	0.12	-	-	6.3	0.090
1-COOH-fluindapyr [b]	22	0.039	8.1	0.092	31	0.049	4.5	0.064
trans-1-COOH-fluindapyr[b]	*13*	*0.023*	*4.6*	*0.052*	*17*	*0.026*	*2.6*	*0.037*
cis-1-COOH-fluindapyr[b]	*9.1*	*0.016*	*3.5*	*0.040*	*14*	*0.023*	*1.9*	*0.027*

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
1-OH-Met-fluindapyr [b,c]	37	0.067	11	0.13	27	0.042	9.2	0.13
	35	*0.062*	*10*	*0.12*	*22*	*0.035*	*7.6*	*0.11*
	2.9	*0.005*	*1.3*	*0.015*	*4.2*	*0.007*	*1.6*	*0.023*
N-DesMet-fluindapyr [b]	3.4	0.006	-	-	4.8	0.008	4.3	0.062
Total identified	76	0.14	90	1.0	76	0.12	87	1.2
Unknown (organo soluble) 1 compound [d,e]	2.0	0.004	-	-	2.3	0.004	-	-
Unknown (water soluble) 2-9 compounds [d,f]	14	0.025	5.0	0.056	13	0.020	9.1	0.13
Total characterized	16	0.028	5.0	0.056	15	0.023	9.1	0.13
PES	5.6	0.010	3.6	0.041	4.7	0.007	2.9	0.041

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.028 mg/kg or 7.2 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 44 Total Radioactive Residues (TRRs), Identification and distribution of radioactivity in rotated carrot tops

	300d PBI			
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.075	100	1.7
Extractable	98	0.073	99	1.7
Fluindapyr [a]	11	0.009	0.24	0.004
3-OH-fluindapyr [a]	6.4	0.005	0.52	0.009
N-DesMet-pyrazole carboxylic acid [b]	-	-	58	0.99
Pyrazole carboxylic acid [b]	-	-	14	0.24
Pyrazole carboxamide [b]	-	-	10	0.17
1-COOH-fluindapyr [b]	29	0.021	4.4	0.074
trans-1-COOH-fluindapyr[b]	*21*	*0.016*	*2.0*	*0.034*
cis-1-COOH-fluindapyr[b]	*7.5*	*0.006*	*2.4*	*0.040*
1-OH-Met-fluindapyr [b,c]	27	0.020	3.5	0.058
	23	*0.017*	*2.5*	*0.042*
	4.4	*0.003*	*1.0*	*0.017*
N-DesMet-fluindapyr [b]	6.3	0.005	1.3	0.022
Total identified	80	0.060	93	1.6
Unknown (organo soluble) 1 compound [d,e]	3.1	0.002	0.84	0.014
Unknown (water soluble) 2-4 compounds [d,f]	15	0.011	5.1	0.086
Total characterized	18	0.013	5.9	0.10
PES	6.5	0.005	2.5	0.043

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.028 mg/kg or 7.2 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 45 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated immature lettuce

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	**100**	**0.070**	**100**	**0.22**	**100**	**0.064**	**100**	**0.24**
Extractable	**93**	**0.065**	**106**	**0.23**	**91**	**0.058**	**101**	**0.24**
Fluindapyr [a]	18	0.013	4.0	0.009	19	0.012	6.4	0.015
3-OH-fluindapyr [a]	8.1	0.006	-	-	8.2	0.005	-	-
N-DesMet-pyrazole carboxylic acid [b]	-	-	76	0.17	-	-	82	0.12
Pyrazole carboxylic acid [b]	-	-	3.6	0.008	-	-	5.2	0.012
Pyrazole carboxamide [b]	-	-	7.5	0.017	-	-	6.8	0.016
1-COOH-fluindapyr [b]	25	0.018	7.6	0.017	24	0.015	-	-
trans-1-COOH-fluindapyr [b]	*15*	*0.010*	*5.3*	*0.012*	*14*	*0.009*	-	-
cis-1-COOH-fluindapyr [b]	*11*	*0.007*	*2.3*	*0.005*	*9.7*	*0.006*	-	-
1-OH-Met-fluindapyr [b,c]	30	0.021	4.0	0.009	23	0.015	-	-
	21	*0.015*	*4.0*	*0.009*	*17*	*0.011*	-	-
	9.0	*0.006*	-	-	*6.0*	*0.004*	-	-
N-DesMet-fluindapyr [b]	<LOQ	<LOQ	0.69	0.002	4.0	0.003	-	-
Total identified	**82**	**0.057**	**103**	**0.23**	**78**	**0.050**	**101**	**0.24**
Unknown (organo soluble) 1 compound [d,e]	2.6	0.002	-	-	2.0	0.001	-	-
Unknown (water soluble) 2-9 compounds [d,f]	8.9	0.006	2.8	0.006	10	0.006	-	-
Total characterized	**12**	**0.008**	**2.8**	**0.006**	**12**	**0.008**	**-**	**-**
PES	7.2	0.005	1.8	0.004	7.0	0.004	3.9	0.009

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.008 mg/kg or 5.8 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

LOQ: 0.01 mg/kg.

Table 46 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated immature lettuce.

	300d PBI			
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.046	100	0.25
Extractable	90	0.041	97	0.25
Fluindapyr [a]	14	0.006	4.0	0.010
3-OH-fluindapyr [a]	7.8	0.004	-	-

	300d PBI			
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.046	100	0.25
N-DesMet-pyrazole carboxylic acid [b]	-	-	64	0.16
Pyrazole carboxylic acid [b]	-	-	-	-
Pyrazole carboxamide [b]	-	-	13	0.032
1-COOH-fluindapyr [b]	34	0.016	3.3	0.008
trans-1-COOH-fluindapyr [b]	*22*	*0.010*	*2.3*	*0.006*
cis-1-COOH-fluindapyr [b]	*13*	*0.006*	*0.93*	*0.002*
1-OH-Met-fluindapyr [b,c]	24	0.011	1.1	0.003
	15	*0.007*	*1.1*	*0.003*
	9.3	*0.004*	-	-
N-DesMet-fluindapyr [b]	3.8	0.002	0.71	0.002
Total identified	84	0.038	86	0.22
Unknown (organo soluble) 1 compound [d,e]	2.2	0.001	-	-
Unknown (water soluble) 2-9 compounds [d,f]	3.8	0.002	11	0.028
Total characterized	6.0	0.003	11	0.028
PES	7.4	0.003	3.9	0.010

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.008 mg/kg or 5.8 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 47 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated mature lettuce

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.081	100	0.23	100	0.071	100	0.26
Extractable	94	0.076	101	0.23	93	0.066	97	0.25
Fluindapyr [a]	18	0.015	3.0	0.007	20	0.014	4.7	0.012
3-OH-fluindapyr [a]	8.1	0.007	-	-	8.5	0.006	2.0	0.005
N-DesMet-pyrazole carboxylic acid [b]	-	-	62	0.14	-	-	63	0.16
Pyrazole carboxylic acid [b]	-	-	9.5	0.022	-	-	12	0.030
Pyrazole carboxamide [b]	-	-	3.0	0.007	-	-	1.9	0.005
1-COOH-fluindapyr [b]	16	0.013	7.5	0.017	17	0.012	3.5	0.009
trans-1-COOH-fluindapyr [b]	*12*	*0.010*	*4.9*	*0.011*	*12*	*0.008*	*2.2*	*0.006*
cis-1-COOH-fluindapyr [b]	*4.3*	*0.003*	*2.6*	*0.006*	*4.8*	*0.003*	*1.3*	*0.003*
1-OH-Met-fluindapyr [b,c]	33	0.027	5.6	0.013	34	0.024	2.8	0.007
	24	*0.019*	*4.4*	*0.010*	*24*	*0.017*	*2.4*	*0.006*
	8.9	*0.007*	*1.2*	*0.003*	*9.8*	*0.007*	*0.4*	*0.001*
N-DesMet-fluindapyr [b]	2.0	0.002	0.8	0.002	3.3	0.002	0.7	0.002
Total identified	78	0.063	91	0.21	82	0.058	90	0.23
Unknown (organo soluble) 1 compound [d,e]	2.2	0.002	-	-	1.7	0.001	-	-

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Unknown (water soluble) 2-5 compounds [d,f]	14	0.012	9.8	0.022	9.9	0.007	6.6	0.017
Total characterized	16	0.013	9.8	0.022	12	0.008	6.6	0.017
PES	5.8	0.005	5.4	0.012	4.6	0.003	8.1	0.021

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.015 mg/kg or 6.4 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 48 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated mature lettuce

	300d PBI			
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.044	100	0.34
Extractable	89	0.039	98	0.34
Fluindapyr [a]	13	0.006	2.8	0.009
3-OH-fluindapyr [a]	7.3	0.003	1.2	0.004
N-DesMet-pyrazole carboxylic acid [b]	-	-	62	0.21
Pyrazole carboxylic acid [b]	-	-	11	0.039
Pyrazole carboxamide [b]	-	-	9.3	0.032
1-COOH-fluindapyr [b]	31	0.014	3.3	0.011
trans-1-COOH-fluindapyr [b]	*21*	*0.009*	*2.2*	*0.007*
cis-1-COOH-fluindapyr [b]	*11*	*0.005*	*1.1*	*0.004*
1-OH-Met-fluindapyr [b,c]	30	0.013	1.6	0.005
	23	*0.010*	*0.84*	*0.003*
	7.8	*0.003*	*0.71*	*0.002*
N-DesMet-fluindapyr [b]	4.6	0.002	0.93	0.003
Total identified	87	0.039	92	0.32
Unknown (organo soluble) 1 compound [d,e]	2.1	0.001	-	-
Unknown (water soluble) 2-5 compounds [d,f]	-	-	5.8	0.020
Total characterized	2.1	0.001	5.8	0.020
PES	7.5	0.003	7.3	0.025

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.015 mg/kg or 6.4 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 49 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated wheat forage

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.35	100	0.54	100	0.41	100	0.62
Extractable	95	0.33	93	0.50	94	0.38	96	0.59
Fluindapyr [a]	32	0.11	22	0.12	30	0.12	22	0.14
3-OH-fluindapyr [a]	17	0.058	11	0.060	18	0.073	11	0.068
N-DesMet-pyrazole carboxylic acid [b]	-	-	3.0	0.016	-	-	5.5	0.034
Pyrazole carboxylic acid [b]	-	-	23	0.12	-	-	14	0.086
Pyrazole carboxamide [b]	-	-	3.0	0.016	-	-	4.1	0.025
1-COOH-fluindapyr [b]	5.5	0.019	3.5	0.019	3.3	0.014	2.0	0.012
trans-1-COOH-fluindapyr [b]	*3.7*	*0.013*	*2.0*	*0.011*	*2.0*	*0.008*	*1.5*	*0.009*
cis-1-COOH-fluindapyr [b]	*1.8*	*0.006*	*1.5*	*0.008*	*1.4*	*0.006*	*0.5*	*0.003*
1-OH-Met-fluindapyr [b,c]	26	0.089	11	0.061	27	0.11	17	0.11
	8.1	*0.028*	*4.7*	*0.025*	*9.7*	*0.040*	*6.3*	*0.039*
	17	*0.061*	*6.6*	*0.036*	*18*	*0.072*	*11*	*0.068*
N-DesMet-fluindapyr [b]	1.1	0.004	2.6	0.014	0.46	0.002	1.9	0.012
Total identified	81	0.28	79	0.43	79	0.32	78	0.48
Unknown (organo soluble) 1 compound [d,e]	1.6	0.005	-	-	2.3	0.010	1.7	0.011
Unknown (water soluble) 2-13 compounds [d,f]	13	0.044	14	0.075	13	0.052	17	0.10
Total characterized	14	0.049	14	0.075	15	0.062	19	0.11
PES	3.1	0.011	2.9	0.016	3.5	0.014	2.7	0.017

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.033 mg/kg or 8.7 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 50 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated wheat forage

	300d PBI			
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.36	100	0.37
Extractable	89	0.32	96	0.36
Fluindapyr [a]	17	0.060	13	0.050
3-OH-fluindapyr [a]	13	0.048	9.6	0.036
N-DesMet-pyrazole carboxylic acid [b]	-	-	3.0	0.011
Pyrazole carboxylic acid [b]	-	-	13	0.050
Pyrazole carboxamide [b]	-	-	1.9	0.007
1-COOH-fluindapyr [b]	9.5	0.034	6.2	0.023
trans-1-COOH-fluindapyr [b]	*5.3*	*0.019*	*3.5*	*0.013*
cis-1-COOH-fluindapyr [b]	*4.2*	*0.015*	*2.7*	*0.010*
1-OH-Met-fluindapyr [b,c]	36	0.13	22	0.084
	13	*0.048*	*8.7*	*0.032*

	300d PBI			
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
	23	*0.082*	*14*	*0.051*
N-DesMet-fluindapyr [b]	1.0	0.004	0.52	0.002
Total identified	76	0.28	70	0.26
Unknown (organo soluble) 1 compound [d,e]	2.1	0.008	-	-
Unknown (water soluble) 2-4 compounds [d,f]	11	0.040	23	0.088
Total characterized	13	0.047	23	0.35
PES	4.6	0.017	2.8	0.011

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.033 mg/kg or 8.7 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 51 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated wheat hay

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	**100**	**0.82**	**100**	**1.5**	**100**	**0.79**	**100**	**1.7**
Extractable	**93**	**0.76**	**96**	**1.4**	**95**	**0.75**	**94**	**1.6**
Fluindapyr [a]	26	0.21	23	0.33	25	0.20	22	0.38
3-OH-fluindapyr [a]	15	0.12	13	0.19	16	0.12	14	0.23
N-DesMet-pyrazole carboxylic acid [b]	-	-	3.5	0.052	-	-	4.2	0.072
Pyrazole carboxylic acid [b]	-	-	15	0.22	-	-	11	0.19
Pyrazole carboxamide [b]	-	-	0.83	0.012	-	-	1.5	0.027
1-COOH-fluindapyr [b]	3.6	0.030	2.7	0.040	4.7	0.037	3.6	0.062
trans-1-COOH-fluindapyr [b]	*2.1*	*0.017*	*2.1*	*0.030*	*3.3*	*0.026*	*2.4*	*0.041*
cis-1-COOH-fluindapyr [b]	*1.5*	*0.013*	*0.65*	*0.010*	*1.4*	*0.011*	*1.2*	*0.021*
1-OH-Met-fluindapyr [b,c]	33	0.27	25	0.36	31	0.24	25	0.42
	12	*0.099*	*7.6*	*0.11*	*11*	*0.089*	*10*	*0.18*
	21	*0.18*	*17*	*0.25*	*20*	*0.15*	*14*	*0.25*
N-DesMet-fluindapyr [b]	<LOQ	<LOQ	-	-	1.0	0.008	0.63	0.011
Total identified	**78**	**0.64**	**82**	**1.2**	**77**	**0.61**	**81**	**1.4**
Unknown (organo soluble) 1 compound [d,e]	1.2	0.010	1.7	0.025	1.6	0.013	1.8	0.031
Unknown (water soluble) 2-12 compounds [d,f]	14	0.12	12	0.18	16	0.130	11	0.18
Total characterized	**15**	**0.13**	**14**	**0.20**	**18**	**0.14**	**12**	**0.21**
PES	4.2	0.035	1.9	0.028	5.0	0.039	1.9	0.033

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.044 mg/kg or 2.9 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

LOQ: 0.01 mg/kg.

Table 52 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated wheat hay

	300d PBI			
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.62	100	1.7
Extractable	92	0.57	96	1.6
Fluindapyr [a]	17	0.11	6.6	0.11
3-OH-fluindapyr [a]	14	0.090	6.2	0.11
N-DesMet-pyrazole carboxylic acid [b]	-	-	6.2	0.11
Pyrazole carboxylic acid [b]	-	-	38	0.65
Pyrazole carboxamide [b]	-	-	2.0	0.034
1-COOH-fluindapyr [b]	7.4	0.046	3.4	0.058
trans-1-COOH-fluindapyr [b]	*4.1*	*0.026*	*2.6*	*0.043*
cis-1-COOH-fluindapyr [b]	*3.3*	*0.020*	*0.87*	*0.015*
1-OH-Met-fluindapyr [b,c]	38	0.23	17	0.29
	15	*0.093*	*7.6*	*0.13*
	23	*0.14*	*9.6*	*0.16*
N-DesMet-fluindapyr [b]	0.92	0.006	1.1	0.018
Total identified	78	0.48	81	1.4
Unknown (organo soluble) 1 compound [d,e]	2.1	0.013	1.3	0.022
Unknown (water soluble) 2-12 compounds [d,f]	12	0.072	14	0.24
Total characterized	14	0.085	15	0.26
PES	5.9	0.037	2.6	0.044

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.044 mg/kg or 2.9 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 53 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated wheat straw

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	**100**	**2.02**	**100**	**3.8**	**100**	**2.3**	**100**	**3.7**
Extractable	**93**	**1.87**	**84**	**3.2**	**90**	**2.0**	**84**	**3.1**
Fluindapyr [a]	17	0.34	9.1	0.34	14	0.31	10	0.38
3-OH-fluindapyr [a]	12	0.24	7.1	0.27	10	0.23	8.4	0.31
N-DesMet-pyrazole carboxylic acid [b]	-	-	5.3	0.20	-	-	15	0.54

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Pyrazole carboxylic acid [b]	-	-	9.1	0.34	-	-	22	0.81
Pyrazole carboxamide [b]	-	-	1.8	0.067	-	-	1.5	0.054
1-COOH-fluindapyr [b]	5.1	0.10	2.1	0.081	4.5	0.10	1.9	0.072
trans-1-COOH-fluindapyr [b]	*3.1*	*0.063*	*1.1*	*0.040*	*2.6*	*0.057*	*1.1*	*0.039*
cis-1-COOH-fluindapyr [b]	*2.0*	*0.040*	*1.1*	*0.040*	*1.9*	*0.044*	*0.88*	*0.033*
1-OH-Met-fluindapyr [b,c]	44	0.88	43	1.6	46	1.0	17	0.61
	17	*0.34*	*18*	*0.66*	*19*	*0.43*	*6.4*	*0.24*
	27	*0.54*	*25*	*0.95*	*27*	*0.60*	*10*	*0.38*
N-DesMet-fluindapyr [b]	1.3	0.027	0.77	0.029	1.2	0.027	0.97	0.036
Total identified	**79**	**1.60**	**78**	**2.9**	**76**	**1.7**	**76**	**2.8**
Unknown (organo soluble) 1 compound [d,e]	1.3	0.027	1.2	0.044	1.7	0.037	1.2	0.045
Unknown (water soluble) 2-9 compounds [d,f]	12	0.25	4.9	0.19	13	0.29	6.4	0.24
PES	7.8	0.16	11	0.41	8.7	0.20	12	0.43
Mild base	0.9	0.018	1.1	0.041	1.0	0.023	1.1	0.041
Surfactant	-	-	0.12	0.004	-	-	0.14	0.005
Cellulase	5.9	0.12	8.8	0.33	6.9	0.16	9.6	0.36
Total characterized	**21**	**0.42**	**16**	**0.61**	**22**	**0.50**	**18**	**0.68**

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.046 mg/kg or 2.4 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 54 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated wheat straw

	300d PBI			
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	**100**	**1.3**	**100**	**3.2**
Extractable	**85**	**1.1**	**91**	**2.9**
Fluindapyr [a]	11	0.14	5.1	0.17
3-OH-fluindapyr [a]	12	0.15	4.8	0.15
N-DesMet-pyrazole carboxylic acid [b]	-	-	20	0.63
Pyrazole carboxylic acid [b]	-	-	28	0.89
Pyrazole carboxamide [b]	-	-	3.0	0.10
1-COOH-fluindapyr [b]	6.3	0.080	1.7	0.056
trans-1-COOH-fluindapyr [b]	*3.8*	*0.049*	*0.72*	*0.023*
cis-1-COOH-fluindapyr [b]	*2.5*	*0.032*	*1.0*	*0.033*
1-OH-Met-fluindapyr [b,c]	38	0.49	19	0.61
	17	*0.21*	*7.6*	*0.24*
	21	*0.27*	*11*	*0.37*
N-DesMet-fluindapyr [b]	1.4	0.017	1.3	0.041
Total identified	**69**	**0.87**	**82**	**2.7**
Unknown (organo soluble)	1.6	0.021	-	-

	300d PBI			
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
1 compound [d,e]				
Unknown (water soluble) 2-9 compounds [d,f]	14	0.18	8.5	0.27
PES	13	0.16	12	0.38
Mild base	1.9	0.025	1.3	0.041
Surfactant	-	-	0.16	0.005
Cellulase	8.3	0.11	10	0.33
Total characterized	**26**	**0.33**	**20**	**0.65**

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.046 mg/kg or 1.4 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 55 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated wheat grain

	30d PBI				120d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	**100**	**1.5**	**100**	**2.9**	**100**	**1.5**	**100**	**3.0**
Extractable	**90**	**1.4**	**89**	**2.6**	**92**	**1.4**	**86**	**2.6**
Fluindapyr [a]	15	0.23	13	0.39	14	0.22	11	0.34
3-OH-fluindapyr [a]	11	0.17	10	0.29	11	0.17	9.0	0.27
N-DesMet-pyrazole carboxylic acid [b]	-	-	3.0	0.087	-	-	10	0.31
Pyrazole carboxylic acid [b]	-	-	12	0.34	-	-	23	0.69
Pyrazole carboxamide [b]	-	-	1.7	0.050	-	-	2.1	0.061
1-COOH-fluindapyr [b]	7.0	0.11	3.5	0.10	5.9	0.091	4.3	0.13
trans-1-COOH-fluindapyr [b]	*3.8*	*0.057*	*2.1*	*0.061*	*3.3*	*0.051*	*2.5*	*0.074*
cis-1-COOH-fluindapyr [b]	*3.2*	*0.048*	*1.4*	*0.041*	*2.6*	*0.040*	*1.9*	*0.056*
1-OH-Met-fluindapyr [b,c]	47	0.71	37	1.1	50	0.78	19	0.56
	19	*0.29*	*16*	*0.47*	*19*	*0.30*	*8.0*	*0.24*
	28	*0.42*	*21*	*0.60*	*31*	*0.48*	*11*	*0.32*
N-DesMet-fluindapyr [b]	1.1	0.016	0.68	0.020	1.4	0.021	0.58	0.017
Total Identified	**82**	**1.2**	**81**	**2.4**	**83**	**1.28**	**80**	**2.4**
Unknown (organo soluble) 1 compound [d,e]	0.83	0.013	0.75	0.022	1.8	0.028	1.5	0.045
Unknown (water soluble) 2-7 compounds [d,f]	7.3	0.11	6.5	0.19	7.3	0.11	5.4	0.16
PES	6.9	0.10	8.2	0.24	4.8	0.074	8.2	0.25
Mild base	1.4	0.021	1.5	0.042	1.1	0.017	1.5	0.046
Surfactant	-	-	0.06	0.002	-	-	0.07	0.002
β-amylase	1.1	0.016	1.0	0.029	0.78	0.012	1.0	0.031
Cellulase	4.0	0.061	5.2	0.15	2.7	0.042	5.3	0.16
Total characterized	**15**	**0.22**	**15**	**0.43**	**14**	**0.21**	**15**	**0.44**

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.044 mg/kg or 3.2 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

Table 56 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in rotated wheat grain

	300d PBI			
	Phenyl-label		Pyrazole-label	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	**100**	**0.80**	**100**	**2.84**
Extractable	**91**	**0.73**	**87**	**2.5**
Fluindapyr [a]	10	0.084	6.1	0.17
3-OH-fluindapyr [a]	11	0.092	5.9	0.17
N-DesMet-pyrazole carboxylic acid [b]	-	-	9.7	0.27
Pyrazole carboxylic acid [b]	-	-	29	0.83
Pyrazole carboxamide [b]	-	-	16	0.45
1-COOH-fluindapyr [b]	10	0.082	2.7	0.076
trans-1-COOH-fluindapyr [b]	*4.8*	*0.039*	*1.5*	*0.041*
cis-1-COOH-fluindapyr [b]	*5.3*	*0.043*	*1.2*	*0.034*
1-OH-Met-fluindapyr [b,c]	52	0.42	12	0.34
	21	*0.17*	*4.8*	*0.14*
	31	*0.25*	*7.0*	*0.20*
N-DesMet-fluindapyr [b]	<LOQ	<LOQ	-	-
Total Identified	**84**	**0.68**	**81**	**2.3**
Unknown (organo soluble) 1 compound [d,e]	3.2	0.025	0.45	0.013
Unknown (water soluble) 2-4 compounds [d,f]	3.5	0.028	5.1	0.14
PES	7.3	0.059	7.4	0.21
Mild base	1.3	0.010	1.4	0.039
Surfactant	-	-	0.07	0.002
β-amylase	0.93	0.008	0.94	0.027
Cellulase	4.2	0.034	4.8	0.14
Total characterized	**13**	**0.11**	**13**	**0.36**

Notes:

[a] Identified in total extracts [pooled extracts I-II-III-IV].

[b] Identified in hydrolysed aqueous extracts.

[c] Pair of diastereomers.

[d] None exceeded 0.044 mg/kg or 3.2 percent TRR.

[e] Detected in total extracts [pooled extracts I-II-III-IV].

[f] Detected in hydrolysed aqueous extracts.

LOQ: 0.01 mg/kg.

Table 57 Enantiomeric ratios of fluindapyr in rotational crops at different PBIs (heptane extract)

	30d PBI				120d PBI				300d PBI			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	S (%)	R (%)	S (%)	R (%)	S (%)	R (%)	S (%)	R (%)	S (%)	R (%)	S (%)	R (%)
Carrot												

Root	35	66	37	63	31	69	38	62	32	68	38	62
Leaves	31	69	40	60	33	67	38	62	33	67	40	60
Lettuce												
Immature	43	57	36	64	42	58	37	63	42	58	37	63
Mature	36	64	33	67	35	66	35	65	37	63	34	66
Wheat												
Forage	36	64	39	61	35	65	39	61	38	62	37	63
Hay	34	66	37	63	34	66	37	63	33	67	37	63
Straw	29	72	26	74	25	75	26	74	28	72	25	75
Grain	27	73	23	77	28	72	23	77	28	72	23	77

Study 3

A third confined rotational crop study was conducted in California, United States, with wheat (variety: Certified WB Patron) planted 30 and 120 days after a single bare-soil application of pyrazole-labelled fluindapyr [Desai, 2017b, 2015MET-IFP2064]. Only wheat has been investigated as rotational crop in this study, to verify the relatively high residue levels in rotational cereals obtained in the previous two confined rotational crop studies. The soil was treated with an actual application rats of 356 g ai/ha for the 30-day PBI and 360 g ai/ha for the 120-day PBI. Wheat seeds were planted into treated sandy loam soil in outdoor wooden boxes.

Forage samples were harvested at growth stage BBCH 17, hay at BBCH 85, and straw and grain at BBCH 89. Crops stored frozen (-20 °C) prior to shipment for analysis. Harvested commodities were processed cryogenically, after which they were combusted to determine the TRR. Processing of the samples occurred within maximally 8 days. Only data on the TRR were investigated, and further identification was considered not required.

The TRR of the samples is shown in Table 58. The TRR in hay and straw increased from the 30-day PBI to the 120-day PBI, while the TRR in forage and grain remained rather constant. Residues in control samples were < 0.01 mg/kg.

Table 58 Total Radioactive Residues (TRRs) in rotational wheat

Commodity	TRR (mg eq/kg)	
	30d PBI	120d PBI
Forage	0.22	0.21
Hay	0.67	1.2
Straw	0.67	1.2
Grain	0.064	0.10

Field rotational crop studies

Study 1

Field rotational crop studies were conducted in 2017 in Southern Europe (Italy and Spain) to provide information on the uptake of fluindapyr and its metabolites in rotational crops [Huaulmé, 2020a, 2017RES-IFP3569]. Representative crops from root and tuber vegetables (carrots/radish), small grain cereals (winter/spring wheat), leafy vegetables (lettuce), brassica vegetables (head cabbage), oilseeds crop group (soya beans), and fruits and fruiting vegetables (tomato) were planted into areas (bare soils) previously treated with a 100 g/L formulation of fluindapyr at interval of 30 (+/-3), 120 (+/-10), and 269 to 273 days (for targeted timing of 270 days) after the application. Fluindapyr was incorporated into the soil

at about 5 cm depth in trial 01-IT and at 5 mg/kg eq 10 cm depth in trial 02-SP with rotavator at actual dose rates of 215–226 g ai/ha (01-IT) and 204–231 g ai/ha (02-SP).

Soil samples (horizon approximately 0–20 cm depth) were collected just before the crops were sown or planted (on the same day). The characteristics of the soil are shown in Table 59. Succeeding crops (carrot/radish, wheat, lettuce, head cabbage, soya bean, and tomato) were sown or transplanted into the plots at PBI of approximately 30, 120 and 270 days. Samples of succeeding crops were harvested at their respective harvest time (Table 60). Commodities were collected from at least 12 plants or from 12 different areas of the plot. Samples for plants included for radish, leaves and roots separately, for cereal forage (whole plants), hay (whole plants), grain and straw, for lettuce, immature whole plants without roots and mature whole plants without roots, for head cabbage, immature whole plants without roots and mature whole plants without roots, for soya bean, forage, hay and seeds, and for tomato, fruits. The minimum weight was at least 1 kg per sample, except 0.3–0.5 kg soya bean hay, 0.5 kg straw and hay, 2 kg lettuce heads (mature), 2 kg tomato fruits, and 2 kg carrot roots and 2–4 kg head cabbage (mature).

Table 59 Soil characteristics

Trial	01-IT [a]		02-SP
	01-IT (Galliera, 40015, Italy)	01-IT (Poggio Renatico, 44028, Italy)*	Biar, 03410, Spain,
Soil type (USDA)	Clay loam	Loam	Sandy loam
-- percent clay	32	21	19
-- percent silt	36	44	11
-- percent sand	32	36	70
pH (water)	7.7	7.8	8.9
CEC (meq/100 g)	24.15	17.85	5.2
Water holding capacity [b]	11.8 percent (on dry soil)	13.4 percent (on dry soil)	3.21
Organic matter (%)	2.58	1.76	0.73

Notes:

[a] The trial site 01-IT was performed into two different test sites: one for carrot, white cabbage and lettuce (Galliera), the second one for the crops wheat, soya bean and tomato (Poggio Renatico).

[b] Moisture capacity (= available water content (AWC)) = Field capacity – Wilting point with Field capacity measured at pF 2.0 and/or 2.5 (this depends on the physical characteristics of soil) and wilting point measured at pF 4.2.

Table 60 Summary of PBI, DAT and DAS in field rotational crops (carrot/radish, wheat, lettuce, head cabbage, soya bean, and tomato) grown in soil at planted 30, 120 and 270 days after treatment of the bare soil with fluindapyr at two field sites in Southern Europe (Italy (01-IT) and Spain (02SP))

Samples and PBI		Galliera, 40015, Italy or Poggio Renatico, 44028, Italy, 01-IT 2 × 215-226 g ai/ha incorporated into bare soil Clay loam/Loam		Samples and PBI		Biar, 03410, Spain 02-SP 2 × 204-231 g ai/ha incorporated into bare soil Sandy loam	
Rotational crop sample	PBI	Harvest DAT	Harvest DAS	Rotational crop sample	PBI	Harvest DAT	Harvest DAS
Carrot root	31	144	113	Radish root	29	92	63
Carrot leaves	31	144	133	Radish leaves	29	92	63
Wheat forage	31	206	175	Wheat forage	32	159	127
Wheat hay	31	241	210	Wheat hay	32	193	161
Wheat grain	31	264	233	Wheat grain	32	241	209
Wheat straw	31	264	233	Wheat straw	32	241	209
Lettuce [i]	32	67	35	Lettuce [i]	31	77	46

Samples and PBI		Galliera, 40015, Italy or Poggio Renatico, 44028, Italy, 01-IT 2 × 215-226 g ai/ha incorporated into bare soil Clay loam/Loam		Samples and PBI		Biar, 03410, Spain 02-SP 2 × 204-231 g ai/ha incorporated into bare soil Sandy loam	
Lettuce [m]	32	77	45	Lettuce [m]	31	118	87
Head cabbage [i]	32	102	70	Head cabbage [i]	31	111	80
Head cabbage [m]	32	113	81	Head cabbage [m]	31	146	115
Soya bean forage	30	102	72	Soya bean forage	28	158	130
Soya bean hay	30	118	88	Soya bean hay	28	195	167
Soya bean seeds	30	150	120	Soya bean seeds	28	228	200
Tomato fruit	32	133	101	Tomato fruit	29	119	90
Carrot root	119	211	92	Radish root	120	222	102
Carrot leaves	119	211	92	Radish leaves	120	222	102
Wheat forage	119	294	175	Wheat forage	125	252	127
Wheat hay	119	329	210	Wheat hay	125	286	161
Wheat grain	119	352	233	Wheat grain	125	334	209
Wheat straw	119	352	233	Wheat straw	125	334	209
Lettuce [i]	117	152	35	Lettuce [i]	124	160	36
Lettuce [m]	117	159	42	Lettuce [m]	124	186	62
Head cabbage [i]	119	193	74	Head cabbage [i]	124	196	72
Head cabbage [m]	119	208	89	Head cabbage [m]	124	228	104
Soya bean forage	119	187	68	Soya bean forage	120	228	108
Soya bean hay	119	200	81	Soya bean hay	120	263	143
Soya bean seeds	119	276	157	Soya bean seeds	120	299	179
Tomato fruit	117	205	88	Tomato fruit	124	216	92
Carrot roots	270	362	92	Radish root	269	371	102
Carrot leaves	270	362	92	Radish leaves	269	371	102
Wheat forage	272	355	83	Wheat forage	273	364	91
Wheat hay	272	376	104	Wheat hay	273	405	132
Wheat grain	272	399	127	Wheat grain	273	434	161
Wheat straw	272	399	127	Wheat straw	273	434	161
Lettuce [i]	276	311	35	Lettuce [i]	273	309	36
Lettuce [m]	276	318	42	Lettuce [m]	273	335	62
Head cabbage [i]	270	356	86	Head cabbage [i]	273	345	72
Head cabbage [m]	270	368	98	Head cabbage [m]	273	377	104
Soya bean forage	270	331	61	Soya bean forage	269	377	108
Soya bean hay	270	344	74	Soya bean hay	269	412	143
Soya bean seeds	270	421	151	Soya bean seeds	269	448	179
Tomato fruit	276	364	88	Tomato fruit	273	365	92

Notes:

PBI = Plant Back Interval; DAT = days after last application; DAS = Days after sowing.

[i] = Immature.

[m] = Mature.

Weather conditions did not generally alter the growth, development and maturity of the rotational crops at the trial sites. Samples were kept frozen (-18 °C) until extraction. Extraction for the analysis of fluindapyr and its metabolites occurred within a maximum of 361 and 421 days after the corresponding harvest for both trials, respectively. This storage period is covered by the storage stability studies for all crop commodities.

Soil samples were analysed using LC/MS-MS methods RA.14.07 (fluindapyr), RA.16.03 (3-OH-fluindapyr, 1-COOH-fluindapyr, and pyrazole carboxamide), and RA.18.11 (pyrazole carboxylic acid and N-DesMet-pyrazole carboxylic acid). The principle of the method was based on extraction using acetone or a mixture of acetone/water 5:1 (v/v). Residue were quantified with LC/MS-MS using ion transitions m/z 325 to 256 for fluindapyr, m/z 366 to 175 for 3-OH-fluindapyr, m/z 382 to 336 for both diastereomers of 1-COOH-fluindapyr, m/z 176 to 136 for pyrazole carboxamide, m/z 177/175 to 137 for pyrazole carboxylic acid and 163/161 to 123/141 for N-DesMet-pyrazole carboxylic acid. LOQs of the methods were 0.010 mg/kg for fluindapyr and for 3-OH-fluindapyr, 0.014 mg/kg for DesMet-F-N1-Gluc, 0.006 and 0.004 mg/kg for the two diastereomers of both 1-OH-Met-F and 1-COOH-fluindapyr (0.010 mg/kg combined), and 0.007 and 0.003 (combined 0.010 mg/kg) for the two stereo isomers of 1-OH-Met-N-DesMet-F, 0.010 mg/kg for pyrazole carboxamide, pyrazole carboxylic acid and N-desmethyl-pyrazole carboxylic acid.

The residues of fluindapyr found in soil were summarized in the table below.

Table 61 Residues of fluindapyr and its metabolites in soil after treatment to bare soil, expressed in mg/kg as such

Sample	PBI	fluindapyr	3-OH-fluindapyr	1-COOH-fluindapyr [a]	pyrazole carboxamide	pyrazole carboxylic acid	N-DesMet-pyrazole carboxylic acid
Galliera, 40015, Italy or Poggio Renatico, 44028, Italy, 01-IT 2 × 215-226 g ai/ha incorporated into bare soil, Clay loam/Loam							
Carrot	31	0.21	0.010	<0.010	<0.010	<0.010	<0.010
	119	0.13	0.010	0.010	<0.010	<0.010	<0.010
	270	0.098	0.011	<0.010	<0.010	<0.010	<0.010
Wheat	31	0.23	<0.010	0.011	<0.010	<0.010	<0.010
	119	0.072	<0.010	0.015	<0.010	<0.010	<0.010
	272	0.16	0.027	<0.010	<0.010	<0.010	<0.010
Lettuce	32	0.11	<0.010	<0.010	<0.010	<0.010	<0.010
	117	0.22	0.011	<0.010	<0.010	<0.010	<0.010
	276	0.16	0.010	0.010	<0.010	<0.010	<0.010
Head cabbage	32	0.16	<0.010	<0.010	<0.010	<0.010	<0.010
	119	0.15	0.014	<0.010	<0.010	<0.010	<0.010
	270	0.12	0.009	<0.010	<0.010	<0.010	<0.010
Soya bean	30	0.16	0.022	<0.010	<0.010	<0.010	<0.010
	119	0.16	0.012	0.010	<0.010	<0.010	<0.010
	270	0.084	0.013	<0.010	<0.010	<0.010	<0.010
Tomato fruit	32	0.17	0.011	<0.010	<0.010	<0.010	<0.010
	117	0.14	0.011	0.010	<0.010	<0.010	<0.010
	276	0.10	0.013	<0.010	<0.010	<0.010	<0.010
Biar, 03410, Spain, 02-SP 2 × 204-231 g ai/ha incorporated into bare soil, sandy loam							
Radish	29	0.032	<0.010	<0.010	<0.010	<0.010	<0.010
	120	0.032	<0.010	<0.010	<0.010	<0.010	<0.010
	269	0.035	<0.010	<0.010	<0.010	<0.010	<0.010
Wheat	32	0.12	<0.010	<0.010	<0.010	<0.010	<0.010
	125	0.086	<0.010	<0.010	<0.010	<0.010	<0.010
	273	0.028	<0.010	<0.010	<0.010	<0.010	<0.010
Lettuce	31	0.090	<0.010	<0.010	<0.010	<0.010	<0.010
	124	0.045	<0.010	<0.010	<0.010	<0.010	<0.010
	273	0.035	<0.010	<0.010	<0.010	<0.010	<0.010
Head	31	0.049	<0.010	<0.010	<0.010	<0.010	<0.010

Sample	PBI	fluindapyr	3-OH-fluindapyr	1-COOH-fluindapyr [a]	pyrazole carboxamide	pyrazole carboxylic acid	N-DesMet-pyrazole carboxylic acid
cabbage							
	124	0.054	<0.010	<0.010	<0.010	<0.010	<0.010
	273	0.040	<0.010	<0.010	<0.010	<0.010	<0.010
Soya bean	28	0.094	<0.010	<0.010	<0.010	<0.010	<0.010
	120	0.029	<0.010	<0.010	<0.010	<0.010	<0.010
	269	0.032	<0.010	<0.010	<0.010	<0.010	<0.010
Tomato fruit	29	0.041	<0.010	<0.010	<0.010	<0.010	<0.010
	124	0.038	<0.010	<0.010	<0.010	<0.010	<0.010
	273	0.036	<0.010	<0.010	<0.010	<0.010	<0.010

Notes:

[a] Sum of diastereomers.

Samples of plant commodities were analysed for fluindapyr, 3-OH-fluindapyr and DesMet-fluindapyr-N1-Glucoside using the pre-hydrolysis method PTRL Europe Study ID P3770 [Stanislowski, 2016a, 2015RES-IFP2155], with an LOQ of 0.01 mg/kg for each compound. The same samples, were analysed for 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr using the post-hydrolysis method RA.17.01 [Riccelli, 2017a, RA.17.01] with an LOQ of 0.01 mg/kg (sum of combined diastereomers) for each analyte. The post-hydrolysis method RA17.19 [Ricelli, 2017b, RA.17.19] was used to determine pyrazole carboxamide, pyrazole carboxylic acid and N-DesMet-pyrazole carboxylic acid with an LOQ of 0.01 mg/kg for each analyte.

A reduced method validation [Soddu, 2018, RA18.01, appendix to Huaulmé, 2020a, 2017RES-IFP3569] in carrot root and leaves and radish roots and leaves, cabbage, tomato fruit, soya bean forage and hay (n=3 at LOQ level and n=1 at 10 × LOQ) showed that recoveries for each matrix ranged between: 70-110 percent for fluindapyr, 3-OH-fluindapyr, DesMet-fluindapyr-N1-glucoside, pyrazole carboxamide, pyrazole carboxylic acid and N-DesMet-pyrazole-carboxylic acid, and for each diastereomer of 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr. The results are summarized in the section on analytical methods. Procedural recoveries also ranged between 70-120 percent for parent fluindapyr and its metabolites (not included in the analytical section). Control samples had residues below 0.3LOQ.

Fluindapyr and its metabolites including their glucoside conjugates, expressed as the free aglycons, were not found (<0.01 mg/kg each) in lettuce, carrot or radish root (except one sample), carrot foliage, head cabbage, wheat grain, or soya bean seed) planted at approximately 30, 120 or 270 days after incorporation of the fluindapyr into the soil. Residues were observed in wheat straw, hay, forage, soya bean forage and hay, and incidentally in radish leaves and roots. The results are summarized in Table 62.

Table 62 Residues of fluindapyr and its metabolites, expressed as such in rotational crop samples of trials performed in Italy and Spain, respectively

Sample	Anticipated PBI	parent	DMNgluc	3-HF	1-HMF [a]	1-HDMF [a]	1-CF [a]	PC	PCA	NDPCA
Wheat straw	30	**0.014** **0.012**	<0.01 <0.01	<0.01 **0.019**	<0.01 **0.031**	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	120	<0.01 **0.022**	<0.01 <0.01	<0.01 **0.034**	<0.01 **0.066**	<0.01 <0.01 [b]	<0.01 <0.01 [b]	<0.01 <0.01	<0.01 <0.01	<0.01 **0.029**
	270	<0.01 <0.01	<0.01 <0.01	**0.012** **0.014**	**0.021** **0.025**	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.027** <0.01

Sample	Anticipated PBI	parent	DMNgluc	3-HF	1-HMF [a]	1-HDMF [a]	1-CF [a]	PC	PCA	NDPCA
Wheat hay	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.011** **0.010**	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.013** <0.01
Wheat forage	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 **0.011**	<0.01 **0.012**
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.012** <0.01
Soya bean forage	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01 [b]	<0.01 <0.01 [b]	<0.01 **0.014**	<0.01 <0.01	<0.01 <0.01	<0.01 **0.011**
	120	<0.01 <0.01	<0.01 <0.01	<0.01 **0.013**	<0.01 <0.01 [b]	<0.01 **0.014**	<0.01 **0.017**	<0.01 <0.01	<0.01 <0.01	<0.01 **0.012**
	270	<0.01 <0.01	<0.01 <0.01	<0.01 0.010	<0.01 <0.01 [b]	<0.01 <0.01 [b]	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.018** 0.010
Soya bean hay	30	<0.01 <0.01	<0.01 <0.01	<0.01 **0.010**	<0.01 <0.01 [b]	<0.01 0.017	<0.01 **0.022**	<0.01 <0.01	<0.01 <0.01	<0.01 **0.015**
	120	<0.01 <0.01	<0.01 **0.010**	<0.01 **0.024**	<0.01 <0.01 [b]	<0.01 **0.027**	<0.01 [b] **0.028**	<0.01 <0.01	<0.01 <0.01	<0.01 **0.025**
	270	<0.01 <0.01	<0.01 <0.01	<0.01 **0.010**	<0.01 <0.01 [b]	<0.01 **0.014**	<0.01 0.017	<0.01 <0.01	<0.01 <0.01	**0.040** **0.012**
Radish leaves	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 **0.014**	<0.01 <0.01 [b]	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 **0.016**	<0.01 <0.01 [b]	<0.01 <0.01 [b]	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Radish roots	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 **0.014**
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01

Notes:

DMN-gluc=DesMet-N1-glucoside; 3-HF=3-OH-fluindapyr; 1-HMF=1-OH-Met-fluindapyr; 1-HDMF=1-OH-Met-N-DesMet-fluindapyr; 1-CF=1-COOH-fluindapyr; PC=pyrazole carboxamide; PCA = pyrazole carboxylic acid, NDPCA = N-DesMet-pyrazole carboxylic acid. All residues are expressed as such (not in parent equivalents).

[a] Sum of both diastereomers.

[b] One of the single diastereomers is above the respective LOQ, but are below LOQ when combined.

Study 2

Field rotational crop studies were conducted in 2017 in Northern Europe (Northern France and Hungary) to provide information on the uptake of fluindapyr and its metabolites in rotational crops [Huaulmé, 2020b, 2017RES-IFP3638]. Representative crops from root and tuber vegetables (radish), small grain cereals (winter/spring wheat), leafy vegetables (lettuce), brassica vegetables (head cabbage), oilseeds crop group (soya beans), and fruits and fruiting vegetables (tomato) were planted into areas (bare soils) previously treated with a 100 g/L formulation of fluindapyr at interval of 30 (+/-3), 120 (+/-10), and 269 to 273 days after the application. Fluindapyr was incorporated into the soil at about 5–10 cm depth in both

trial BPL17/69401/RC-01-FR and trial BPL/694/RC-02-HU with rotavator at actual dose rates of 204–226 g ai/ha (01-FR) and 203–232 g ai/ha (02-HU).

Soil samples (horizon approximately 0–20 cm depth) were collected just before the crops were sown or planted (on the same day). The characteristics of the soil are shown in Table 63. Succeeding crops (carrot/radish, wheat, lettuce, head cabbage, soya bean, and tomato) were sown or transplanted into the plots at PBI of approximately 30, 120 and 270 days. Samples of succeeding crops were harvested at their respective harvest time (Table 64). Commodities were collected from at least 12 plants or from 12 different areas of the plot. Samples for plants included for radish, leaves and roots separately, for cereal forage (whole plants), hay (whole plants), grain and straw, for lettuce, immature whole plants without roots and mature whole plants without roots, for head cabbage, immature whole plants without roots and mature whole plants without roots, for soya bean, forage, hay and seeds, and for tomato, fruits. The minimum weight was at least 1 kg per sample, except 0.3–0.5 kg soya bean hay, 0.5 kg straw and hay, 2 kg lettuce heads (mature), 2 kg tomato fruits, and 2–4 kg head cabbage (mature).

Table 63 Soil characteristics

Trial	BPL17/69401/RC-01-FR [a]		BPL/694/RC-02-HU
	01-FRA (Vauchrétien, 49320, France)	01-FRB (Saint Georges de Sept voies, 49350, France)	02-HU (Jászfényszaru, 5126, Hungary)
Soil type (USDA)	Loam	Silt loam (soil with 27 percent $CaCO_3$)	Sandy loam
-- percent clay	19.8	21.5	14.3
-- percent silt	50.0	43.1	8.1
-- percent sand	30.2	35.3	77.6
pH (water)	5.5	8.5	7.9
CEC (meq/100 g)	9.1	14.2	Not reported
Water holding capacity [b]	15.2 (on dry soil)	18.9 (on dry soil)	Not reported
Organic matter (%)	2.3	1.9	1.9

Notes:

[a] The trial site 01-FR was performed into two different test sites: one for wheat and the other for the remaining crops.

[b] Moisture capacity (= available water content (AWC)) = Field capacity – Wilting point with Field capacity measured at pF 2.0 and/or 2.5 (this depends on the physical characteristics of soil) and wilting point measured at pF 4.2.

Table 64 Summary of PBI, DAT and DAS in field rotational crops (radish, wheat, lettuce, head cabbage, soya bean, and tomato) grown in soil at planted 30, 120 and 270 days after treatment of the bare soil with fluindapyr at two field sites in Northern Europe (France (01-FRA and 01-FRB) and Hungary (02-HU))

Samples and PBI		01-FRA (Vauchrétien, 49320, France) and 01-FRB (Saint Georges de Sept voies, 49350, France) [a] 1 × 204-226 g ai/ha incorporated into bare soil Loam/Silt loam		Samples and PBI		02-HU (Jászfényszaru, 5126, Hungary) 1 × 203-232 g ai/ha incorporated into bare soil Sandy loam	
Rotational crop sample	PBI	Harvest DAT	Harvest DAS	Rotational crop sample	PBI	Harvest DAT	Harvest DAS
Carrot root	35 [b]	73	38	Radish root	33	118	85
Carrot leaves	35 [b]	73	38	Radish leaves	33	118	35
Wheat forage	29	206	177	Wheat forage	32	229	197
Wheat hay	29	260	231	Wheat hay	32	260	228
Wheat grain	29	284	255	Wheat grain	32	299	267
Wheat straw	29	284	255	Wheat straw	32	299	267

Samples and PBI		01-FRA (Vauchrétien, 49320, France) and 01-FRB (Saint Georges de Sept voies, 49350, France) [a] 1 × 204-226 g ai/ha incorporated into bare soil Loam/Silt loam		Samples and PBI		02-HU (Jászfényszaru, 5126, Hungary) 1 × 203-232 g ai/ha incorporated into bare soil Sandy loam	
Lettuce [i]	31	58	27	Lettuce [i]	33	56	23
Lettuce [m]	31	73	42	Lettuce [m]	33	69	36
Head cabbage [i]	30	87	57	Head cabbage [i]	31	88	57
Head cabbage [m]	30	158	128	Head cabbage [m]	31	132	101
Soya bean forage	35 [b]	107	72	Soya bean forage	31	90	59
Soya bean hay	35 [b]	127	92	Soya bean hay	31	108	77
Soya bean seeds	35 [b]	169	134	Soya bean seeds	31	147	116
Tomato fruit	35 [b]	109	78	Tomato fruit	30	118	88
Carrot root	118	156	38	Radish root	123	195	72
Carrot leaves	118	156	38	Radish leaves	123	195	72
Wheat forage	120	297	177	Wheat forage	122	319	197
Wheat hay	120	351	231	Wheat hay	122	350	228
Wheat grain	120	375	255	Wheat grain	122	389	267
Wheat straw	120	375	255	Wheat straw	122	389	267
Lettuce [i]	114	141	27	Lettuce [i]	117	138	21
Lettuce [m]	114	156	42	Lettuce [m]	117	152	35
Head cabbage [i]	113	170	57	Head cabbage [i]	128	192	64
Head cabbage [m]	113	241	128	Head cabbage [m]	128	215	87
Soya bean forage	118	190	72	Soya bean forage	119	178	59
Soya bean hay	118	210	92	Soya bean hay	119	196	77
Soya bean seeds	118	252	134	Soya bean seeds	119	235	116
Tomato fruit	114	192	78	Tomato fruit	118	206	88
Carrot roots	273	311	38	Radish root	279	351	72
Carrot leaves	273	311	38	Radish leaves	279	351	72
Wheat forage	296 [b]	368	72	Wheat forage	301 [b]	353	52
Wheat hay	296 [b]	386	90	Wheat hay	301 [b]	375	74
Wheat grain	296 [b]	409	113	Wheat grain	301 [b]	409	108
Wheat straw	296 [b]	409	113	Wheat straw	301 [b]	409	108
Lettuce [i]	279	311	32	Lettuce [i]	273	294	21
Lettuce [m]	279	322	43	Lettuce [m]	273	308	35
Head cabbage [i]	279	334	55	Head cabbage [i]	284	348	64
Head cabbage [m]	279	385	106	Head cabbage [m]	284	371	87
Soya bean forage	273	334	61	Soya bean forage	275	334	59
Soya bean hay	273	354	81	Soya bean hay	275	352	77
Soya bean seeds	273	396	123	Soya bean seeds	275	391	116
Tomato fruit	273	361	88	Tomato fruit	274	362	88

Notes:

PBI = Plant Back Interval; DAT = days after last application; DAS = Days after sowing.

[a] The trial site 01-FR was performed into two different test sites: one for wheat and the other for the remaining crops.

[b] deviation from protocol 30 days +/- 3 days and 207 +/- 15 days.

[i] = Immature.

[m] = Mature.

Weather conditions did not generally alter the growth, development and maturity of the rotational crops at the trial sites. Samples were kept frozen (-18 °C) until extraction. Extraction for the analysis of fluindapyr and its metabolites occurred within a maximum of 275 and 337 days after the corresponding harvest for both trials, respectively. This storage period is covered by the storage stability studies for all crop commodities.

Soil samples were analysed using LC/MS-MS methods RA.14.07 (fluindapyr), RA.16.03 (3-OH-fluindapyr, 1-COOH-fluindapyr, and pyrazole carboxamide), and RA.18.11 (pyrazole carboxylic acid and N-desmethyl-pyrazole carboxylic acid). The principle of the methods was based on extraction using acetone or a mixture of acetone/water 5:1 (v/v). Residue were quantified with LC/MS-MS using ion transitions m/z 325 to 256 for fluindapyr, m/z 366 to 175 for 3-OH-fluindapyr, m/z 382 to 336 for both diastereomers of 1-COOH-fluindapyr, m/z 176 to 136 for pyrazole carboxamide, m/z 177/175 to 137 for pyrazole carboxylic acid and 163/161 to 123/141 for N-DesMet-pyrazole carboxylic acid. Text can be removed after inclusion in the analytical section.

Response applicant: The LC/MS/MS method used to analyse test soil samples in 2017RES-IFP3569 is summarized in the document “2017RES-IFP3569_soil method_JMPR_08052022”.

LOQs of the methods were 0.010 mg/kg for fluindapyr, 0.005 mg/kg for 3-OH-fluindapyr, 0.003 and 0.002 mg/kg for the two diastereomers of 1-COOH-fluindapyr (0.005 mg/kg combined), 0.005 mg/kg for pyrazole carboxamide, pyrazole carboxylic acid and N-desmethyl-pyrazole carboxylic acid.

Table 65 Residues of fluindapyr and its metabolites, expressed as such, in soil after treatment to bare soil

Sample	PBI	fluindapyr	3-OH-fluindapyr	1-COOH-fluindapyr [a]	pyrazole carboxamide	pyrazole carboxylic acid	N-DesMet-pyrazole carboxylic acid
01-FRA (Vauchrétien, 49320, France) and 01-FRB (Saint Georges de Sept voies, 49350, France) [a]; 1 × 204-226 g ai/ha incorporated into bare soil; loam/silt loam							
Radish	29	0.062	<0.005	0.012	<0.005	<0.005	<0.005
	112	0.040	<0.005	0.006	<0.005	<0.005	<0.005
	272	0.039	0.009	<0.005	<0.005	<0.005	<0.005
Wheat	29	0.076	<0.005	<0.005	<0.005	<0.005	<0.005
	120	0.054	<0.005	<0.005	<0.005	<0.005	<0.005
	295	0.032	<0.005	<0.005	<0.005	<0.005	<0.005
Lettuce	29	0.075	0.005	0.012	<0.005	<0.005	<0.005
	112	0.051	0.005	0.007	<0.005	<0.005	<0.005
	279	0.034	0.009	<0.005	<0.005	<0.005	<0.005
Head cabbage	29	0.079	0.005	0.011	<0.005	<0.005	<0.005
	112	0.056	0.005	0.007	<0.005	<0.005	<0.005
	279	0.044	0.010	<0.005	<0.005	<0.005	<0.005
Soya bean	29	0.070	<0.005	0.012	<0.005	<0.005	<0.005
	112	0.035	<0.005	0.006	<0.005	<0.005	<0.005
	272	0.033	0.007	<0.005	<0.005	<0.005	<0.005
Tomato fruit	29	0.097	0.006	0.011	<0.005	<0.005	<0.005
	112	0.056	0.005	0.007	<0.005	<0.005	<0.005
	273	0.024	<0.005	<0.005	<0.005	<0.005	<0.005
02-HU (Jászfényszaru, 5126, Hungary); 1 × 203-232 g ai/ha incorporated into bare soil; sandy loam							
Radish	33	0.11	<0.005	<0.005	<0.005	<0.005	<0.005
	123	0.098	0.010	0.008	<0.005	<0.005	<0.005
	279	0.072	0.013	<0.005 [b]	<0.005	<0.005	<0.005
Wheat	32	0.081	<0.005	<0.005	<0.005	<0.005	<0.005
	122	0.065	0.007	<0.005	<0.005	<0.005	<0.005
	301	0.049	0.010	<0.005	<0.005	<0.005	<0.005
Lettuce	33	0.078	<0.005	<0.005	<0.005	<0.005	<0.005

Sample	PBI	fluindapyr	3-OH-fluindapyr	1-COOH-fluindapyr [a]	pyrazole carboxamide	pyrazole carboxylic acid	N-DesMet-pyrazole carboxylic acid
	117	0.12	0.009	0.010	<0.005	<0.005	<0.005
	273	0.12	0.017	<0.005	<0.005	<0.005	<0.005
Head cabbage	31	0.11	0.007	<0.005 [b]	<0.005	<0.005	<0.005
	128	0.095	0.008	<0.005	<0.005	<0.005	<0.005
	284	0.090	0.016	<0.005	<0.005	<0.005	<0.005
Soya bean	31	0.11	0.006	<0.005 [b]	<0.005	<0.005	<0.005
	119	0.074	0.007	0.007	<0.005	<0.005	<0.005
	275	0.086	0.014	<0.005 [b]	<0.005	<0.005	<0.005
Tomato fruit	30	0.086	<0.005	<0.005 [b]	<0.005	<0.005	<0.005
	118	0.086	0.007	0.009	<0.005	<0.005	<0.005
	274	0.064	0.011	<0.005	<0.005	<0.005	<0.005

Notes:

[a] Sum of diastereomers.

[b] One of the single diastereomers is above the respective LOQ, but are below LOQ when combined.

Samples of plant commodities were analysed for fluindapyr, 3-hydroxy-fluindapyr and desmethyl-fluindapyr-N1-Glucoside using the pre-hydrolysis method PTRL Europe Study ID P3770 [Stanislowski, 2016a, 2015RES-IFP2155], with an LOQ of 0.01 mg/kg for each compound. The same samples, were analysed for 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr using the post-hydrolysis method RA.17.01 [Riccelli, 2017a, RA.17.01] with an LOQ of 0.01 mg/kg (sum of combined diastereomers) for each analyte. The post-hydrolysis method RA17.19 [Ricelli, 2017b, RA.17.19] was used to determine pyrazole carboxamide, pyrazole carboxylic acid and N-DesMet-pyrazole carboxylic acid. Except for DesMet-F-N1-Gluc, for which an LOQ of 0.014 mg/kg was established, for all other metabolites an LOQ of 0.01 mg/kg (combined for diastereomers) applied. Residues of the metabolites were expressed as such (not in parent equivalents).

A reduced method validation [Soddu, 2018, RA18.01, appendix to Huaulmé, 2020a, 2017RES-IFP3569] in carrot root and leaves and radish roots and leaves, cabbage, tomato fruit, soya bean forage and hay (n=3 at LOQ level and n=1 at 10 × LOQ) showed that recoveries for each matrix ranged between: 70–110 percent for fluindapyr, 3-OH-fluindapyr, DesMet-fluindapyr-N1-glucoside, pyrazole carboxamide, pyrazole carboxylic acid and N-desmethyl-pyrazole-carboxylic acid, and for each diastereomer of 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr. The results are summarized in the section on analytical methods. Procedural recoveries also ranged between 70–120 percent for parent fluindapyr and its metabolites (not included in the analytical section). Control samples had residues below LOQ.

Fluindapyr and metabolites DesMet-N1-glucoside, 1-OH-Met-fluindapyr (apart from one finding), 3-OH-fluindapyr and 1-OH-Met-N-DesMet-fluindapyr, pyrazole carboxamide; were not observed in any of the matrices. Metabolite 1-COOH-fluindapyr was found in radish leaves at PBI 30 and 120 days, immature head cabbage (PBI 30 days) and soya bean forage and hay (all PBIs). Residues of 1-COOH-fluindapyr, pyrazole carboxylic acid, and N-DesMet-pyrazole carboxylic acid were observed in more plant matrices, predominantly soya bean forage and hay. The results are summarized in Table 66.

Table 66 Residues of fluindapyr and its metabolites, expressed as such, in mg/kg in rotational crop samples of trials performed in Northern France and Hungary, respectively

Sample	Anticipated PBI	fluidapyr	DMNgluc	3-HF	1-HMF [a]	1-HDMF [a]	1-CF [a]	PC	PCA	NDPCA

Sample	Anticipated PBI	fluidapyr	DMNgluc	3-HF	1-HMF [a]	1-HDMF [a]	1-CF [a]	PC	PCA	NDPCA
Radish leaves	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.035** <0.01	<0.01 <0.01	<0.01 <0.01	**0.020** <0.01
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.015** <0.01	<0.01 <0.01	<0.01 <0.01	**0.012** <0.01
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Radish roots	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.27** <0.01	<0.01 <0.01
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.12** <0.01	<0.01 <0.01
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.024** <0.01	<0.01 <0.01
Head cabbage immature	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.020** <0.01	<0.01 <0.01	<0.01 <0.01	**0.016** <0.01
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.025** <0.01
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.02** <0.01
Lettuce immature	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01 [b]	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 **0.028 [c]**
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01 [b]	<0.01 <0.01	<0.01 <0.01	<0.01 **0.028 [d]**
Lettuce mature	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 **0.010**
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Wheat straw	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 [b] **0.021**	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 [b] <0.01 [b]	<0.01 <0.01	<0.01 [b] <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Wheat forage	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 **0.021**	<0.01 **0.082**
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Soya bean forage	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.086** **0.049**	<0.01 <0.01	<0.01 <0.01	**0.027** **0.033**
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.019** **0.020**	<0.01 <0.01	<0.01 <0.01	**0.039** **0.014**
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 **0.043**	<0.01 <0.01	<0.01 <0.01	**0.030** **0.025**
Soya bean hay	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.087** **0.066**	<0.01 <0.01	<0.01 <0.01	**0.033** **0.021**
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.084** **0.044**	<0.01 <0.01	<0.01 <0.01	**0.062** **0.020**
	270	<0.01	<0.01	<0.01	<0.01	<0.01	**0.016**	<0.01	<0.01	**0.037**

Sample	Anticipated PBI	fluidapyr	DMNgluc	3-HF	1-HMF [a]	1-HDMF [a]	1-CF [a]	PC	PCA	NDPCA
		<0.01	<0.01	<0.01	<0.01	<0.01	**0.092**	<0.01	<0.01	**0.018**
Soya bean seeds	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.058** **0.065**
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.073** **0.020**
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.028** **0.036**

Notes:

DMN-gluc=DesMet-N1-glucoside; 3-HF=3-OH-fluindapyr; 1-HMF=1-OH-Met-fluindapyr; 1-HDMF=1-OH-Met-N-DesMet-fluindapyr; 1-CF=1-COOH-fluindapyr; PC=pyrazole carboxamide; PCA = pyrazole carboxylic acid, NDPCA = N-DesMet-pyrazole carboxylic acid.

[a] Sum of both diastereomers.

[b] One of the single diastereomers is above the respective LOQ, but are below LOQ when combined.

[c] Mean of three analyses.

[d] Mean of four analyses.

Study 3

Field rotational crop studies were conducted in 2015-2016 in the United States to provide information on the uptake of fluindapyr and its metabolites in rotational crops [Schreier, 2018, 2015RES-IFP1902]. Representative crops from root and tuber vegetables (radish), small grain cereals (wheat), leafy vegetables (mustard) were used. Nine untreated control plots and nine treated plots were established with soya beans as the primary crop at each trial site. At the appropriate timing, the treated plots received two applications of test substance formulated as a water soluble liquid. Each application was at an actual rate of 124–128 g ai/ha per application (168 to 224 L/ha). The first application was scheduled to occur 35±2 days prior to typical harvest of soya bean seed with the second application occurring 12 to 15 days following the first application (21±2 days prior to typical harvest of soya bean seed). For the Nebraska site (trial number 01) the soya bean seed was harvested with a combine which removed the seed but allowed the plant debris to return to the field. At the Georgia site location (Trial 02) both the soya bean seed and the soya bean straw were removed from the field. Following removal of the primary soya bean crop, the plot was planted with the follow-on rotational crop of mustard, radish, or wheat at target plant back intervals of 30, 60, and 210 days following the last application (DALA).

Soil was characterized as silt loam (Trail 01 from Nebraska) and loamy sand (Trial 02 from Georgia), but specifics were not reported. No soil samples were taken. Samples of succeeding crops were harvested at their respective normal commercial harvest time (Table 67). Commodities were collected from at least 12 plants or from 12 different areas of the plot and stored frozen within 3 hours of harvest. Samples for plants included for tops (0.5 kg) and roots (24 roots, minimum of 2 kg) separately for radish, for cereal forage (1 kg), hay (0.5 kg), grain (1 kg) and straw (0.5 kg), for mustard (2 kg).

Table 67 Summary of PBI, DAT and DAS in field rotational crops (radish, mustard and wheat) grown in soil at planted 30, 60 and 210 days after a double treatment of fluindapyr to the primary crop soya bean at two field sites in the United States (Nebraska (01) and Georgia (02))

Samples and PBI		01-Nebraska (United States) 2 × 124-128 g ai/ha to soya bean as primary crop Silt loam		Samples and PBI		02-Georgia (United States) 2 × 124-128 g ai/ha to soya bean as primary crop Loamy and	
Rotational crop	PBI	Harvest DAT	Harvest DAS	Rotational crop	PBI	Harvest DAT	Harvest

Samples and PBI		01-Nebraska (United States) 2 × 124-128 g ai/ha to soya bean as primary crop Silt loam		Samples and PBI		02-Georgia (United States) 2 × 124-128 g ai/ha to soya bean as primary crop Loamy and	
sample		[a]	[a]	sample		[a]	DAS [a]
Radish root	30	76	44	Radish root	30	65	35
Radish leaves	30	76	44	Radish leaves	30	65	35
Mustard	30	68	36	Mustard	30	91	61
Wheat forage	30	296	264	Wheat forage	30	153	93
Wheat hay	30	296	264	Wheat hay	30	213	153
Wheat grain	30	334	302	Wheat grain	30	261	201
Wheat straw	30	334	302	Wheat straw	30	261	201
Radish root	60	90	37	Radish root	60	114	54
Radish leaves	60	90	37	Radish leaves	60	114	54
Mustard	60	90	37	Mustard	60	114	54
Wheat forage	60	268	291	Wheat forage	60	153	93
Wheat hay	60	300	289	Wheat hay	60	213	153
Wheat grain	60	345	303	Wheat grain	60	261	201
Wheat straw	60	345	303	Wheat straw	60	261	201
Radish root	210	248	42	Radish root	210	251	41
Radish leaves	210	248	42	Radish leaves	210	251	41
Mustard	210	248	42	Mustard	210	258	48
Wheat forage	210	248	42	Wheat forage	210	252	49
Wheat hay	210	281	75	Wheat hay	210	261	58
Wheat grain	210	328	122	Wheat grain	210	305	102
Wheat straw	210	328	122	Wheat straw	210	305	102

Notes:

PBI = Plant Back Interval; DAT = days after last application; DAS = Days after sowing.

[a] Calculated by the reviewer based on the data provided in the report.

The farming practices or environmental conditions did not adversely impact the residue trials of wheat, mustard and radish. The crops were grown and maintained per typical agricultural practices for each geographical region. The crop varieties selected were typical for commercial production in each area. The actual temperature and rainfall were generally within normal parameters during the residue study period. The trials received irrigation as required during the trial period. The maximum storage interval of the rotational crop samples, from harvest to extraction for analysis, was 717 days (frozen, ~24 months) for the analysis of fluindapyr residues and its metabolites.

All samples were analysed for residues of fluindapyr and the metabolites, 3-hydroxy-fluindapyr, 1-hydroxymethyl-fluindapyr, and 1-carboxy-fluindapyr. Residues of fluindapyr and 3-hydroxy- fluindapyr in study samples were determined using PTRL Europe method P3770G [Stanislowski, 2016a, 2015RES-IFP2155]. Residues of 1-OH-Met-fluindapyr and 1-COOH-fluindapyr in study samples were determined using the Isagro method RA.17.01 [Riccelli, 2017a, 2017RESIFP3206]. For both methods, the limit of quantitation (LOQ) was 0.01 mg/kg (expressed as fluindapyr equivalents). Mean procedural recoveries and relative standard deviations (RSDs) for fluindapyr, 3-hydroxyfluindapyr, 1-hydroyxmethyl-fluindapyr and 1-carboxy-fluindapyr in all crop matrices, at each fortification level, were within the range of 70–120 percent for recoveries and a RSD of ≤ 20 percent. No apparent residues were detected in any of the control samples used for fortification recovery above the method LOQ (expressed as fluindapyr equivalents) for all analytes.

The results show that average residues of fluindapyr in radish (tops and roots), mustard leaves, and wheat (forage, hay, straw and grain) reached a maximum of 0.022 mg/kg for residues of fluindapyr (210 days, radish tops). Average residues of 3-OH-fluindapyr were <LOQ for all RACs at all plant back intervals. Average residues if 1-OH-Met-fluindapyr reached a maximum of 0.012 mg/kg (60 days, wheat straw) and the maximum average residues of 1-COOH-fluindapyr reached 0.054 mg/kg (210 days, radish tops). The results are summarized in Table 68.

Table 68 Residues of fluindapyr and its metabolites in mg/kg parent equivalents in rotational crop samples of trials performed in Nebraska and Georgia (United States), respectively

Sample	PBI	Fluindapyr [a]	3-OH-fluindapyr [a]	1-OH-Met-fluindapyr [a]	1-COOH-fluindapyr [a]
Radish tops	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.016 [b]** <0.01
	60	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.012 [b]** <0.01
	210	**0.022** <0.01	<0.01 <0.01	<0.01 <0.01	**0.020** **0.054**
Radish roots	30	**0.022** <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	60	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	210	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Mustard	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	**0.018** <0.01
	60	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	210	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 [c] <0.01
Wheat forage	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	60	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	210	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Wheat hay	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	270	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Wheat grain	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	60	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	210	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Wheat straw	30	<0.01 <0.01	<0.01 <0.01	**0.010** **0.012 [b]**	<0.01 <0.01
	60	<0.01 <0.01	<0.01 <0.01	**0.012** <0.01	<0.01 <0.01
	210	<0.01 <0.01 [c]	<0.01 <0.01	<0.01 **0.010 [a]**	**0.013** <0.01

Notes:

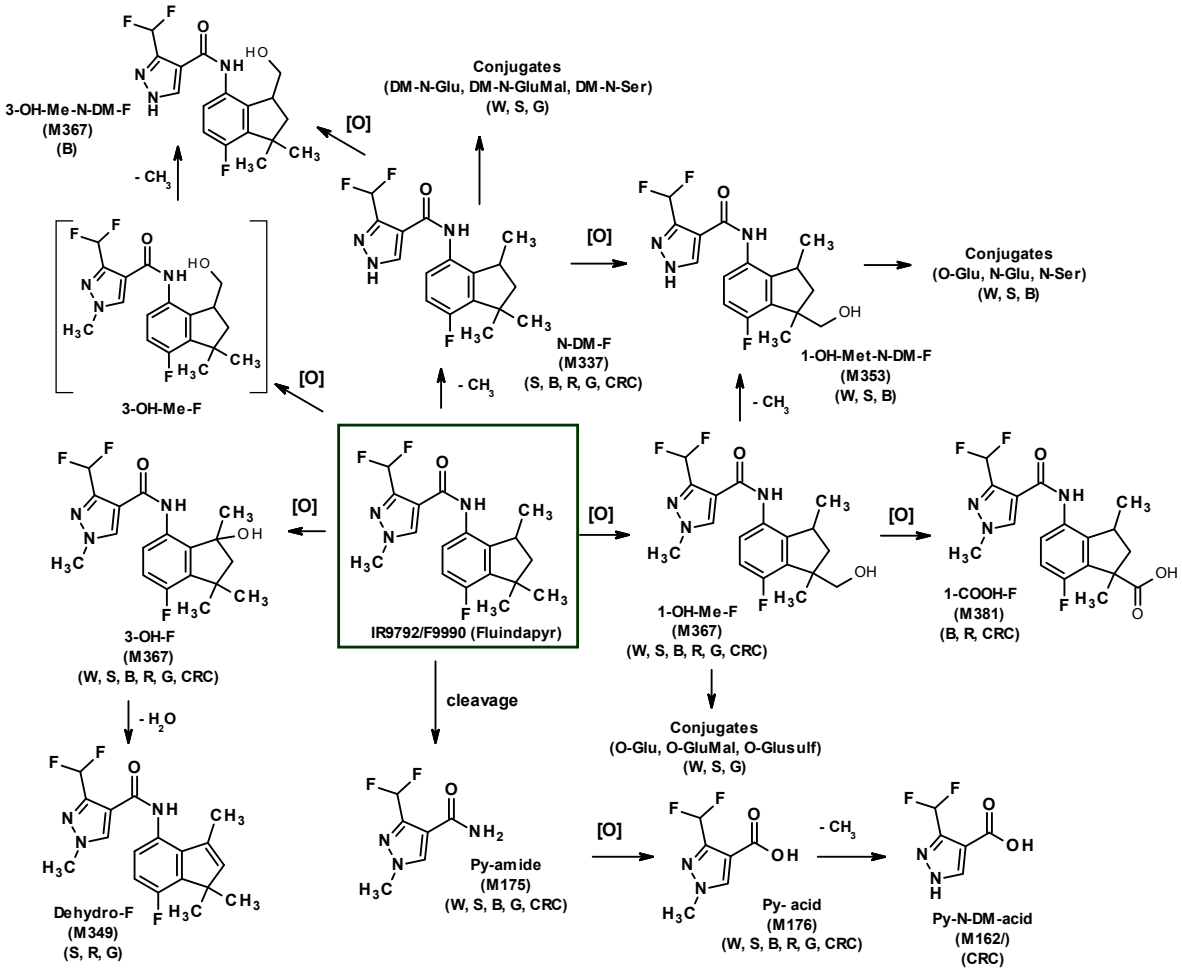
[a] Residues are means of two samples, expressed as parent equivalents.

[b] One of the duplicate samples had levels below LOQ, but combined the values are still above LOQ.

[c] One of the duplicate samples had levels above LOQ, but combined the values were below LOQ.

Overview of the metabolic pathway of fluindapyr in rotational crops

The proposed confined rotational crop metabolism (depicted in Figure 2 and again Figure 5) studies have been presented covering the crop categories root and tuber vegetables (carrots), leafy vegetables (lettuce), and cereal grains (wheat). Note that the figure below also includes the proposed metabolic pathways of the primary crops.



F: fluidapyr; Me: methyl; DM: desmethyl; py: pyrazole; Glu: glucoside; GluMal: malonylglucoside; Ser: serine; Glusulf: glucosyl sulfate
M: Mol. Wt.
Primary crops: W: wheat, S: soybean; B: sugarbeet; R: rice, G: grape
CRC: confined rotational crops (carrot, lettuce and wheat)

Figure 5 Metabolic pathway of fluindapyr in rotational crops

The enantiomeric ratio of the remaining fluindapyr slightly changed to 32/68 in both root and leaves, 42/58 in lettuce immature samples and 36/64 in lettuce mature samples. In wheat the S/R ratio was 36/64 in forage, 34/66 in hay and 27/73 in mature RACs (grain and straw). Generally a higher change in the enantiomeric ratio was observed in mature RACs than in the immature RACs. These conclusions are based on the confined rotational crop studies.

Parent was present in all commodities and the majority of the extractable metabolites were conjugated with the major (deconjugated) metabolites being 3-OH-fluindapyr, 1-COOH-fluindapyr, 1-OH-Met-fluindapyr, and N-DesMet-fluindapyr.

Based on the identified residues taken up from the soil by crops, and/or generated in the crops by further metabolism of those residues a metabolic pathway common to all rotated crops can be derived. The proposed metabolic pathway of rotational crops is similar to that in primary crops.

Metabolism in livestock

Lactating goats

The metabolism of fluindapyr was studied in lactating goats [Thomas, 2019a, 2015MET-IFP2176]. Two goats were orally dosed by capsule once daily for 7–8 consecutive days with either pyrazole-labelled fluindapyr or phenyl-labelled fluindapyr at 7.3 or 7.5 mg/kg feed/day, respectively. This is corresponding to 0.35 or 0.23 mg/kg bw/day, respectively. The actual dose received by the treated goats was based on the mean daily feed consumption of the treated goats over at least 3 days of acclimation (i.e. 1.9–2.0 kg feed/day). Goats were 7–9 years old, and body weights were 72 and 52 kg at the start of the study, and 81 kg and 48 kg at sacrifice, for the goats receiving phenyl-labelled and pyrazole-labelled fluindapyr respectively.

Goats were milked twice daily at regular intervals (in the morning prior to dosing and late afternoon). Milk was separated into aqueous and milk fat fractions for analysis. Urine and feces were collected twice daily. All goats were euthanized approximately 6 hours after the last dose. Select tissue samples were collected from each goat: liver, kidneys, loin and flank muscle, subcutaneous, renal and omental fat.

Milk was separated into cream and skimmed milk by centrifugation. Tissue samples and excreta were homogenized. All samples were stored frozen at about -20 °C until analysis. Initial sample analysis and metabolite profiling, along with LC-MS characterization/identification, were performed within 6 months of sample collection. Some samples (primarily liver, bile, urine, and feces) were re-extracted and/or re-analysed up to 15 months after collection for the confirmation of metabolite identification (qualitative analysis). In some cases, the metabolite profiles generated during the subsequent analyses were compared with the initial analyses to confirm that the profiles were qualitatively similar and no significant degradation of metabolites had taken place. Total radioactive residues (TRR) were determined by liquid scintillation counting (LSC).

Skimmed milk was extracted three times with ethyl acetate. All extracts were combined and analysed by LSC and LC-MS/MS. Composite fat samples (renal, omental, subcutaneous) and composite cream samples from several collection days were extracted by homogenization with hexane. After centrifugation, the hexane phase was separated and the remaining residue was extracted three times with acetonitrile. The hexane phase was extracted three times with acetonitrile as well, and all acetonitrile phases were combined and analysed by LSC and LC-MS/MS. Liver and kidney were extracted three times with acetonitrile followed by two extractions with an acetonitrile/water mixture (50:50; v/v). All extracts were combined and analysed by LSC and LC-MS/MS. Alternatively, three extractions with an acetone/water mixture (90:10; v/v) were conducted, followed by 2 extractions with acetone/water mixture (50:50; v/v). All acetone/water extracts were combined and analysed by LSC and LC-MS/MS. Composite muscle samples (loin and flank) were extracted three times with acetonitrile followed by two extractions with an acetonitrile/water mixture (50:50; v/v). All extracts were combined and analysed by LSC. The remainder was re-extracted three times with ethyl acetate. The combined ethyl acetate phases were concentrated and residues were determined by LSC and LC-MS/MS.

Liver extracts from the extraction process performed with acetone/water mixtures (see above) were lyophilized and re-suspended in acetate buffer at pH 5.0. Bile samples were diluted in acetate buffer at pH 5.0. Liver and bile samples were treated with β-glucuronidase by incubation for approximately 24 hours at 37 °C. Afterwards, the incubation was continued for an additional 21 hours with fresh β-glucuronidase. After incubation the whole samples were subjected to a C-18 SPE column clean-up. All fractions containing radioactivity were combined and residues were determined by LC-MS/MS.

Reference standards used were fluindapyr, 5'-OH-fluindapyr, 1-OH-Met-fluindapyr, 3-OH-Met-N-DesMet-fluindapyr, 3-OH-Met-fluindapyr, 3-OH-fluindapyr, 2-dehydro-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, 3-COOH-N-DesMet-fluindapyr, 2-OH-fluindapyr, 1-COOH-fluindapyr, 1-COOH-N-DesMet-fluindapyr, N-DesMet-fluindapyr, and DesMet-N-OH-fluindapyr.

The ratio of the two chiral isomers of the intact fluindapyr was determined in body fat and cream. Samples were extracted according to the same extraction procedure as mentioned above. The combined acetonitrile extracts were further processed to isolate the fluindapyr fractions on an HPLC. The combined fractions were partitioned into hexane. The hexane phase was analysed by a chiral HPLC method.

The overall recovery was approximately 76 percent of the total administered phenyl-labelled dose, while 94 percent was recovered of the total administered pyrazole-labelled dose. Total radioactivity recovered in the excreta (urine and faeces) accounted for 65–81 percent of the total dose. After sacrifice, 11–13 percent of the dose remained unextracted in the gastrointestinal (GI) tract. For the phenyl-label, 0.017 percent was recovered in milk and 0.34 percent in tissues at necropsy, with the highest amount in liver (0.27 percent). For the pyrazole-label, 0.088 percent of the total dose was found in milk, while 0.20 percent was recovered from tissues at necropsy, with 0.16 percent in liver.

The total residue levels determined in milk, both fat (cream) and aqueous fraction (skimmed milk), versus sampling times (AM and PM) are given in Table 69. The concentration in milk reached the maximum concentration by day 2 or day 3. Residues were higher in cream compared to skimmed milk. The concentrations in the PM samples (collected in the afternoon after capsule administration) were higher than the concentration in the AM sample collected just prior to administration of the next capsule.

The TRR, distribution, and identification of radioactivity are shown in Table 70 to Table 72. The highest TRR was observed in liver, followed by kidney. The TRRs in the different fat (renal, omental, subcutaneous) and muscle (loin, flank) samples were comparable, and these were pooled for further analysis. Extractability was at least 78 percent. Comparable radioactivity was extracted from liver and kidney using the two different extraction procedures (with acetone or acetonitrile). The PES from all matrices was found to contain <10 percent TRR or a low residue level, therefore, no further characterization of bound residue was necessary.

Fluindapyr was extensively metabolized, and no big differences between the metabolic profiles were observed between the phenyl and pyrazole label. Parent was the predominant compound with regard to the percent TRR in cream (0.045–0.057 mg/kg, 75-93 percent TRR), fat (0.024–0.042 mg/kg, 74–75 percent TRR), and muscle (0.004–0.006 mg/kg, 32–39 percent TRR), while it ranged from not detected to 8.4 percent TRR in the other samples. Glucuronidated metabolites were identified in kidney and liver; The primary metabolites identified in the liver were 1-OH-Met-fluindapyr and its glucuronides (up to 52 percent TRR and 0.13 mg eq/kg), 1-COOH-fluindapyr plus glucuronides (up to 27 percent TRR and 0.075 mg eq/kg), and 1-OH-Met-N-DesMet-fluindapyr plus glucuronides (up to 8.9 percent TRR and 0.025 mg eq/kg). In the kidney, 1-OH-Met-fluindapyr (up to 57 percent TRR and 0.059 mg eq/kg), and 1-OH-Met-N-DesMet-fluindapyr plus their glucuronides (up to 24 percent TRR and 0.029 mg eq/kg), were major metabolites with a notable concentration of 1-COOH-fluindapyr plus glucuronides (up to 11 percent TRR and 0.011 mg.kg eq). No individual metabolite in the remaining edible tissues (skimmed milk, cream,

fat, or muscle) exceeded 0.01 mg eq/kg in concentration. In skimmed milk, 1-OH-Met-fluindapyr and di-hydroxylated species represented a large part of the residue in terms of percent TRR (16 percent to 43 percent TRR), however, their individual concentrations were very low, ranging between <0.001 and 0.005 mg eq/kg. In muscle, 1-OH-Met-fluindapyr, found at the very low concentration of max. 0.006 mg eq/kg, represented the main metabolite in terms of TRR (up to 41 percent TRR).

The acid and enzymatic hydrolysis experiments, which were performed on selected samples, confirmed the identity of the glucuronidated metabolites. Chiral analysis of fluindapyr isolated from fat and cream, which showed high levels of unchanged parent compound compared to other tissues, showed that the S/R enantiomeric ratio changed from about 50/50, as observed in the dosing formulations, to 34/66 and 35/65 in milk and fat with the respective labels.

Table 69 Residue levels in skimmed milk and cream

	Skimmed milk		Cream	
	Phenyl-label	Pyrazole-label	Phenyl-label	Pyrazole-label
	mg eq/kg	mg eq/kg	mg eq/kg	mg eq/kg
Day 0 PM	0.005	0.007	0.048	0.072
Day 1 AM	0.004	0.006	0.030	0.049
Day 1 PM	0.010	0.014	0.070	0.088
Day 2 AM	0.005	0.010	0.044	0.048
Day 2 PM	0.009	0.014	0.051	0.062
Day 3 AM	0.005	0.010	0.028	0.036
Day 3 PM	0.009	0.014	0.055	0.057
Day 4 AM	0.006	0.008	0.032	0.040
Day 4 PM	0.009	0.014	0.079	0.082
Day 5 AM	0.005	0.009	0.034	0.041
Day 5 PM	0.009	0.015	0.062	0.067
Day 6 AM	0.005	0.009	0.032	0.040
Day 6 PM	0.010	0.013	0.062	0.060
Day 7 AM	n.a.	0.007	n.a.	0.029
Day 7 PM	n.a.	0.012	n.a.	0.055

Table 70 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in goat samples

	Skimmed milk [c]				Cream [d]				Fat			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR
TRR	0.010	100	0.012	100	0.060	100	0.061	100	0.056	100	0.033	100
Extracted	0.008	88	0.012	96	0.063	105	0.071	116	0.053	95	0.029	86
Fluindapyr	<0.001	0.1	<0.001	0.5	0.045	75	0.057	93	0.042	75	0.024	74
Tri-OH-dehydro-fluindapyr [a]	<0.001	5.0	0.001	5.6	-	-	-	-	-	-	-	-
Di-OH-fluindapyr [a]	0.003	28	0.005	43	-	-	-	-	-	-	-	-
1-OH-Met-N-DesMet-dehydro-fluindapyr	<0.001	2.3	0.001	11	-	-	-	-	-	-	-	-
1-OH-Met-N-DesMet-fluindapyr [b]	<0.001	1.9	<0.001	2.1	-	-	-	-	-	-	-	-
1-OH-Met-dehydro-fluindapyr	0.001	15	0.002	15	-	-	-	-	-	-	-	-
1-OH-Met-fluindapyr [b]	0.003	31	0.002	16	0.001	2.1	0.004	6.4	0.006	11	0.002	7.2
2-OH-fluindapyr	<0.001	5.1	<0.001	2.8	<0.001	0.2	0.001	1.2	0.001	1.1	0.001	2.5
N-DesMet-fluindapyr	-	-	-	-	0.001	2.0	0.003	4.7	0.002	3.7	0.001	3.9

3-OH-fluindapyr	-	-	-	-	0.001	1.5	0.002	3.0	0.002	3.0	0.002	4.8
Unknown [e]	-	-	-	-	0.012	19	-	-	0.001	2	-	-
Total identified or characterized	0.008	88	0.012	96	0.048	81	0.067	109	0.052	93	0.030	92
PES	No PES		No PES		0	0.5	0.000	0.4	0.002	3.9	0.001	4.2

Notes:

[a] Includes two separate metabolites.

[b] Sum of two diastereomers.

[c] For the phenyl-label, the milk sample from day 6 has been used, while for the pyrazole-label, the milk sample from day 7 has been used.

[d] For both labels, a composite sample of day 3, 5 and 6 has been used.

[e] Determined by calculation.

Table 71 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in goat samples

	Muscle				Liver (acetonitrile)				Liver (acetone)			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR
TRR	0.016	100	0.013	100	0.28	100	0.22	100	0.28	100	0.22	100
Extracted	0.017	103	0.013	98	0.24	88	0.19	86	0.23	82	0.17	78
Fluindapyr	0.006	39	0.004	32	0.023	8.4	0.001	0.3	0.007	2.7	0.001	0.3
Di-OH- fluindapyr [a]	0.001	8.6	0.001	9.4	-	-	-	-	-	-	-	-
1-OH-Met-N-DesMet-fluindapyr [b]	0.001	4.7	0.001	5.2	-	-	-	-	-	-	-	-
1-OH-Met-N-DesMet-fluindapyr glucuronide [a]	-	-	-	-	0.017	6.2	0.012	5.2	0.025	8.9	0.010	4.3
1-OH-Met-N-DesMet-dehydro-fluindapyr	<0.001	2.9	<0.001	0.8	-	-	-	-	-	-	-	-
1-OH-Met-fluindapyr [b]	0.006	41	0.004	34	0.13 [c]	47 [c]	0.11 [c]	52 [c]	0.12 [c]	42 [c]	0.086 [c]	39 [c]
1-COOH-fluindapyr [b]	-	-	-	-	0.075 [d]	27 [d]	0.057 [d]	26 [d]	0.055 [d]	20 [d]	0.045 [d]	21 [d]
2-OH-fluindapyr	0.001	8.5	0.001	6.6	-	-	-	-	0.003	1.0	0.003	1.3
N-DesMet-fluindapyr	<0.001	2.5	<0.001	2.5	-	-	-	-	-	-	-	-
3-OH-fluindapyr	0.001	7.4	<0.001	2.8	-	-	-	-	-	-	-	-
3-OH-Met-N-DesMet-fluindapyr	-	-	-	-	0.002	0.9	-	-	<0.001	0.1	-	-
Unknown [e]	-	-	0.001	4.0	-	-	0.01	2.0	0.02	7.0	0.02	12
Total identified or characterized	0.016	115	0.012	94	0.25	90	0.18	84	0.21	75	0.15	66
PES	0.001	3.3	0.002	14	0.016	5.6	0.017	7.6	0.020	7.4	0.024	11

Notes:

[a] Includes two separate metabolites.

[b] Sum of two diastereomers.

[c] Including glucuronides, for phenyl-label: 0.11 mg eq/kg, 38 percent TRR in acetonitrile-liver; 0.10 mg eq/kg, 36 percent TRR in acetone-liver; for pyrazole-label: 0.11 mg eq/kg, 49 percent TRR in acetonitrile-liver; 0.079 mg eq/kg, 37 percent TRR in acetone-liver.

[d] Including glucuronides, for phenyl-label: 0.020 mg eq/kg, 7.3 percent TRR in acetonitrile-liver; 0.016 mg eq/kg, 5.8 percent TRR in acetone-liver; for pyrazole-label: 0.013 mg eq/kg, 5.8 percent TRR in acetonitrile-liver; 0.018 mg eq/kg, 8.3 percent TRR in acetone-liver.

[e] Determined by calculation.

Table 72 Total Radioactive Residues (TRRs), identification and distribution of radioactivity in goat samples

	Kidney (acetonitrile)				Kidney (acetone)			
	Phenyl-label		Pyrazole-label		Phenyl-label		Pyrazole-label	
	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR
TRR	0.12	100	0.087	100	0.12	100	0.087	100
Extracted	0.11	89	0.078	89	0.10	86	0.077	88
Fluindapyr	-	-	0.001	0.7	-	-	-	-
N-DesMet-fluindapyr glucuronide [a]	0.004	2.8	0.005	6.3	0.003	2.3	-	-
N-DesMet-OH-fluindapyr glucuronide	0.001	0.9	0.001	0.7	0.001	0.6	-	-
1-OH-Met-N-DesMet-fluindapyr glucuronide [a]	0.023	19	0.011	13	0.029	24	0.010	12
1-COOH-fluindapyr [b]	0.011	11	0.010 [d]	9.4 [d]	0.010	8.4	0.005	5.3
1-OH-Met-fluindapyr [b] [c]	0.059	51	0.046	51	0.052	45	0.050	57
2-OH-fluindapyr	-	-	-	-	<0.001	0.3	-	-
Di-OH-N-DesMet-fluindapyr	<0.001	0.3	-	-	-	-	0.001	1.2
Unknown [e]	0.01	4.0	0.004	8.0	0.004	5.0	0.011	12
Total identified or characterized	0.10	85	0.074	81	0.096	81	0.066	76
PES	0.004	3.7	0.002	2.2	0.005	3.9	0.002	1.8

Notes:

[a] Includes two separate metabolites.

[b] Sum of two diastereomers.

[c] Including glucuronides, for phenyl-label: 0.055 mg eq/kg, 47 percent TRR in acetonitrile-kidney; 0.049 mg eq/kg, 42 percent TRR in acetone-kidney; for pyrazole-label: 0.044 mg eq/kg, 50 percent TRR in acetonitrile-kidney; 0.050 mg eq/kg, 57 percent TRR in acetone-kidney.

[d] Including glucuronides, for pyrazole-label: 0.001 mg eq/kg, 0.7 percent TRR in acetonitrile-kidney.

[e] Determined by calculation.

Laying hens

The metabolism and excretion of [phenyl-^{14}C]-fluindapyr or [^{14}C-5-pyrazole]-fluindapyr was investigated in laying hens [Thomas., 2019b, 2015MET-IFP2135]. The study consisted of a preliminary phase using 4 hens per label to determine the tissue collection time points for the main study phase after one single dose of 0.74–0.82 mg/kg body weight. In the main study ten hens per label group were used and 4 in the control group. Hens were 29–31 weeks of age and weighed between 1629 and 2135 g at the time of dosing. In both the preliminary and main test the test compound was orally administered using gelatine

capsules containing the ^{14}C-labeled compound mixed with cellulose at a cumulative dose of 4.95–6.42 mg/kg body weight (mean daily dose of 0.64–0.66 mg/kg bw). Based on the daily feed consumption, the dose level corresponded to *ca.* 10 ppm (mg ai/kg dry feed/day). The hens received 9 consecutive doses at 24-hour intervals in the morning and were sacrificed *ca.* 6 hours after the last dose. This time point was selected based on the fluindapyr-equivalent concentration levels in whole blood, where maximum concentrations were observed between 0.5 and 1 hours after dosing, being 0.31 and 0.35 µg/g for the phenyl and pyrazole label, respectively. By 12 hours post dose, the mean concentration in blood was ≤ 0.01 µg/g for both groups.

The eggs were collected twice daily; egg whites and yolks were combined for analysis. Following the last dose, the treated and control hens were sacrificed and muscle tissue (2 locations), fat tissue (3 locations), skin with adhering fat, partially formed shelled eggs, liver, and gastrointestinal tract with contents were collected. Total radioactive residue was measured in all eggs, tissues, and excreta by LSC (after combustion, depending on sample). All matrices were exhaustively extracted by organic solvent-water mixtures and the radioactivity remaining in post-extraction solids was quantified by LSC after combustion. The extracts were analysed by HPLC with radiometric and mass spectrometry detection to determine the metabolite distribution and nature of the incurred residue. Unchanged parent compound was isolated from representative extracts and analysed by chiral HPLC to determine the enantiomeric composition.

The overall recovery was 96.6 percent of the total administered dose. Until sacrifice, 93.2–95.7 percent of the total administered radioactivity (TAR) was excreted and only a very low amount of 0.113–0.117 percent TAR was quantified in the eggs. At the sacrifice, 6 hours after the 9th dose, the residue in the edible organs and tissues was very low and accounted for 0.146–0.158 percent TAR.

The TRR in the laid eggs ranged from 0.0073–0.0142 mg eq/kg (Day 1 AM) to 0.045–0.047 mg eq/kg (Day 8 AM). A gradual increase was observed until Day 6 when a residue plateau of ca. 0.05 mg eq/kg with both labels was reached.

The highest TRR in edible tissues was measured in the liver (0.107–0.117 mg eq/kg, 0.043 percent TAR) followed in decreasing order by those measured in fat (0.079–0.104 mg eq/kg, 0–0.043 percent TAR) and skin (0.044–0.057 mg eq/kg, 0.024–0.037 percent TAR). The lowest TRR was measured in muscle (0.010–0.013 mg eq/kg, 0.012–0.016 percent TAR). The TRR and the percent of total administered dose quantified in each tissue is summarized in Table 73.

Table 73 Distribution of total administered dose

Sample Description	Total radioactive residues			
	Phenyl-label		Pyrazole-label	
	percent of dose	**mg/kg**	**percent of dose**	**mg/kg**
Fat (renal)	0.000	0.079	0.001	0.085
Fat (omental)	0.038	0.094	0.043	0.098
Fat (Subcutaneous)	0.014	0.104	0.012	0.087
Liver	0.043	0.117	0.038	0.107
Muscle (breast)	0.012	0.010	0.012	0.010
Muscle (thigh and leg)	0.014	0.012	0.016	0.013
Skin	0.024	0.044	0.037	0.057
GI tract	3.284	2.427	2.739	1.985
Egg, day 1	0.005	0.0186	0.006	0.0296
Egg, day 2	0.009	0.0186	0.009	0.0346
Egg, day 3	0.009	0.0436	0.011	0.0444
Egg, day 4	0.009	0.0524	0.012	0.0541
Egg, day 5	0.014	0.0602	0.014	0.0658

Sample Description	Total radioactive residues			
	Phenyl-label		Pyrazole-label	
Egg, day 6	0.019	0.105	0.015	0.0742
Egg, day 7	0.017	0.082	0.020	0.0771
Egg, day 8	0.017	0.0819	0.018	0.0846
Egg, necropsy	0.015	0.0588	0.011	0.0593
Eggs, total	0.114	0.521	0.116	0.524
Excreta	89.4	137.1	92.8	129.6
Cage rinse	3.40	2.168	2.70	1.863
Cage wash	0.352	0.191	0.206	0.117
Total	**96.6**	142.9	**98.6**	134.6

Because the concentration of radioactivity in each tissue exceeded 0.01 ppm following dosing of both [phenyl-^{14}C]-fluindapyr and [^{14}C-5-pyrazole]-fluindapyr, sub-samples from each tissue type were extracted to characterize the nature of the radioactive residues, obtain metabolite profiles, and identify their molecular structure, where possible.

Fat and skin samples were extracted by homogenization with hexane. After centrifugation, the hexane phase was separated and the remaining residue was extracted three times with acetonitrile. The hexane phase was extracted three times with acetonitrile as well, and all acetonitrile phases were combined and analysed by LSC and LC-MS/MS.

Pooled shelled eggs were extracted by homogenization with hexane. After centrifugation, the hexane phase was separated and the remaining residue was extracted an approximate equivalent volume of acetone:DI water (9:1, [v/v]). This was repeated two times followed by 2 times with acetone:DI water (1:1, [v/v]). For the first extraction, the volume of each supernatant fraction was measured individually before pooling and the radio-concentration determined by LSC. All acetone:DI water extractions were pooled and evaporated in a rotary evaporator at room temperature. The remaining aqueous fraction was extracted 3 times with ethyl acetate. Ethyl acetate extracts and residual aqueous phase were quantified by LSC and residues identified by LC-MS/MS.

Liver samples were equally extracted as egg samples (see above). In addition, the aqueous fraction of liver samples contained a significant amount of radioactivity and was concentrated by evaporation with ACN, diluted, separated by solid phase extraction, concentrated and analysed by HPLC.

Muscle samples from the breast and thigh were pooled; approximate equal masses from each location were used in the pools. A portion of muscle was extracted by homogenization with of ACN. The extraction was repeated 2 additional times with ACN and 2 times with ACN:DI water (1:1, [v/v]) as the solvent. Radio-concentration was determined by LSC (first extraction only). All ACN and ACN:DI water (1:1, [v/v]) extractions were pooled and concentrated and the radioconcentration determined by LSC. The concentrated extracts were re-extracted from the aqueous phase with ethyl acetate, 3 times. The combined ethyl acetate phases were concentrated and residues were determined by LSC and LC-MS/MS.

Reference standards used were fluindapyr, 5'-OH-fluindapyr, 1-OH-Met-fluindapyr, 3-OH-Met-N-DesMet-fluindapyr, 3-OH-Met-fluindapyr, 3-OH-fluindapyr, 2-dehydro-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, 3-COOH-N-DesMet-fluindapyr, 2-OH-fluindapyr, 1-COOH-fluindapyr, 1-COOH-N-DesMet-fluindapyr, N-DesMet-fluindapyr, and DesMet-N-OH-fluindapyr.

The ratio of the two chiral isomers of the intact fluindapyr was determined in hen fat and skin. Samples were extracted according to the same extraction procedure as mentioned above. The combined acetonitrile extracts were further processed to isolate the fluindapyr fractions on an HPLC. The combined fractions were partitioned into hexane. The hexane phase was analysed by a chiral HPLC method.

Radioactive residues were efficiently extracted from eggs, edible organ and tissues (>89 percent of the TRR) prior to HPLC analysis. The PES were found to contain <10 percent TRR and ≤ 0.01 mg/kg; therefore, no additional work was conducted on the post-extraction residue. The distribution of radioactivity in eggs and edible tissues is presented in Table 74.

Table 74 Distribution of radioactivity in the extracts of eggs and edible tissues of hens following oral dosing with phenyl or pyrazole-labelled fluindapyr for 9 consecutive days at 10 ppm in feed

Sample	Eggs [a]		Muscle		Fat		Liver		Skin	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Label	Phenyl-label									
TRR (mg eq/kg)	0.059		0.011		0.096		0.117		0.044	
Total extracted	96.8	0.057	91.3	0.010	106	0.101	93.4	0.110	110	0.049
PES	3.4	0.002	7.5	0.001	0.3	0	7.0	0.008	7	0.03
Total recovery	100	0.059	99	0.011	106	0.102	100	0.118	118	0.052
Total analysed	87	0.051	89	0.010	96	0.092	85	0.100	110	0.049
Label	Pyrazole-label									
TRR (mg eq/kg)	0.059		0.011		0.095		0.107		0.057	
Total extracted	97.8	0.058	88.9	0.010	108.6	0.103	96.3	0.103	98.7	0.056
PES	5.2	0.003	5.5	0.001	0.45	0.000	9.5	0.010	1.5	0.001
Total recovery	103	0.061	94	0.011	109	0.103	106	0.114	100	0.057
Total analysed	81	0.048	90	0.010	103	0.098	98	0.105	97	0.055

Notes:

[a] At necropsy.

The parent compound, fluindapyr was the major residue identified in skin, fat, egg, and muscle extracts, ranging from 30.8 percent (egg) to 93.5 percent (skin) TRR. In liver, it represented only about 5 percent TRR. The concentration of fluindapyr was 0.041 mg eq/kg and 0.050 mg eq/kg in skin, 0.073 mg eq/kg and 0.090 mg eq/kg in fat, 0.028 mg eq/kg and 0.018 mg eq/kg in egg, 0.006 mg eq/kg and 0.005 mg eq/kg in liver, and 0.005 mg eq/kg and 0.005 mg eq/kg in muscle for Group 4 (phenyl label) and Group 5 (pyrazole label), respectively.

The N-DesMet-fluindapyr metabolite was the residue of greatest concentration in the liver and represented up to 62 percent of the TRR and 0.067 mg eq/kg. A minor fraction of N-DesMet-fluindapyr was present as sulfate conjugate (< 2 percent of TRR). The N-DesMet-fluindapyr metabolite was detected at much lower levels in the egg. (up to 0.004 mg eq/kg and 6.8 percent TRR), in fat (up to 0.002 mg eq/kg and 2.0 percent TRR), in skin (up to 0.001 mg eq/kg and 1.6 percent TRR), and in muscle (<0.001 mg eq/kg and 4.5 percent of TRR). The 2 diastereomers of 1-OH-Met-IR9792/F9990 and the corresponding conjugates 1-SO_4-Met-fluindapyr represented significant fractions of TRR in liver, at 22.2 percent TRR and 0.026 mg eq/kg, in egg, at 31.8 percent TRR and 0.019 mg eq/kg, and in fat, at 10.6 percent TRR and 0.010 mg eq/kg, and in muscle, at 14.5 percent TRR and 0.002 mg eq/kg. The 1-OH-Met-fluindapyr was a minor metabolite in skin, at 0.004 mg eq/kg and 9.8 percent TRR. The 1-COOH-fluindapyr

metabolite reached 12.1 percent TRR and 0.001 ppm in muscle and 7.2 percent TRR and 0.008 mg eq/kg in liver. The 2 diastereomers of 1-OH-Met-N-DesMet-fluindapyr and/or the corresponding conjugates 1 SO_4-Met-N-DesMet-fluindapyr reached 8.5 percent of TRR and 0.010 mg eq/kg in liver and 4 percent of TRR and <0.001 mg eq/kg in muscle.

Several minor metabolites, generally representing less than 10 percent TRR and always found at concentrations lower than 0.01 mg eq/kg, were identified/characterized in edible tissues from hen. Among them, 2-OH-fluindapyr, 5'-OH-fluindapyr, 3-OH-fluindapyr, and 3-OH-Met-fluindapyr were identified and in both phenyl and pyrazole extracts. A glycine conjugate of pyrazole carboxylic acid was detected at a trace level of 0.001 mg eq/kg (11.4 percent TRR) in muscle.

The metabolic profile of fluindapyr in eggs and the edible tissues of hens is presented in Table 75 and Table 76.

Chiral analysis of fluindapyr isolated from fat and skin, which showed the higher level of unchanged parent compound compared to other tissues, showed that the S/R enantiomeric ratio changed from about 50/50, as observed in the dosing formulations, to 20/80 (to 18:82 in fat (phenyl and pyrazole group) and to 23:77 in the skin (pyrazole group only)).

Table 75 Distribution of parent compound and metabolites in the extracts of eggs, fat and skin of hens following oral administration of [phenyl-^{14}C]-fluindapyr or [^{14}C-5-pyrazole]-fluindapyr for 9 consecutive days at 10 ppm ai in feed

Component / Sample	Eggs [a]		Eggs [a]		Fat		Fat		Skin		Skin	
	Phenyl		Pyrazole		Phenyl		Pyrazole		Phenyl		Pyrazole	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.059	100	0.059	100	0.096	100	0.095	100	0.044	100	0.057
Extracted	96.8	0.057	97.8	0.058	105.7	0.101	108.6	0.103	110	0.049	98.7	0.056
Fluindapyr [a]	48.2	0.028	30.8	0.018	76	0.073	94.9	0.090	93.5	0.041	88.3	0.050
1-OH-Met-fluindapyr [b]	26.1	0.015	31.8	0.019	10.6	0.010	1.3	0.001	9.8	0.004	5.2	0.003
N-DesMet-fluindapyr [c]	4.8	0.003	6.8	0.004	2.0	0.002	1.3	0.001	1.2	0.001	1.6	0.001
2-OH-fluindapyr	1.5	0.001	3.3	0.002	2.1	0.002	0.2	<0.001	0.9	<0.001	0.1	<0.001
OH-fluindapyr	-	-	1.5	0.00	-	-	-	-	-	-	-	-
3-OH-fluindapyr	1.0	0.001	0.7	<0.001	0.3	<0.001	0.6	0.001	0.5	<0.001	0.1	<0.001
5'-OH-fluindapyr	5.9	0.003	6.4	0.004	3.0	0.003	4.9	0.005	4.2	0.002	1.5	0.001
3-OH-Met-fluindapyr	n.d.	n.d.	0.1	<0.001	-	-	-	-	-	-	-	-
COOH-fluindapyr	-	-	-	-	2.0	0.002	-	-	-	-	-	-
1-COOH-fluindapyr	-	-	-	-	-	-	-	-	-	-	-	-
1-OH-Met-N-DesMet-fluindapyr [d]	-	-	-	-	-	-	-	-	-	-	-	
Total identified	87.5	0.051	81.4	0.049	95.9	0.093	103.2	0.1	110	0.050	96.8	0.057
Unknown/loss [c]	9.1	0.006	16.5	0.010	9.8	0.012	5.4	0.006	0.6	0.001	1.7	0.001
PES	3.4	0.002	5.2	0.003	0.3	0	0.4	0	7.4	0.003	1.5	0.001
Total recovery	100	0.059	103	0.062	106	0.102	109	0.11	118	0.054	100	0.059

Notes:

[a] Includes a glucuronide conjugate.

[b] Two diastereomers and their sulfate conjugates.

[c] Includes two minor sulfate conjugates.

[d] Result of 2 diastereomers present as a sulfate and a glucuronide conjugate.

Table 76 Distribution of parent compound and metabolites in the extracts liver and muscle of hens following oral administration of [phenyl-^{14}C]-fluindapyr or [^{14}C-5-pyrazole]-fluindapyr for 9 consecutive days at 10 ppm ai in feed

Component / Sample	Liver		Liver		Muscle		Muscle	
	Phenyl		Pyrazole		Phenyl		Pyrazole	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
TRR	100	0.117	100	0.107	100	0.011	100	0.011
Extracted	93.4	0.110	96.3	0.103	91.3	0.010	88.9	0.010
Fluindapyr [a]	5.4	0.006	4.7	0.005	45.1	0.005	45.8	0.005
1-OH-Met-fluindapyr [b]	22.2	0.026	18.8	0.020	14.5	0.002	6.4	0.001
1-COOH-fluindapyr [c]	7.2	0.008	4.7	0.005	12.1	<0.001	11.1	0.001
N-DesMet-fluindapyr [d]	37.5	0.044	62.2	0.067	4.5	<0.001	3.8	<0.001
2-OH-fluindapyr	0.8	0.001	0.3	<0.001	5.7	0.001	4.0	<0.001
OH-fluindapyr	1.2	0.001	1.2	0.001	-	-	-	-
3-OH-fluindapyr	0.3	<0.001	0.1	<0.001	0.3	<0.001	0.2	<0.001
5'-OH-fluindapyr	1.3	0.002	1.0	0.001	5.3	0.001	3.4	<0.001
3-OH-Met-fluindapyr	0.3	<0.001	0.0	<0.001	-	-	-	-
1-OH-Met-N-DesMet-fluindapyr [e]	8.5	0.10	4.0	0.004	1.8	<0.001	4.0	<0.001
Total identified	84.7	0.19	97	0.106	89.3	0.013	78.7	0.012
Unknown/loss [d]	7.7	0.008	-	0	2.2	0	-1.5	0.099
PES	7	0.008	9.5	0.010	7.5	0.001	5.5	0.001
Total recovery	100	0.117	107	0.114	99	0.011	94	0.11

Notes:

[a] Includes a glucuronide conjugate.

[b] Two diastereomers and their sulfate conjugates.

[c] Two diastereomers.

[d] includes two minor sulfate conjugates.

[e] Result of 2 diastereomers present as a sulfate and a glucuronide conjugate.

Overview of the metabolic pathway of fluindapyr in livestock

In goats the primary metabolic pathways for fluindapyr involve oxidation to form alcohols (a.o. 1-OH-Met-fluindapyr (diastereomers M24 and M26), 2-OH-fluindapyr (M27), Di-OH-fluindapyr) and carboxylic acids (1-COOH-fluindapyr (diastereomers M23 and M25)). A less pronounced metabolic pathway is N-demethylation of the parent to form N-DesMet-fluindapyr (M33) or further demethylation of the alcohols to form 1-OH-Met-DesMet-fluindapyr. The primary site of oxidation is at the 1-Met-fluindapyr position. The alcohols and carboxylic acids can also undergo extensive glucuronide conjugation. No sulfate conjugates were detected.

A shift of R/S-ratio of enantiomers was recognized for parent fluindapyr from a 50:50 ratio in the applied compound to a 35:65 ratio for S:R in body fat and cream (milk fat), indicating a higher metabolism rate for the S-enantiomer.

The primary metabolic pathways for fluindapyr in hens also involve N-demethylation (N-DesMet-fluindapyr (M33)) and oxidation to form alcohols (1-OH-Met-fluindapyr (diastereomers M24 and M26), 2-OH-fluindapyr (M27), and 5'-OH-fluindapyr (M35)). The 1-OH-Met-fluindapyr alcohols are further oxidized to form carboxylic acids (1-COOH-fluindapyr (diastereomers M23 and M25)). The 1-OH-Met-fluindapyr alcohols also undergo sulfation to form 1-SO_4-Met-fluindapyr sulfates (M41 and M42). These can

subsequently undergo demethylation to form 1-SO_4-Met-N-DesMet-fluindapyr (diastereomers M39 and M40).

Apart from the more extensive glucuronide conjugations in goat and sulfation steps in hens, the primary metabolic steps prior to glucuronidation or sulfation are similar for goat and hen.

The metabolic pathways of fluindapyr in goats and hens are depicted in Figure 6 and Figure 7. A combined overview of the metabolic pathways of fluindapyr (F9990/IR9792) in goat and hen is presented at the end of the evaluation (landscape required).

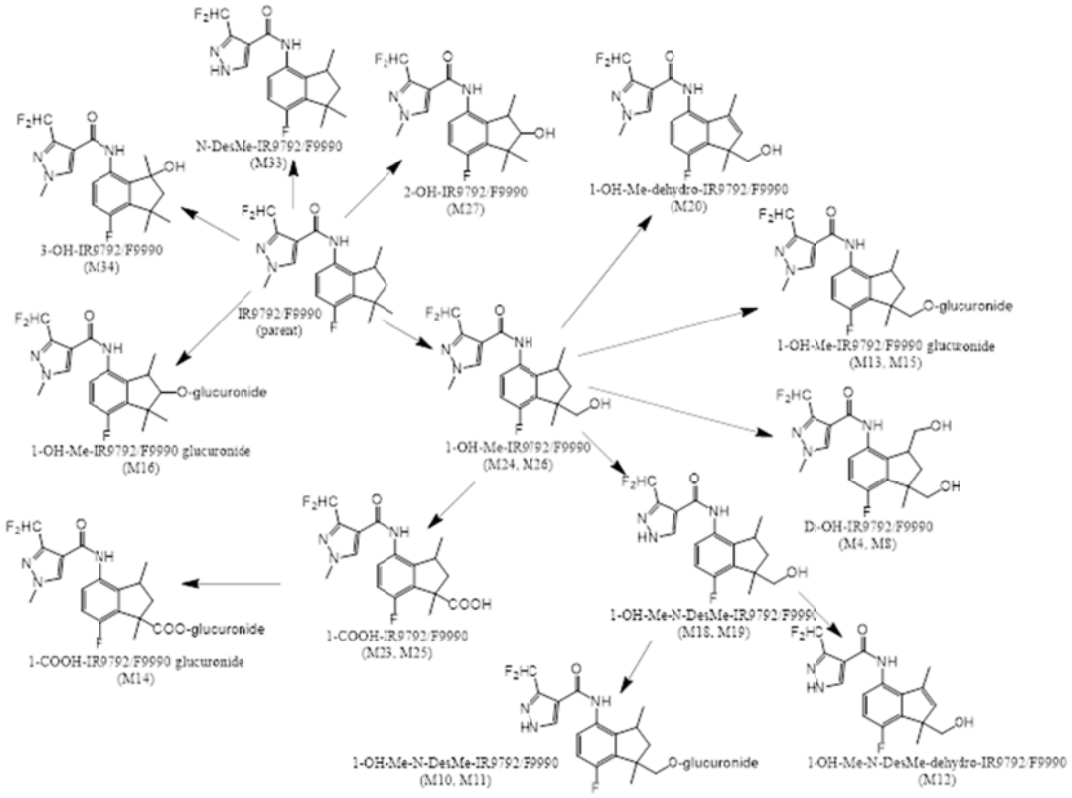


Figure 6 Proposed metabolic pathways of fluindapyr in goat

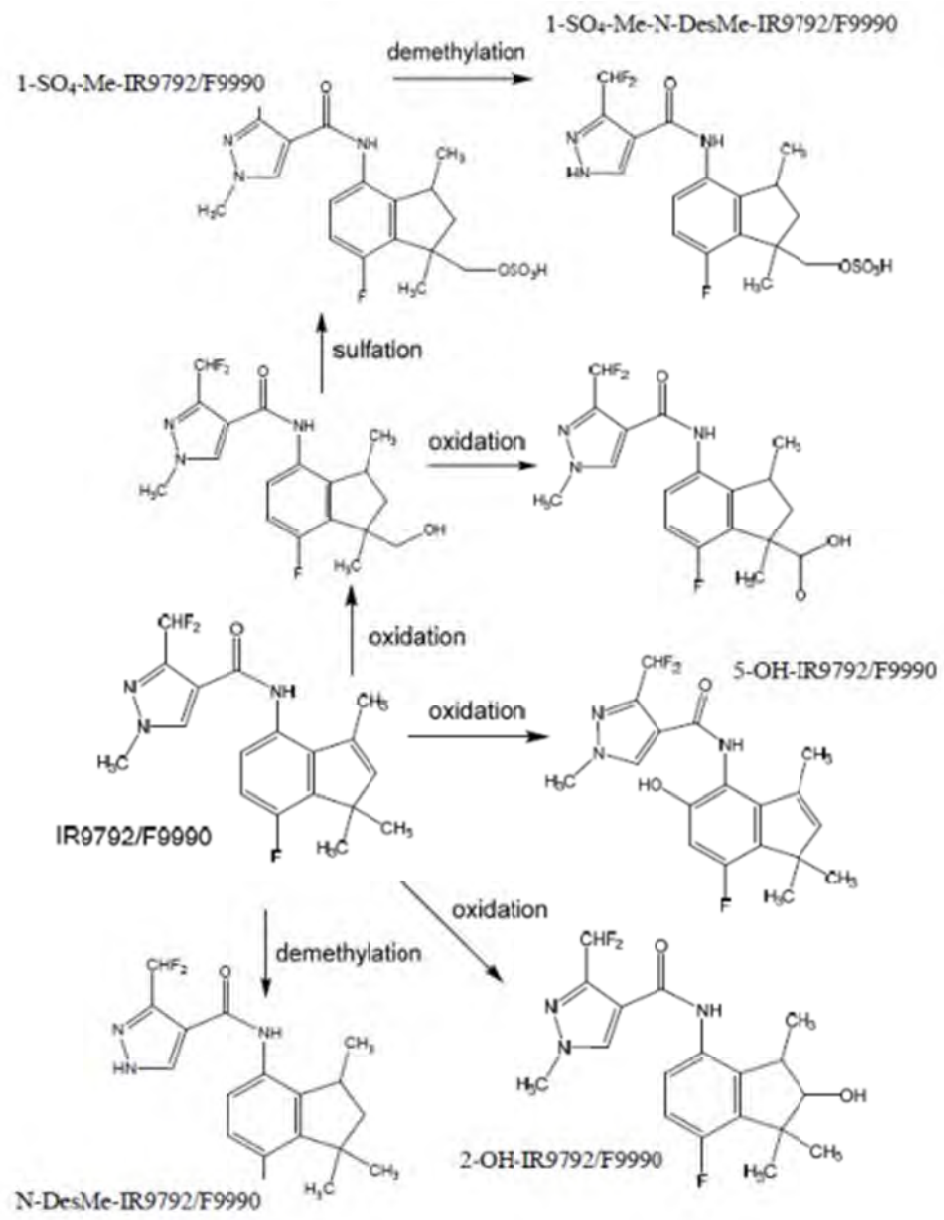


Figure 7 Proposed metabolic pathways of fluindapyr in hens

RESIDUE ANALYSIS

The Meeting received information on enforcement/monitoring methods for the determination of fluindapyr and its metabolites in plant and animal commodities. In addition the Meeting received information on analytical methods for the determination of fluindapyr and its metabolites as used in the various study reports (supervised residue trials, storage stability studies, processing studies, feeding studies). The analytical residue methods have been evaluated according to the guidance provided by OECD (Series on Pesticides number 39) as indicated on page 25 of the FAO manual 2009.

Radiovalidation for plant commodities

The extraction efficiency of the residue analytical methods PTRL P 3770 G and Isagro RA17.01 for fluindapyr and its metabolites was determined using radiolabelled samples from wheat and soya bean [Mainolfi&Garau, 2017, 2017RES-IFP3209]. Method PTRL P3770 is used for determination of parent fluindapyr, 3-OH-fluindapyr and DesMet-fluindapyr-N1-Glu. Method Isagro RA.17.01 is used for determination of 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluinpdayr, 1-COOH-fluindapyr, Pyrazole carboxylica acid, N-DesMet-pyrazole carboxylic-acid and pyrazole amide.

Radiolabelled samples were obtained from a wheat metabolism study [Mainolfi & Garau, 2016, 2013MET-IFP0694], soya bean metabolism study [Desai, 2016,2013MET-IFP0730] and two confined rotational crop metabolism studies [Mainolfi & Colombini, 2017, 2013MET-IFP0693; Vanini, 2013MET-IFP0717]. In these studies pyrazole-^{14}C- of phenyl-^{14}C-labelled fluindapyr was applied. The characteristics of the studies are summarized in the table below.

Table 77 Study characteristics of the studies used providing the radiolabelled samples

Study	Study type	Method used in study	Treatment and timing	Crop samples	PHI	Analyte
2013MET-IFP0694	PCM	MEF.13.14	2 × 125-130 g ai/ha or 2 ×601-625 g ai/ha at BBCH 31-33 and BBCH 65	Wheat forage	18-22	fluindapyr 3-OH-fluindapyr 1-OH-Met-fluindapyr 1-OH-Met-N-DesMet-fluindapyr
				Wheat grain	41-42	fluindapyr 3-OH-fluindapyr
				Wheat straw	41-42	fluindapyr 3-OH-fluindapyr 1-OH-Met-fluindapyr 1-OH-Met-N-DesMet-fluindapyr
2013MET-IFP0730	PCM	XBL 13027	3 × 125-130 g ai/ha at BBCH 15-16, BBCH 60, and BBCH 79, or 1 × 625 g ai/ha at BBCH 60	Soya bean hay	7 (after 2nd application)	DesMet-fluindapyr-N1-Gluc
2013MET-IFP0693	CRCM	MEF.13.08	366-375 g ai/ha to bare soil, 30, 120 and 300 days before planting	Wheat hay	n.a.	1-COOH-fluindapyr
				Wheat straw	n.a.	1-COOH-fluindapyr
				Wheat grain	n.a.	1-OH-Met-fluindapyr 1-COOH-fluindapyr
2013MET-IFP0717	CRCM	MEF.13.09	360-387 g ai/ha to bare soil 30, 120 and 300 days before planting	Wheat forage, straw and grain	n.a.	Pyr-acid N-DesMet-Pyr-acid Pyr-amide
				Wheat straw	n.a.	Pyr-acid N-DesMet-Pyr-acid Pyr-amide
				Wheat grain	n.a.	Pyr-acid N-DesMet-Pyr-acid Pyr-amide

Notes:

PCM = primary crop metabolism; CRCM = confined rotational crop metabolism; n.a. = not applicable because it is a rotational crop study.

The crop samples were collected during the in-life part of the relevant crop metabolism studies, homogenized and stored frozen. Wheat and soybean RACs were exhaustively extracted and analysed according to the methods of the metabolism studies. These RACs were also extracted and analysed by the residue analytical methods to check the extraction efficiency of the residues as reported for PTRL P 3770 G and Isagro RA.17.01. An overview of the extraction schemes and of the various relevant metabolites analysed with the different methods is given in Table 78 and Table 79.

Table 78 Extraction schemes of the various methods

Study	Method	Extraction	Partitioning	Samples
2013MET-IFP0694	MEF.13.14	Ultra-Turrax Forage and straw I: acetone:water 70:30 v/v II: acetone:water 50:50 v/v III: acetone:water 50:50 v/v IV: acetone V (straw only): hydrolysis by acetone/HCl (0.1N) 50:50 v/v → neutralized with 0.5 N HN_3 Grain I-III + step V, evaporated and solubilized in methanol	Extracts combined and evaporated. Aqueous phase extracted twice with n-heptane (A extract) and twice with ethyl acetate (B-extract incubated with HCl and C -aqueous phase) also incubated with HCl.	14 g wheat forage 13 g wheat straw 10 g wheat grain
2013MET-IFP0730	XBL 13027	Ultra-Turrax 3 × acetone:water 1-1 (v/v) 3 × methanol:water 1-1 (v/v/)	No partitioning, pooled extract (extract A) are evaporated and re-dissolved with CH_3OH-H_2O 1-1 (v/v) – Extract B	9 g soya bean hay
2013MET-IFP0693 and 2013MET-IFP0717	MEF.13.08 and MEF.13.09	Ultra-Turrax I: acetone:water 70:30 v/v II: acetone:water 50:50 v/v III: acetone:water 50:50 v/v IV: acetone	Extracts combined and evaporated. Aqueous phase extracted three times with n-heptane (extract A). 12 N HCl was added to the aqueous phase (extract B) for hydrolysis and neutralize with NH_3. Dried and resolved in methanol (extract C)	40-50 g wheat forage 20 g wheat straw 20-24 g wheat grain
	PTRL P 3770 G	Addition of water, same amount of acetonitrile, vigorous shaking 1 minute, centrifugation 5 minutes at 4000 rpm	Extract A	5 g wheat forage 2.5 g wheat straw 5 g wheat grain 2.5 g soya bean hay
	Isagro RA.17.01	Addition of water (shake), same amount of acetonitrile (shake), hydrolysis with HCl 4 N (80° C, 2 hrs) neutralization with 10 N NAOH, dilution with acetone and water followed by centrifugation	Extract A Cleanup on CHEMELUT cartridge (Extract B) Evaporation to dryness followed by redissolution with acetone-water 50-60 (v/v)	1 g wheat forage 1 g wheat straw 1 g wheat grain

Table 79 Overview of relevant metabolites covered by the different methods

Relevant residue	Residue method	Metabolism method	RACs
Fluindapyr	PTRL P 3770 G	MEF.13.14	wheat straw, wheat grain and wheat forage
3-OH-fluindapyr			
DesMet-fluindapyr -N1-Glu		XBL 13027	soybean hay
1-OH-Met-fluindapyr	Isagro RA.17.01	MEF.13.14	wheat straw and wheat forage
1-OH-Met-N-DesMet-fluindapyr			
1-COOH-fluindapyr		MEF.13.08	wheat straw, wheat grain and wheat forage
Pyr-acid		MEF.13.09	wheat straw, wheat grain and wheat forage
N-DesMet-Pyr-acid			
Pyr-amide			

All the extracts were assayed for radioactivity content by LSC and radio-chromatographic profiles were obtained by TLC or HPLC. The amounts of the residues obtained by residues and metabolism analytical methods were determined and the extraction efficiency as percentage of recovery (percentR) was calculated. The results of the radio-validation were reported in Table 80 and Table 81.

PTRL P 3770 G residue analytical method efficiently extracted fluindapyr, 3-OH-fluindapyr

and DesMet-fluindapyr-N1-Glu residues from crop matrices. Recovery was in the range 105 percent to 113 percent for fluindapyr, 81.7 to 110 percent for 3-OH-fluindapyr and amounted to 72.9 percent for DesMet-fluindapyr-N1-Glu.

Isagro RA.17.01 residue analytical method efficiently extracted 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, 1-COOH-fluindapyr, N-DesMet-Pyr-acid, Pyr-acid and Pyr-amide.

Recovery was in the range 79.0 to 86.7 percent for 1-OH-Met-fluindapyr, 95.0 to 95.3 percent for 1-OH-Met-N-DesMet-fluindapyr, 88.1 to 112 percent for 1-COOH-fluindapyr, 73.2 to 79 percent for N-DesMet-Pyr-acid, 73.4 to 85.5 percent for Pyr-acid and 72.2 to 81.4 percent for Pyr-amide.

Table 80 Radiovalidation of residue analytical method PTRL P 3770 G

Compound	RACs	Residue method (mg/kg) [a]		Metabolism method (mg/kg) [a]		percent R
Fluindapyr	wheat grain	PTRL P 3770G	0.0202	MEF.13.14	0.0193	105
	wheat forage		0.7901		0.7022	113
	wheat straw		3.4556		3.1423	110
3-OH-fluindapyr	wheat grain	PTRL P 3770G	0.0083	MEF.13.14	0.0102	81.7
	wheat forage		0.1255		0.1142	110
	wheat straw		1.4978		1.6789	89.2
DesMet-fluindapyr-N1-Glu	soybean hay	PTRL P 3770 G	0.1478	XBL 13027	0.2034	72.7

Notes:

[a] Data are expressed as mg/kg of fluindapyr equivalents per kg of plant material weight.

Table 81 Radiovalidation of residue analytical method Isagro RA.17.10

Compound	RACs	Residue method (mg/kg) [a]		Metabolism method (mg/kg) [a]		percent R
1-OH-Met-fluindapyr (a)	wheat forage	Isagro RA.17.01	0.6565	MEF.13.14	0.8331	79.0
1-OH-Met-fluindapyr (b)			0.3432		0.4329	
1-OH-Met-fluindapyr (a)	wheat straw		3.4556		3.1423	86.7

Compound	RACs	Residue method (mg/kg) [a]		Metabolism method (mg/kg) [a]		percent R
1-OH-Met- fluindapyr (b)			1.4978		1.6789	
1-OH-Met-N-DesMet-fluindapyr (a)	wheat forage	Isagro RA.17.01	0.0354	MEF.13.14	0.0356	95.3
1-OH-Met-N-DesMet-fluindapyr (b)			0.0358		0.0391	
1-OH-Met-N-DesMet-fluindapyr (a)	wheat straw		0.0318		0.0349	95.0
1-OH-Met-N-DesMet-fluindapyr (b)			0.0361		0.0366	
trans -1-COOH-fluindapyr	wheat grain	Isagro RA.17.01	0.0473	MEF.13.08	0.0608	88.1
cis -1-COOH-fluindapyr			0.0470		0.0463	
trans -1-COOH-fluindapyr	wheat forage		0.0201		0.0189	95.7
cis -1-COOH-fluindapyr			0.0096		0.0121	
trans -1-COOH-fluindapyr	wheat straw		0.0528		0.0448	112
cis -1-COOH-fluindapyr			0.0390		0.0369	
N-DesMet-Pyr-acid	Wheat grain	Isagro RA.17.01	0.0866	MEF.13.09	0.1183	73.2
	Wheat forage		0.0255		0.0328	77.7
	Wheat straw		0.1591		0.2014	79.0
Pyr-acid	Wheat grain	Isagro RA.17.01	0.2849	MEF.13.09	0.3331	85.5
	Wheat forage		0.0789		0.1024	77.0
	Wheat straw		0.2438		0.3323	73.4
Pyr-amide	Wheat grain	Isagro RA.17.01	0.0400	MEF.13.09	0.0491	81.4
	Wheat forage		0.0195		0.0270	72.2
	Wheat straw		0.0465		0.0585	79.4

Notes:

[a] Data are expressed as mg/kg of fluindapyr equivalents per kg of plant material weight.

The analytical methods used in the supervised trials use the following extraction methods:

- Method PTRL P 3770 G uses water & same amount of acetonitrile and 5 g (wheat forage or grain) or 2.5 g (wheat straw or soya bean hay) samples, shaking vigorously for 1 minute, centrifuge for 5 minutes at 4000 rpm.
- Method Isagro RA.17.01 uses water (shaking for 30 minutes), same amount of acetonitrile (shaking for 30 minutes) to extract 1 g of samples (wheat forage straw and grain), hydrolysis with HCl 4 N (80° C, 2 hours), neutralization with 10 N NaOH, dilution with acetone and water followed by centrifugation and clean-up on CHEMELUT cartridge, after drying re-dissolution with acetone-water 50–60 (v/v)

The extraction efficiency for these methods is shown to be sufficient.

Radiovalidation for animal commodities

A radiovalidation study was carried out to determine the extraction efficiency of SynTech Analytical Method 133SRUS16R0208 as a data collection method for determination of residues of fluindapyr, N-DesMet-fluindapyr, 1-OH-Met-fluindapyr, fluindapyr-1-carboxylate and 1-OH-Met-N-DesMet-fluindapyr in livestock matrices [Ray, 2018, 2016RES-IFP2944]. ^{14}C-labeled goat liver, muscle, milk-aqueous, milk-fat and ^{14}C-labeled hen fat and egg samples were obtained from two metabolism studies [Thomas, 2019a and 2019b, 2015MET-IFP2176 and 2015MET-IFP2135].

Extractions used in metabolism studies performed at Charles River Laboratories (CRL)

In the goat metabolism study aqueous milk samples were extracted 3 times with equivalent volumes of EtOAc, extracts were combined and concentrated and analysed by LSC and LC-MS/MS. Subsamples of other samples were extracted by polytron homogenization (Brinkmann Homogenizer).

Samples of goat fat, milk fat and hen fat were homogenized using a 2-fold volume of hexane; after shaking, sonification and centrifugation, supernatant was separated from PES. PES was extracted 3 times with acetonitrile; The radio-concentration in hexane and combined acetonitrile supernatants was determined by LSC. The hexane fractions were extracted 3 times with acetonitrile in equal volumes and the radioactivity of the combined extracts was determined by LSC. All acetonitrile fractions and extracts were combined, concentrated and analysed by LSC and LC-MS/MS.

Subsamples of goat liver and kidney were homogenized with equivalent volumes of acetonitrile; after sonification. shaking, and centrifugation, supernatant was separated from PES and PES was extracted another 2 times with acetonitrile and 2 times with acetonitrile/water (1:1, v/v/). All extractions were pooled and concentrated and radio-concentration determined by LSC.

Another set of subsamples of goat liver and kidney were homogenized with equivalent volumes of acetonitrile:water (9:1, v/v); after sonification. shaking, and centrifugation, supernatant was separated from PES and PES was extracted another 2 times with acetonitrile:water (9:1, v/v) and 2 times with acetone/water (1:1, v/v/). All acetone:water extractions were pooled and concentrated and radio-concentration determined by LSC. Subsequent liver and kidney extractions used the acetone:water methods.

Samples of goat muscle were homogenized with equivalent volumes of acetonitrile; after shaking, sonification, and centrifugation, supernatant was separated from PES and PES was extracted another 2 times with acetonitrile and 2 times with acetonitrile:water (1:1, v/v). All acetonitrile and acetonitrile:water extractions were pooled and concentrated and radio-concentration determined by LSC and LC-MS/MS.

Samples of eggs were homogenized using a 2-fold volume of hexane; after shaking, sonification and centrifugation, supernatant was separated from PES. PES was extracted with acetone:water (9:1, v/v) and 2 additional times with acetone:water (1:1, v/v); All acetone:water extracts were combined, concentrated. The remaining aqueous fraction was extracted 3 times with ethyl acetate. Ethyl acetate extracts and residual aqueous phase were quantified by LSC. The ethyl acetate fractions were pooled and concentrated under a steady stream of nitrogen and injected into HPLC for metabolite profiling and identification.

Hydrolysis of the glucuronides in goat liver and kidney samples was performed by addition of 10 N HCl and overnight incubation at 70 °C. Samples were neutralized using ammonium hydroxide and followed by SPE column clean-up. Fractions containing radioactivity were pooled, concentrated, and analysed by LSC and LC-MS/MS.

Enzyme hydrolysis of goat liver samples was performed on acetone:water extracts of liver by addition of β-glucuronidase. After SPE-clean-up the fractions containing radioactivity were concentrated and analysed by LC-MS/MS.

Extractions used in radio-validation study by Symbiotic Research LLC (Method 133SRUS16R0208)

For the radio-validation study subsamples (4 g) of ^{14}C-labeled goat liver and muscle and hen egg were weighed in triplicates into HDPE bottles (125 mL) and 50 mL of acetonitrile was added to each. The samples were shaken and centrifuged and the supernatants transferred to a graduated cylinder.

Acetonitrile:water (7:3) was added to the solids and the samples mixed and centrifuged again. The supernatants were combined and diluted acetonitrile.

Subsamples of (4 g) ^{14}C-labeled hen fat and goat milk fat were weighed in triplicates into HDPE bottles (125 mL) and 100 mL of acetonitrile:hexane (1:1) added to each. The samples were blended and the phases allowed to separate.

A subsample (4 g) of ^{14}C-labeled goat aqueous milk was weighed in triplicate into HDPE bottles (125 mL) and 40 mL of acetonitrile was added to each. The samples were shaken and centrifuged and the supernatants diluted to 50 mL with acetonitrile. Further extraction steps were followed depending on the analyte/matrix as detailed below.

In the radio-validation study the individual supernatants for each sample were radio-assayed by a liquid scintillation counter (Beckman LS6500) in triplicate. The samples were combusted in the previous goat and hen metabolism studies and the total radioactive residues (TRR) were determined.

For the current radio-validation a HPLC system was used to profile the sample extract and for profiling of the metabolites as described in the previous goat and hen metabolism studies. The HPLC system was also used as a means of comparing reference standard to ^{14}C-components by comparison of retention times.

For determination of fluindapyr and N-DesMet-fluindapyr, 4 mL methanol/water (1:3) was added to the 1 mL extract of each of the matrices (except fat and milk fat) before injection on the LC-MS/MS.

For determination of fluindapyr and N-DesMet-fluindapyr in milk fat and fat, water was added to the acetonitrile layer, mixed and cleaned up with SPE, dried, re-dissolved in MeOH/water (1:3) and injected onto the LC-MS/MS.

For determination of 1-OH-Met-fluindapyr, 1-COOH-fluindapyr and 1-OH-Met-N-DesMet-fluindapyr in goat liver, hen fat and eggs 4 NHL was added to the acetonitrile layer, mixed, incubated (60 min at 80 °C). After SPE clean-up, and drying, the samples were re-dissolved in MeOH/water (1:4) and injected onto the LC-MS/MS.

Comparison of the profiles of the extracted ^{14}C-labeled goat and hen samples showed that the retention times for the reference standards are shorter compared to the results of CRL, as different HPLC systems were used. Goat milk (aqueous and fat) profiles were similar using both methods. Parent fluindapyr is the only analyte of concern in milk and is only observed in the milk fat. The goat muscle profiles were similar across both methods with parent also being the only major analyte. The profiles for goat liver show parent and two major clusters of several peaks. These peaks were better resolved by Symbiotic Research LC system and consisted of the major metabolites 1-OH-Met-fluindayr and 1-COOH-fluindapyr. While 1-OH-Met-N-DesMet-fluindapyr was not considered a major metabolite in the goat liver, it was included in the analytical method due to the presence of liver as a representative matrix for kidney in the validation. The hen egg extracts show similar profiles between both methods with the parent and 1-OH-Met-fluindapyr being the major analytes observed. The hen fat profiles essentially exhibit one major peak for parent for both methods. Overall, similar profile patterns are exhibited for each extracted sample for CRL and Symbiotic Research. This demonstrates that the extraction used for data collection is capable of extracting the residues of concern from livestock matrices.

Table 82 Extraction efficiency and accountability of radioactivity

Matrix	Component	TRR by combustion or LSC (ppm) [a]	RR in sample extract by LSC (ppm) [b]	Extraction efficiency (%) [c]	HPLC/LSC (ppm) [a]	Determined by 133SRUS16R0208 (ppm) [d]	Accountability 133SRUS16R0208 [e]
Goat $milk_{Aq}$ [f]	fluindapyr	0.012	0.011	92	n.d.	n.d.	n.a.
Goat $milk_{Fat}$[g]	fluindapyr	0.055	0.046	84	0.057	0.013	23 percent
Goat muscle [h]	fluindapyr	0.013	0.013	100	0.004	<LOQ	n.a.
Goat liver [i]	fluindapyr	0.219	0.217	99	0.001	0.014	1400 percent
Hen egg [j]	fluindapyr	0.048	0.045	94	0.018	<LOQ	n.a.
Hen fat [k]	fluindapyr	0.098	0.081	83	0.0990	0.025	28 percent
Goat liver [i]	1-OH-Met-F [l]	0.219	0.217	99	0.086	0.095	110 percent
Hen egg [j]	1-OH-Met-F [l]	0.048	0.045	94	0.019	0.022	116 percent
Hen fat [k]	1-OH-Met-F [l]	0.098	0.081	83	0.001	<LOQ	n.a.
Goat liver [i]	1-OH-Met-N-DesMet-F [l]	0.219	0.217	99	0.010	0.009	90 percent
Goat liver [i]	1-COOH-F [l]	0.219	0.217	99	0.046	0.129	280 percent
Hen egg [j]	N-DesMet-F	0.048	0.045	94	0.004	<LOQ	n.a.

Notes:

F= fluindapyr; n.d. = not detected; n.a. = not analysed.

[a] Goat samples transferred from goat metabolism study [Thomas, 2019a, 2015MET-IFP2176] and hen samples transferred from hen metabolism study [Thomas, 2019b, 2015MET-IFP2135]; the total radioactive residues (TRR) were determined by combustion analysis of ^{14}C-labeled samples in both metabolism studies.

[b] LSC (ppm) values were determined by Symbiotic Research after extractions in current radio-validation study.

[c] Method extraction efficiency = (LSC results ÷ TRR values) × 100 percent.

[d] Averaged from triplicate analysis of ^{14}C-labeled samples using Analytical Method 133SRUS16R0208 LC-MS/MS analysis.

[e] Method accountability = (result from LC-MS/MS analysis ÷ results from HPLC beta ram detection/LSC) × 100 percent.

[f] Data from CRL Study [Thomas, 2019a, 2015MET-IFP2176] for Group 3: Day 7 (same sample sent to Symbiotic Research).

[g] Data from CRL Study [Thomas, 2019a, 2015MET-IFP2176] for Group 3: Days 3,5,6 (Day 7 sample sent to Symbiotic Research).

[h] Data from CRL Study [Thomas, 2019a, 2015MET-IFP2176] Group 3 (same sample sent to Symbiotic Research).

[i] Data from CRL Study [Thomas, 2019a, 2015MET-IFP2176] for Group 3 Acetone Extract, sum of metabolites and their glucuronides (same sample sent to Symbiotic Research).

[j] Data from CRL Study [Thomas, 2019b, 2015MET-IFP2135] Group 5, Necropsy (Day 8 sample sent Symbiotic Research).

[k] Data from CRL Study [Thomas, 2019b, 2015MET-IFP2135] Group 5 (same sample sent to Symbiotic Research).

[l] Expressed as sum of both isomers.

The method extraction efficiencies were determined to be: goat milk-aqueous (92 percent), goat milk-fat (84 percent), goat muscle (100 percent), goat liver (99 percent), hen egg (94 percent) and hen fat (83 percent). The full method accountability was variable due to the low absolute value of radioactivity present in some of the matrices and ranged from 23 percent to 1400 percent as determined by triplicate analyses of the goat and hen samples. The results demonstrate that the analytical method used for livestock residue analysis and tolerance enforcement is capable of extracting and analysing the analytes 1-OH-Met-F and 1-OH-Met-N-DesMet-F. The results do not demonstrate that the method used in the

dietary feedings studies is capable of extracting the main analyte of concern, being parent fluindapyr and 1-COOH-fluindapyr.

The analytical methods used in the dietary feeding studies [Brungardt, 2018, 2016RES-IFP2942 and Brungardt&Dixon 2018, 2016RES-IFP2943] use the following extraction methods:

- 133SRUS16R0208 [Moore& Shephard, 2018, as Appendix 3 of both feeding studies] Samples of muscle, liver, and egg were first extracted by blending with acetonitrile followed by extraction with acetonitrile/water (7:3). Extracts were pooled and diluted with acetonitrile. For analysis of fluindapyr and N-DesMet-fluindapyr a 1 mL sample was diluted with 4 mL of methanol/water (1:3), mixed and injected on LC-MS/MS. For analysis of 1-OH-Met-fluindapyr and 1-OH-Met-N-DesMet-fluindapyr, a 1 mL sample was mixed with 2.5 mL of 4 N HCL and incubated (80°C, 60 min). The samples were cleaned-up by SPE, concentrated and reconstituted in methanol/water (1:4) for injection on LC-MS/MS.
- Samples of fat were extracted with acetonitrile/hexane (1:1) and the phases were separated. For analysis of fluindapyr and N-DesMet-fluindapyr, part of the acetonitrile layer was mixed with water, cleaned-up by SPE, concentrated and reconstituted in methanol/water (1:4) for injection on LC-MS/MS.
- For analysis of 1-OH-Met-fluindapyr and 1-OH-Met-N-DesMet-fluindapyr, part of the acetonitrile layer mixed with 4 N HCL and incubated (80°C, 60 min), and subsequently cleaned-up, concentrated, reconstituted in methanol/water (1:4) for injection on LC-MS/MS.

Analytical methods for enforcement in plant commodities

Four methods are provided for the analysis of fluindapyr and/or metabolites in crops. The method selected for enforcement will depend on the compounds that are being analysed.

The first QuEChERS-based method [Stanislowski, 2016a, 2015RES-IFP2155] consists of a solvent/water extraction followed by analysis with LC-MS/MS. This method is to be used for analysis of fluindapyr plus metabolites 3-OH-fluindapyr and fluindapyr-DesMet-N-glucoside. An ILV of this study was conducted [Sahvorost, 2018a, 2017AMT-IFP3871] to qualify this procedure as an enforcement method.

While this method was validated for the metabolites 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr, corresponding radio-validation experiments demonstrated that the method was not suitable for the analysis of incurred residues and could only be used for the analysis of spiked residues. The validation data is included here to support the crop storage stability [Soddu, 2020, 2016RES-IFP2653] that was performed using this method.

A separate LC-MS/MS method [Riccelli, 2017a, 2017RES-IFP3206] was developed to analyse for fluindapyr metabolites 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr and should be used to analyse for incurred residues of these compounds. This method consists of solvent/water extraction followed by acid hydrolysis to hydrolyse sugar conjugates and allow for analysis of the metabolite aglycones.

An independent method validation of this procedure was conducted in [Sahvorost, 2018b, 2017AMT-IFP3872]. The acid hydrolysis method was further validated [Sahvorost, 2018b, 2017AMT-IFP3872] to include the common pyrazole metabolites pyrazole carboxamide, pyrazole carboxylic acid, and desmethyl-pyrazole carboxylic acid. A chiral method is also presented [Stanislowski, 2016b, 2016RES-IFP2666] to analyse the individual enantiomers of fluindapyr in crop matrices. Finally, a QuEChERS-based method [Soddu&Sicbaldi, 2014c, 2014-RES-IFP1239] is presented for the analysis of

fluindapyr in wheat matrices to support a storage stability study performed on wheat [Soddu, 2017, 2014RES-IFP1459].

LC-MS/MS method P3770G (fluindapyr, 3-OH-fluindapyr and fluindapyr-DesMet-N-glucoside)

A QuEChERS-based residue analytical method (including hydrolytic deconjugation) was developed and validated for determination of fluindapyr, and its metabolites 3-OH-fluindapyr, 1-OH-Met-fluindapyr (present in plants as a glucoside conjugate), 1-OH-Met-N-DesMet-fluindapyr (present in plant as a glucoside conjugate), 1-COOH-fluindapyr (presumably present as a glucoside conjugate) and the conjugate fluindapyr-N-DesMet-glucoside (stable to hydrolysis) in various crop materials, using LC-MS/MS for quantification and confirmation. The method was validated for sugar beet leaves (high water content), sugar beet root (high water and carbohydrate content), grapes (high acid content), soya bean seeds (high oil content), dry beans (high protein and starch content), and wheat straw (dry and difficult matrix) [Stanislowski, 2016a, 2015RES-IFP2155].

Initially parent fluindapyr and its metabolites/conjugates are extracted from various crop materials (sample size 5.0 g, except straw: 2.5 g) with a mixture of acetonitrile and water (about 1/1 v/v) by shaking vigorously for 1 min, followed by centrifugation. This extraction solvent composition is based on the initial extraction procedure of the European QuEChERS (EN 15662:2009-2) multi-residue method.

Before hydrolysis and phase separation (determination of fluindapyr, 3-OH-fluindapyr, and fluindapyr-N-DesMet-glucoside)

A small aliquot of the raw extract supernatant (acetonitrile/water about 1/1 v/v) is diluted with acetonitrile/water (1/1, v/v, containing 0.1 percent formic acid) for subsequent LC-MS/MS determination of fluindapyr, 3-OH-fluindapyr, and fluindapyr-N-desmethyl-glucoside, monitoring daughter ions for quantification and confirmation for all three analytes. The transitions are listed in Table 83.

After hydrolysis and phase separation (determination of 1-OH-Met-fluindapyr, l-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr)

In a second branch of the method, the raw extract with homogenized crop sample material still present is acidified with 10N HCl_{aq}, vortexed and hydrolysed at about 60° C for 1 hour. Then 10 N $NaOH_{aq}$ is added and the pH adjusted to slightly acidic (pH 4–6). The contents of the QuEChERS dispersive SPE (dSPE) citrate (buffer/salt) extraction tubes are added and the mixture is shaken vigorously for 1 min, followed by separation of the acetonitrile and water/salt phases supported by centrifugation.

For all matrices except straw and other dry, difficult matrices, a small aliquot of the hydrolysed upper acetonitrile extract is taken and diluted with acetonitrile/water (1/1, v/v, 0.1 percent formic acid) for subsequent LC-MS/MS determination of the deconjugated (by hydrolysis) metabolites 1-OH-Met-fluindapyr, l-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr, monitoring daughter ions for quantitation and confirmation for all three analytes. For straw a larger aliquot of the acetonitrile extracts obtained after hydrolysis and salt-induced phase separation is cleaned-up by liquid/liquid partition based on SPE for final LC-MS/MS determination of the deconjugated (by hydrolysis) metabolites 1-OH-Met-fluindapyr, l-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr. The transitions are listed in Table 83.

Table 83 Ion transitions

Analyte	MS/MS transition	
	Quantification	Confirmation
Fluindapyr	m/z 352 → 332	m/z 352 → 312
3-OH-fluindapyr	m/z 366 → 175	m/z 366 → 91
Fluindapyr-N-DesMet-glucoside	m/z 500 → 338	m/z 500 → 242

1-OH-Met-fluindapyr	m/z 368 → 310	m/z 368 → 330
1-OH-Met-N-DesMet-fluindapyr	m/z 352 → 312	m/z 352 → 332
1-COOH-fluindapyr	m/z 382 → 336	m/z 382 → 296 and m/z 382 → 281

The limit of quantitation (LOQ) was 0.010 mg/kg for all analytes, expressed as fluindapyr equivalents. The molecular weight (MW) ratios of the analytes in relation to the MW of parent fluindapyr resulted in individual LOQs for the analytes as listed below (Table 84):

Table 84 Overview of diastereomer ratio, molecular weights, LOQ and conversion factors

Analyte	Diastereomer ratio	Molecular Weight	LOQ (F-eq.)	LOQ (analyte)	Conversion Factor
Fluindapyr	n.a.	351.37	0.010	0.0100	1.000
3-OH-fluindapyr	n.a.	367.37	0.010	0.0105	1.046
Fluindapyr-N-DesMet-glucoside	n.a.	499.49	0.010	0.0142	1.422
1-OH-Met-N-DesMet-fluindapyr	1.28:1	353.35	0.010 (a+b) 0.0069 (a) 0.0031 (b)	0.010	1.046
1-OH-Met-fluindapyr	2.2:1	367.35	0.010 (a+b) 0.0056 (a) 0.0044 (b)	0.0109	1.006
1-COOH-fluindapyr	1.72:1	381.36	0.010 (a+b) 0.0063 (a) 0.037 (b)	0.0105	1.085

Notes:

n.a. = Not applicable

Calibration diagrams/functions obtained from injections of calibration solutions in matrix with at least 5 different concentrations were used to evaluate the diluted extracts. Matrix matched controls ranged from 0.0125 ng/mL (lowest in straw) to 0.50 ng/mL. Calibration functions were calculated and plotted by regression analysis. Regression coefficients (r) were > 0.99. In all cases linear regression was employed using the LC-MS/MS evaluation software, with one exception. For 3-OH-fluindapyr in soya bean the 366 m/z → 91 m/z mass transitions was evaluated using a slightly quadratic response/calibration curve. The validation results are summarized in Table 86.

A reduced validation for the analytes in dry crop matrices (soya bean seeds, dry bean, and wheat straw) was performed to prove that addition of water before fortification (full validation) has no impact on recovery results compared to addition of water after fortification (reduced validation) [Stanislowski, 2016a, 2015RES-IFP2155]. The reduced validation recovery results ranged from 70 to 107 percent with RSDs ranging from 0 to 10 percent per fortification level (n=3/fortification level) in the various matrices. Since the results are similar, they are not included in the tables below.

The method allows the determination and confirmation of all analytes with limits of quantification (LOQ) of 0.01 mg/kg, expressed as parent equivalents. No detectable analyte residues in any of the blank control samples were observed. A signal observed in the blank chromatogram of fluindapyr-*N*-DesMet-glucoside in soya bean at the retention time expected for the analyte was present at approximately 35 percent of the peak observed for the LOQ fortified sample. It was presumably caused by matrix interference and was not present in the confirmatory mass transition.

The analytical method P3770G [Stanislowski, 2016a, 2015RES-IFP2155] was subjected to independent validation for the determination of fluindapyr and metabolites 3-OH-fluindapyr, and

fluindapyr-N-glucoside in/on wheat straw (difficult matrix), wheat forage (high water content), wheat grain (high starch content), soybean seed (high oil content), grape (high acid content) and dry bean (high protein content) [Sahvorost, 2018a, 2017AMT-IFTP3871]. A sample aliquot was extracted once by shaking with water and acetonitrile. After centrifugation, the raw extract was diluted in acetonitrile/water (1/1, v/v) containing 0.1 percent formic acid prior LC-MS/MS analysis. The limit of quantification (LOQ) is 0.010 mg/kg for each analyte/matrix expressed as parent equivalents. Validation results are shown in Table 86 to Table 91.

LC-MS/MS method RA.17.01 (1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and 1-COOH-fluindapyr)

During the assessment of the extraction efficiency it was determined that for metabolites 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and 1-COOH-fluindapyr alteration of the original method P3770 G was required. An adjusted method was developed and validated for 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and 1-COOH-fluindapyr and their isomers in various wheat matrices [Ricelli, 2017a, 2017RES-IFP3206]. Samples were retrieved from different sources and included wheat dry gluten (high protein), grapes (high acid), oil seed rape (OSR) crude oil (high oil content), OSR straw (difficult matrices), OSR whole plant (high water content), wheat grain (high starch content) and wheat straw.

For this method water is added to the crop samples (1 g) and shaken for 30 minutes followed by addition of acetonitrile (10 mL) and shaking for 30 minutes (except for wheat straw where both acetonitrile and water are added at the same time and for dry gluten (high protein) where first acetonitrile is added followed by water). After addition of 37 percent hydrochloric acid (7 mL) samples are hydrolysed in a water bath at 80 °C for 2 hours, after cooling and pH adjustment (pH 4–5) with 10N NaOH (8 mL), acetone (25 mL) and water (20 mL) are added with mixing and sonification for 5 minutes, followed by centrifugation for 10 minutes at 4000 rpm. The sample is cleaned-up with a Chem Elute Cartridge (first 20 min soak) and elution with ethyl acetate (25 mL). The elution is and evaporated to dryness at 40 °C, reconstituted in 40:60, v/v, acetone/water (5 mL) with sonication and analysed by LC-MS/MS in positive ion mode (negative for 1-OH-Met-N-DesMet-fluindapyr) using a Phenomenex Kinetex 2.6µ C18 column (50 × 4.6 mm, 5 µm particle size), a SecurityGuard Ultra Cartridge UHPLC C18 guard column and gradient elution with mobile phases of 10mM ammonium acetate and 0.2 percent formic acid in water, and 0.2 percent formic acid in methanol. Calibration was performed using matrix matched external reference standards. The ion transitions monitored for quantification are included in Table 83.

Specificity and linearity was established. The limit of quantification for the method for all analytes in the tested matrices was 0.01 mg/kg (sum of diastereomers). The limit of quantification for the individual diastereomers is given in Table 84. Matrix effects for all analytes were found to be significant. Therefore matrix matched standards were used for all determinations. The stability of the calibration solutions and the final extracts was determined. The recovery findings of the individual diastereomers are included in Table 89, Table 90, and Table 91.

An independent method validation (ILV) for determination of 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and 1-COOH-fluindapyr was performed [Sahvorost, 2018b, 2017AMT-IFP3872]. Similar as in the primary method validation [Ricelli, 2017a, 2017RES-IFP3206] a sample aliquot was extracted by shaking with water and acetonitrile. After addition of concentrated hydrochloric acid, the raw extract was hydrolysed in a water bath at 80 °C for 2 hours with sonication. After cooling, neutralization with 10N sodium hydroxide solution, addition of acetone and water, sonication and centrifugation, the clean-up steps were carried out using a ChemElute cartridge. After evaporation, the remaining residues were reconstituted in acetone/water (40/60, v/v), mixed and sonicated prior to LC-MS/MS analysis. Matrix matched standards were used in the calibration ranges as indicated Table 85. The validation results for

the individual diastereomers are included in Table 89, Table 90, and Table 91. The recovery findings of the sum of the diastereomers were reported, but not included in the tables.

Table 85 Calibration ranges of matrix matched standards

Analyte	Calibration range (ng/mL)	Calibration range (mg/kg)
1-OH-Met-fluindapyr, diastereomer a	0.0225-1.12	0.0015-0.07
1-OH-Met-fluindapyr, diastereomer b	0.0175-0.877	0.0013-0.06
1-OH-Met-N-DesMet-fluindapyr, diastereomer a	0.0275-1.38	0.0021-0.09
1-OH-Met-N-DesMet-fluindapyr, diastereomer b	0.0125-0.625	0.0009-0.04
1-COOH-fluindapyr, diastereomer a	0.0253-1.26	0.0019-0.08
1-COOH-fluindapyr, diastereomer b	0.0147-0.735	0.0011-0.05

LC-MS/MS multi-residue method QuEChERS for determination of fluindapyr (RA.14.04)

[QuEChERS, 2007] is a multi-residue method of the Official Collection of Test Methods. The method describes the analytical procedures for the determination of pesticide residues in foods of plant origin with a low fat content such as fruits, vegetables, cereals and cereal products, herbs, spices, tea and tobacco using GC-MS and/or LC-MS/MS following acetonitrile extraction and clean-up by dispersive SPE. The method was validated for determination of fluindapyr in wheat grain, forage, hay and straw [Soddu&Sicbaldi, 2014c, 201RES-IFP1239].

The principle of the method is based on extraction in test tubes containing, anhydrous magnesium sulfate, sodium chloride, sodium citrate tribasic dihydrate, or sodium citrate dibasic sesquihydrate. A further purification is carried out in test tubes containing anhydrous magnesium citrate, PSA or C18. The mean recoveries of the tested levels (LOQ of 0.01 mg/kg (n=7) and 10 × LOQ at 0.1 m/kg (n=5)) were within 70–120 percent range, with percentRSD ranging from 1.2–7.9 per fortification level and for each matrix. The results are not included in the tables below.

Matrix effects were investigated for wheat grain, forage, grain, and wheat straw [Soddu&Sicbaldi, 2014b, 201RES-IFP1238] by comparing peak areas of solvent standard solution with peak areas of matrix matched standard solutions. Significant matrix effects (>20 percent) were observed for wheat hay (-24 to -26 percent) and wheat straw (-27 to -28 percent). For wheat grain and wheat forage, the matrix effects were reduced to -14 to -16 percent and -12 to -15 percent, respectively.

The stability of fluindapyr in wheat forage, grain, hay and straw extracts for two weeks under controlled storage conditions was determined [Soddu&Sicbaldi, 2014a, 2014RES-IFP1236]. Two spiking levels (LOQ and 10× LOQ) were tested. The mean recoveries were 75-80 percent in wheat grain (14 days), 86-99 percent in wheat forage (15 days), 72 percent in wheat hay (15 days) and 73-75 percent in wheat straw (16 days). In addition, the stability of fluindapyr in standard calibration solutions was determined [Soddu&Sicbaldi, 2014d, 2014RES-IFP1240]. Concentrations of 0.001, 0.005 and 0.01 mg/L were tested over a period of 0–90 days. Uncorrected recoveries ranged from 75–106 percent. Recoveries corrected for recovery in fresh solution ranged from 76–117 percent.

Enantioselective (chiral) RP-HPLC-MS/MS method (P 3928 G) for determination of fluindapyr

A QuEChERS-based residue analytical method for the enantio-selective (chiral) determination of fluindapyr in various crop materials was developed and validated, with a target limit of quantitation (LOQ) of 0.010 mg/kg, using chiral reversed-phase (RP-)HPLC/MS/MS for quantitation and confirmation [Stanislowski, 2016bb, 2016RES-IFP2666].

The analytical method was validated for oilseed rape plant (high water content), grape (high acid content), wheat grain (dry, high starch content), oilseed rape seeds (high oil content), dry bean (high protein content) and straw (difficult matrix), obtained locally or from a related field study. The analytical method is derived from the QuEChERS (EN 156621) multi-residue method. Fluindapyr residues were extracted from various crop materials (sample size 5.0 g, except straw:2.5 g) with a mixture of acetonitrile and water (about 1/1, v/v) by shaking vigorously for 1 minute. After addition of $MgSO_4$, NaCl and buffering citrate salts (pH 5–5.5), the mixture is shaken intensively and centrifuged for phase separation. After freezing out fat (only for oilseed rape matrix), an aliquot of the organic extract was cleaned-up by dispersive SPE with PSA and $MgSO_4$. An aliquot of the raw extract supernatant was further diluted with acetonitrile/water (1/1, v/v, containing 0.1 percent formic acid) for subsequent chiral RP-HPLC/MS/MS determination of the two enantiomers of fluindapyr, monitoring daughter ions for quantitation and confirmation for both enantiomers.

Chiral separation was achieved on a Daicel Chiralcel OX-3 (250 mm × 4.6 mm i.d., 3 µm particle size) column, operated at 25 °C with a mobile phase consisting of aqueous 20 mM ammonium hydrogen carbonate (pH 9 adjusted with ammonia) and acetonitrile (containing 0.1 percent water).

For method validation, homogenized plant/crop samples were fortified (5 replicates per matrix and fortification level) at LOQ (0.01 mg/kg) and at 0.10 mg/kg, or 0.005 and 0.05 mg/kg per enantiomer. In addition, unfortified samples were used as blank controls. Residues in all blank control specimens were below 30 percent of the LOQ (<0.003 mg/kg). The average recoveries (n=5 per fortification level, per enantiomer, per matrix), for the two parent-daughter ion transitions monitored ranged from 95 percent to 110 percent with relative standard deviations (RSD) ≤10 percent and were within the acceptable range of 70-110 percent, with RSD ≤20 percent. The results are not included in the tables below.

Note by the reviewer:

LC-MS/MS method P3370G is considered

- valid (full validation (n=5) for the determination of fluindapyr, 3-OH-fluindapyr, fluindapyr-DesMet-N-glucoside in the range 0.01-0.1 mg/kg in sugar beet leaves (high water content), sugar beet root (high starch content), grapes (high acid content), almond, pecan and soya bean seeds (high oil content), dry beans (high protein content), and wheat grain (high starch content), wheat forage (high water content) wheat straw and (dry and difficult matrix) .
- valid (reduced validation (n=3) for the determination of fluindapyr, 3-OH-fluindapyr, fluindapyr-DesMet-N-glucoside in the range 0.01-0.1 mg/kg in maize and sorghum forage and stover, maize grain, wheat hay, and reduced validation at 0.01 mg/kg in cabbage, carrot roots and leaves and radish roots and leaves, soya bean hay and forage.
- Limited recovery experiments (n=1-2) suggest validity of method P3370G for the determination of fluindapyr, 3-OH-fluindapyr, fluindapyr-DesMet-N-glucoside in the range 0.01-0.1 mg/kg in maize AGF, flour, forage, grits, meal, oil and starch, as well as I mustard greens, peakon nutmeat, sorghum AGF and flour, sweet corn K+CWHR, forage, and stover. In addition, limited recovery experiments with fluindapyr were available at higher levels in almond hulls (5.0 and 15 mg/kg), maize forage (7.8 mg/kg), maize stover (0.2-2.0 mg/kg), sorghum forage (0.25-16.0 mg/kg), grain (0.25-8.0 mg/kg), stover (0.40-2.0 mg/kg) and AGF (1.0-26.0 mg/kg), sweet corn forage (1.0-10.0 mg/kg) and stover (1.0-20.8 mg/kg), wheat forage (1.0-20 mg/kg), wheat grain (1.0 mg/kg), wheat hay (1.0-20 mg/kg), and wheat straw (1.0-20 mg/kg). Similarly, incidental limited recoveries were available for some metabolites.

LC-MS/MS method RA.17.01 is considered

- valid (full validation (n=5) for the determination of the (sum of) diastereomers 1-OH-Met-fluindapyr in almond and pecan nutmeat (high oil content), almond hulls, dry beans (high protein content), grapes (high acid content), soya bean seed (high protein content), sugar beet roots (high starch content) and sugar beet leaves and wheat forage (both high water content), wheat straw, wheat grain and dry gluten (high starch)
- valid (full validation (n=5) for the determination of 1-OH-Met-N-DesMet-fluindapyr and 1-COOH-fluindapyr in the range 0.01-0.1 mg/kg (sum) in dry beans and soya bean seeds (high protein content), grapes (high water content), oil seed rape (OSR) crude oil (high oil content content), OSR whole plant (high water content), OSR straw, grapes (high acid content), sugar beet leaves (high water content) and roots (high starch content), wheat forage (high water content), wheat grain (high starch content), wheat dry gluten (high protein content), wheat hay and wheat straw.
- valid (reduced validation (n=3) for the determination of the (sum of) diastereomers 1-OH-Met-fluindapyr was available for concentration level 0.01 mg/kg (sum) in cabbage immature, carrot roots and leaves, maize grain, maize forage (also 0.1 mg/kg), radish leaves, radish roots, sorghum forage, sorghum grain and stover (both also 0.1 mg/kg), soya bean forage and hay, tomato and wheat hay.
- valid (reduced validation (n=3) for the determination of the (sum of) diastereomers 1-OH-Met-N-DesMet-fluindapyr and 1-COOH-fluindapyr was available for concentration level 0.01 mg/kg (sum) in in cabbage immature, carrot roots and leaves, radish roots and leaves, soya bean forage and hay, and tomato.
- Limited recovery experiments (n=1-2) suggest validity of method RA.17.01 for the determination of the (sum of) diastereomers 1-OH-Met-fluindapyr in the range 0.01-0.1 mg/kg in almond nutmeat, almond hulls, maize AGF, maize flour, maize grits, maize meal, oil, starch and stover, mustard greens sorghum AGF and flour, sweet corm K+CWHR, forage, stover
- Limited recovery experiments (n=1-2) suggest validity of method RA.17.01 for the determination of the (sum of) diastereomers 1-COOH-fluindapyr in the range 0.01-0.1 mg/kg in mustard greens and wheat hay.

Table 86 Validation result for fluindapyr with LC-MS/MS methods RA.17.01 or P3770G

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
Parent									
Almond nutmeat	0.01	0.01 0.1	5 5	109 90	107-111 88-91	1.6 1.3	<LOD (5) [a]	matrix matched standards 0.050-25 ng/mL 1/× weighted R^2 > 0.995 m/z 352 → 256	Webber, 2017a, 2016RES-FNF2450 (P3770G)
	0.01	0.01 0.1	5 5	113 88	111-116 86-91	2.1 2.4	<LOD (5) [a]	m/z 352 → 312	
Almond nutmeat	0.01	0.01 0.1	2 2	112 103	96, 128 87, 118	- -	<LOD (5) [a]	Concurrent recoveries field trials	Webber, 2017a, 2016RES-FNF2450 (RA.17.01)
Almond hulls	0.01	0.01 0.1	5 5	105 86	98-109 85-87	4 1	<LOD (5) [a]	matrix matched standards 0.050-25 ng/mL	Webber, 2017a, 2016RES-

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
								1/× weighted R^2 > 0.995 m/z 352 → 256	FNF2450 (P3770G)
	0.01	0.01 0.1	5 5	113 90	107-120 89-91	4.5 0.9	<LOD (5) [a]	m/z 352 → 312	
Almond hulls	0.01	0.01 0.1 5.0 15	2 2 1 2	110 97 - 107	110, 110 97, 97 97 105, 108	- -	<LOD (5) [a]	Concurrent recoveries field trials	Webber, 2017a, 2016RES-FNF2450 (RA.17.01)
Cabbage immature	0.01	0.01 0.1	3 1	102 -	95-109 100	6.9 -		m/z 352 → 312	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	99 -	90-107 112	8.5 -		m/z 352 → 332	
Carrot leaves	0.01	0.01 0.1	3 1	97 -	95-98 87	2.1 -		m/z 352 → 312	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
Carrot leaves	0.01	0.01 0.1	3 1	95 -	94-96 86	1.2 -		m/z 352 → 332	
Carrot roots	0.01	0.01 0.1	3 1	107 -	107-107 113	0.5 -		m/z 352 → 312	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	106 -	105-107 111	0.9 -		m/z 352 → 332	
Dry bean	0.01	0.01 0.1	5 5	91 87	87-94 85-89	3 2	<0.01 (3)	Matrix matched standards 0.025-5.0 ng/mL 1/× weighed r ≥0.99 m/z 352 → 332	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	89 85	87-91 82-90	1 3		m/z 352 → 312	
Dry bean	0.01	0.01 0.1	5 5	87 86	84-95 84-87	4.3 1.3	<0.01 (3)	6 matrix matched standards 0.050-5.0 ng/mL 1/× weighted r > 0.998 m/z 352 → 332	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	88 86	85-95 85-87	4.0 0.9	<0.01 (3)	m/z 352 → 312	
Grape	0.01	0.01 0.1	5 5	104 106	99-108 99-113	4 5	<0.01 (3)	Matrix matched standards 0.050-5.0 ng/mL 1/× weighed r ≥0.99 m/z 352 → 332	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	101 106	900-106 100-110	6 4		m/z 352 → 312	
Grape	0.01	0.01 0.1	5 5	102 101	98-105 99-103	2.7 2.0	<0.01 (3)	6 matrix matched standards 0.050-5.0 ng/mL 1/× weighted r > 0.998	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
								m/z 352 → 332	
	0.01	0.01 0.1	5 5	106 100	100-105 97-102	2.0 2.3	<0.01 (3)	m/z 352 → 312	
Maize AGF	0.01	0.01 0.1 1.0	1 1 1	- - -	107 101 127	- - -	<LOD (3)	Concurrent recoveries field trials	Webber, 2018a, 2015RS-FNF-1900 (P 3370G)
Maize flour	0.01	0.01 0.1	1 1	-	101 94	- -	<LOD (2)	Concurrent recoveries field trials	
Maize forage	0.01	0.01 0.1 8.0 15.5	2 2 1 1	84 80 - -	77, 90 75, 85 100 87	- -	<LOD (6)	Concurrent recoveries field trials	
Maize germ	0.01	0.01 0.1	1 1	- -	97 91	- -	<LOD (2)	Concurrent recoveries field trials	
Maize grain	0.01	0.01 0.1	2 2	100 101	103, 97 105, 96	- -	<LOD (4)	Concurrent recoveries field trials	
Maize grits	0.01	0.01 0.1	1 1	- -	84 93	- -	<LOD (2)	Concurrent recoveries field trials	
Maize meal	0.01	0.01 0.1	1 1	- -	129 66	- -	<LOD (2)	Concurrent recoveries field trials	
Maize oil	0.01	0.01 0.1	1 1	- -	94 92	- -	<LOD (2)	Concurrent recoveries field trials	
Maize starch	0.01	0.01 0.1	1 1	- -	141 119	- -	<LOD (2)	Concurrent recoveries field trials	
Maize stover	0.01	0.01 0.1 0.2 2.0	2 2 1 1	111 109 - -	115, 106 113, 105 100 123	- - - -	<LOD (5)	Concurrent recoveries field trials	
Maize forage	0.01	0.01 0.1 7.8 [c]	3 3 1	104 97 -	103-106 94-102 100	1.5 4.4 -	<LOD (7)	Concurrent recoveries field trials	Webber, 2018b 2016RES-FNF-2453
Maize grain	0.01	0.01 0.1	3 3	94 94	83-103 90-97	11 3.7	<LOD (6)	Concurrent recoveries field trials	
Maize stover	0.01	0.01 0.1 0.2 [c] 2.0 [c]	4 4 1 1	101 92 - -	80-115 85-97 100 123	15 5.6 - -	<LOD (9)	Concurrent recoveries field trials	
Mustard greens	0.01	0.01 0.1	2 2	113 106	109, 117 103, 108	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Pecan nutmeat	0.01	0.01 0.1	5 5	96 89	94-97 87-90	1.4 1.3	<0.5LOQ (5) [a]	matrix matched standards 0.050-25 ng/mL 1/× weighted	Webber, 2017a, 2016RES-FNF2450

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
								R^2 > 0.995 m/z 352 → 256	(RA.17.01)
	0.01	0.01 0.1	5 5	98 88	95-100 86-90	1.9 1.7	<0.5LOQ (5) [a]	m/z 352 → 312	
Pecan nutmeat	0.01	0.01 0.1	2 2	106 98	104, 107 89, 106	- -	<0.5LOQ (5) [a]	Concurrent recoveries field trials	Webber, 2017a, 2016RES-FNF2450 (RA.17.01)
Radish leaves	0.01	0.01 0.1	2 2	114 102	108, 120 98, 106	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Radish leaves	0.01	0.01 0.1	3 1	107 -	101-113 120	5.6 -		312 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	105 -	102-108 118	2.9 -		332 m/z	
Radish roots	0.01	0.01 0.1	2 2	115 100	111, 119 97, 104	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Radish roots	0.01	0.01 0.1	3 1	106 -	97-111 113	7.1 -		312 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	104 -	95-110 114	7.6 -		332 m/z	
Sorghum forage	0.01	0.01 0.10 0.25 16.0	2 2 1 1	118 113 - -	111, 124 112, 113 85 90	- - - -	<LOD (5)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (P3770G)
Sorghum forage	0.01	0.01 0.10 0.25 15.8	3 3 1 1	103 103 - -	73-124 84-113 85 90	26 16 - -	<LOD (7)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF-2455 (P3770G)
Sorghum grain	0.01	0.01 0.10 0.25 8.0	2 2 1 1	85 100 - -	80, 90 89, 111 80 135	- - - -	<LOD (5)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (P3770G)
Sorghum grain	0.01	0.01 0.10 0.25 8.0	3 3 1 1	103 106 - -	80, 140 89-118 80 135	31 14 - -	<LOD (7)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF-2455 (P3770G)
Sorghum stover	0.01	0.01 0.10 0.40 0.50	2 2 1 1	107 - - -	100, 114 103 91 112	- - - -	<LOD (5)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
		2.0	1	-	130				(P3770G)
Sorhum stover	0.01	0.01 0.10 0.40 0.50 2.0	3 2 1 1 1	106 108 - - -	100-114 103-112 91 112 130	- - - - -	<LOD (7)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF-2455 (P3770G)
Sorghum AGF	0.01	0.01 0.10 1.0 26	1 1 1 1	- - - -	127 112 102 85	- - - - -	<LOD (4)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (P3770G)
Sorghum flour	0.01	0.01 0.10 3.0	1 1 1	- - -	87 135 78	- - -	<LOD (3)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (P3770G)
Soya bean forage	0.01	0.01 0.1	3 1	91 -	88-94 102	3.3 -		312 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	96 -	95-97 98	3.3 -		332 m/z	
Soya bean hay	0.01	0.01 0.1	3 1	82 -	79-87 89	5.6 -		312 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	84 -	83-84 87	0.7 -		332 m/z	
Soya bean seeds	0.01	0.01 0.1	5 5	91 90	82-102 80-98	8 7	<0.01 (3)	m/z 352 → 332	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	92 88	85-97 78-95	5 8		m/z 352 → 312	
Soya bean seeds	0.01	0.01 0.1	5 5	85 84	83-87 82-86	1.4 1.3	<0.01 (3)	6 matrix matched standards 0.050-5.0 ng/mL 1/× weighted r > 0.998 m/z 352 → 332	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5				<0.01 (3)	m/z 352 → 312	
Sugar beet leaves	0.01	0.01 0.1	5 5	110 108	107-114 98-115	3 6	<0.01 (3)	Matrix matched standards 0.025-5.0 ng/mL 1/× weighed r ≥0.99 m/z 352 → 332	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	112 11	107-116 107-114	3 2		m/z 352 → 312	
Sugar beet roots	0.01	0.01 0.1	5 5	100 101	94-107 99-106	5 3	<0.01 (3)	Matrix matched standards 0.025-5.0 ng/mL 1/× weighed r ≥0.99 m/z 352 → 332	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01	5	96	91-102	5		m/z 352 → 312	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
		0.1	5	100	92-102	5			
Sweet corn K+CWHR	0.01	0.01 0.1 1.0	2 1 1	99 - 	87, 111 89 99	- - -	<LOD (3)	Concurrent recoveries field trials	Webber, 2018c, 2016RES-FNF2454 (P 3770 G)
Sweet corn forage	0.01	0.01 0.1 1.0 10	2 1 1 1	108 - - -	100, 116 107 103 86	- - - -	<LOD (3)	Concurrent recoveries field trials	
Sweet corn stover	0.01	0.01 0.1 1.0 20.8	2 1 1 1	105 - - -	96, 113 102 112 81	- - - -	<LOD (3)	Concurrent recoveries field trials	
Tomato mature	0.01	0.01 0.1	3 1	99 -	92-105 107	6.6 -		312 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	98 -	92-103 108	5.7 -		332 m/z	
Wheat forage	0.01	0.01 0.1	6 6	114 112	111-118 106-117	2.1 3.2	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Wheat forage	0.01	0.01 0.1	5 5	96 91	92-101 89-92	3.0 1.3	<0.01 (3)	6 matrix matched standards 0.0125-2.0 ng/mL 1/× weighted r > 0.998 m/z 352 → 332	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	96 91	93-101 90-91	3.8 0.5	<0.01 (3)	m/z 352 → 312	
Wheat forage	0.01	0.01 0.10 1.0 20	4 3 1 1	100 97 - -	92-112 92-101 112 94	8.6 4.6 - -	<LOD (9)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)
Wheat grain	0.01	0.01 0.1	7 7	112 107	72-113 72-125	17 16	<LOD (6)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Wheat grain	0.01	0.01 0.1	5 5	107 100	105-111 98-102	2.4 4.1	<0.01 (3)	6 matrix matched standards 0.0125-2.0 ng/mL 1/× weighted r > 0.998 m/z 352 → 332	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	107 101	105-109 97-103	1.5 2.2	<0.01 (3)	m/z 352 → 312	
Wheat grain	0.01	0.01 0.10 1.0	4 3 1	103 92 -	98-106 90-94 113	3.3 2.3 -	<LOD (8)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)
Wheat hay	0.01	0.01	2	106	99, 112	-	<LOD (4)	10 matrix matched	Schreier,

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
		0.1	2	102	92, 113	-		standards 0.10-25 ng/mL 1/× weighted r > 0.990	2018, 2015RES-IFP1902 (P3770G)
Wheat hay	0.01	0.01 0.10 1.0 20	4 3 1 1	112 104 - -	105-118 100-104 104 79	5.4 3.4 - -	<LOD (9)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)
Wheat straw	0.01	0.01 0.1	6 6	106 103	94-118 99-113	9.4 5.0	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Wheat straw	0.01	0.01 0.1	5 5	102 106	98-106 97-114	3 6	<0.01 (3)	Matrix matched standards 0.0125-5.0 ng/mL 1/× weighed ≥0.99 m/z 352 → 332	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	102 103	93-109 100-106	6 2		m/z 352 → 312	
Wheat straw	0.01	0.01 0.1	5 5	93 89	89-99 85-93	4.2 3.3	<0.01 (3)	6 matrix matched standards 0.0125-2.0 ng/mL 1/× weighted r > 0.998 m/z 352 → 332	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	96 90	87-101 84-95	5.4 4.0	<0.01 (3)	m/z 352 → 312	
Wheat straw	0.01	0.01 0.10 1.0 20	4 3 1 1	108 90 - -	93-114 86-93 99 85	9.2 3.9 - -	<LOD (9)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)

Notes:

[a] At least one control (untreated field sample) per trial was analysed for each matrix.

Table 87 Validation result for 3-OH-fluindapyr with LC-MS/MS methods RA.17.01 or P3770G

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
3-OH-fluindapyr									
Almond nutmeat	0.01	0.01 0.1	5 5	86 78	81-91 74-81	4.3 4.2	<LOD (5) [a]	matrix matched standards 0.050-25 ng/mL 1/× weighted R^2 > 0.995 m/z 366 → 175	Webber, 2017a, 2016RES-FNF2450 (P3770G)
	0.01	0.01 0.1	5 5	86 79	79-91 76-83	5.8 4.1	<LOD (5) [a]	m/z 366 → 131	
Almond	0.01	0.01	2	86	72, 99	-	<LOD (5)	Concurrent	Webber,

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
nutmeat		0.1	2	87	78, 96	-	[a]	recoveries field trials	2017a, 2016RES-FNF2450 (RA.17.01)
Almond hulls	0.01	0.01 0.1	5 5	82 71	77-89 70-75	5.6 3.7	<LOD (5) [a]	matrix matched standards 0.050-25 ng/mL 1/× weighted R^2 > 0.995 m/z 366 → 175	Webber, 2017a, 2016RES-FNF2450 (P3770G)
	0.01	0.01 0.1	5 5	85 75	82-91 72-76	4.6 2.2	<LOD (5) [a]	m/z 366 → 131	
Almond hulls	0.01	0.01 0.1 5.0	2 2 1	102 105 -	97, 107 99, 111 94	- - -	<LOD (5) [a]	Concurrent recoveries field trials	Webber, 2017a, 2016RES-FNF2450 (RA.17.01)
Cabbage immature	0.01	0.01 0.1	3 1	1047 -	102-112 109	4.6 -		366 → 175 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	109 -	99-116 113	8.1 -		366 → 91 m/z	
Carrot leaves	0.01	0.01 0.1	3 1	106 -	103-110 91	3.3 -		366 → 175 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	105 -	102-110 91	3.7 -		366 → 91 m/z	
Carrot roots	0.01	0.01 0.1	3 1	94 -	92-97 89	2.6 -		366 → 175 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	892 -	88-95 89	3.7 -		366 → 91 m/z	
Dry beans	0.01	0.01 0.1	5 5	78 72	73-81 70-75	4 3	<0.01 (3)	Matrix matched standards 0.0125-5.0 ng/mL m/z 366 → 175	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	82 75	78-86 73-77	4 2	<0.01 (3)	m/z 366 → 91	
Dry beans	0.01	0.01 0.1	5 5	89 84	87-95 82-86	3.4 1.5	<0.01 (3)	6 matrix matched standards 0.050-5.0 ng/mL 1/× weighted r > 0.998 m/z 366 → 175	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	90 86	86-90 84-89	4.1 2.0	<0.01 (3)	m/z 366 → 91	
Grapes	0.01	0.01 0.1	5 5	93 108	84-101 102-112	7 4	<0.01 (3)	Matrix matched standards 0.050-5.0 ng/mL 1/× weighed r ≥0.99 m/z 366 → 175	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	98 107	87-106 92-119	7 9	<0.01 (3)	m/z 366 → 91	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
Grapes	0.01	0.01 0.1	5 5	103 97	101-104 94-98	1.4 1.9	<0.01 (3)	6 matrix matched standards 0.050-5.0 ng/mL 1/× weighted r > 0.998 m/z 366 → 175	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	103 97	100-106 95-99	2.0 1.5	<0.01 (3)	m/z 366 → 91	
Maize AGF	0.01	0.01 0.1 1.0	1 1 1	- - -	126 113 118	- - -	<LOD (3)	Concurrent recoveries field trials	Webber, 2018a, 2015RS-FNF-1900 (P 3370G)
Maize flour	0.01	0.01 0.1	1 1	- -	104 83-	- -	<LOD (2)	Concurrent recoveries field trials	
Maize forage	0.01	0.01 0.1	2 2	94 94	106, 82 93, 94	- -	<LOD (4)	Concurrent recoveries field trials	
Maize germ	0.01	0.01 0.1	1 1	- -	109 109	- -	<LOD (2)	Concurrent recoveries field trials	
Maize grain	0.01	0.01 0.1	2 2	101 91	108, 94 92, 90	- -	<LOD (4)	Concurrent recoveries field trials	
Maize grits	0.01	0.01 0.1	1 1	- -	70 78	- -	<LOD (2)	Concurrent recoveries field trials	
Maize meal	0.01	0.01 0.1	1 1	- -	112 68	- -	<LOD (2)	Concurrent recoveries field trials	
Maize oil	0.01	0.01 0.1	1 1	- -	98 102	- -	<LOD (2)	Concurrent recoveries field trials	
Maize starch	0.01	0.01 0.1	1 1	- -	122 112	- -	<LOD (2)	Concurrent recoveries field trials	
Maize stover	0.01	0.01 0.1 0.2 2.0	2 2 1 1	87 92 - -	92,82 91, 92 91 92	- - - -	<LOD (5)	Concurrent recoveries field trials	
Maize forage	0.01	0.01 0.1	3 3	104 97	97-112 96-99	7.2 1.6	<LOD (7)	Concurrent recoveries field trials	Webber, 2018b 2016RES-FNF-2453
Maize grain	0.01	0.01 0.1	3 3	94 95	91-96 92-97	2.8 2.8	<LOD (6)	Concurrent recoveries field trials	
Maize stover	0.01	0.01 0.1 0.2 [c] 2.0 [c]	4 4 1 1	99 92 - -	90-107 89-94 91 92	8.6 2.2 - -	<LOD (9)	Concurrent recoveries field trials	
Mustard greens	0.01	0.01 0.1	2 2	92 106	87, 103 104, 108	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
Pecan nutmeat	0.01	0.01 0.1	5 5	97 97	94-100 96-100	2.5 1.6	<0.5LOQ (5) [a]	matrix matched standards 0.050-25 ng/mL 1/× weighted R^2 > 0.995 m/z 366→ 175	Webber, 2017b, 2016RES-FNF2451 (RA.17.01)
	0.01	0.01 0.1	5 5	97 97	95-100 96-99	2.5 1.2	<0.5LOQ (5) [a]	m/z 366 → 131	
Pecan nutmeat	0.01	0.01 0.1	2 2	103 102	101, 104 99, 105	- -	<0.5LOQ (5) [a]	Concurrent recoveries field trials	Webber, 2017b, 2016RES-FNF2451 (RA.17.01)
Radish leaves	0.01	0.01 0.1	2 2	106 104	97, 116 101, 106	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Radish leaves	0.01	0.01 0.1	3 1	103 -	97-107 118	4.9 -		m/z 366 → 175	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	103 -	96-108 118	5.8 -		m/z 366 → 91	
Radish roots	0.01	0.01 0.1	2 2	90 86	77, 104 71, 101	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Radish roots	0.01	0.01 0.1	3 1	107 -	100-112 104	5.9 -		m/z 366 → 175	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	104 -	99-111 104	5.6 -		m/z 366 → 91	
Sorghum forage	0.01	0.01 0.10 0.20	2 2 1	108 108 -	98, 117 105, 110 88	- - -	<LOD (5)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (P3770G)
Sorghum forage	0.01	0.01 0.10 0.25	3 3 1	96 98 -	72, 117 80-110 88	24 16 - -	<LOD (7)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF-2455 (P3770G)
Sorghum grain	0.01	0.01 0.10 0.25	2 2 1	90 99 -	85, 94 89, 108 74	- - -	<LOD (5)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (P3770G)
Sorghum grain	0.01	0.01 0.10 0.25	3 3 1	93 97 -	85-100 89-108 74	8.1 10 -	<LOD (7)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF-2455 (P3770G)

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
Sorghum stover	0.01	0.01 0.10 0.40 0.50 2.0	2 1 1 1 1	107 - - - -	101, 112 95 82 93 90	- - - - -	<LOD (5)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (P3770G)
Sorhum stover	0.01	0.01 0.10 0.40 0.50 2.0	3 3 1 1 1	108 100 - - -	101-112 95-105 82 93 90	- - - - -	<LOD (7)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF-2455 (P3770G)
Sorghum AGF	0.01	0.01 0.10 1.0	1 1 1	- - -	100 108 103	- - -	<LOD (4)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (P3770G)
Sorghum flour	0.01	0.01 0.10 3.0	1 1 1	- - -	96 142 80	- - -	<LOD (3)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (P3770G)
Soya bean forage	0.01	0.01 0.1	3 1	103 -	99-107 103	3.8 -		m/z 366 → 175	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	103 -	100-105 102	2.5 -		m/z 366 → 91	
Soya bean hay	0.01	0.01 0.1	3 1	77	76-79-92	1.9 -		m/z 366 → 175	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	78 -	84-81 94	4.4 -		m/z 366 → 91	
Soya bean seeds	0.01	0.01 0.1	5 5	92 88	84-95 82-94	5 6	<0.01 (3)	Matrix matched standards 0.025-5.0 ng/mL 1/× weighed r ≥0.99 m/z 366 → 175	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	91 92	88-95 83-99	3 6	<0.01 (3)	m/z 366 → 91	
Soya bean seed	0.01	0.01 0.1	5 5	79 81	75-82 79-82	2.7 1.3	<0.01 (3)	6 matrix matched standards 0.050-5.0 ng/mL 1/× weighted r > 0.998 m/z 366 → 175	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	85 82	81-91 80-84	4.1 1.5	<0.01 (3)	m/z 366 → 91	
Sugar beet leaves	0.01	0.01 0.1	5 5	100 100	84-114 96-107	12 4	<0.01 (3)	Matrix matched standards 0.025-5.0 ng/mL 1/× weighed r ≥0.99 m/z 366 → 175	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01	5	101	84-112	11	<0.01 (3)	m/z 366 → 91	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
		0.1	5	106	93-112	7			
Sugar beet roots	0.01	0.01 0.1	5 5	81 111	71-93 92-126	10 11	<0.01 (3)	Matrix matched standards 0.025-5.0 ng/mL 1/× weighed r ≥0.99 m/z 366 → 175	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	76 108	67-92 103-114	14 5	<0.01 (3)	m/z 366 → 91	
Sweet corn K+CWHR	0.01	0.01 0.1 1.0	2 1 1	97 -	92, 101 92 93	- - -	<LOD (3)	Fortified controls	Webber, 2018c, 2016RES-FNF2454 (P 3770 G) Concurrent recoveries
Sweet corn forage	0.01	0.01 0.1 1.0	2 1 1	106 - -	105, 106 103 100	- - -	<LOD (3)	Fortified controls	
Sweet corn stover	0.01	0.01 0.1 1.0	2 1 1	105 - -	103, 106 102 98	- - -	<LOD (3)	Fortified controls	
Tomato mature	0.01	0.01 0.1	3 1	107 -	102-115 104	7.0 -		366 → 175 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	107 -	104-112 103	4.2 -		366 → 91 m/z	
Wheat forage	0.01	0.01 0.1	6 6	103 99	97-106 97-104	3.1 2.4	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Wheat forage	0.01	0.01 0.1	5 5	95 93	92-100 92-94	3.0 0.8	<0.01 (3)	6 matrix matched standards 0.0125-2.0 ng/mL 1/× weighted r > 0.998 m/z 366 → 175	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	98 93	94-103 91-93	3.5 1.0	<0.01 (3)	m/z 366 → 91	
Wheat forage	0.01	0.01 0.10 1.0	4 3 1	103 96 -	95-108 91-100 108	6.1 4.7 -	<LOD (9)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)
Wheat grain	0.01	0.01 0.1	7 7	99 103	92-107 94-111	17 16	<LOD (6)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Wheat grain	0.01	0.01 0.1	5 5	98 96	93-102 93-99	3.3 2.3	<0.01 (3)	6 matrix matched standards 0.0125-2.0 ng/mL 1/× weighted r > 0.998 m/z 366 → 175	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	96 95	94-102 92-97	3.3 2.3	<0.01 (3)	m/z 366 → 91	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
Wheat grain	0.01	0.01 0.10 1.0	4 3 1	105 99 -	101-111 95-106 87	4.2 5.9 -	<LOD (8)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)
Wheat hay	0.01	0.01 0.1	2 2	90 98	88, 93 94, 102	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Wheat hay	0.01	0.01 0.10 1.0	4 3 1	102 96 -	91-109 95-96 102	7.9 0.6 -	<LOD (9)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)
Wheat straw	0.01	0.01 0.1	6 6	94 95	83-102 92-97	7.1 1.9	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990	Schreier, 2018, 2015RES-IFP1902 (P3770G)
Wheat straw	0.01	0.01 0.1	5 5	106 106	102-110 101-111	4 4	<0.01 (3)	Matrix matched standards 0.0125-5.0 ng/mL 1/× weighed ≥0.99 m/z 366 → 175	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	97 99	91-101 93-105	5 5	<0.01 (3)	m/z 366 → 91	
Wheat straw	0.01	0.01 0.1	5 5	91 91	84-95 86-96	4.8 3.8	<0.01 (3)	6 matrix matched standards 0.0125-2.0 ng/mL 1/× weighted r > 0.998 m/z 366 → 175	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	90 89	82-98 83-94	5.6 4.2	<0.01 (3)	m/z 366 → 91	
Wheat straw	0.01	0.01 0.10 1.0	4 3 1	112 97 -	108-114 91-101 100	2.3 5.3 -	<LOD (9)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)

Notes:

[a] At least one control (untreated field sample) per trial was analysed for each matrix.

Table 88 Validation result for DesMet-fluindapyr-N1-glucoside with LC-MS/MS methods RA.17.01 or P3770G

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
Desmethyl-fluindapyr-N1-glucoside/fluindapyr-N-glucoside									

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
Cabbage immature	0.01	0.01 0.1	3 1	104 -	99-112 105	7.0 -		m/z 500 → 242	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	100 -	91-111 112	10 -		m/z 500 → 338	
Carrot leaves	0.01	0.01 0.1	3 1	97 -	96-98 87	1.1 -		m/z 500 → 242	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	97 -	94-100 99	2.9 -		m/z 500 → 338	
Carrot roots	0.01	0.01 0.1	3 1	99 -	97-100 103	1.6 -		m/z 500 → 242	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	96 -	92-98 103	2.9 -		m/z 500 → 338	
Dry bean	0.01	0.01 0.1	5 5	83 83	78-86 81-85	4 2	<0.01 (3)	Matrix matched standards 0.025-5.0 ng/mL 1/× weighed r ≥0.99 m/z 500 → 338	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	77 81	74-82 79-85	4 3	<0.01 (3)	m/z 500 → 242	
Dry bean	0.01	0.01 0.1	5 5	84 86	79-89 84-88	3.8 1.5	<0.01 (3)	6 matrix matched standards 0.050-5.0 ng/mL 1/× weighted r > 0.998 m/z 500 → 338	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	84 88	80-92 86-89	5.2 1.2	<0.01 (3)	m/z 500 → 242	
Grape	0.01	0.01 0.1	5 5	98 115	92-105 107-128	6 7	<0.01 (3)	Matrix matched standards 0.050-5.0 ng/mL 1/× weighed r ≥0.99 m/z 500 → 338	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	104 112	94-113 107-115	7 3	<0.01 (3)	m/z 500 → 242	
Grape	0.01	0.01 0.1	5 5	99 104	96-102 101-106	2.8 2.1	<0.01 (3)	6 matrix matched standards 0.050-5.0 ng/mL 1/× weighted r > 0.998 m/z 500 → 338	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	98 102	94-101 100-104	3.0 1.6	<0.01 (3)	m/z 500 → 242	
Radish leaves	0.01	0.01 0.1	3 1	108 -	105-112 119	3.5 -		m/z 500 → 242	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	105 -	104-106 118	0.9 -		m/z 500 → 338	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
Radish roots	0.01	0.01 0.1	3 1	99 -	92-105 108	6.7 -		m/z 500 → 242	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	97 -	92-103 108	5.8 -		m/z 500 → 338	
Soya bean forage	0.01	0.01 0.1	3 1	85 -	82-87 93	2.9 -		m/z 500 → 242	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	98 -	92-106 96	7.8 -		m/z 500 → 338	
Soya bean hay	0.01	0.01 0.1	3 1	79 -	76-82 82	4.1 -		m/z 500 → 242	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	74 -	70-77 82	4.5 -		m/z 500 → 338	
Soya bean seeds	0.01	0.01 0.1	5 5	82 82	74-92 73-86	9 7	<0.01 (3)	Matrix matched standards 0.025-5.0 ng/mL 1/× weighed r ≥0.99 m/z 500 → 338	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	81 84	76-88 77-87	6 5	<0.01 (3)	m/z 500 → 242	
Sugar beet leaves	0.01	0.01 0.1	5 5	93 110	86-100 108-112	6 2	<0.01 (3)	Matrix matched standards 0.025-5.0 ng/mL 1/× weighed r ≥0.99 m/z 500 → 338	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	89 110	83-100 106-117	7 4	<0.01 (3)	m/z 500 → 242	
Sugar beet roots	0.01	0.01 0.1	5 5	84 105	78-88 102-107	2 2	<0.01 (3)	Matrix matched standards 0.025-5.0 ng/mL 1/× weighed r ≥0.99 m/z 500 → 338	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	89 107	86-91 104-110	2 2	<0.01 (3)	m/z 500 → 242	
Soya bean seed	0.01	0.01 0.1	5 5	83 85	80-85 82-88	2.1 2.2	<0.01 (3)	6 matrix matched standards 0.050-5.0 ng/mL 1/× weighted r > 0.998 m/z 500 → 338	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	84 85	81-87 82-88	2.5 2.0	<0.01 (3)	m/z 500 → 242	
Tomato mature	0.01	0.01 0.1	3 1	98 -	95-101 106	2.9 -		m/z 500 → 242	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	96 -	92-99 111	4.2 -		m/z 500 → 338	
Wheat forage	0.01	0.01	5	95	90-100	3.9	<0.01	6 matrix matched	Sahvorost,

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, mass transition	reference (method)
[a]		0.1	5	103	101-106	1.6	(3)	standards 0.0125-2.0 ng/mL 1/× weighted r > 0.998 m/z 500 → 338	2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	96 104	91-101 101-107	4.9 2.2	<0.01 (3)	m/z 500 → 242	
Wheat grain [a]	0.01	0.01 0.1	5 5	100 103	97-102 98-106	2.1 3.6	<0.01 (3)	6 matrix matched standards 0.0125-2.0 ng/mL 1/× weighted r > 0.998 m/z 500 → 338	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	104 104	101-110 98-108	3.5 4.0	<0.01 (3)	m/z 500 → 242	
Wheat straw	0.01	0.01 0.1	5 5	98 102	92-105 98-105	5 3	<0.01 (3)	Matrix matched standards 0.0125-5.0 ng/mL 1/× weighed ≥0.99 m/z 500 → 338	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	103 101	94-110 98-103	7 2	<0.01 (3)	m/z 500 → 242	
Wheat straw [a]	0.01	0.01 0.1	5 5	99 98	93-104 92-103	3.9 3.8	<0.01 (3)	6 matrix matched standards 0.0125-2.0 ng/mL 1/× weighted r > 0.998 m/z 500 → 338	Sahvorost, 2018a, 2017AMT-IFP3871 (P 3370G)
	0.01	0.01 0.1	5 5	93 99	89-95 94-102	2.6 3.5	<0.01 (3)	m/z 500 → 242	

Notes:

[a] Result of the second attempt with a correct mixed intermediate solution after a first attempt where the recoveries were out of range for this component.

Table 89 Validation result for 1-OH-Met-fluindapyr with LC-MS/MS methods RA.17.01 or P3770G

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
1-OH-Met-fluindapyr									
Almond nutmeat	0.01	0.01 0.1	5 5	96 87	93-101 84-92	3.4 3.9	<LOD (5) [a]	matrix matched standards 0.050-25 ng/mL 1/× weighted R^2 > 0.995 m/z 368 → 310	Webber, 2017a, 2016RES-FNF2450 (RA.17.01)
	0.01	0.01 0.1	5 5	97 86	91-101 83-90	4.2 3.6	<LOD (5) [a]	m/z 368 → 330	
Almond nutmeat	0.01	0.01 0.1	2 2	88 94	85, 91 87, 100	- -	<LOD (5) [a]	Concurrent recoveries field trials	Webber, 2017a, 2016RES-FNF2450 (RA.17.01)
Almond hulls	0.01	0.01 0.1	5 5	97 88	94-102 85-92	3.5 3.0	<LOD (5) [a]	matrix matched standards 0.050-25 ng/mL 1/× weighted R^2 > 0.995 m/z 368 → 310	Webber, 2017a, 2016RES-FNF2450 (RA.17.01)
	0.01	0.01 0.1	5 5	94 89	91-98 88-91	3.1 1.6	<LOD (5) [a]	m/z 368 → 330	
Almond hulls	0.01	0.01 0.1 1.0	2 2 1	96 95 -	95, 97 85, 105 83	- -	<LOD (5) [a]	Concurrent recoveries field trials	Webber, 2017a, 2016RES-FNF2450 (RA.17.01)
Cabbage immature	0.0056	0.0056 0.056	3 1	77 -	73-82 88	5.8 -		m/z 368 → 310 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0056	0.0056 0.056	3 1	78 -	75-82 84	4.3 -		m/z 368 → 330 diastereomer a	
	0.0044	0.0044 0.044	3 1	91 -	79-83 84	2.7 -		m/z 368 → 310 diastereomer b	
	0.0044	0.0044 0.044	3 1	73 -	70-77 90	4.5 -		m/z 368 → 330 diastereomer b	
Carrot leaves	0.0056	0.0056 0.056	3 1	97 -	87-103 119	9.1 -		m/z 368 → 310 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0056	0.0056 0.056	3 1	94 -	92-98 120	3.6 -		m/z 368 → 330 diastereomer a	
	0.0044	0.0044 0.044	3 1	94 -	92-96 118	2.2 -		m/z 368 → 310 diastereomer b	
	0.0044	0.0044 0.044	3 1	96 -	74-108 115	19 -		m/z 368 → 330 diastereomer b	
Carrot roots	0.0056	0.0056 0.056	3 1	86 -	82-87 84	1.1 -		m/z 368 → 310 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0056	0.0056 0.056	3 1	79 -	70-85 76	10 -		m/z 368 → 330 diastereomer a	
	0.0044	0.0044 0.044	3 1	82 -	79-83 82	3.0 -		m/z 368 → 310 diastereomer b	
	0.0044	0.0044 0.044	3 1	85 -	79-85 80	7.5 -		m/z 368 → 330 diastereomer b	
Dry beans	0.01	0.01 0.1	5 5	107 103	103-113 98-110	4 4	<0.01 (2)	m/z 368 → 310 ≥5 matrix matched	Stanislowski, 2016a,

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
								standards 0.025-0.50 ng/mL r≥0.99 (sum a+b)	2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	106 102	100-114 99-109	5 4	<0.01 (2)	m/z 368 → 330 (sum a+b)	
Dry beans	0.0056	0.0056 0.056	5 5	89 87	81-93 84-88	5.2 1.8	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.0056	0.0056 0.056	5 5	97 88	91-104 84-90	5.4 2.7	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	5 5	97 90	93-104 88-93	4.6 2.6	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	5 5	90 89	87-92 86-91	3.5 2.3	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Grapes	0.0056	0.0056 0.056	7 5	86 70	76-97 65-76	8.1 5.7	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.0056	0.0056 0.056	7 5	88 71	81-104 65-76	8.6 5.6	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	7 5	84 70	72-93 66-74	7.7 4.2	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	7 5	83 71	75-86 63-76	4.7 6.8	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Grapes	0.0056	0.0056 0.056	5 5	86 82	81-90 79-84	4.6 2.5	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.0056	0.0056 0.056	5 5	87 81	85-89 78-82	1.7 2.2	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	5 5	84 83	80-87 81-85	3.7 1.7	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	5 5	90 82	79-111 80-85	14 2.7	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Grapes	0.01	0.01 0.1	5 5	99 99	91-104 91-102	5 5	<0.01 (2)	m/z 368 → 310 ≥5 matrix matched standards 0.050-0.50 ng/mL r≥0.99 (sum a+b)	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	102 101	98-108 87-107	5 9	<0.01 (2)	m/z 368 → 330 (sum a+b)	
Maize AGF	0.01	0.01 0.1 0.5	1 1 1	- - -	74 75 85	- - -	<LOD (3)	Concurrent recoveries field trials	Webber, 2018a, 2015RS-FNF-1900 (RA.17.01)
Maize flour	0.01	0.01 0.1	1 1	- -	83 85	- -	<LOD (2)	Concurrent recoveries field trials	
Maize forage	0.01	0.01 0.1 1.0	2 2	78 80 -	78, 77 81, 79 70	- - -	<LOD (5)	Concurrent recoveries field trials	
Maize germ	0.01	0.01 0.1	1 1	- -	75 73	- -	<LOD (2)	Concurrent recoveries field trials	
Maize grain	0.01	0.01 0.1	2 2	73 82	70, 76 83, 80	- -	<LOD (4)	Concurrent recoveries field trials	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
Maize grits	0.01	0.01 0.1	1 1	- -	78 76	- -	<LOD (2)	Concurrent recoveries field trials	
Maize meal	0.01	0.01 0.1	1 1	- -	112 68	- -	<LOD (2)	Concurrent recoveries field trials	
Maize oil	0.01	0.01 0.1	1 1	- -	83 80	- -	<LOD (2)	Concurrent recoveries field trials	
Maize starch	0.01	0.01 0.1	1 1	- -	70 83	- -	<LOD (2)	Concurrent recoveries field trials	
Maize stover	0.01	0.01 0.1 2.0	2 2 1	76 79 -	78, 73 74, 83 83	- - -	<LOD (5)	Concurrent recoveries field trials	
Maize forage	0.01	0.01 0.1 1.0 [c]	3 3 1	80 82 -	70-86 81-83 70	11 1.2	<LOD (7)	Concurrent recoveries field trials	Webber, 2018b 2016RES-FNF-2453 (RA.17.01)
Maize grain	0.01	0.01 0.1	3 3	95 75	86-100 65-81	8.2 12	<LOD (6)	Concurrent recoveries field trials	
Maize stover	0.01	0.01 0.1 2.0 [c]	4 4 1	88 79 -	75-105 71-87 83	14 10	<LOD (9)	Concurrent recoveries field trials	
Mustard greens	0.01	0.01 0.1	2 2	86 86	84, 88 81, 90	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Oil seed rape crude oil	0.0056	0.0056 0.056	7 5	112 103	97-126 90-114	8.2 9.1	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.0056	0.0056 0.056	7 5	114 108	106-119 103-115	4.7 4.5	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	7 5	117 110	106-129 102-119	8.0 6.3	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	7 5	113 110	102-124 101-118	7.1 6.9	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Oil seed rape straw	0.0056	0.0056 0.056	7 5	84 92	78-89 83-97	4.0 6.3	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.0056	0.0056 0.056	7 5	88 86	78-99 80-96	8.2 8.1	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	7 5	86 86	77-93 77-90	7.2 6.1	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	7 5	117 96	113-127 84-109	4.5 9.3	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Oil seed rape whole plant	0.0056	0.0056 0.056	7 5	101 92	92-112 88-99	8.2 4.3	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.0056	0.0056 0.056	7 5	102 94	97-113 90-100	6.7 4.1	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	7 5	104 98	97-109 95-103	6.2 3.2	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
	0.0044	0.0044 0.044	7 5	105 100	97-113 98-105	5.4 3.1	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Pecan nutmeat	0.01	0.01 0.1	5 5	85 91	79-90 89-92	4.6 1.4	<0.5LOQ (5) [a]	matrix matched standards 0.050-25 ng/mL 1/× weighted R^2 > 0.995 m/z 368 → 310	Webber, 2017a, 2016RES-FNF2450 (RA.17.01)
	0.01	0.01 0.1	5 5	87 91	80-94 91-92	6.6 0.5	<0.5LOQ (5) [a]	m/z 368 → 330	
Pecan nutmeat	0.01	0.01 0.1	2 2	74 74	73, 75 71, 76	- -	<0.5LOQ (5) [a]	Concurrent recoveries field trials	Webber, 2017a, 2016RES-FNF2450 (RA.17.01)
Radish leaves	0.01	0.01 0.1	2 2	90 86	88-91 75-97	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Radish leaves	0.0056	0.0056 0.056	3 1	84 -	77-95 74	11 -		m/z 368 → 310 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0056	0.0056 0.056	3 1	88	77-105-73	17 -		m/z 368 → 330 diastereomer a	
	0.0044	0.0044 0.044	3 1	91 -	83-104 74	13 -		m/z 368 → 310 diastereomer b	
	0.0044	0.0044 0.044	3 1	84 -	70-100 77	18 -		m/z 368 → 330 diastereomer b	
Radish roots	0.01	0.01 0.1	2 2	82 74	72-92 73-74	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Radish roots	0.0056	0.0056 0.056	3 1	100 -	95-105 88	5.0 -		m/z 368 → 310 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0056	0.0056 0.056	3 1	88 -	85-90 89	2.9 -		m/z 368 → 330 diastereomer a	
	0.0044	0.0044 0.044	3 1	99 -	94-104 92	5.5 -		m/z 368 → 310 diastereomer b	
	0.0044	0.0044 0.044	3 1	95 -	94-98 91	2.6 -		m/z 368 → 330 diastereomer b	
Sorghum forage	0.01	0.01 0.10 0.20	2 2 1	108 94 -	96, 119 79, 109 93	- - -	<LOD (5)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (RA.17.01)
Sorghum forage	0.01	0.01 0.10 0.20	3 3 1	107 96 -	96-119 79-109 93	11 16 -	<LOD (7)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF-2455 (RA.17.01)
Sorghum grain	0.01	0.01 0.10	2 2	84 77	79, 119 71, 83	- -	<LOD (5)	Concurrent recoveries field	Webber, 2018d, 2015RES-FNF-

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
		0.30	1	-	92	-		trials	1901 (RA.17.01)
Sorghum grain	0.01	0.01 0.100.30	3 3 1	88 83 -	81-98 71-96 92	9.9 15 -	<LOD (7)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF-2455 (RA.17.01)
Sorghum stover	0.01	0.01 0.10 0.80	2 1 1	85 82 -	84, 85 80, 84 72	- - -	<LOD (5)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (RA.17.01)
Sorhum stover	0.01	0.01 0.10 0.80	3 3 1	85 82 -	84-85 80-84 72	0.7 2.5 -	<LOD (7)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF-2455 (RA.17.01)
Sorghum AGF	0.01	0.01 0.10 3.0	1 1 1	- - -	82 79 70	- - -	<LOD (3)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (RA.17.01)
Sorghum flour	0.01	0.01 0.10 0.30	1 1 1	- - -	94 78 75	- - -	<LOD (3)	Concurrent recoveries field trials	Webber, 2018d, 2015RES-FNF-1901 (RA.17.01)
Soya bean forage	0.0056	0.0056 0.056	3 1	102 -	90-117 88	13-		m/z 368 → 310 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0056	0.0056 0.056	3 1	108 -	92-128 88	17 -		m/z 368 → 330 diastereomer a	
	0.0044	0.0044 0.044	3 1	102 -	87-113 84	13 -		m/z 368 → 310 diastereomer b	
	0.0044	0.0044 0.044	3 1	99 -	77-113 83	20 -		m/z 368 → 330 diastereomer b	
Soya bean hay	0.0056	0.0056 0.056	3 1	94 -	88-105 72	9.7 -		m/z 368 → 310 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0056	0.0056 0.056	3 1	88 -	78-98 76	11 -		m/z 368 → 330 diastereomer a	
	0.0044	0.0044 0.044	3 1	109 -	100-119 80	8.8 -		m/z 368 → 310 diastereomer b	
	0.0044	0.0044 0.044	3 1	87 -	79-94 78	8.8 -		m/z 368 → 330 diastereomer b	
Soya bean seed	0.01	0.01 0.1	5 5	111 115	106-119 111-118	3 3	<0.01 (2)	≥5 matrix matched standards 0.050-0.50 ng/mL r≥0.99 m/z 368 → 310 (sum a+b)	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	115 115	111-122 100-121	4 4	<0.01 (2)	m/z 368 → 330 (sum a+b)	
Soya bean seed	0.0056	0.0056 0.056	5 5	73 74	72-76 68-84	3.1 8.1	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.0056	0.0056 0.056	5 5	79 75	73-85 70-83	5.3 6.8	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	5 5	79 76	76-82 71-87	3.2 8.2	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044	5	82	75-85	4.7	<0.3LOQ	m/z 368 → 310	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
		0.044	5	75	70-87	8.7	(n=2)	diastereomer b	
Sugar beet leaves	0.01	0.01 0.1	5 4	107 102	101-112 98-106 [a]	4 4	<0.01 (2)	≥5 matrix matched standards 0.050-0.50 ng/mL r≥0.99 m/z 368 → 310 (sum a+b)	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 4	106 101	97-115 96-105 [b]	7 6	<0.01 (2)	m/z 368 → 330 (sum a+b)	
Sugar beet roots	0.01	0.01 0.1	5 5	108 112	102-113 107-117	4 3	<0.01 (2)	≥5 matrix matched standards 0.050-0.50 ng/mL r≥0.99 m/z 368 → 310 (sum a+b)	Stanislowski, 2016, 2015RES-IFP2155 (P 3770G)
	0.01	0.01 0.1	5 5	109 111	104-113 107-114	4 3	<0.01 (2)	m/z 368 → 330 (sum a+b)	
Sweet corn K+CWHR	0.01	0.01 0.1 1.0	2 1 1	87 - 	84, 89 77 78	- - -	<LOD (3)	Concurrent recoveries field trials	Webber, 2018c, 2016RES-FNF2454 (RA.17.01)
Sweet corn forage	0.01	0.01 0.1 1.0	2 1 1	99 - -	93, 104 82 80	- - -	<LOD (3)	Concurrent recoveries field trials	
Sweet corn stover	0.01	0.01 0.1 1.0	2 1 1	99 - -	80, 104 96 79	- - -	<LOD (3)	Concurrent recoveries field trials	
Tomato mature	0.0056	0.0056 0.056	3 1	88 -	97-90 93	2.2 -		m/z 368 → 310 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0056	0.0056 0.056	3 1	89 -	85-93 96	4.7 -		m/z 368 → 330 diastereomer a	
	0.0044	0.0044 0.044	3 1	92 -	92-94 100	1.3 -		m/z 368 → 310 diastereomer b	
	0.0044	0.0044 0.044	3 1	91 -	83-98 102	8.2 -		m/z 368 → 330 diastereomer b	
Wheat forage	0.01	0.01 0.1	6 6	80 81	75-84 74-85	4.3 5.2	<0.3LOQ (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Wheat forage	0.0056	0.0056 0.056	5 5	84 79	78-89 77-83	5.1 3.2	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.0056	0.0056 0.056	5 5	81 80	73-84 78-84	3.6 3.2	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	5 5	83 81	79-88 79-85	3.8 3.5	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	5 5	85 82	82-89 78-86	3.2 3.4	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Wheat forage	0.01	0.01 0.10 1.0	4 4 1	85 83 -	74, 104 77-92 76	16 7.8 -	<LOD (9)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)
Wheat dry	0.0056	0.0056	7	96	71-115	16	<0.3LOQ	m/z 368 → 330	Riccelli, 2017a,

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
gluten		0.056	5	92	79-118	16	(n=2)	diastereomer a	2017RES-IFP3206 (RA.17.01)
	0.0056	0.0056 0.056	7 5	99 91	73-117 76-119	16 18	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	7 5	92 88	77-106 77-119	12 20	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	7 5	100 89	77-115 71-118	14 20	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Wheat grain	0.01	0.01 0.1	7 7	86 82	78-101 79-86	9.3 3.2	<0.3LOQ (6)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Wheat grain	0.0056	0.0056 0.056	7 5	108 108	103-112 105-112	3.3 2.8	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.0056	0.0056 0.056	7 5	110 108	103-117 106-111	5.3 2.5	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	7 5	110 108	97-115 105-111	5.9 2.3	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	7 5	108 108	97-115 104-112	6.1 2.7	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Wheat grain	0.0056	0.0056 0.056	5 5	76 72	68-80 67-80	6.4 7.4	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.0056	0.0056 0.056	5 5	103 74	92-110 69-80	6.6 6.4	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	5 5	87 75	80-99 71-81	8.1 5.9	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	5 5	84 73	80-90 68-71	5.0 6.6	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Wheat grain	0.01	0.01 0.10	4 3	90 73	82-107 60-85	14 14	<LOD (10)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)
Wheat hay	0.01	0.01 0.1	2 2	74 73	73, 76 72, 74	- -	<0.3LOQ (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Wheat hay	0.01	0.01 0.10 1.0	4 3 1	84 72 -	70-91 70-74 71	12 2.9 -	<LOD (8)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)
Wheat straw	0.01	0.01 0.1	6 6	92 89	84-104 77-94	8.7 7.1	<0.3LOQ (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Wheat straw	0.0056	0.0056 0.056	7 5	82 73	76-90 70-79	7.6 4.9	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.0056	0.0056 0.056	7 5	85 75	73-97 70-80	8.4 5.1	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
	0.0044	0.0044 0.044	7 5	88 77	77-97 73-81	8.4 4.0	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	7 5	84 77	79-95 73-80	7.3 4.0	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Wheat straw	0.0056	0.0056 0.056	5 5	82 79	79-84 76-83	2.4 3.6	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.0056	0.0056 0.056	5 5	89 79	8293 76-83	4.8 3.8	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer a	
	0.0044	0.0044 0.044	5 5	84 80	81-87 75-85	3.2 4.7	<0.3LOQ (n=2)	m/z 368 → 330 diastereomer b	
	0.0044	0.0044 0.044	5 5	80 81	73-87 77-83	6.8 3.1	<0.3LOQ (n=2)	m/z 368 → 310 diastereomer b	
Wheat straw	0.01	0.01 0.1	5 5	104 104	103-106 103-106	1 1	<0.01 (2)	≥5 matrix matched standards 0.0125-0.50 ng/mL r≥0.99 m/z 368 → 310 (sum a+b)	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	105 104	100-107 103-106	3 1	<0.01 (2)	m/z 368 → 330 (sum a+b)	
Wheat straw	0.01	0.01 0.10 1.0	4 3 1	96 70 -	74-113 66-72 71	18 19 -	<LOD (10)	Concurrent recoveries field trials	Webber, 2018e, 2016RES-FNF2456 (P 3370 G)

Notes:

[a] At least one control (untreated field sample) per trial was analysed for each matrix.

[a] One value of 147 percent was excluded. If included (n=5), mean = 111 percent and RSD = 18 percent.

[b] One value of 148 percent was excluded. If included (n=5), mean = 111 percent and RSD = 19 percent

[c] This fortification was conducted within the 2015 field corn study entitled "Magnitude and Decline of Residues of F9944 and Metabolites in/on Field Corn and Processed Fractions Following Applications of F9944-6" (Thorn 2018) that was run simultaneously with this study.

Table 90 Validation result for 1-COOH-fluindapyr with LC-MS/MS methods RA.17.01 or P3770G

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration, mass transition	reference (method)
1-COOH-fluindapyr									
Cabbage immature	0.0063	0.0063 0.063	3 1	84 -	77-92 97	9.2 -		m/z 382 → 336 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0063	0.0063 0.063	3 1	83 -	78-88 96	5.7 -		m/z 382 → 296 diastereomer a	
	0.0037	0.0037 0.037	3 1	78 -	73-84 93	6.9 -		m/z 382 → 336 diastereomer b	
	0.0037	0.0037 0.037	3 1	101 -	95-111 103	8.6 -		m/z 382 → 296 diastereomer b	
Carrot leaves	0.0063	0.0063 0.063	3 1	94 -	81-106 119	13 -		m/z 382 → 336 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569
	0.0063	0.0063 0.063	3 1	90 -	80-106 119	16 -		m/z 382 → 281 diastereomer a	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration, mass transition	reference (method)
	0.0037	0.0037 0.037	3 1	96 -	89-105 118	9.1 -		m/z 382 → 336 diastereomer b	(RA.17.01)
	0.0037	0.0037 0.037	3 1	87 -	81-92 113	6.4 -		m/z 382 → 281 diastereomer b	
Carrot roots	0.0063	0.0063 0.063	3 1	95 -	91-102 82	5.9 -		m/z 382 → 336 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0063	0.0063 0.063	3 1	82 -	78-88 81	6.2 -		m/z 382 → 296 diastereomer a	
	0.0037	0.0037 0.037	3 1	87 -	78-95 84	9.4 -		m/z 382 → 336 diastereomer b	
	0.0037	0.0037 0.037	3 1	78 -	76-81 86	3.4 -		m/z 382 → 296 diastereomer b	
Dry beans	0.01	0.01 0.1	5 5	113 110	100-124 104-115	8 4	<0.01 (2)	≥5 matrix matched standards 0.025-0.50 ng/mL r≥0.99 m/z 382 → 336 (sum a+b)	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	115 113	106-130 110-116	9 2	<0.01 (2)	m/z 382 → 296 (sum a+b)	
Dry beans	0.063	0.0063 0.063	5 5	131 111	111-149 107-116	11 3.1	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.063	0.0063 0.063	5 5	117 110	113-120 106-113	2.0 2.6	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	
	0.063	0.0063 0.063	5 5	126 110	105-136 107-112	10 1.8	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.037	0.0037 0.037	5 5	109 111	98-124 109-114	9.8 2.3	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
	0.037	0.0037 0.037	5 5	116 109	101-135 108-112	12 1.5	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	5 5	113 112	100-119 107-118	6.8 3.7	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
Grapes	0.063	0.0063 0.063	7 5	83 76	76-88 71-80	4.9 4.4	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.063	0.0063 0.063	7 5	83 82	72-88 77-91	6.1 7.0	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.063	0.0063 0.063	7 5	74 71	66-82 65-76	6.5 5.5	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	
	0.037	0.0037 0.037	7 5	86 81	70-98 73-94	9.7 9.6	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	7 5	84 79	73-95 70-90	8.4 8.8	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
	0.037	0.0037 0.037	7 5	76 76	54-95 69-85	16 8.2	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
Grapes	0.063	0.0063 0.063	5 5	100 98	93-108 92-101	5.8 3.6	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.063	0.0063 0.063	5 5	102 97	92-109 92-101	6.2 3.7	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	
	0.063	0.0063 0.063	5 5	118 101	108-130 94-104	7.3 4.0	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.037	0.0037 0.037	5 5	110 99	97-119 93-102	7.8 3.5	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
	0.037	0.0037	5	96	89-104	6.0	<0.3LOQ	m/z 382 → 296	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration, mass transition	reference (method)
		0.037	5	95	91-98	2.9	(n=2)	diastereomer b	
	0.037	0.0037 0.037	5 5	100 100	93-109 94-102	6.6 3.3	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
Grapes	0.01	0.01 0.1	5 5	109 110	95-118 104-117	8 4	<0.01 (2)	≥5 matrix matched standards 0.050-0.50 ng/mL r≥0.99 m/z 382 → 336 (sum a+b)	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	108 107	94-116 103-114	8 4	<0.01 (2)	m/z 382 → 296 (sum a+b)	
Mustard greens	0.01	0.01 0.1	2 2	101 103	99, 103 101, 105	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Oil seed rape crude oil	0.063	0.0063 0.063	7 5	113 118	101-128 114-122	8.4 2.6	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.063	0.0063 0.063	7 5	115 119	109-124 114-123	5.4 2.9	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.063	0.0063 0.063	7 5	111 118	99-129 116-121	9.3 2.1	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	
	0.037	0.0037 0.037	7 5	100 112	95-103 108-116	5.0 3.3	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	7 5	103 113	95-111 106-117	6.0 4.3	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
	0.037	0.0037 0.037	7 5	101 112	98-106 106-116	5.7 3.5	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
Oil seed rape whole plant	0.063	0.0063 0.063	7 5	111 102	104-120 97-109	5.6 4.3	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.063	0.0063 0.063	7 5	116 103	107-123 99-11	5.3 4.8	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.063	0.0063 0.063	7 5	103 104	80-116 99-110	16 4.1	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	
	0.037	0.0037 0.037	7 5	103 104	87-114 98-115	10 6.4	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	7 5	95 102	84-106 98-110	9.7 5.1	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
	0.037	0.0037 0.037	7 5	84 106	73-100 99-116	13 7.9	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
Oil seed rape straw	0.063	0.0063 0.063	7 5	90 88	74-102 78-102	12 11	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.063	0.0063 0.063	7 5	88 91	72-96 79-103	11 9.7	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.063	0.0063 0.063	7 5	96 91	79-106 80-106	10 11	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	
	0.037	0.0037 0.037	7 5	80 79	70-92 73-95	9.5 12	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	7 5	79 80	62-92 73-100	12 14	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
	0.037	0.0037 0.037	7 5	90 81	70-103 72-98	13 13	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration, mass transition	reference (method)
Radish leaves	0.01	0.01 0.1	2 2	102 100	97-108 97-102	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Radish leaves	0.0063	0.0063 0.063	3 1	86 -	78-102 79	16 -		m/z 382 → 336 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0063	0.0063 0.063	3 1	98 -	89-112 79	13 -		m/z 382 → 296 diastereomer a	
	0.0037	0.0037 0.037	3 1	96 -	89-108 94	11 -		m/z 382 → 336 diastereomer b	
	0.0037	0.0037 0.037	3 1	100 -	86-114 83	14 -		m/z 382 → 296 diastereomer b	
Radish roots	0.01	0.01 0.1	2 2	96 87	92, 99 80, 94	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Radish roots	0.0063	0.0063 0.063	3 1	91 -	89-92 88	1.7 -		m/z 382 → 336 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0063	0.0063 0.063	3 1	94 -	91-95 89	2.9 -		m/z 382 → 296 diastereomer a	
	0.0037	0.0037 0.037	3 1	103 -	92-114 94	10 -		m/z 382 → 336 diastereomer b	
	0.0037	0.0037 0.037	3 1	93 -	86-97 93	6.1 -		m/z 382 → 296 diastereomer b	
Soya bean forage	0.0063	0.0063 0.063	3 1	100 -	92-109 89	8.6 -		m/z 382 → 336 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0063	0.0063 0.063	3 1	107 -	94-116 87	11 -		m/z 382 → 296 diastereomer a	
	0.0037	0.0037 0.037	3 1	105 -	100-111 85	5.1 -		m/z 382 → 336 diastereomer b	
	0.0037	0.0037 0.037	3 1	92 -	84-103 90	11 -		m/z 382 → 296 diastereomer b	
Soya bean hay	0.0063	0.0063 0.063	3 1	96 -	89-106 81	9.2 -		m/z 382 → 336 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0063	0.0063 0.063	3 1	96 -	94-98 87	2.4 -		m/z 382 → 296 diastereomer a	
	0.0037	0.0037 0.037	3 1	86 -	78-97 82	11 -		m/z 382 → 336 diastereomer b	
	0.0037	0.0037 0.037	3 1	96 -	89-105 82	9.1 -		m/z 382 → 296 diastereomer b	
Soya bean seeds	0.01	0.01 0.1	5 5	103 115	100-106 106-125	2 7	<0.01 (2)	≥5 matrix matched standards 0.025-0.50 ng/m L r≥0.99 m/z 382 → 336 (sum a+b)	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	112 116	110-116 108-124	2 5	<0.01 (2)	m/z 382 → 296 (sum a+b)	
Soya bean seeds	0.063	0.0063 0.063	5 5	91 94	85-99 87-106	7.1 8.1	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	Sahvorost, 2018b

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration, mass transition	reference (method)
	0.063	0.0063 0.063	5 5	95 94	87-102 90-103	5.7 5.8	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	2017AMT-IFP3872 (RA.17.01)
	0.063	0.0063 0.063	5 5	98 94	93-114 88-104	9.3 6.5	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.037	0.0037 0.037	5 5	93 98	81-106 92-113	14 8.8	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
	0.037	0.0037 0.037	5 5	99 97	84-104 92-108	8.6 6.9	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	5 5	92 97	81-102 92-103	8.7 4.8	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
Sugar beet leaves	0.01	0.01 0.1	5 4	107 105	98-113 103-107 [a]	5 2	<0.01 (2)	≥5 matrix matched standards 0.025-0.50 ng/mL r≥0.99 m/z 382 → 336 (sum a+b)	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 4	108 107	104-110 106-110 [b]	2 2	<0.01 (2)	m/z 382 → 296 (sum a+b)	
Sugar beet roots	0.01	0.01 0.1	5 5	106 108	102-109 101-113	3 5	<0.01 (2)	≥5 matrix matched standards 0.025-0.50 ng/mL r≥0.99 m/z 382 → 336 (sum a+b)	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	107 1078	105-110 104-110	2 2	<0.01 (2)	m/z 382 → 296	
Tomato mature	0.0063	0.0063 0.063	3 1	99 -	92-103 91	6.0 -		m/z 382 → 336 diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.0063	0.0063 0.063	3 1	92 -	89-94 91	2.9 -		m/z 382 → 281 diastereomer a	
	0.0037	0.0037 0.037	3 1	106 -	103-108 91	2.9 -		m/z 382 → 336 diastereomer b	
	0.0037	0.0037 0.037	3 1	93 -	89-97 88	4.4 -		m/z 382 → 281 diastereomer b	
Wheat forage	0.01	0.01 0.1	6 6	98 102	88-106 93-109	6.3 5.8	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Wheat forage	0.063	0.0063 0.063	5 5	90 92	85-96 89-99	5.1 4.6	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.063	0.0063 0.063	5 5	91 91	88-95 89-98	3.1 4.1	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	
	0.063	0.0063 0.063	5 5	87 91	85-91 89-97	3.0 3.6	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.037	0.0037 0.037	5 5	92 92	88-110 90-98	11 3.9	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
	0.037	0.0037 0.037	5 5	86 91	84-88 89-96	2.1 3	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	5 5	91 92	85-93 88-96	3.4 3.4	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
Wheat grain	0.01	0.01 0.1	7 7	92 103	86-108 96-112	8.7 5.1	<LOD (6)	10 matrix matched standards	Schreier, 2018, 2015RES-

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration, mass transition	reference (method)
								0.10-25 ng/mL 1/× weighted r > 0.990	IFP1902 (RA.17.01)
Wheat grain	0.063	0.0063 0.063	5 5	101 96	95-117 91-101	10 4.4	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.063	0.0063 0.063	5 5	109 93	97-124 88-100	11 5.2	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	
	0.063	0.0063 0.063	5 5	92 94	88-100 91-98	5.6 2.5	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.037	0.0037 0.037	5 5	85 95	75-97 87-103	13 6.8	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
	0.037	0.0037 0.037	5 5	92 93	86-97 85-101	5.0 6.5	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	5 5	98 95	88-106 88-103	7.6 6.4	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
Wheat dry gluten	0.063	0.0063 0.063	7 5	107 101	79-121 87-134	15 19	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.063	0.0063 0.063	7 5	103 103	74-124 85-136	18 19	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	
	0.037	0.0037 0.037	7 5	103 110	81-136 95-136	17 14	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	7 5	110 106	92-133 91-122	13 11	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
	0.037	0.0037 0.037	7 5	108 103	89-114 92-115	14 8.1	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
Wheat grain	0.063	0.0063 0.063	7 5	110 110	98-120 108-113	7.0 1.8	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.063	0.0063 0.063	7 5	109 110	98-120 108-112	7.5 1.5	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.063	0.0063 0.063	7 5	107 110	96-120 108-112	7.6 1.5	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	
	0.037	0.0037 0.037	7 5	83 108	70-92 103-113	9.7 3.9	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	7 5	81 108	65-87 104-112	9.6 3.3	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
	0.037	0.0037 0.037	7 5	81 108	68-92 105-111	11 2.5	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
Wheat hay	0.01	0.01 0.1	2 2	104 100	104, 105 96, 104	- -	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Wheat straw	0.01	0.01 0.1	6 6	115 118	104-122 102-125	5.6 7.0	<LOD (4)	10 matrix matched standards 0.10-25 ng/mL 1/× weighted r > 0.990 (sum a+b)	Schreier, 2018, 2015RES-IFP1902 (RA.17.01)
Wheat straw	0.063	0.0063 0.063	7 5	87 82	76-98 78-87	8.1 4.9	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.063	0.0063 0.063	7 5	86 81	72-96 76-88	9.0 6.5	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration, mass transition	reference (method)
	0.063	0.0063 0.063	7 5	82 84	72-93 82-87	8.7 2.3	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	
	0.037	0.0037 0.037	7 5	87 87	73-100 82-100	11 8.9	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	7 5	101 87	92-116 79-101	11 10	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
	0.037	0.0037 0.037	7 5	98 90	76-116 84-102	13 8.2	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
Wheat straw	0.063	0.0063 0.063	5 5	88 95	75-93 88-100	9.2 4.4	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.063	0.0063 0.063	5 5	93 92	88-96 88-95	3.2 3.3	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer a	
	0.063	0.0063 0.063	5 5	92 93	84-102 90-98	7.7 3.4	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer a	
	0.037	0.0037 0.037	5 5	93 92	73-109 88-94	16 2.5	<0.3LOQ (n=2)	m/z 382 → 281 diastereomer b	
	0.037	0.0037 0.037	5 5	94 91	88-101 88-93	5.2 2.5	<0.3LOQ (n=2)	m/z 382 → 296 diastereomer b	
	0.037	0.0037 0.037	5 5	88 91	71-94 87-94	11 3.4	<0.3LOQ (n=2)	m/z 382 → 336 diastereomer b	
Wheat straw	0.01	0.01 0.1	5 5	107 108	100-117 107-109	6 1	<0.01 (2)	≥5 matrix matched standards 0.0125-0.50 ng/mL r≥0.99 m/z 382 → 336 (sum a+b)	Stanislowski, 2016a, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	105 108	100-110 105-110	3 2	<0.01 (2)	m/z 382 → 296 (sum a+b)	

Notes:

[a] One value of 174 percent was excluded. If included (n=5), mean = 121 percent and RSD = 25 percent.

[b] One value of 176 percent was excluded. If included (n=5), mean = 119 percent and RSD = 26 percent.

Table 91 Validation result for 1-OH-Met-N-DesMet-fluindapyr with LC-MS/MS method RA.17.01

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
1-OH-Met-N-DesMet-fluindapyr									
Cabbage immature	0.01	0.01 0.1	3 1	83 -	78-86 91	4.6 -		312 m/z diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	81 -	77-83 90	4.2 -		332 m/z diastereomer a	
	0.01	0.01 0.1	3 1	83 -	77-87 95	6.0 -		312 m/z diastereomer b	
	0.01	0.01 0.1	3 1	81 -	71-90 93	12 -		332 m/z diastereomer b	
Carrot leaves	0.01	0.01 0.1	3 1	97 -	80-110 118	16 -		312 m/z diastereomer a	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
	0.01	0.01 0.1	3 1	99 -	80-113 118	17 -		332 m/z diastereomer a	
	0.01	0.01	3	100	87-110	12		312 m/z	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
		0.1	1	-	119	-		diastereomer b	
	0.01	0.01	3	95	84-100	9.8		332 m/z	
		0.1	1	-	115	-		diastereomer b	
Carrot roots	0.01	0.01	3	89	83-94	6.6		312 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
		0.1	1	-	95	-		diastereomer a	
	0.01	0.01	3	93	87-97	5.6		332 m/z	
		0.1	1	-	95	-		diastereomer a	
	0.01	0.01	3	92	84-100	8.8		312 m/z	
		0.1	1	-	98	-		diastereomer b	
	0.01	0.01	3	91	84-97	7.3		332 m/z	
		0.1	1	-	96	-		diastereomer b	
Dry beans	0.01	0.01	5	101	83-121	14	<0.01 (2)	m/z 352 → 312	Stanislowski, 2016, 2015RES-IFP2155 (P 3770G)
		0.1	5	95	90-104	6		(sum a+b)	
	0.01	0.01	5	101	92-116	13	<0.01 (2)	m/z 352 → 332	
		0.1	5	97	90-103	6		(sum a+b)	
Dry beans	0.0069	0.0069	5	84	73-96	11	<0.3LOQ	m/z 352 → 312	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
		0.069	5	83	79-86	3	(n=2)	diastereomer a	
	0.0069	0.0069	5	90	71-119	22	<0.3LOQ	m/z 352 → 332	
		0.069	5	82	77-90	5.8	(n=2)	diastereomer a	
	0.0013	0.0013	5	104	81-129	17	<0.3LOQ	m/z 352 → 312	
		0.013	5	85	72-104	15	(n=2)	diastereomer b	
	0.0013	0.0013	5	95	63-120	25	<0.3LOQ	m/z 352 → 332	
		0.013	5	78	75-82	3.3	(n=2)	diastereomer b	
Grapes	0.0069	0.0069	7	101	92-108	6.6	<0.3LOQ	m/z 352 → 332	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
		0.069	5	94	87-101	5.6	(n=2)	diastereomer a	
	0.0069	0.0069	7	95	88-104	6.3	<0.3LOQ	m/z 352 → 312	
		0.069	5	93	85-99	5.8	(n=2)	diastereomer a	
	0.0013	0.0013	7	105	94-112	8.0	<0.3LOQ	m/z 352 → 332	
		0.013	5	89	82-95	6.7	(n=2)	diastereomer b	
	0.0013	0.0013	7	104	91-112	8.6	<0.3LOQ	m/z 352 → 312	
		0.013	5	99	82-100	8.2	(n=2)	diastereomer b	
Grapes	0.0069	0.0069	5	77	71-84	9.7	<0.3LOQ	m/z 352 → 312	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
		0.069	5	71	68-74	3.5	(n=2)	diastereomer a	
	0.0069	0.0069	5	70	62-84	12	<0.3LOQ	m/z 352 → 332	
		0.069	5	73	69-74	2.8	(n=2)	diastereomer a	
	0.0013	0.0013	5	78	70-92	11	<0.3LOQ	m/z 352 → 312	
		0.013	5	71	69-72	1.7	(n=2)	diastereomer b	
	0.0013	0.0013	5	82	67-98	14	<0.3LOQ	m/z 352 → 332	
		0.013	5	73	66-75	9	(n=2)	diastereomer b	
Grapes	0.01	0.01	5	106	95-111	7	<0.01 (2)	m/z 352 → 312	Stanislowski, 2016, 2015RES-IFP2155 (P 3770)
		0.1	5	102	92-109	8		(sum a+b)	
	0.01	0.01	5	105	85-118	13	<0.01 (2)	m/z 352 → 332	
		0.1	5	97	88-106	7		(sum a+b)	
Oil seed rape crude oil	0.0069	0.0069	7	113	98-119	7.6	<0.3LOQ	m/z 352 → 332	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
		0.069	5	112	108-117	3.4	(n=2)	diastereomer a	
	0.0069	0.0069	7	114	102-122	7.0	<0.3LOQ	m/z 352 → 312	
		0.069	5	114	110-118	2.5	(n=2)	diastereomer a	
	0.0013	0.0013	5	78	69-88	11	<0.3LOQ	m/z 352 → 332	
		0.013	5	86	78-107	14	(n=2)	diastereomer b	
	0.0013	0.0013	7	84	75-91	7.9	<0.3LOQ	m/z 352 → 312	
		0.013	5	84	81-87	3.8	(n=2)	diastereomer b	
Oil seed	0.0069	0.0069	7	80	71-86	7.6	<0.3LOQ	m/z 352 → 332	Riccelli, 2017a,

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
rape straw		0.069	5	75	71-83	7.2	(n=2)	diastereomer a	2017RES-IFP3206 (RA.17.01)
	0.0069	0.0069	7	81	71-92	8.5	<0.3LOQ	m/z 352 → 312	
		0.069	5	78	73-84	7.6	(n=2)	diastereomer a	
	0.0013	0.0013	7	84	72-94	12	<0.3LOQ	m/z 352 → 332	
		0.013	5	77	70-83	7.5	(n=2)	diastereomer b	
	0.0013	0.0013	7	81	72-94	10	<0.3LOQ	m/z 352 → 312	
		0.013	5	77	70-85	8.4	(n=2)	diastereomer b	
Oil seed rape whole plant	0.0069	0.0069	7	82	74-95	8.8	<0.3LOQ	m/z 352 → 332	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
		0.069	5	74	70-78	4.2	(n=2)	diastereomer a	
	0.0069	0.0069	7	81	75-94	8.2	<0.3LOQ	m/z 352 → 312	
		0.069	5	74	70-78	4.5	(n=2)	diastereomer a	
	0.0013	0.0013	7	87	78-103	10	<0.3LOQ	m/z 352 → 332	
		0.013	5	77	75-81	3.2	(n=2)	diastereomer b	
	0.0013	0.0013	7	89	78-106	10	<0.3LOQ	m/z 352 → 312	
		0.013	5	77	75-82	3.7	(n=2)	diastereomer b	
Radish leaves	0.01	0.01	3	96	83-116	19		312 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
		0.1	1	-	82	-		diastereomer a	
	0.01	0.01	3	96	87-114	17		332 m/z	
		0.1	1	-	83	-		diastereomer a	
	0.01	0.01	3	98	87-119	19		312 m/z	
		0.1	1	-	86	-		diastereomer b	
	0.01	0.01	3	99	87-119	18		332 m/z	
		0.1	1	-	88	-		diastereomer b	
Radish roots	0.01	0.01	3	91	86-96	5.6		312 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
		0.1	1	-	90	-		diastereomer a	
	0.01	0.01	3	90	94-88	3.7		332 m/z	
		0.1	1	-	90	-		diastereomer a	
	0.01	0.01	3	95	90-97	4.0		312 m/z	
		0.1	1	-	87	-		diastereomer b	
	0.01	0.01	3	94	84-103	10		332 m/z	
		0.1	1	-	88	-		diastereomer b	
Soya bean forage	0.01	0.01	3	94	91-97	3.2		312 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
		0.1	1	-	85	-		diastereomer a	
	0.01	0.01	3	95	91-97	3.2		332 m/z	
		0.1	1	-	85	-		diastereomer a	
	0.01	0.01	3	90	87-97	6.2		312 m/z	
		0.1	1	-	84	-		diastereomer b	
	0.01	0.01	3	96	90-100	5.2		332 m/z	
		0.1	1	-	83	-		diastereomer b	
Soya bean hay	0.01	0.01	3	82	81-83	1.0		312 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
		0.1	1	-	74	-		diastereomer a	
	0.01	0.01	3	84	83-84	1.0		332 m/z	
		0.1	1	-	73	-		diastereomer a	
	0.01	0.01	3	85	81-87	4.4		312 m/z	
		0.1	1	-	80	-		diastereomer b	
	0.01	0.01	3	88	87-90	2.1		332 m/z	
		0.1	1	-	79	-		diastereomer b	
Soya bean seed [a]	0.0069	0.0069	5	66	55-77	14	<0.3LOQ	m/z 352 → 312	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
		0.069	5	72	68-77	5.7	(n=2)	diastereomer a	
	0.0069	0.0069	5	67	65-93[b]	3.2	<0.3LOQ	m/z 352 → 332	
		0.069	5	74	67-78	5.6	(n=2)	diastereomer a	
	0.0013	0.0013	5	72	44-94	29	<0.3LOQ	m/z 352 → 312	
		0.013	5	71	66-78	7.4	(n=2)	diastereomer b	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
	0.0013	0.0013	5	78	47-106	27	<0.3LOQ	m/z 352 → 332	
		0.013	5	75	64-82	10	(n=2)	diastereomer b	
Soya bean seeds	0.01	0.01	5	97	74-118	18	<0.01 (2)	m/z 352 → 312	Stanislowski, 2016, 2015RES-IFP2155 (P 3770G)
		0.1	5	87	75-92	8		(sum a+b)	
	0.01	0.01	5	96	84-112	11	<0.01 (2)	m/z 352 → 332	
		0.1	5	87	91-93	6		(sum a+b)	
Sugar beet leaves	0.01	0.01	5	97	90-107	9	<0.01 (2)	m/z 352 → 312	Stanislowski, 2016, 2015RES-IFP2155 (P 3770)
		0.1	5	106	104-110	3		(sum a+b)	
	0.01	0.01	5	110	101-119	7	<0.01 (2)	m/z 352 → 332	
		0.1	5	105	96-114	6		(sum a+b)	
Sugar beet roots	0.01	0.01	5	103	97-111	6	<0.01 (2)	m/z 352 → 312	Stanislowski, 2016, 2015RES-IFP2155 (P 3770G)
		0.1	5	110	109-114	4		(sum a+b)	
	0.01	0.01	5	110	100-124	9	<0.01 (2)	m/z 352 → 332	
		0.1	5	105	101-108	3		(sum a+b)	
Tomato mature	0.01	0.01	3	87	86-90	2.9		m/z 352 → 312	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.01)
		0.1	1	-	102	-		diastereomer a	
	0.01	0.01	3	86	84-90	3.5		m/z 352 → 332	
		0.1	1	-	100	-		diastereomer a	
	0.01	0.01	3	85	74-94	12		m/z 352 → 312	
		0.1	1	-	98	-		diastereomer b	
	0.01	0.01	3	86	84-87	2.1		m/z 352 → 332	
		0.1	1	-	98	-		diastereomer b	
Wheat forage	0.0069	0.0069	5	67	60-73	8.6	<0.3LOQ	m/z 352 → 312	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
		0.069	5	78	74-82	2.9	(n=2)	diastereomer a	
	0.0069	0.0069	5	76	73-81	4.7	<0.3LOQ	m/z 352 → 332	
		0.069	5	79	75-86	5.2	(n=2)	diastereomer a	
	0.0013	0.0013	5	80	66-97	15	<0.3LOQ	m/z 352 → 312	
		0.013	5	79	73-88	6.7	(n=2)	diastereomer b	
	0.0013	0.0013	5	75	65-86	11	<0.3LOQ	m/z 352 → 332	
		0.013	5	82	77-86	4.4	(n=2)	diastereomer b	
Wheat dry gluten	0.0069	0.0069	7	93	77-101	9.6	<0.3LOQ	m/z 352 → 332	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
		0.069	5	85	74-107	15	(n=2)	diastereomer a	
	0.0069	0.0069	7	94	78-102	9.5	<0.3LOQ	m/z 352 → 312	
		0.069	5	84	72-107	16	(n=2)	diastereomer a	
	0.0013	0.0013	7	98	84-109	9.2	<0.3LOQ	m/z 352 → 332	
		0.013	5	90	77-111	15	(n=2)	diastereomer b	
	0.0013	0.0013	7	99	88-109	9.3	<0.3LOQ	m/z 352 → 312	
		0.013	5	90	78-108	13	(n=2)	diastereomer b	
Wheat grain	0.0069	0.0069	7	110	105-116	4.9	<0.3LOQ	m/z 352 → 332	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
		0.069	5	106	103-109	2.7	(n=2)	diastereomer a	
	0.0069	0.0069	7	106	98-115	5.6	<0.3LOQ	m/z 352 → 312	
		0.069	5	103	99-106	2.4	(n=2)	diastereomer a	
	0.0013	0.0013	7	110	106-119	5.8	<0.3LOQ	m/z 352 → 332	
		0.013	5	107	103-111	2.7	(n=2)	diastereomer b	
	0.0013	0.0013	7	110	100-119	5.6	<0.3LOQ	m/z 352 → 312	
		0.013	5	106	102-109	2.6	(n=2)	diastereomer b	
Wheat grain [a]	0.0069	0.0069	5	62	50-71	15	<0.3LOQ	m/z 352 → 312	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
		0.069	5	74	39[b]-76	3.9	(n=2)	diastereomer a	
	0.0069	0.0069	5	68	53-80	15	<0.3LOQ	m/z 352 → 332	
		0.069	5	70	49[b]-75	5.4	(n=2)	diastereomer a	
	0.0013	0.0013	5	59	41-86	28	<0.3LOQ	m/z 352 → 312	
		0.013	5	77	69-80	5.9	(n=2)	diastereomer b	
	0.0013	0.0013	5	78	65-105	20	<0.3LOQ	m/z 352 → 332	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
		0.013	5	73	67-84	10	(n=2)	diastereomer b	
Wheat straw	0.0069	0.0069 0.069	7 5	75 71	71-79 66-75	4.1 5.3	<0.3LOQ (n=2)	m/z 352 → 332 diastereomer a	Riccelli, 2017a, 2017RES-IFP3206 (RA.17.01)
	0.0069	0.0069 0.069	7 5	76 74	70-82 68-75	5.2 3.7	<0.3LOQ (n=2)	m/z 352 → 312 diastereomer a	
	0.0013	0.0013 0.013	7 5	81 70	75-88 68-75	5.2 4.4	<0.3LOQ (n=2)	m/z 352 → 332 diastereomer b	
	0.0013	0.0013 0.013	7 5	77 70	72-81 68-72	4.5 2.1	<0.3LOQ (n=2)	m/z 352 → 312 diastereomer b	
Wheat straw	0.0069	0.0069 0.069	5 5	68 69	58-74 65-72	8.9 5.0	<0.3LOQ (n=2)	m/z 352 → 312 diastereomer a	Sahvorost, 2018b 2017AMT-IFP3872 (RA.17.01)
	0.0069	0.0069 0.069	5 5	75 68	61-93 64-72	18 4.7	<0.3LOQ (n=2)	m/z 352 → 332 diastereomer a	
	0.0013	0.0013 0.013	5 5	67 66	46-88 63-72	23 5.0	<0.3LOQ (n=2)	m/z 352 → 312 diastereomer b	
	0.0013	0.0013 0.013	5 5	76 68	62-91 66-70	19 3.1	<0.3LOQ (n=2)	m/z 352 → 332 diastereomer b	
Wheat straw	0.01	0.01 0.1	5 5	110 95	94-132 91-100	14 4	<0.01 (2)	m/z 352 → 312 (sum a+b)	Stanislowski, 2016, 2015RES-IFP2155 (P 3770)
	0.01	0.01 0.1	5 5	89 98	74-108 95-106	15 5	<0.01 (2)	m/z 352 → 332 (sum a+b)	

Notes:

[a] second attempt

[b] considered an outlier

LC-MS/MS method RA.17.19 for determination of pyrazole carboxamide, pyrazole carboxylic acid and N-desmethyl-pyrazole carboxylic acid

An analytical method was developed and validated for the determination of pyrazole carboxamide, pyrazole carboxylic acid and N-desmethyl-pyrazole-caborboxylic acid in plant matrices [Ricelli, 2017b, 2017AMT-IFP3922]. A full validation was performed for wheat grain (high starch content), lettuce (high water content), soya bean seed (high oil content), dry bean (high protein content), strawberry (high acid content), wheat straw (difficult matrix). This method is an extension of method RA.17.01.

Samples of each matrix (1.0 g) were extracted with water (5 mL) and acetonitrile (10 mL) added one after the other. For high protein matrices (dry bean) acetonitrile was added prior to water and for wheat straw, water and acetonitrile were added simultaneously. HCl was added to 4N HCl and left to hydrolysed at 80 °C for 2 hours with sonication. NaOH was added to neutralize (pH 4). Acetone (5 mL) and water (20 mL) were added and mixed subsequently. After sonication and centrifugation samples were cleaned-up by ChemElute cartridge (20 minutes soaking and elution with ethyl acetate). After evaporation the residue was reconstituted in acetone/water (40/60 v/v), stirred and sonicated. Where applicable matrix matched standards where added and analysed by LC-MS/MS.

The limit of quantitation (LOQ) was 0.010 mg/kg for all analytes. The molecular weight (MW) ratios of the analytes in relation to the MW of parent fluindapyr as listed below can be used to calculate LOQs expressed as parent equivalents where needed.

Table 92 Overview of molecular weights, LOQ and conversion factors

Analyte	Transition (m/z) [a]	Molecular Weight	LOQ (analyte)	Conversion Factor
Fluindapyr	-	351.37	0.010	1.000
Pyrazole carboxylic acid	177 → 137 (+) 175 → 91 (-) 175 → 111 (-)	176.12	0.010	1.995
Pyrazole carboxamide	176 → 136 (+) 176 → 156 (+)	175.14	0.010	2.006
N-desmethyl-pyrazole carboxylic acid	163 → 123 (+) at H 3 161 → 141 (-)	162.10	0.010	2.168

Notes:

[a] Positive (+) or negative (-) polarity.

An independent method validation was carried out within a rotational crop field trial [Skaggs, 2019, 2018RES-IFP4200]. Field samples from radish tops, radish roots, mustard greens, wheat forage, wheat hay, wheat grain and wheat straw were collected, extracted and analysed for pyrazole carboxamide, pyrazole carboxylic acid and N-desmethyl-pyrazole-caborboxylic acid.

The metabolites were extracted by solvent:water, followed by addition of hydrochloric acid and subsequent hydrolysis with 4N HCl at 75 °C for 2 hours. After adjustment of pH (to approximately 4) with NaOH and dilution, analysis was performed using LC-MS/MS with primary and confirmatory mass transition. Since in the primary study matrix effects were found to be significant, matrix matched standards were included in the method to correct for potential matrix effect.

In addition, validation studies were carried out for determination of pyrazole carboxamide in almond nutmeat [Skaggs, 2019a, 2018RES-FNF4542] and soya bean seeds, hulls, meal, and refined oil [Skaggs, 2019b, 2018RES-IFP4183]. The results are summarized in Table 93, Table 94 and Table 95 for pyrazole carboxamide, pyrazole carboxylic acid and N-DesMet-pyrazole carboxylic acid.

Table 93 Validation results for pyrazole carboxamide with LC-MS/MS method RA.17.19

Commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, ion transition	reference (method)
Pyrazole carboxamide									
Almond nutmeat	0.01	0.01 0.1	5 5	84 93	80-87 91-94	3.8 1.2	0<0.2LOQ (n=nr)	matrix matched standards 0.04-10 ng/mL r≥0.99 1/× weighted 176 → 136	Skaggs, 2019a, 2018RES-FNF4542 (RA.17.19)
	0.01	0.01 0.1	5 5	85 92	82-86 91-94	1.6 0.9	0<0.2LOQ (n=nr)	176 → 156	
Cabbage immature	0.01	0.01 0.1	3 1	106 -	101-109 112	4.2 -		176 → 156 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	108 -	105-112 113	3.6 -		176 → 136 m/z	
Carrot leaves	0.01	0.01 0.1	3 1	80 -	73-89 96	10 -		176 → 156 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	77 -	70-86 96	11 -		176 → 136 m/z	

Commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, ion transition	reference (method)
Carrot roots	0.01	0.01 0.1	3 1	85	82-88-85	3.3 -		176 → 156 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	88 -	83-92 86	4.9 -		176 → 136 m/z	
Dry bean	0.01	0.01 0.1	7 5	79 74	78-82 71-75	3 2.2	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.1 ng/mL r≥0.999 1/× weighted 176 → 156	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	80 73	78-84 70-75	3.5 2.9	<0.3LOQ (n=2)	176 → 136	
Leafy vegetable	0.01	0.01 0.1	5 5	78 81	76-80 78-85	2.1 3.2	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 176 → 136	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	76 78	75-78 77-80	1.7 1.6	<0.3LOQ (n=2)	176 → 156	
Lettuce	0.01	0.01 0.1	7 5	76 90	69-82 90-91	5.3 0.8	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.1 ng/mL r≥0.999 1/× weighted 176 → 156	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	76 90	70-82 89-92	5.1 1.1	<0.3LOQ (n=2)	176 → 136	
Radish leaves	0.01	0.01 0.1	5 5	102 104	96-11 101-107	5.7 2.2	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 176 → 136	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	101 104	95-107 101-106	5.5 1.8	<0.3LOQ (n=2)	176 → 156	
Radish leaves	0.01	0.01 0.1	3 1	104 -	100-109 88	4.7 -		156 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	98 -	92-102 86	3.6		136 m/z	
Radish roots	0.01	0.01 0.1	5 5	75 75	72-77 71-81	2.8 5.7	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 176 → 136	Skaggs, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	74 75	71-77 72-81	3.5 5.3	<0.3LOQ (n=2)	176 → 156	
Radish roots	0.01	0.01 0.1	3 1	88 -	84-94 82	6.1 -		156 m/z	Huaulmé, 2020a, 2017RES-IFP3569
	0.01	0.01 0.1	3 1	84 -	80-90 80	6.1 -		136 m/z	

Commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, ion transition	reference (method)
									(RA.17.19)
Soya bean hulls	0.01	0.01 0.1	5 5	84 83	81-90 80-86	3.9 2.9	0<0.2LOQ (n=nr)	matrix matched standards 0.04-10 ng/mL r≥0.99 1/× weighted 176 → 136	Skaggs, 2019b, 2018RES-IFP4183 (RA.17.19)
	0.01	0.01 0.1	5 5	81 83	79-86 80-86	3.5 3.4	0<0.2LOQ (n=nr)	176 → 156	
Soya bean meal	0.01	0.01 0.1	5 5	73 83	71-81 73-90	5.5 7.7	0<0.2LOQ (n=nr)	matrix matched standards 0.04-10 ng/mL r≥0.99 1/× weighted 176 → 136	Skaggs, 2019b, 2018RES-IFP4183 (RA.17.19)
	0.01	0.01 0.1	5 5	74 83	71-80 73-90	5.2 7.4	0<0.2LOQ (n=nr)	176 → 156	
Soya bean refined oil	0.01	0.01 0.1	5 5	82 81	81-84 80-83	1.2 1.8	0<0.2LOQ (n=nr)	matrix matched standards 0.04-10 ng/mL r≥0.99 1/× weighted 176 → 136	Skaggs, 2019b, 2018RES-IFP4183 (RA.17.19)
	0.01	0.01 0.1	5 5	82 81	81-82 80-82	0.6 1.5	0<0.2LOQ (n=nr)	176 → 156	
Soya bean seeds	0.01	0.01 0.1	7 5	78 91	68-87 84-94	10 7	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.1 ng/mL r≥0.999 1/× weighted 176 → 156	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	79 91	68-90 85-100	12 7	<0.3LOQ (n=2)	176 → 136	
Soya bean seeds	0.01	0.01 0.1	5 5	87 75	85-89 74-76	1.8 1.2	0<0.2LOQ (n=nr)	matrix matched standards 0.04-10 ng/mL r≥0.99 1/× weighted 176 → 136	Skaggs, 2019b, 2018RES-IFP4183 (RA.17.19)
	0.01	0.01 0.1	5 5	75 76	71-77 74-77	3.8 1.7	0<0.2LOQ (n=nr)	176 → 156	
Soya bean forage	0.01	0.01 0.1	3 1	100 -	91-107 83	8.6 -		156 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA17.19)
	0.01	0.01 0.1	3 1	99 -	89-106 84	9.2 -		136 m/z	
Soya bean hay	0.01	0.01 0.1	3 1	74 -	70-79 77	5.6 -		156 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	74 -	70-77 76	4.4 -		136 m/z	
Strawberry	0.01	0.01 0.1	7 5	76 78	69-80 75-80	4.8 2.7	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.1 ng/mL	Riccelli, 2017b, 2017AMT-IFP3922

Commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, ion transition	reference (method)
								r≥0.999 1/× weighted 176 → 156	(RA.17.19)
	0.01	0.01 0.1	7 5	77 77	71-80 74-80	4.3 3.2	<0.3LOQ (n=2)	176 → 136	
Tomato mature	0.01	0.01 0.1	3 1	86 -	79-92 108	7.6 -		156 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	83 -	77-93 109	9.8 -		136 m/z	
Wheat forage	0.01	0.01 0.1	5 5	85 83	81-91 79-87	4.7 3.9	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 176 → 136	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	87 84	81-90 81-87	4.3 3.4	<0.3LOQ (n=2)	176 → 156	
Wheat grain	0.01	0.01 0.1	7 5	76 81	69-82 76-83	5.4 3.5	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.1 ng/mL r≥0.999 1/× weighted 176 → 156	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	78 81	70-83 76-83	5.9 3.4	<0.3LOQ (n=2)	176 → 136	
Wheat grain	0.01	0.01 0.1	5 5	77 74	75-81 72-76	3.2 2.5	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 176 → 136	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	78 74	75-83 72-77	5.1 3.5	<0.3LOQ (n=2)	176 → 156	
Wheat hay	0.01	0.01 0.1	5 5	77 74	74-80 71-76	3.0 2.5	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 176 → 136	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	79 76	75-83 73-78	5.8 2.6	<0.3LOQ (n=2)	176 → 156	
Wheat straw	0.01	0.01 0.1	7 5	76 108	70-82 99-114	6.1 7.8	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.1 ng/mL r≥0.999 1/× weighted 176 → 156	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	92 112	82-110 99-126	10 9.3	<0.3LOQ (n=2)	176 → 136	
Wheat straw	0.01	0.01 0.1	5 5	79 76	77-80 75-77	1.4 0.9	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)

Commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, ion transition	reference (method)
								1/× weighted 176 → 136	
	0.01	0.01 0.1	5 5	102 77	95-108 75-79	4.6 2.0	<0.3LOQ (n=2)	176 → 156	

Notes:

nr = Not reported

Table 94 Validation results for pyrazole carboxylic acid with LC-MS/MS method RA.17.19

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
Pyrazole carboxylic acid									
Cabbage immature	0.01	0.01 0.1	3 1	96 -	93-98 104	2.6 -		177 → 137 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	82 -	72-87 107	8.3 -		175 → 91 m/z	
Carrot leaves	0.01	0.01 0.1	3 1	80 -	75-86 94	6.5 -		177 → 137 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	93 -	86-105 107	11 -		175 → 91 m/z	
Carrot roots	0.01	0.01 0.1	3 1	90 -	84-95 95	6.4 -		177 → 137 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	90 -	71-101 106	18 -		175 → 91 m/z	
Dry bean	0.01	0.01 0.1	7 5	66 68	58-82 60-74	12 8.2	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.2 ng/mL r≥0.999 1/× weighted 177 → 137	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	82 80	75-99 70-105	10 18	<0.3LOQ (n=2)	175 → 91	
	0.01	0.01 0.1	7 5	85 78	74-100 70-98	10 15	<0.3LOQ (n=2)	175 → 111	
Lettuce	0.01	0.01 0.1	7 5	86 80	77-96 76-87	7.3 5.4	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.2 ng/mL r≥0.999 1/× weighted 177 → 137	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	102 83	98-108 80-89	7.1 4.6	<0.3LOQ (n=2)	175 → 91	
	0.01	0.01 0.1	7 5	95 82	87-109 78-88	8.8 4.6	<0.3LOQ (n=2)	175 → 111	
Leafy vegetables	0.01	0.01 0.1	5 5	116 116	109-118 111-119	3.4 3.1	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL	Skaggs&Afedi, 2019, 2018RES-IFP4200

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
								r≥0.999 1/× weighted 175 → 91	(RA.17.19)
	0.01	0.01 0.1	5 5	105 113	102-110 109-117	2.9 3	<0.3LOQ (n=2)	175 → 111	
Radish leaves	0.01	0.01 0.1	5 5	104 106	99-108 87-117	3.3 12	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 175 → 91	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	103 105	100-106 89-114	2.7 9.4	<0.3LOQ (n=2)	175 → 111	
Radish leaves	0.01	0.01 0.1	3 1	87 -	85-89 91	2.3 -		177 → 137 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	106 -	100-112 97	5.9-		175 → 91 m/z	
Radish roots	0.01	0.01 0.1	5 5	98 117	88-108 14-118	7.3 1.7	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 175 → 91	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	106 113	98-109 111-114	4.5 1.3	<0.3LOQ (n=2)	175 → 111	
Radish roots	0.01	0.01 0.1	3 1	89 -	86-96 81	6.8 -		177 → 137 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	92 -	90-93 93	1.6 -		175 → 91 m/z	
Soya beans seeds	0.01	0.01 0.1	7 5	87 110	63-108 106-115	17 3.7	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.2 ng/mL r≥0.999 1/× weighted 177 → 137	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	85 108	69-107 99-114	17 6.3	<0.3LOQ (n=2)	175 → 91	
	0.01	0.01 0.1	7 5	87 96	74-95 90-108	9.5 8.3	<0.3LOQ (n=2)	175 → 111	
Soya bean forage	0.01	0.01 0.1	3 1	82 -	70-89 113	12 -		177 → 137 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA17.19)
	0.01	0.01 0.1	3 1	99 -	83-110 105	14 -		175 → 91 m/z	
Soya bean hay	0.01	0.01 0.1	3 1	73 -	70-75 81	3.2 -		177 → 137 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	72 -	70-75 81	3.4 -		175 → 91 m/z	
Strawberry	0.01	0.01 0.1	7 5	79 98	73-85 87-106	6.3 7.5	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.2 ng/mL	Riccelli, 2017b, 2017AMT-IFP3922

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
								r≥0.999 1/× weighted 177 → 137	(RA.17.19)
	0.01	0.01 0.1	7 5	106 119	90-116 101-129	8.7 8.7	<0.3LOQ (n=2)	175 → 91	
	0.01	0.01 0.1	7 5	102 120	94-115 106-130	8.1 7.5	<0.3LOQ (n=2)	175 → 111	
Tomato mature	0.01	0.01 0.1	3 1	88 -	86-90 98	2.7 -		177 → 137 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	81 -	79-83 107	2.5 -		175 → 91 m/z	
Wheat forage	0.01	0.01 0.1	5 5	114 110	106-117 101-115	3.8 5.5	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 175 → 91	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	104 109	88-111 99-115	8.9 6.4	<0.3LOQ (n=2)	175 → 111	
Wheat grain	0.01	0.01 0.1	7 5	84 91	73-90 86-94	8 3.1	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.2 ng/mL r≥0.999 1/× weighted 177 → 137	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	100 116	90-110 112-120	8.6 2.7	<0.3LOQ (n=2)	175 → 91	
	0.01	0.01 0.1	7 5	96 113	82-106 108-116	8.3 3.3	<0.3LOQ (n=2)	175 → 111	
Wheat grain	0.01	0.01 0.1	5 5	112 113	102-115 109-115	4.9 2.3	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 175 → 91	Skaggs, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	114 113	109-116 108-115	2.6 2.6	<0.3LOQ (n=2)	175 → 111	
Wheat hay	0.01	0.01 0.1	5 5	102 112	93-106 106-117	5.2 3.9	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 175 → 91	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	105 113	100-110 110-115	4.5 1.7	<0.3LOQ (n=2)	175 → 111	
Wheat straw	0.01	0.01 0.1	7 5	84 94	77-96 84-108	7.8 10	<0.3LOQ (n=2)	≥3 matrix matched standards 0.041-5.2 ng/mL r≥0.999 1/× weighted 177 → 137	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01	7	127	115-130	6.2	<0.3LOQ	175 → 91	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
		0.1	5	113	106-119	5.3	(n=2)		
	0.01	0.01 0.1	7 5	119 115	101-140 112-120	11 2.7	<0.3LOQ (n=2)	175 → 111	
Wheat straw	0.01	0.01 0.1	5 5	107 111	92-112 106-114	7.8 3.1	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 175 → 91	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	107 113	95-116 106-116	9.1 3.5	<0.3LOQ (n=2)	175 → 111	

Table 95 Validation results for N-desmethyl-pyrazole carboxylic acid with LC-MS/MS method RA.17.19

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
N-desmethyl-pyrazole carboxylic acid									
Cabbage immature	0.01	0.01 0.1	3 1	75 -	70-78 77	5.7 -		163 → 123 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	100	92-108-92	7.5 -		161 → 141 m/z	
Carrot leaves	0.01	0.01 0.1	3 1	90 -	82-99 94	9.5 -		163 → 123 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	85 -	92 94	12 -		161 → 141 m/z	
Carrot roots	0.01	0.01 0.1	3 1	78 -	72-91 91	13 -		163 → 123 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	85 -	78-92 90	8.2 -		161 → 141 m/z	
Dry bean	0.01	0.01 0.1	7 5	89 81	79-111 74-88	12 8.4	<0.3LOQ (n=2)	≥3 matrix matched standards 0.042-5.2 ng/mL r≥0.999 1/× weighted 163 → 123	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	108 97	98-128 88-103	10 8.4	<0.3LOQ (n=2)	161 → 141 (pH 3)	
Leafy vegetables	0.01	0.01 0.1	5 5	98 97	93-102 94-99	4.0 2.0	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 161 → 141	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	99	94-10296 94-99	3.7 2.3	<0.3LOQ (n=2)	163 → 123	
Lettuce	0.01	0.01 0.1	7 5	76 66	69-79 64-69	5.9 3.2	<0.3LOQ (n=2)	≥3 matrix matched standards 0.042-5.2 ng/mL r≥0.999	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
								1/× weighted 163 → 123	
	0.01	0.01 0.1	7 5	78 64	74-82 61-71	4.4 6.5	<0.3LOQ (n=2)	161 → 141	
Radish leaves	0.01	0.01 0.1	5 5	87 85	77-94 70-91	7.5 11	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 161 → 141	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	84 85	73-91 71-93	8.3 9.8	<0.3LOQ (n=2)	163 → 123	
Radish leaves	0.01	0.01 0.1	3 1	89 -	86-94 83	4.4 -		163 → 123 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	96 -	94-97 84	2.0 -		161 → 141 m/z	
Radish roots	0.01	0.01 0.1	5 5	86 95	75-108 93-97	15 2.0	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 161 → 141	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	87 95	75-108 93-98	15 2.0	<0.3LOQ (n=2)	163 → 123	
Radish roots	0.01	0.01 0.1	3 1	84 -	82-85 64	1.7 -		163 → 123 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	81 -	78-85 70	4.1 -		161 → 141 m/z	
Soya beans seeds	0.01	0.01 0.1	7 5	71 71	63-80 62-75	8.8 8.5	<0.3LOQ (n=2)	≥3 matrix matched standards 0.042-5.2 ng/mL r≥0.999 1/× weighted 163 → 123	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	83 85	68-89 81-94	11 6.4	<0.3LOQ (n=2)	161 → 141	
Soya bean forage	0.01	0.01 0.1	3 1	76 -	70-84 88	9.3 -		163 → 123 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA17.19)
	0.01	0.01 0.1	3 1	70 -	64-73 87	7.7 -		161 → 141 m/z	
Soya bean hay	0.01	0.01 0.1	3 1	72 -	71-74 82	2.2 -		163 → 123 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	73 -	70-75 81	3.3 -		161 → 141 m/z	
Strawberry	0.01	0.01 0.1	7 5	70 81	92-77 73-93	8.7 9.4	<0.3LOQ (n=2)	≥3 matrix matched standards 0.042-5.2 ng/mL r≥0.999 1/× weighted 163 → 123	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
	0.01	0.01 0.1	7 5	92 95	83-100 86-105	7.6 7.7	<0.3LOQ (n=2)	161 → 141	
Tomato mature	0.01	0.01 0.1	3 1	91 -	86-95 96	5.2 -		163 → 123 m/z	Huaulmé, 2020a, 2017RES-IFP3569 (RA.17.19)
	0.01	0.01 0.1	3 1	92 -	87-96 91	5.1 -		161 → 141 m/z	
Wheat forage	0.01	0.01 0.1	5 5	92 85	75-106 79-91	12 5.9	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 161 → 141	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	93 85	79-107 79-92	11 6.2	<0.3LOQ (n=2)	163 → 123	
Wheat grain	0.01	0.01 0.1	7 5	76 87	68-81 81-88	6.4 3.7	<0.3LOQ (n=2)	≥3 matrix matched standards 0.042-5.2 ng/mL r≥0.999 1/× weighted 163 → 123	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	79 97	69-88 88-104	9.3 6.7	<0.3LOQ (n=2)	161 → 141	
Wheat grain	0.01	0.01 0.1	5 5	95 90	89-103 85-92	6.3 3.1	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 161 → 141	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	94 88	88-102 83-91	6.5 3.5	<0.3LOQ (n=2)	163 → 123	
Wheat hay	0.01	0.01 0.1	5 5	79 82	75-82 78-87	3.4 4.3	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 161 → 141	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01 0.1	5 5	78 81	72-82 77-86	4.7 4.4	<0.3LOQ (n=2)	163 → 123	
Wheat straw	0.01	0.01 0.1	7 5	74 96	63-90 86-105	14 7.4	<0.3LOQ (n=2)	≥3 matrix matched standards 0.042-5.2 ng/mL r≥0.999 1/× weighted 163 → 123	Riccelli, 2017b, 2017AMT-IFP3922 (RA.17.19)
	0.01	0.01 0.1	7 5	90 104	74-107 97-112	14 5.8	<0.3LOQ (n=2)	161 → 141	
Wheat straw	0.01	0.01 0.1	5 5	86 82	80-95 72-90	6.8 8.4	<0.3LOQ (n=2)	9 matrix matched standards 0.02-10 ng/mL r≥0.999 1/× weighted 161 → 141	Skaggs&Afedi, 2019, 2018RES-IFP4200 (RA.17.19)
	0.01	0.01	5	85	79-96	7.9	<0.3LOQ	163 → 123	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference (method)
		0.1	5	82	72-89	7.9	(n=2)		

Analytical methods for enforcement in animal commodities

The Meeting received the description and validation for an analytical method for the determination of fluindapyr, and its metabolites N-DesMet-fluindapyr, 1-OH-Met-fluindapyr, 1-COOH-fluindapyr and 1-OH-Met-N-DesMet-fluindapyr in animal commodities. These studies are summarized in the section analytical methods used in study reports on animal commodities. No validation results for existing multi-residue methods were submitted.

Analytical methods used in study reports in plant commodities

Field residue trials on field corn [Webber, 2018a, 2015RES-FNF1900, Webber, 2018b, 2016RES-FNF2453] were performed using the analytical methods PTRL Method P3770G for determination of fluindapyr, 3-OH-fluindapyr and fluindapyr-N-DesMet-glucoside and method RA.17.01 for determination of 1-OH-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and 1-COOH-fluindapyr. The same applies for the field residue trials on sorghum [Webber, 2018d, 2015RES-FNF1901, Webber, 2018e, 2016RES-FNF2455] and wheat [Webber, 2018f, 2016RES-FNF2456, Peterek, 2018, 2015RES-IFP1968, Ricelli, 2018, 2015RESIFP1950] and tree nuts [Webber, 2017a, 2016RES-FNF2450 and Webber, 2017b, 2016RES-FNF2451].

Analytical methods used in study reports in animal commodities

The Meeting received the description and validation for an analytical method, Method 133SRUS16R0208, for the determination of fluindapyr, and its metabolites N-DesMet-fluindapyr, 1-OH-Met-fluindapyr, 1-COOH-fluindapyr and 1-OH-Met-N-DesMet-fluindapyr in animal commodities.

HPLC-MS/MS Method 133SRUS16R0208 for the determination of fluindapyr, N-DesMet-fluindapyr, 1-OH-Met-fluindapyr, 1-COOH-fluindapyr and 1-OH-Met-N-DesMet-fluindapyr

HPLC-MS/MS Method 133SRUS16R0208 determines fluindapyr, N-DesMet-fluindapyr, 1-OH-Met-fluindapyr, 1-COOH-fluindapyr and 1-OH-Met-N-DesMet-fluindapyr in animal commodities. The method was used in the animal feeding studies [Brungardt, 2018, 2016RES-IFP2942 and Brungardt&Dixon, 2018, 2016RES-IFP2943] and storage stability study [Moore&Shepherd, 2018, 2016RES-IFP2945]. The development and validation of the method is described in Moore&Shepherd [2018, 2016RES-IFP2941].

Residues of fluindapyr and its metabolites were extracted from muscle, liver, and kidney by blending first with acetonitrile (2 ×). The extract was centrifuged. The samples were blended a second time with acetonitrile/water (7:3, v/v), centrifuged, and the extracts pooled. The extracts were brought up to a final volume of 100 mL with acetonitrile. For analysis of fluindapyr, a 1 mL aliquot was diluted with 4 mL of methanol/water (1:3, v/v), mixed well, and injected on LC-MS/MS. For analysis of fluindapyr-1-COOH, 1-OH-Met-fluindapyr, and 1-OH-Met-N-DesMet-fluindapyr, the extract was mixed with 4 N HCL and allowed to hydrolyse (at 80 °C for 60 minutes). After cooling, the sample was processed through an Oasis HLB cartridge and the analytes were eluted with acetonitrile/water (7:3, v/v). The sample were concentrated to dryness and reconstituted in 1 mL of methanol/water (1:4, v/v) for injection on LC-MS/MS.

Residues of fluindapyr were extracted from milk by shaking with acetonitrile. The extract was centrifuged. The extracts were brought up to a final volume of 50 mL with acetonitrile. A 1 mL aliquot was diluted with 4 mL of methanol/water (1:3, v/v), mixed well, and injected on LC-MS/MS.

Residues of fluindapyr and its metabolites were extracted from fat by blending with acetonitrile/hexane (1:1, v/v). After phase separation the procedure was split for analyses of the various analytes. For analysis of fluindapyr, a 0.5 mL aliquot of the acetonitrile layer was diluted and mixed with 2.5 mL of water. The sample was then processed through an Oasis HLB cartridge and the analytes were eluted with acetonitrile/water (7:3, v/v). The sample was concentrated to dryness and reconstituted in 1 mL of methanol/water (1:4, v/v) for injection on LC-MS/MS. For analysis of 1-OH-Met-fluindapyr, a 0.5 mL aliquot of the acetonitrile layer was mixed with 2.5 mL of 4 N HCL and allowed to hydrolyse (80 °C for 60 minutes). After cooling down, the sample was processed through an Oasis HLB cartridge and the analytes were eluted with acetonitrile/water (7:3, v/v). The sample was concentrated to dryness and reconstituted in 1 mL of methanol/water (1:4, v/v) for injection on LC-MS/MS.

Samples were analysed by LC-MS/MS at different primary transitions for quantitation and confirmation as summarized in Table 96. The linearity of detector response for fluindapyr and its metabolites was evaluated using matrix-matched standard solutions. The reported LOQ for each analyte was 0.01 mg/kg in all tissues and 0.005 mg/kg in milk. Validation results are shown in Table 97

Table 96 Ion transitions

Analyte	Ion transition (quantitation)	Ion transition (confirmation)
Fluindapyr	352 → 332	352 → 256
N-DesMet-fluindapyr	338 → 262	338 → 242
1-OH-Met-fluindapyr	368 → 310	368 → 330
1-OH-Met-N-DesMet-fluindapyr	354 → 296	354 → 145
Fluindapyr-1-COOH	382 → 336	382 → 296

An independent laboratory validation (ILV) was performed for residues in several animal matrices [Sahvorost, 2018c, 2017AMT-IFP3873]: fluindapyr in bovine, muscle, cow milk, bovine fat, bovine liver and poultry eggs; N-DesMet-fluindapyr in bovine liver and poultry eggs; diastereomers of 1-OH-Met-fluindapyr in bovine fat and liver and poultry eggs; diatereomers of 1-OH-Met-N-DesMet-fluindapyr in bovine liver and poultry eggs, and diastereomers of 1-carboxy-fluindapyr in bovine liver. Linearity was demonstrated by determination of matrix matched standards at 6 concentration levels covering the range from no more than 20 percent of the LOQ and at least +20 percent of the highest analyte concentration. The calibration curves obtained for both mass transitions were linear with the target correlation coefficients $r \geq 0.995$. Linear regression was performed with 1/× weighting. Validation results are shown in Table 97 to Table 101.

Note by the reviewer:

HPLC-MS/MS method 133SRUS16R0208 is considered

- valid (full validation) for the determination of fluindapyr, N-DesMet-fluindapyr, 1-OH-Met-fluindapyr (sum of both diastereomers), 1-OH-Met-N-DesMet-fluindapyr (sum of both diastereomers) in the range 0.01–0.1 mg/kg in bovine muscle, bovine fat, bovine liver, bovine kidney, poultry muscle (breast & thigh), poultry fat, poultry liver and eggs and in the range 0.005-0.05 mg/kg in milk.
- Valid (full validation) for the determination of 1-COOH-fluindapyr (sum of both diastereomers) in the range 0.01–0.1 mg/kg in bovine liver and bovine kidney.

Table 97 Validation results for fluindapyr with HPLC-MS/MS method 133SRUS16R0208

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
bovine muscle	0.01	0.01 0.1	5 5	99 94	96-106 92-96	4.2 1.6	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 352 → 332	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	100 96	96-108 95-98	4.6 1.2	<0.3LOQ (2)	352 → 256	
bovine muscle	0.01	0.01 0.1	5 5	101 96	98-102 90-99	1.5 3.8	<0.3LOQ (2)	352 → 332	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	99 97	96-101 92-100	1.8 3.3	<0.3LOQ (2)	352 → 256	
bovine muscle	0.01	0.01 0.1	5 5	89 92	86-94 89-96	3.3 3.4	<02LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine fat	0.01	0.01 0.1	5 5	80 79	72-84 78-82	5.8 2.4	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 352 → 332	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	80 80	73-85 78-84	6.2 3.0	<0.3LOQ (2)	352 → 256	
bovine fat [a]	0.01	0.01 0.1	5 5	77 74	53-96 64-83	2.1 9.4	<0.3LOQ (2)	352 → 332	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	75 70	51-95 63-81	22 9.5	<0.3LOQ (2)	352 → 256	
bovine mesenterial fat	0.01	0.01 0.1	5 5	80 84	65-88 76-89	11 6.9	<02LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine perirenal fat	0.01	0.01 0.1	5 5	80 76	65-98 70-86	16 9.0	<02LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
Bovine subcutaneous fat	0.01	0.01 0.1	5 5	76 83	73-79 82-85	3.0 1.4	<02LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine liver	0.01	0.01 0.1	5 5	96 96	91-101 95-98	3.5 1.0	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 352 → 332	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	98 98	97-100 96-99	0.9 1.2	<0.3LOQ (2)	352 → 256	
bovine liver	0.01	0.01 0.1	5 5	103 104 101	102- 99-102	1.1 0.9	<0.3LOQ (2)	352 → 332	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	102 101	99-104 100-	2.6 1.2	<0.3LOQ (2)	352 → 256	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
				103					
bovine liver	0.01	0.01 0.1	5 5	99 97	95-103 95-100	3.2 2.4	<02LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine kidney	0.01	0.01 0.1	5 5	95 98	92-98 94-101	2.5 2.9	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 352 → 332	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	95 97	92-98 95-99	2.9 1.9	<0.3LOQ (2)	352 → 256	
bovine kidney	0.01	0.01 0.1	5 5	92 94	73-109 81-103	14 9.4	<02LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine milk	0.005	0.005 0.05	5 5	87 87	78-94 84-90	7.3 2.4	<0.3LOQ (2)	7 standards matrix matched 0.0008-0.25 µg/mL $1/x^2$ weighted r^2>0.99	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.005	0.005 0.05	5 5	87 87	82-93 82-91	7.5 3.1	<0.3LOQ (2)	352 → 256	
bovine milk	0.005	0.005 0.05	5 5	99 96	92-102 89-104	3.8 6.0	<0.3LOQ (2)	352 → 332	Sahvorost, 2018c, 2017AMT-IFP3873
	0.005	0.005 0.05	5 5	100 96	96-103 90-105	2.6 6.2	<0.3LOQ (2)	352 → 256	
bovine whole milk	0.005	0.005 0.05	8 8	89 92	71-100 81-99	12 7.2	<0.2LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine skim milk	0.005	0.005 0.05	1 1	- -	98 96	- -	<0.2LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine milk cream	0.005	0.005 0.05	1 1	- -	101 88	- -	<0.2LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
poultry breast	0.01	0.01 0.1	5 5	102 101	98-104 99-102	2.3 1.1	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 352 → 332	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	103 100	99-106 99-100	2.2 0.8	<0.3LOQ (2)	352 → 256	
poultry tigh	0.01	0.01 0.1	5 5	107 110 101 102	101- 100-	3.1 1.1	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 352 → 332	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01	5	105	103-	1.2	<0.3LOQ	352 → 256	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
		0.1	5	107		1.4	(2)		
				100	99-102				
poultry muscle	0.01	0.01	5	98	95-105	4.2	<0.2LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943
		0.1	5	91	90-94	1.8			
poultry fat	0.01	0.01	5	82	79-84	2.4	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r2>0.99 352 → 332	Moore&Shepherd, 2018, 2016RES-IFP2941
		0.1	5	80	72-86	6.3			
	0.01	0.01	5	81	78-84	3.0	<0.3LOQ (2)	352 → 256	
		0.1	5	78	69-85	7.0			
poultry fat	0.01	0.01	5	83	74-88	6.7	<02LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943
		0.1	5	90	87-93	2.5			
poultry liver	0.01	0.01	5	101	95-103	3.5	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 352 → 332	Moore&Shepherd, 2018, 2016RES-IFP2941
		0.1	5	97	95-98	1.1			
	0.01	0.01	5	103	98-106	2.9	<0.3LOQ (2)	352 → 256	
		0.1	5	99	96-100	1.6			
poultry liver	0.01	0.01	5	94	94-95	0.6	<0.2LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943
		0.1	5	93	91-100	4.0			
		0.4	5	97	93-101	3.9			
poultry egg	0.01	0.01	5	103	91-109	6.7	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r2>0.99 352 → 332	Moore&Shepherd, 2018, 2016RES-IFP2941
		0.1	5	98	89-102	5.2			
	0.01	0.01	5	104	94-111	6.0	<0.3LOQ (2)	352 → 256	
		0.1	5	99	90-102	5.1			
poultry egg	0.01	0.01	5	98	90-104	6.0	<0.3LOQ (2)	352 → 332	Sahvorost, 2018c, 2017AMT-IFP3873
		0.1	5	96	85-107	10			
	0.01	0.01	5	98	90-107	6.4	<0.3LOQ (2)	352 → 256	
		0.1	5	96	85-107	10			
poultry egg	0.01	0.01	11	100	91-108	4.9	<0.2LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943
		0.1	11	97	91-103	4.3			

Notes:

[a] Second attempt.

Table 98 Validation results for N-DesMet-fluindapyr with HPLC-MS/MS method 133SRUS16R0208

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
bovine muscle	0.01	0.01 0.1	5 5	102 98	99 - 109 95 - 100	3.8 2.1	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 338 → 262	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	102 97	99-107 94-100	3.7 2.8	<0.3LOQ (2)	338 → 242	
bovine fat	0.01	0.01 0.1	5 5	65 62	61 - 67 61 - 64	4.1 2.0	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 338 → 262	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	64 63	59-67 62-66	4.8 2.7	<0.3LOQ (2)	338 → 242	
bovine liver	0.01	0.01 0.1	5 5	105 103	100 - 107 101 - 105	2.5 1.8	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 338 → 262	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	105 103	103-105 100-104	0.8 1.7	<0.3LOQ (2)	338 → 242	
bovine liver	0.01	0.01 0.1	5 5	103 101	99-108 100-102	3.1 1.1	<0.3LOQ (2)	338 → 242	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	101 99	98-103 98-101	2.1 1.1	<0.3LOQ (2)	338 → 262	
bovine kidney	0.01	0.01 0.1	5 5	103 102	100-105 100-105	2.0 1.8	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 338 → 262	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	103 103	101-106 100-106	2.1 2.8	<0.3LOQ (2)	338 → 242	
bovine milk	0.005	0.005 0.05	5 5	94 95	85 - 97 92 - 99	5.4 3.0	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 338 → 262	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.005	0.005 0.05	5 5	95 95	88-98 92-99	4.4 3.0	<0.3LOQ (2)	338 → 242	
poultry breast	0.01	0.01 0.1	5 5	97 100	93- 00 99- 01	3.0 0.6	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 338 → 262	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	97 100	95-100 99-100	2.3 0.6	<0.3LOQ (2)	338 → 242	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
poultry tigh	0.01	0.01 0.1	5 5	107 105	104-111 104-106	2.6 1.2	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 338 → 262	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	106- 105	104-110 104-107	2.5 1.1	<0.3LOQ (2)	338 → 242	
poultry fat	0.01	0.01 0.1	5 5	77 76	75-82 65-82	4.3 9.4	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 338 → 262	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	79 75	76-80 65-82	2.2 8.9	<0.3LOQ (2)	338 → 242	
poultry liver	0.01	0.01 0.1	5 5	107 102	106-109 101-103	1.3 0.7	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 338 → 262	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	106 103	103-109 101-103	2.6 0.8	<0.3LOQ (2)	338 → 242	
poultry liver	0.01	0.01 0.1 0.4	5 5 5	105 98 98	100-111 93-106 93-102	3.9 5.1 3.9	<0.2LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943
poultry egg	0.01	0.01 0.1	5 5	100 99	90-103 88-103	5.8 6.1	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 338 → 262	Moore&Shepherd, 2018, 2016RES-IFP2941
	0.01	0.01 0.1	5 5	98 97	87-103 87-101	6.4 5.7	<0.3LOQ (2)	338 → 242	
poultry egg	0.01	0.01 0.1	5 5	100 97	93-109 85-107	8.0 9.9	<0.3LOQ (2)	338 → 242	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	100 97	93-110 86-108	6.8 9.8	<0.3LOQ (2)	338 → 262	
poultry egg	0.01	0.01 0.1	11 11	100 99	84-112 87-107	7.6 6.1	<0.2LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943

Table 99 Validation results for 1-OH-Met-fluindapyr with HPLC-MS/MS method 133SRUS16R0208

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
bovine muscle diastereomer a	0.01	0.01 0.1	5 5	97 94	95-98 91-96	1.3 2.2	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted	Moore&Shepherd [2018, 2016RES-IFP2941] Moore&Shepherd [2018, 2016RES-

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
								r^2>0.99 368 → 332	IFP2941]
	0.01	0.01 0.1	5 5	95 93	93-97 89-95	1.5 2.8	<0.3LOQ (2)	368 → 330	
bovine muscle diastereomer b	0.01	0.01 0.1	5 5	97 93	95-101 90-95	2.5 2.2	<0.3LOQ (2)	368 → 332	
	0.01	0.01 0.1	5 5	95 92	93-97 89-94	1.5 2.3	<0.3LOQ (2)	368 → 330	
bovine fat diastereomer a	0.01	0.01 0.1	5 5	87 88	85-91 85-92	2.8 3.3	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 368 → 310	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	86 89	82-90 85-94	3.6 4.1	<0.3LOQ (2)	368 → 330	
bovine fat diastereomer b	0.01	0.01 0.1	5 5	79 84	75-88 78-91	6.2 8.2	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	78 85	73-86 79-93	6.6 8.4	<0.3LOQ (2)	368 → 330	
bovine fat diastereomer a	0.01	0.01 0.1	5 5	77 78	76-80 74-83	2.2 4.7	<0.3LOQ (2)	368 → 310	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	76 79	74-78 75-83	2.3 4.3	<0.3LOQ (2)	368 → 330	
bovine fat diastereomer b	0.01	0.01 0.1	5 5	72 77	70-74 72-82	2.1 4.7	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	71 77	67-73 72-82	3.3 5.0	<0.3LOQ (2)	368 → 330	
bovine liver diastereomer a	0.01	0.01 0.1	5 5	101 99	99-103 97-101	1.7 1.5	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 368 → 310	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	102 100	98-105 99-105	2.4 1.9	<0.3LOQ (2)	368 → 330	
bovine liver diastereomer b	0.01	0.01 0.1	5 5	100 102 104	97-103 100-	2.1 1.7	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	100 103 105	97-103 101-	2.4 1.5	<0.3LOQ (2)	368 → 330	
bovine liver diastereomer a	0.01	0.01 0.1	5 5	90 86	85-100 82-90	6.6 3.6	<0.3LOQ (2)	368 → 310	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	89 85	86-99 81-91	6.6 4.4	<0.3LOQ (2)	368 → 330	
bovine liver diastereomer b	0.01	0.01 0.1	5 5	90 89	82-94 87-91	5.1 1.6	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	91 89	83-94 86-90	4.9 1.7	<0.3LOQ (2)	368 → 330	
bovine liver	0.01	0.01	5	89	76-98	9.3	<0.2LOQ	Concurrent	Brungardt, 2018,

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
(sum of diastereomers)		0.1 0.4	5 5	90 91	84-94 88-94	4.9 2.9		recoveries feeding study	2016RES-IFP2942
bovine kidney diastereomer a	0.01	0.01 0.1	5 5	101 100	97-112 98-101	2.6 6.5	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 368 → 310	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	101 100	95-112 98-102	6.5 1.7	<0.3LOQ (2)	368 → 330	
bovine kidney diastereomer b	0.01	0.01 0.1	5 5	100 101	94-110 98-104	6.2 2.3	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	100 101	95-113 98-104	7.2 2.3	<0.3LOQ (2)	368 → 330	
bovine kidney (sum of diastereomers)	0.01	0.01 0.1 0.4	5 5 5	102 85 94	86-112 75-100 88-102	9.7 11 6.8	<0.2LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine milk diastereomer a	0.005	0.005 0.05	5 5	96 95	94-99 93-97	2.5 1.9	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 368 → 310	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.005	0.005 0.05	5 5	95 95	92-98 94-97	3.3 1.4	<0.3LOQ (2)	368 → 330	
bovine milk diastereomer b	0.005	0.005 0.05	5 5	96 96	94-100 93-98	2.6 2.5	<0.3LOQ (2)	368 → 310	
	0.005	0.005 0.05	5 5	96 96	94-101 94-99	2.9 2.5	<0.3LOQ (2)	368 → 330	
poultry breast diastereomer a	0.01	0.01 0.1	5 5	96 93	93-98 92-95	2.5 1.2	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 368 → 310	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	95 92	92-98 91-92	2.9 0.6	<0.3LOQ (2)	368 → 330	
poultry breast diastereomer b	0.01	0.01 0.1	5 5	96 93	93-98 93-94	2.6 0.5	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	94 92	89-98 91-93	4.1 0.7	<0.3LOQ (2)	368 → 330	
poultry thigh diastereomer a	0.01	0.01 0.1	5 5	97 94	95-99 93-96	1.6 1.2	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 368 → 310	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	97 94	94-99 93-96	1.7 1.4	<0.3LOQ (2)	368 → 330	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
poultry tigh diastereomer b	0.01	0.01 0.1	5 5	97 93	93-98 90-95	0.7 2.1	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	97 92	95-98 91-94	1.3 1.9	<0.3LOQ (2)	368 → 330	
poultry fat diastereomer a	0.01	0.01 0.1	5 5	90 87	88-94 81-91	2.7 4.6	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 368 → 310	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	89 87	85-91 82-92	2.7 4.2	<0.3LOQ (2)	368 → 330	
poultry fat diastereomer b	0.01	0.01 0.1	5 5	86 83	82-91 79-88	4.1 4.0	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	85 84	82-89 78-85	3.6 4.3	<0.3LOQ (2)	368 → 330	
poultry fat (sum of diastereomers)	0.01	0.01 0.1	5 5	88 85	85-93 83-90	3.8 3.4	<0.2LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943
poultry liver diastereomer a	0.01	0.01 0.1	5 5	99 98	92-105 97-100	4.7 1.5	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 368 → 310	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	96 96	91-104 94-97	4.8 1.8	<0.3LOQ (2)	368 → 330	
poultry liver diastereomer b	0.01	0.01 0.1	5 5	98 97	90-104 95-99	5.4 1.6	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	97 96	90-102 95-98	5.1 1.1	<0.3LOQ (2)	368 → 330	
poultry liver (sum of diastereomers)	0.01	0.01 0.1 0.4	5 5 5	91 90 97	82-96 85-92 95-100	6.1 3.3 1.9	<0.2LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943
poultry egg diastereomer a	0.01	0.01 0.1	5 5	101 96	99-103 94-99	1.4 1.8	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 368 → 310	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	101 97	99-103 97-98	1.5 1.7	<0.3LOQ (2)	368 → 330	
poultry egg diastereomer b	0.01	0.01 0.1	5 5	102 105 100	100- 99-103	2.2 1.9	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	102 101	97-103 99-104	3.1 1.9	<0.3LOQ (2)	368 → 330	
poultry egg diastereomer a	0.01	0.01 0.1	5 5	91 85	84-94 78-89	6.0 4.8	<0.3LOQ (2)	368 → 310	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	89 85	82-94 79-89	5.2 4.6	<0.3LOQ (2)	368 → 330	

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
poultry egg diastereomer b	0.01	0.01 0.1	5 5	93 88	89-97 86-91	3.7 2.3	<0.3LOQ (2)	368 → 310	
	0.01	0.01 0.1	5 5	94 87	91-100 85-91	3.9 2.5	<0.3LOQ (2)	368 → 330	
poultry egg (sum of diastereomers)	0.01	0.01 0.1	11 11	97 92	92-104 86-97	3.8 3.7	<0.2LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943

Table 100 Validation results for 1-OH-Met-N-DesMet-fluindapyr with HPLC-MS/MS method 133SRUS16R0208

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
bovine muscle diastereomer a	0.01	0.01 0.1	5 5	85 84	69-89 72-90	10 8.2	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 354 → 296	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	86 83	70-91 71-89	10 8.7	<0.3LOQ (2)	354 → 145	
bovine muscle diastereomer b	0.01	0.01 0.1	5 5	82 82	65-87 70-88	12 8.6	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	86 82	68-95 72-89	13 7.8	<0.3LOQ (2)	354 → 145	
bovine fat diastereomer a	0.01	0.01 0.1	5 5	77 83	70-82 80-86	5.7 2.5	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 354 → 296	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	79 82	66-80 79-85	7.1 2.7	<0.3LOQ (2)	354 → 145	
bovine muscle diastereomer b	0.01	0.01 0.1	5 5	73 79	63-80 77-83	8.5 3.3	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	74 79	65-77 75-83	7.1 4.5	<0.3LOQ (2)	354 → 145	
bovine mesenterial fat (sum of diastereomers)	0.01	0.01 0.1	5 5	81 80	76-86 73-86	6.1 8.2	<0.2LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine perirenal fat (sum of diastereomers)	0.01	0.01 0.1	5 5	100 99	98-101 96-101	1.3 1.9	<0.2LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine subcutaneous fat (sum of diastereomers)	0.01	0.01 0.1	5 5	90 88	90-91 87-89	0.5 1.2	<0.2LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine liver	0.01	0.01	5	80	71-88	8.1	<0.3LOQ (2)	7 standards	Moore&Shepherd

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
diastereomer a		0.1	5	85	79-94	6.7		matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 354 → 296	[2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	79 84	68-87 79-94	8.9 7.0	<0.3LOQ (2)	354 → 145	
bovine liver diastereomer b	0.01	0.01 0.1	5 5	76 86	66-82 80-93	8.4 5.5	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	87 84	77-94 79-89	7.6 4.8	<0.3LOQ (2)	354 → 145	
bovine liver diastereomer a	0.01	0.01 0.1	5 5	76 78	69-81 69-86	5.7 9.8	<0.3LOQ (2)	354 → 296	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	77 78	70-81 68-87	6.0 11	<0.3LOQ (2)	354 → 145	
bovine liver diastereomer b	0.01	0.01 0.1	5 5	69 69	67-74 62-79	4.2 11	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	66 69	62-69 61-80	3.9 11	<0.3LOQ (2)	354 → 145	
bovine liver (sum of diastereomers)	0.01	0.01 0.1 0.4	5 5 5	79 83 82	69-86 80-86 78-84	7.8 2.8 3.7	<0.2LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine kidney diastereomer a	0.01	0.01 0.1	5 5	86 80	81-95 58-88	6.4 16	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 354 → 296	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	87 80	82-95 58-88	5.6 16	<0.3LOQ (2)	354 → 145	
bovine kidney diastereomer b	0.01	0.01 0.1	5 5	86 81	81-93 58-90	5.3 16	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	86 81	82-92 58-90	4.3 16	<0.3LOQ (2)	354 → 145	
bovine kidney (sum of diastereomers)	0.01	0.01 0.1 0.4	5 5 5	90 82 79	79-100 72-102 74-83	9.9 14 4.4	<0.2LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942
bovine milk diastereomer a	0.005	0.005 0.05	5 5	83 88	69-93 80-94	12 6.6	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 354 → 296	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.005	0.005 0.05	5 5	85 86	75-93 78-92	8.7 6.2	<0.3LOQ (2)	354 → 145	
bovine milk diastereomer b	0.005	0.005 0.05	5 5	83 86	73-93 78-91	9.2 6.0	<0.3LOQ (2)	354 → 296	
	0.005	0.005 0.05	5 5	83 86	72-91 78-91	10 5.9	<0.3LOQ (2)	354 → 145	
poultry breast diastereomer a	0.01	0.01 0.1	5 5	82 86	77-86 83-87	4.1 2.1	<0.3LOQ (2)	7 standards matrix matched	Moore&Shepherd [2018, 2016RES-

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
								0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 354 → 296	IFP2941]
	0.01	0.01 0.1	5 5	80 85	78-82 82-87	2.3 2.8	<0.3LOQ (2)	354 → 145	
poultry breast diastereomer b	0.01	0.01 0.1	5 5	81 84	74-86 81-85	5.9 1.8	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	79 83	76-84 80-87	4.8 3.1	<0.3LOQ (2)	354 → 145	
poultry tigh diastereomer a	0.01	0.01 0.1	5 5	86 87	83-89 79-92	2.8 5.6	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 354 → 296	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	87 87	83-91 80-91	2.9 5.0	<0.3LOQ (2)	354 → 145	
poultry tigh diastereomer b	0.01	0.01 0.1	5 5	82 84	81-83 78-87	0.9 4.5	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	89 83	83-100 78-86	7.8 3.8	<0.3LOQ (2)	354 → 145	
poultry fat diastereomer a	0.01	0.01 0.1	5 5	79 79	74-82 73-82	4.1 4.3	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 354 → 296	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	80 79	73-84 74-82	5.2 4.1	<0.3LOQ (2)	354 → 145	
poultry tigh diastereomer b	0.01	0.01 0.1	5 5	76 78	73-78 73-81	2.6 4.2	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	77 77	72-80 71-81	4.6 5.1	<0.3LOQ (2)	354 → 145	
poultry liver diastereomer a	0.01	0.01 0.1	5 5	83 86	64-91 83-91	13 3.4	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 354 → 296	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	83 85	62-91 81-90	14 3.6	<0.3LOQ (2)	354 → 145	
poultry liver diastereomer b	0.01	0.01 0.1	5 5	81 84	60-98 78-88	17 4.9	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	81 83	56-97 78-88	18 4.4	<0.3LOQ (2)	354 → 145	
poultry liver (sum of diastereomers)	0.01	0.01 0.1 0.4	5 5 5	76 72 81	73-80 71-74 79-84	3.4 1.6 2.8	<0.2LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943
poultry egg	0.01	0.01	5	87	84-88	2.3	<0.3LOQ (2)	7 standards	Moore&Shepherd

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
diastereomer a		0.1	5	87	84-90	2.4		matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 354 → 296	[2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	88 88	85-89 85-91	1.8 3.2	<0.3LOQ (2)	354 → 145	
poultry egg diastereomer b	0.01	0.01 0.1	5 5	85 86	82-87 85-89	2.1 2.0	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	86 87	84-88 86-89	3.0 1.5	<0.3LOQ (2)	354 → 145	
poultry egg diastereomer a	0.01	0.01 0.1	5 5	70 61	64-77 57-68	7.1 7.6	<0.3LOQ (2)	354 → 296	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	71 60	67-79 57-68	6.4 7.7	<0.3LOQ (2)	354 → 145	
poultry egg diastereomer b	0.01	0.01 0.1	5 5	74 63	69-80 59-68	5.6 5.4	<0.3LOQ (2)	354 → 296	
	0.01	0.01 0.1	5 5	72 63	67-75 60-69	4.6 5.7	<0.3LOQ (2)	354 → 145	
poultry egg (sum of diastereomers)	0.01	0.01 0.1	11 11	82 83	76-87 76-89	4.7 5.3	<0.2LOQ	Concurrent recoveries feeding study	Brungardt & Dixon, 2018, 2016RES-IFP2943

Table 101 Validation results for fluindapyr-1-COOH with HPLC-MS/MS method 133SRUS16R0208

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
bovine liver diastereomer a	0.01	0.01 0.1	5 5	103 105 104 106	100- 100-	2.3 2.5	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 382 → 336	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	100 102	99-102 99-106	1.4 2.6	<0.3LOQ (2)	382 → 296	
bovine liver diastereomer b	0.01	0.01 0.1	5 5	102 103 109	99-107 100-	3.1 3.1	<0.3LOQ (2)	382 → 336	
	0.01	0.01 0.1	5 5	101 105 111	98-104 101-	2.5 3.7	<0.3LOQ (2)	382 → 296	
bovine liver diastereomer a	0.01	0.01 0.1	5 5	92 92	88-95 90-94	2.9 1.6	<0.3LOQ (2)	382 → 336	Sahvorost, 2018c, 2017AMT-IFP3873
	0.01	0.01 0.1	5 5	94 92	91-97 91-93	2.7 1.1	<0.3LOQ (2)	382 → 296	
bovine liver diastereomer b	0.01	0.01 0.1	5 5	92 90	85-96 89-90	4.5 0.7	<0.3LOQ (2)	382 → 336	
	0.01	0.01 0.1	5 5	91 90	85-95 89-91	4.3 1.0	<0.3LOQ (2)	382 → 296	
bovine liver	0.01	0.01	5	83	71-100	16.4	<0.2LOQ	Concurrent	Brungardt, 2018,

commodity	reported LOQ mg/kg	spike level mg/kg	n	percent recovery mean	range	RSD_r	control samples mg/kg (n)	Calibration, m/z transition	reference, method
(sum of diastereomers)		0.1	5	88	79-98	9.7		recoveries feeding study	2016RES-IFP2942
bovine kidney diastereomer a	0.01	0.01 0.1	5 5	94 96	89-97 90-100	3.1 3.7	<0.3LOQ (2)	7 standards matrix matched 0.004-0.25 µg/mL $1/x^2$ weighted r^2>0.99 382 → 336	Moore&Shepherd [2018, 2016RES-IFP2941]
	0.01	0.01 0.1	5 5	94 96	90-98 90-100	2.8 3.6	<0.3LOQ (2)	382 → 296	
bovine kidney diastereomer b	0.01	0.01 0.1	5 5	90 95	88-94 92-99	2.7 2.8	<0.3LOQ (2)	382 → 336	
	0.01	0.01 0.1	5 5	90 96	87-94 93-99	3.4 2.1	<0.3LOQ (2)	382 → 296	
bovine kidney (sum of diastereomers)	0.01	0.01 0.1	5 5	94 94	93-96 90-102	1.2 5.0	<0.2LOQ	Concurrent recoveries feeding study	Brungardt, 2018, 2016RES-IFP2942

Analytical methods used in study reports on soil

The Meeting received the description and validation for an analytical methods for the determination of fluindapyr, 3-OH-fluindapyr, 1-COOH-fluindapyr (sum of diastereomers), and pyrazole carboxamide in soil.

LC-MS/MS method (fluindapyr, 3-OH-fluindapyr, 1-COOH-fluindapyr (both isomers), and pyrazole carboxamide)

The LC-MS/MS method that is part of a terrestrial field dissipation study [Schreier, 2017, 2014EFT-IFP1203] determines fluindapyr, cis-1-COOH-fluindapyr, trans-1-COOH-fluindapyr, 3-OH-fluindapyr, and pyrazole carboxamide and was validated in two representative soils, one from Nebraska and one from New York [Sahvorost, 2018d, 2017AMT-IFP3870].

Soil samples (5 g) were extracted twice using acetone:water (9:1, v/v), followed by a single extraction utilizing acetone:0.5 N HCl (1:1, v/v). The resulting solution was concentrated under nitrogen to remove the acetone and diluted with methanol. Following methanol dilution the sample was analysed for fluindapyr, cis-1-COOH-fluindapyr, trans-1-COOH-fluindapyr, 3-OH-fluindapyr, and pyrazole carboxamide content using a validated LC-MS/MS method. The samples are analysed by high performance liquid chromatography with tandem mass specific detection (LC-MS/MS) in positive polarity mode (negative for 3 hydroxy-fluindapyr), using a Phenomenex Kinetex C18 100A column (50 × 4.6 mm, 2.6 µm particle size) and gradient elution with mobile phases of 10mM ammonium acetate and 0.2 percent formic acid in water, and 0.2 percent formic acid in methanol. Calibration was performed using external reference standards. Detection was at m/z 352 to 332 (quantitation) and 257 (confirmation) for fluindapyr; at m/z 366 to 175 or 131 for 3-OH-fluindapyr; at m/z 382 to 336 or 296 for the 1-COOH-fluindapyr diastereomers, and at m/z 176 to 136 and 156 for pyrazole carboxamide. The method had a limit of quantitation (LOQ) of 0.005 mg/kg for fluindapyr, the combined diastereomers of 1-COOH-fluindapyr, 3-OH-fluindapyr, and pyrazole carboxamide.

The method was independently validated [Sahvorost, 2018d, 2017AMT-IFP3870]. In brief, the soil samples were extracted three times with acetone/water (90/10, v/v), acetone/water (50/50, v/v) and acetone/0.5 N HCl (50/50, v/v) using a sonicator and/or a reciprocal shaker. After centrifugation and

evaporating off the acetone, the remaining water part was diluted with methanol for HPLC-MS/MS analysis. Quantification was performed by use of HPLC-MS/MS detection. Two mass transitions for each analyte were evaluated in order to demonstrate that the method achieves a high level of specificity. No significant interference above 20 percent of LOQ was detected in any of the reagent blanks or the control sample extracts of each soil type, so that a high level of selectivity was demonstrated.

Matrix effects on the detection of fluindapyr, 3-OH-fluindapyr, 1-COOH-fluindapyr, and pyrazole carboxamide in final extracts of both types of soil were found to be insignificant (< ±20 percent), except 3-hydroxy-IR9792/F9990 in silt loam soil from New York. However, matrix-matched standards were used for quantification. Linearity of the response was demonstrated with six (6) matrix matched calibration standards (corresponding with 0.002–0.20 mg/kg (fluindapyr, 3-OH-fliundpayr and pyrazole carboxamide) and 0.0013–0.13 mg/kg and 0.0007–0.07 mg/kg for both diastereomer a and b from 1-COOH-fluindapyr, respectively). The calibration curves obtained for both mass transitions were linear with correlation coefficients ($r \geq 0.995$). Linear regression was performed with 1/x-weighting.

The validation results (primary transitions) for the determination of each of the analytes in soil from both reports are summarised in Table 102

Note by the reviewer: The LC-MS/MS method for the determination of fluindapyr, 3-OH-fluindapyr, 1-COOH-fluindapyr (both diastereomers), and pyrazole carboxamide in soil is considered valid in the range 0.005-0.5 mg/kg for each analyte.

Table 102 Validation results for fluindapyr and its metabolites with HPLC-MS/MS method from the original study (OS) [Schreier, 2017, 2014EFT-IFP1203] and the independent laboratory validation (ILV) [Sahvorost, 2018d, 2017AMT-IFP3870]

Matrix	Fortification Level (mg/kg	n	Recovery Range	Average recovery (%)	percent RSD	Recovery Range	Average recovery (%)	percent RSD
			fluindapyr			3-hydroxy-fluindapyr		
Nebraska soil (OS)	0.005	5	84-92	89	4.1	73-95	85	9.8
	0.05	5	89-94	92	2.3	90-97	94	2.8
Nebraska soil (ILV)	0.005	5	101 - 105	103	1.6	101 - 106	103	2.8
	0.05	5	98 - 104	101	2.5	92 - 104	97	5.1
New York soil (OS)	0.005	5	82-92	89	3.5	73-80	77	3.9
	0.05	5	86-96	93	4.2	76-88	82	5.9
New York soil (ILV)	0.005	5	103 - 107	105	1.8	92 - 110	98	7.3
	0.05	5	100 - 106	102	3.3	94 - 102	97	3.6
Matrix	Fortification Level (mg/kg)	n	1-carboxy-fluindapyr (sum of diastereomers)			pyrazole carboxamide		
Nebraska soil (OS)	0.005	5	82-90 [a]	86 [a]	3.6 [a]	84-92	88	4.0
	0.05	5	73-93 [a]	80 [a]	10 [a]	89-99	93	4.0
Nebraska soil (ILV)	0.005	5	98 – 109	104 [a]	4.4 [a]	98 - 107	103	3.6
	0.05	5	93 - 107	101 [a]	2.5 [a]	96 - 104	99	3.4
New York soil (OS)	0.005	5	74-87 [a]	81	7.9	76-90	82	6.5
	0.05	5	87-101 [a]	94	5.4	98-106	101	3.2
New York soil (ILV)	0.005	5	92 - 98	95	2.5	103 - 106	104	0.9
	0.05	5	92 - 99	96	3.5	98 - 104	100	2.4

Notes:

[a] Diastereomer a only. Results with diastereomer b are similar.

STABILITY OF [PESTICIDES RESIDUES IN STORED ANALYTICAL SAMPLES

The Meeting received information on the storage stability of parent, 3-OH-fluindapyr, fluindapyr-glucoside, 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr in wheat matrices (grain, forage, hay and straw, dry gluten), oilseed rape (seed and whole plant) and grapes. Storage stability of fluindapyr, N-DesMet-fluindapyr, 1-OH-Met-fluindapyr, 1-COOH-fluindapyr and 1-OH-Met-N-DesMet-fluindapyr in animal tissues (muscle, fat, liver, kidney), egg, and milk were also received, as well as data on the storage stability of parent, 3-OH-fluindapyr, 1-COOH-fluindapyr diastereomers, and 3-(difluoromethyl)- l-methyl-1H-pyrazole-4-carboxamide in different soils.

Storage stability of spiked residues in plant commodities

Storage stability was investigated by **spiking** oil seed rape (OSR) whole plant (high water), wheat dry gluten (high protein), wheat grain (high starch), grapes (high acid), and OSR seed (high oil) with 0.10 mg/kg of parent, 3-OH-fluindapyr, DesMet-fluindapyr-1N-glucoside, 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr [Soddu, 2020, 2016RES-IFP2653]. Samples were stored for 36 months at -20 ºC and were analysed in duplicate at various intervals.

The analytes were quantified by LC-MS/MS method based on method P3770G [2015RES-IFP2155]. The LOQ was 0.01 mg/kg.

In a second study, wheat matrices (grain, forage, straw and hay) were spiked with 0.1 mg/kg fluindapyr [Soddu, 2017, 2014 RES-IFP1459]. Samples were stored for 36 months at -20 ± ºC and were analysed in duplicate at various intervals. The analytical method was based on a QuEChERS-extraction method RA14.04 as validated in report 2014RES-IFP1239. The LOQ of the method was 0.01 mg/kg.

Storage stability results (not corrected for concurrent recovery) and concurrent recoveries for parent fluindapyr and its metabolites are shown in Table 103 to Table 106.

Note by the reviewer:

- The analytical methods used in both studies are valid for the purpose of these studies (commodity type and concentration level of the analytes).
- The results from the storage stability investigations demonstrated that parent fluindapyr, and metabolites 3-OH-fluindapyr, DesMet-fluindapyr-1N-glucoside, 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr are stable over a period of 3 years in matrices covering high water, high protein, high starch, high acid, and high oil contents.

Table 103 Storage stability at ≤ -20 °C in commodities spiked with 0.1 mg/kg of parent and 3-OH-fluindapyr using method P3770G

Matrix	Storage period (months/days)	fluindapyr mean percent [a]	Fluindapyr Mean percent remaining [b]	fluindapyr Mean concurrent recovery (%)	3-OH-F mean percent	3-OH-F Mean percent remaining [a]	3-OH-F Mean concurrent recovery (%)	[Reference], method
Wheat grain	0/0	134	100	-	114	100	-	[2016RES-IFP2653], P3370G
	1/26	128	96	134	119	104	114	
	3/101	93	69	92	96	84	98	
	6/246	103	77	115	114	100	116	
	12/395	90	67	87	106	93	101	
	24 /819	97	72	97	112	98	113	

Matrix	Storage period (months/days)	fluindapyr mean percent [a]	Fluindapyr Mean percent remaining [b]	fluindapyr Mean concurrent recovery (%)	3-OH-F mean percent	3-OH-F Mean percent remaining [a]	3-OH-F Mean concurrent recovery (%)	[Reference], method
	36/1148	110	82	99	113	99	111	
Wheat straw	0/0	100	100	-	91	100	-	[2016RES-IFP2653]
	1/55	108	108	100	81	89	91	
	3/105	139	139	126	100	110	88	
	6/207	114	114	104	119	131	104	
	12/385	102	102	89	100	110	86	
	24/789	113	113	111	118	130	117	
	36/1117	104	104	85	112	123	91	
Grape	0/0	98	100	-	87	100	-	[2016RES-IFP2653]
	1/34	113	115	98	87	100	87	
	3/105	148	151	110	107	123	95	
	6/207	101	103	95	104	120	93	
	12/383	103	105	99	111	128	107	
	24/789	100	102	99	108	124	104	
	36/1117	119	121	104	120	138	102	
OSR seed	0/0	102	100	-	119	100	-	[2016RES-IFP2653]
	1/26	105	103	102	119	100	119	
	3/101	110	108	97	120	101	103	
	6/248	95	93	94	114	96	98	
	12/395	106	104	96	119	100	102	
	24/819	120	118	96	122	103	121	
	36/1148	119	117	91	111	93	122	
OSR whole plant	0/	110	100	-	104	100	-	[2016RES-IFP2653]
	1/34	129	117	110	102	98	104	
	3/105	122	111	104	94	90	98	
	6/207	103	97	106	105	101	103	
	12/385	99	90	97	105	101	104	
	24/789	101	92	107	102	98	109	
	36/1117	103	94	99	94	90	88	
Wheat dry gluten	0/0	103	100	-	100	100	-	[2016RES-IFP2653]
	1/26	109	106	103	109	109	100	
	3/101	93	90	113	97	97	86	
	6/248	107	104	93	112	112	96	
	12/395	110	107	112	116	116	97	
	24/819	114	111	99	116	116	97	
	36/1163	94	91	97	104	104	93	

Notes:

[a] Mean recovery over fortification based on two or three samples.

[b] The percent remaining is indicated as the percentage of the initial, which is calculated by dividing the mean recovery of stored samples at each interval by the mean recovery at day 0. The values are calculated by the reviewer and not corrected with the concurrent fresh recoveries.

Table 104 Storage stability at ≤ -20 °C in wheat commodities spiked with 0.1 mg/kg of parent using method RA.14.04 [Soddu, 2017, 2014RES-IFP1459]

Matrix	fluindapyr mean percent [a]	fluindapyr Mean percent remaining [b]	fluindapyr Mean concurrent recovery (%)	Storage period (months)	Matrix	fluindapyr mean percent [a]	fluindapyr Mean percent remaining [b]	fluindapyr Mean concurrent recovery (%)
Wheat grain	86	100	-	0	Wheat straw	86	100	-
	77	89	79	1		77	90	81
	73	85	79	3		87	101	94
	73	85	86	6		82	96	78
	81	93	93	12		96	112	90
	77	89	85	24		88	103	77
	75	86	86	36		80	93	87
Wheat forage	71	100	-	0	Wheat hay	83	100	-
	81	115	77	1		99	119	94
	73	103	75	3		90	108	96
	78	109	79	6		86	103	86
	74	104	74	12		86	103	86
	77	108	80	24		99	119	93
	71	100	71	36		93	112	99

Table 105 Storage stability at ≤ -20 °C in commodities spiked with 0.1 mg/kg of DesMet-fluindapyr-N1-glucoside and 1-OH-Met-fluindapyr determined with method P3770G

Matrix	Storage period (months)	DesMet-fluindapyr-N1-gluc mean percent [a]	DesMet-fluindapyr-N1-gluc Mean percent remaining [b]	DesMet-fluindapyr-N1-gluc Mean concurrent recovery (%)	1-OH-Met-fluindapyr mean percent	1-OH-Met-fluindapyr Mean percent remaining [a]	1-OH-Met-fluindapyr Mean concurr recovery (%)	Reference
Wheat grain	0/0	120	100	-	120	100	-	[2016RES-IFP2653]
	1/36	119	99	120	120	100	120	
	3/101	94	78	103	109	91	118	
	6/246	97	81	100	105	87	107	
	12/395	91	76	96	115	96	111	
	24 /819	99	83	99	117	98	111	
	36/1148	94	78	101	111	93	107	
Wheat straw	0/0	85	100	-	89	100	-	[2016RES-IFP2653]
	1/55	77	91	85	109	122	89	
	3/105	94	111	85	85	96	77	
	6/207	117	138	105	104	117	88	
	12/385	95	112	87	95	107	88	
	24/789	113	133	120	100	112	92	
	36/1117	91	107	81	91	102	78	
Grape	0/0	92	100	-	106	100	-	[2016RES-IFP2653]
	1/34	91	99	92	114	108	106	
	3/105	113	123	99	112	106	104	
	6/207	101	110	90	114	108	101	
	12/383	100	109	97	122	115	99	
	24/789	101	110	101	121	114	117	

Matrix	Storage period (months)	DesMet-fluindapyr-N1-gluc mean percent [a]	DesMet-fluindapyr-N1-gluc Mean percent remaining [b]	DesMet-fluindapyr-N1-gluc Mean concurrent recovery (%)	1-OH-Met-fluindapyr mean percent	1-OH-Met-fluindapyr Mean percent remaining [a]	1-OH-Met-fluindapyr Mean concurr recovery (%)	Reference
	36/1117	106	115	101	124	117	105	
OSR seed	0/0	107	100	-	102	100	-	[2016RES-IFP2653]
	1/26	103	97	107	99	97	102	
	3/101	115	108	97	123	121	103	
	6/248	90	84	96	102	100	98	
	12/395	111	104	95	133	130	111	
	24/819	128	120	102	137	134	107	
	36/1148	106	99	97	111	109	87	
OSR whole plant	0/	105	100	-	107	100	-	[2016RES-IFP2653]
	1/31	99	94	105	110	103	107	
	3/105	101	96	109	109	102	102	
	6/207	106	101	107	105	98	113	
	12/385	90	86	98	100	93	105	
	24/789	89	85	106	103	96	106	
	36/1117	90	86	101	101	94	105	
Wheat dry gluten	0/0	90	100	-	97	100	-	[2016RES-IFP2653]
	1/26	97	108	90	101	104	97	
	3/101	90	100	104	105	108	94	
	6/248	89	99	82	109	112	97	
	12/395	94	104	100	110	113	103	
	24/819	102	113	94	121	125	100	
	36/1163	92	102	84	111	114	102	

Notes:

[a] Mean recovery over fortification based on two or three samples.

[b] The percent remaining is indicated as the percentage of the initial, which is calculated by dividing the mean recovery of stored samples at each interval by the mean recovery at day 0. The values are calculated by the reviewer and not corrected with the concurrent fresh recoveries.

Table 106 Storage stability at ≤ -20 °C in commodities spiked with 0.1 mg/kg of 1-OH-Met-N-DesMet-fluindapyr and 1-COOH-fluindapyr using method P3770G

Matrix	Storage period (months)	1-OH-Met-N-DesMet-fluindapyr mean percent [a]	1-OH-Met-N-DesMet-fluindapyr Mean percent remaining [b]	1-OH-Met-N-DesMet-fluindapyr Mean concurrent recovery (%)	1-COOH-fluindapyr mean percent	1-COOH - fluindapyr Mean percent remaining [a]	1-COOH-fluindapyr Mean concurr recovery (%)	Reference
Wheat grain	0/0	129	100	-	114	100	-	[2016RES-IFP2653]
	1/36	131	102	129	119	104	114	
	3/101	117	91	116	111	97	119	
	6/246	102	79	122	109	96	114	
	12/395	112	87	104	119	104	112	
	24 /819	104	81	106	98	86	111	
	36/1148	116	90	115	107	94	109	
Wheat	0/0	88	100	-	91	100	-	[2016RES-

Matrix	Storage period (months)	1-OH-Met-N-DesMet-fluindapyr mean percent [a]	1-OH-Met-N-DesMet-fluindapyr Mean percent remaining [b]	1-OH-Met-N-DesMet-fluindapyr Mean concurrent recovery (%)	1-COOH-fluindapyr mean percent	1-COOH - fluindapyr Mean percent remaining [a]	1-COOH-fluindapyr Mean concurr recovery (%)	Reference
straw	1/55	104	117	88	105	115	91	IFP2653]
	3/105	67	75	75	77	85	74	
	6/207	91	103	92	105	115	90	
	12/385	74	84	78	101	111	95	
	24/789	85	97	91	104	114	93	
	36/1117	83	94	78	81	89	75	
Grape	0/0	102	100	-	107	100	-	[2016RES-IFP2653]
	1/34	101	99	102	109	102	107	
	3/105	102	100	100	104	97	98	
	6/207	103	101	105	111	104	103	
	12/383	102	100	113	119	111	102	
	24/789	114	112	132	118	110	116	
	36/1117	103	101	98	103	96	106	
OSR seed	0/0	90	100	-	105	100	-	[2016RES-IFP2653]
	1/26	88	97	90	102	98	105	
	3/101	105	117	97	118	113	97	
	6/248	92	102	87	105	100	101	
	12/395	97	108	85	126	120	117	
	24/819	113	106	105	108	103	103	
	36/1148	95	100	88	105	100	90	
OSR whole plant	0/	105	100	-	107	100	-	[2016RES-IFP2653]
	1/31	100	95	105	113	106	107	
	3/105	97	92	91	110	103	110	
	6/207	94	89	107	106	99	111	
	12/385	86	82	107	106	99	110	
	24/789	90	107	105	105	98	109	
	36/1117	88	107	104	88	82	104	
Wheat dry gluten	0/0	88	100	-	88	100	-	[2016RES-IFP2653]
	1/26	98	111	88	96	109	88	
	3/101	102	116	93	102	116	89	
	6/248	95	108	96	94	107	90	
	12/395	103	117	90	101	115	98	
	24/819	105	119	97	93	106	91	
	36/1163	91	103	105	86	98	86	

Notes:

[a] Mean recovery over fortification based on two or three samples.

[b] The percent remaining is indicated as the percentage of the initial, which is calculated by dividing the mean recovery of stored samples at each interval by the mean recovery at day 0. The values are calculated by the reviewer and not corrected with the concurrent fresh recoveries.

Storage stability of spiked residues in animal commodities

The Meeting received storage stability studies for fluindapyr in animal tissues, milk, and eggs.

The storage stability of fluindapyr and its metabolites N-DesMet-fluindapyr, 1-OH-Met-fluindapyr, 1-COOH-fluindapyr, and 1-OH-Met-N-DesMet-fluindapyr was evaluated in representative animal matrices.

The stability of fluindapyr and its metabolites metabolites N-DesMet-fluindapyr, 1-OH-Met-fluindapyr, 1-COOH-fluindapyr, and 1-OH-Met-N-DesMet-fluindapyr was evaluated in liver (hen). The stability of all analytes except 1-COOH-fluindapyr was evaluated in eggs. The stability of all analytes except N-DesMet-fluindapyr was evaluated in kidney (cow). The stability of fluindapyr and 1-OH-Met-fluindapyr was evaluated in fat (cow). The stability of fluindapyr only was evaluated in milk and muscle (hen). Storage conditions and duration were chosen to closely mimic those utilized for storage of residue samples during the fluindapyr cattle and poultry feeding studies [Brundgardt, 2018, 2016RES-IFP2942 and Brungardt& Dixon, 2018, 2016RES-IFP2943].

Samples were prepared by fortifying homogenized control matrices with the analytes of interest for that matrix at 10× the method LOQ. Samples were removed from frozen storage and analysed at intervals of 2 months in milk, kidney, muscle, and egg, and at intervals of 2 and 3 months in liver. Control and fresh fortification recovery samples were analysed at each of these time points as well as at Day 0.

Samples were analysed for fluindapyr and its metabolites using HPLC-MS/MS Method 133SRUS16R0208 with an LOQ of 0.01 mg/kg for each analyte. Average recoveries for these fresh spikes fortified on the day of extraction were within 70 percent to 120 percent. Control samples had residues below 0.3LOQ.

The results are summarised in Table 107. The results provided are the results of two (2 and 3 months data) or three (day 0) replicate samples.

Notes by the reviewer:

- The analytical method used is considered valid for the purpose of this study (commodity type and concentration level of the analytes).
- The results from the storage stability investigations demonstrated that:
    - fluindapyr was stable in all animal matrices (bovine muscle (55 days), fat (77 days), kidney (62 days), and liver (91 days)), eggs (64 days) and milk (55 days);
    - N-DesMet-fluindapyr was stable in bovine liver (91 days), bovine kidney and eggs (64 days);
    - 1-OH-Met-fluindapyr was stable in bovine fat (70 days), bovine kidney (62 days), bovine liver (58 days) and eggs (64 days);
    - 1-OH-N-DesMet-fluindapyr was stable in bovine liver (58/91 days), bovine kidney (62 days) and eggs (64 days);
    - 1-COOH-fluindapyr was stable in bovine kidney (62 days) and liver (91 days).

Table 107 Storage stability of 0.10 mg/kg fluindapyr and its metabolites, in liver, kidney, muscle, fat, milk and eggs stored at -20 °C using method 133SRUS16R0208

Analyte	Commodity	Storage time (days)	Residue in spiked samples (percent of spiking level)	Mean recovery (%)	Normalized to day 0 (%) [a]	Concurrent recovery (%) [b]
Fluindapyr	Milk	0	101, 92, 98	97	100	97
		55	98, 90 90	93	96	93
	Liver	0	103, 103, 103	103	100	103
		58	85, 84, 86	85	82	101
		91	88, 82, 78	83	80	93
	Kidney	0	100, 101, 103	101	100	101

Analyte	Commodity	Storage time (days)	Residue in spiked samples (percent of spiking level)	Mean recovery (%)	Normalized to day 0 (%) [a]	Concurrent recovery (%) [b]
		62	89, 91, 89	89	88	99
	Muscle	0	100, 97, 93	97	100	97
		55	104, 104, 104	104	107	105
	Fat	0	86, 89, 88	88	100	88
		77	72, 73, 69	71	82	76
	Eggs	0	100, 100, 94	98	100	98
		64	99, 99, 98	99	100	105
N-DesMet-fluindapyr	Liver	0	109, 108, 106	108	100	108
		58	86, 85, 86	85	79	103
		91	87, 78, 77	81	75	96
	Eggs	0	105, 104, 93	101	100	101
		64	100, 100, 99	100	99	106
1-OH-Met-fluindapyr (diastereomer 1)	Liver	0	98, 101, 100	100	100	100
		58	91, 85, 91	89	89	92
		91	71, 67, 62	67	**67**	87
1-OH-Met-fluindapyr (diastereomer 2)	Liver	0	97, 100, 99	99	100	99
		58	86, 84, 87	86	87	93
		91	71, 67, 62	67	**68**	87
1-OH-Met-fluindapyr (diastereomer 1)	Kidney	0	99, 104, 99	101	100	101
		62	94, 91, 90	92	91	97
1-OH-Met-fluindapyr (diastereomer 2)	Kidney	0	98, 103, 99	100	100	100
		62	96, 93, 92	94	93	98
1-OH-Met-fluindapyr (diastereomer 1)	Fat	0	85, 80, 88	84	100	84
		77	94, 91, 90	92	109	97
1-OH-Met-fluindapyr (diastereomer 2)	Fat	0	83, 78, 85	82	100	82
		77	96, 93, 92	94	114	98
1-OH-Met-fluindapyr (diastereomer 1)	Eggs	0	88, 86, 93	89	100	89
		64	97, 101, 98	99	111	100
1-OH-Met-fluindapyr (diastereomer 2)	Eggs	0	88, 84, 93	88	100	88
		64	98, 101, 99	99	113	100
1-COOH-fluinapyr (diastereomer 1)	Liver	0	95, 99, 95	96	100	96
		58	99, 84, 95	93	96	88
		91	75, 79, 72	76	79	80
1-COOH-fluinapyr (diastereomer 2)	Liver	0	96, 98, 97	97	100	97
		58	95, 78, 94	89	92	84
		91	73, 74, 72	73	75	81
1-COOH-fluinapyr (diastereomer 1)	Kidney	0	100, 96, 99	98	100	98
		62	91, 90, 92	91	93	97
1-COOH-fluinapyr (diastereomer 2)	Kidney	0	99, 94, 98	97	100	97
		92	95, 84, 85	85	88	95
1-OH-Met-N-DesMet-fluinapyr (diastereomer 1)	Liver	0	86, 84, 82	84	100	84
		58	86, 77, 92	85	101	80
		91	63, 59, 56	59	71	77
1-OH-Met-N-DesMet-fluinapyr (diastereomer 2)	Liver	0	87, 83, 83	84	100	84
		58	79, 73, 85	79	94	81
		91	60, 57, 51	56	**67**	74
1-OH-Met-N-DesMet-fluinapyr (diastereomer 1)	Kidney	0	73, 73, 74	73	100	3
		62	83, 82, 74	80	108	75
1-OH-Met-N-DesMet-fluinapyr (diastereomer 2)	Kidney	0	72, 74, 72	73	100	73
		62	81, 81, 73	78	108	76

Analyte	Commodity	Storage time (days)	Residue in spiked samples (percent of spiking level)	Mean recovery (%)	Normalized to day 0 (%) [a]	Concurrent recovery (%) [b]
1-OH-Met-N-DesMet-fluinapyr (diastereomer 1)	Eggs	0	77, 73, 89	80	100	80
		64	80, 94, 79	85	100	83
1-OH-Met-N-DesMet-fluinapyr (diastereomer 2)	Eggs	0	76, 71, 89	78	100	78
		64	78, 93, 79	83	100	81

Notes:

[a] Normalized Recovery is indicated as the percentage of the initial, which is calculated by dividing the mean recovery of stored samples at each interval by the mean recovery at day 0 = (Average recovery / average recovery at day 0) × 100 percent. The values are not corrected for the concurrent recoveries.

[b] Mean of 2 or 3 samples.

Storage stability of spiked residues in soils

Storage stability of fluindapyr and its soil degradates, 3-OH-fluindapyr, 1-COOH-fluindapyr diastereomers, and 3-(difluoromethyl)-l-methyl-1H-pyrazole-4-carboxamide during frozen storage was investigated in two United States soils (Nebraska and New York) [Skaggs, 2018, 2015EFT-IFP1940]. Both unfortified and fortified samples of soil were kept in cold storage (-20 °C ± l 0 °C) and in dark conditions for a period of approximately two years. Beginning at 0-day, a total of seven time intervals (0-day, 3, 6, 9, 12, 18, and 24 months) were analysed for the presence of fluindapyr and its metabolites.

The method for soil was validated at a limit of quantitation (LOQ) of 0.005 mg/kg for all analytes. The method validation results included in this report are the same as presented in terrestrial field dissipation study [Schreier, 2017, 2014EFT-IFP1203] used for development and validation of the analytical method. The method consisted of a series of three solvent: water extractions followed by a solvent reconstitution. Analysis was performed using HPLC-MS/MS with primary and confirmatory mass transitions. Individual recoveries were 70–120 percent for all analytes at every stability interval, with one exception. Spike "Replicate l" for 3-OH-fluindapyr in New York soil recovered at 69 percent. Mean recoveries of the stability fortifications for all analytes were 70–120 percent at every stability interval. The percentRSD for all analytes was ≤ 20 percent at every stability interval.

Control samples at each timepoint had residues below 0.2LOQ. The recoveries of the residues after storage (uncorrected for concurrent recoveries) and concurrent recoveries are shown in Table 108.

Note by the reviewer:

The method is considered fit for the purpose of this study.

The results demonstrate that fluindapyr, 3-OH-fluindapyr, 1-COOH-fluindapyr diastereomers, and 3-(difluoromethyl)-l-methyl-1H-pyrazole-4-carboxamide are stable in soil when stored at -20 °C or lower for a period of at least 24 months.

Table 108 Storage stability at -20 °C of fluindapyr, 3-OH-fluindapyr, 1-COOH-fluindapyr diastereomers, and 3-(difluoromethyl)-l-methyl-1H-pyrazole-4-carboxamide in soil spiked with 0.1 mg/kg of each analyte

Storage period	fluindapyr		3-OH-fluindapyr		1-COOH-fluindapyr diastereomer 1		1-COOH-fluindapyr diastereomer 2		pyrazole-4-carboxamide	
(days)	Recovery (%) [a]	Conc. Recov. (%) [b]	Recovery (%) [a]	Conc. Recov. (%) [b]	Recovery (%) [a]	Conc. Recov. (%) [b]	Recovery (%) [a]	Conc. Recov. (%) [b]	Recovery (%) [a]	Conc. Recov. (%) [b]
Nebraska soil										

Storage period	fluindapyr		3-OH-fluindapyr		1-COOH-fluindapyr diastereomer 1		1-COOH-fluindapyr diastereomer 2		pyrazole-4-carboxamide	
(days)	Recovery (%) [a]	Conc. Recov. (%) [b]	Recovery (%) [a]	Conc. Recov. (%) [b]	Recovery (%) [a]	Conc. Recov. (%) [b]	Recovery (%) [a]	Conc. Recov. (%) [b]	Recovery (%) [a]	Conc. Recov. (%) [b]
0	107	109 114	115	73 85	101	97 108	101	97 108	94	97 79
3	105	96 96	16	71 76	105	106 101	103	111 99	116	102 115
6	106	86 94	94	86 80	113	107 111	111	93 103	93	100 108
9	102	84 86	91	86 94	103	106 92	102	97 89	84	76 91
12	106	90 88	82	82 78	96	94 107	97	113 106	108	93 106
18	89	77 100	79	113 86	82	96 84	86	110 91	76	102 93
24	94	83 96	78	78 79	84	104 93	88	111 72	80	79 79
New York soil										
0	109	71 104	80	74 76	100	94 108	100	94 108	97	104 93
3	112	87 98	93	79 76	104	81 112	105	88 104	106	90 113
6	99	96 91	89	98 82	101	107 103	103	91 109	88	91 101
9	100	93 83	94	83 83	109	104 112	114	101 116	81	78 98
12	109	92 113	92	79 84	99	105 105	92	92 95	103	92 11
18	77	71 80	71 [c]	83 81	82	119 100	86	105 100	74	88 96
24	91	87 92	76	85 82	81	82 95	87	92 104	80	96 78

Notes:

[a] Mean of 5 replicates.

[b] Same day spikes at LOQ and 10 × LOQ, respectively.

USE PATTERN

The meeting received labels from the United States for a 480 g/L formulation to be applied as foliar application either by ground or aerial application. See Table 109.

Table 109 Registered pre-harvest uses of fluindapyr

Crop	Country	F/G	Form	Application				PHI, days
				Method	Rate g ai/ha	Spray conc, g ai/hL	Number (RTI)	
Cereal grains, except rice [a]	United States	F	480 g/L SC [b]	Foliar (ground or aerial)	90-150 (max total seasonal rate 300)	Ground: 96-156 [c] Aerial: 480-780	1-2 (10 days)	7 (forage) 14 (hay) 30 (grain/straw)
Grain sorghum	United States	F	480 g/L SC [b]	Foliar (ground or aerial)	90-150 (max total seasonal rate	Ground: 96-156 [c] Aerial: 313-	1-2 (10 days)	7 (forage) 30 (stover/grain)

Crop	Country	F/G	Form	Application				PHI, days
					300)	540		
Maize (field corn, popcorn)	United States	F	480 g/L SC [b][d]	Foliar (ground or aerial)	90-150 (max total seasonal rate 300)	Ground: 96-156 [c] Aerial: 480-780	1-2 (10-14 days)	7 (forage) 30 (stover/grain)
Corn (sweet corn)	United States	F	480 g/L SC [b][d][e]	Foliar (ground or aerial)	90-150 (max total seasonal rate 300)	Ground: 96-156 [c] Aerial: 480-780	1-2 (10-14 days)	14
Tree nuts [f]	United States	F	480 g/L SC [b]	Foliar (ground or aerial) [g]	123-168 (max total seasonal rate 506)	Ground: 131-180 [c] Aerial: 131-180	1-3 (7-14 days)	30
Almonds	United States	F	480 g/L SC [b]	Foliar (ground or aerial) [g]	123-168 (max total seasonal rate 506) [h]	Ground: 131-180 [c] Aerial: 131-180	1-3 (7-14 days)	30

Notes:

F= field; G = Greenhouse; RTI = ReTreatment Interval; PHI: Post Harvest Interval.

[a] Barley, Buckwheat, Millet, pearl, Millet, proso, Oats, Rye, Teosinte, Triticale, Wheat (spring and winter).

[b] An adjuvant may be used, unless specified in the crop use directions.

[c] Sufficient water volume to ensure thorough coverage as a foliar application for good disease control. Ground, air-blast, or aerial equipment may be used, so long as adequate spray coverage is achieved, unless prohibited in crop directions for use. For ground application, apply a minimum of 93.5 L/ha (10 gallons/acre) of spray solution. For aerial application, apply a minimum of 93.5 L/ha (10 gallons/acre) of spray solution for tree nut crops, and a minimum of 18.7 L/ha (2 gallons/acre) of spray solution for all other crops.

[d] Do not use an adjuvant after the V8 stage and prior to the VT stage of the corn. An adjuvant may be used in all other growth stages. V8 is when 8th trifoliate leave is unfolded=BBCH18; VT = tassel fully emerged and separated=BBCH59-61.

[e] Do not apply to sweet corn by mechanically pressurized handgun.

[f] African nut-tree, Almond, Beechnut, Brazil nut, Brazilian pine, Bunya, Bur oak, Butternut, Cajou nut, Candlenut, Cashew, Chestnut, Chinquapin, Coconut, Coquito nut, Dika nut, Ginkgo, Guiana chestnut, Hazelnut, Heartnut, Hickory nut, Japanese horse-chestnut, Macadamia nut, Mongongo nut, Monkey-pot, Monkey puzzle nut, Okari nut, Pachira nut, Peach palm nut, Pecan, Pequi, Pili nut, Pine nut, Pistachio, Sapucaia nut, Tropical almond, Walnut, black, Walnut, English, Yellowhorn, cultivars, varieties, and/or hybrids of these.

[g] Do not apply by handheld sprayer.

[h] Do not apply on almond until after petal fall.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received supervised trial data on wheat, sorghum, field corn, sweet corn and tree nuts.

Cereal grains

Wheat–Unite States trials

Supervised residue trials were conducted in the United States in 2016 to measure the magnitude of fluindapyr residues in/on wheat agricultural commodities following two foliar applications of a fluindapyr SC formulation [Webber, 2018e, 2016RES-FNF2456]. At each site one plot (T2) received two foliar applications at an application rate of 147–157 g ai/ha, with an interval of 6–11 days for forage and hay samples with the last application made at BBCH 21–61. At the second plot (T3) application rates ranged from 146-160 g ai/ha each, with application interval of 7–14 days for the generation of grain and straw

sample with the last applications made at BBCH 54-87. Treated plot (T2) received applications targeted at 17±2 days and 7±1 days prior to forage collection. Treated plot (T3) received applications targeted at 40±2 days and 30±2 days prior to grain harvest.

The wheat (forage, hay, grain, and straw) samples were harvested at proper times to yield commercially representative samples. At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 12 separate areas within the plots so that each sample yielded a minimum of 1 kilogram of forage and grain and 0.5 kilogram of hay and straw. The wheat forage samples were harvested at 7–8 days after the last application and the hay samples were collected from the same plots at 14–15 days after the last treatment. The grain and straw samples were harvested 26–31 days after the last application. Decline samples were collected at two trial locations. At these locations samples were collected at 0, 3, 7±1, 10±1 and 14±1 days after last application for treated forage and 7±1, 10±1, 14±2, 21±2, and 28±2 days after last application for treated hay and 20±2, 25±2, 30±2, 35±2, and 40±2 days after the last application for treated grain and straw.

Wheat raw agricultural commodity samples were maintained frozen after collection through analysis for up to 610 days. Frozen RAC samples were transferred from the field facility to the analytical laboratory in Norwell, MA, for preparation/homogenization and analysis. Samples were prepared by chopping and homogenizing the entire field sample, then removing a subsample for analysis. Samples were maintained frozen from receipt at the analytical facility until extraction for analysis.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-Met-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method for 1-OH-Met-F-O-glucoside (RA.17.01) utilizes acid hydrolysis so that this conjugate would be hydrolysed to the aglycone 1-OH-Met-F

The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 3-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from the LOQ (0.01 mg/kg) to 20.0 mg/kg for forage, 20.0 mg/kg for hay, 1.0 mg/kg for grain, and 20.0 mg/kg for straw. Fortified control samples were included in each analysis set for method verification.

Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from wheat forage were 100 ± 8 percent (n = 9), 101 ± 7 percent (n = 8) and 83 ± 12 percent (n = 9), respectively. Recoveries from wheat hay were 104 ± 11 percent (n = 9), 100 ± 6 percent (n = 8) and 78 ± 12 percent (n = 8), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from wheat grain were 100 ± 8 percent (n = 8), 100 ± 7 percent (n = 8) and 82 ± 18 percent (n = 10), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from wheat straw were 98 ± 12 percent (n = 9), 105 ± 8 percent (n = 8) and 83 ± 22 percent (n = 10), respectively.

Wheat–European trials

Supervised residue trials were conducted in Northern (United Kingdom and Germany) and Southern (Italy and France) Europe in 2015 to measure the magnitude of fluindapyr residues in/on wheat agricultural commodities following two foliar applications of a fluindapyr EC formulation [Peterek, 2018, 2015RES-IFP1968 and Ricelli, 2018, 2015RES-IFP1950]. At each site one plot was left untreated (U) and one plot received two foliar applications (T1) at an application rate of 138-160 g ai/ha, with an interval of 13–15 days [Peterek, 2018, 2015RES-IFP1968] and 9–15 days [Ricelli, 2018, 2015RES-IFP1950] with the last application made at BBCH 69. In harvest + processing phase trials a third plot was included (T2) which was applied with the same timing as plot T1, but the target dose was 5 × the normal dose 689–794 g ai/ha.

For harvest trials, straw (at least 0,5 kg) and grain (at least 1 kg) were taken in plots U and T1 at normal commercial harvest. After sampling, the specimens for analysis were all stored in a freezer within 8 hours after collection. Specimen storage was done in the requested frozen conditions. For harvest + processing phase trials, at least 50 kg of grain were taken in plot U and T2, and were sent at ambient temperature to the processing site.

For decline trials, whole plants (≥1 kg) were sampled just before application 2 and also at 0, 7 (±1), 14 (±1) and 28 (±2) days after application 2; ears and rest of plants (≥1 kg) were taken at 35 (±2) days after application 2; straw (≥0.5 kg) and grain (≥1 kg) were sampled at normal commercial harvest.

Treated raw and processed crop commodity specimens for this study were frozen upon collection, shipped frozen, and stored frozen for less than 814 days (27 months) [Peterek, 2018, 2015RES-IFP1968] and less than 687 days (23 months) [Ricelli, 2018, 2015RES-IFP1950] between sample collection and analysis (<-18 °C at the analytical facility).

Analytical methods used were PTRL Method P3770G for fluindapyr, 3-OH-fluindapyr, and fluindapyr-N-desmethyl-glucoside and method RA.17.01 for determination of 1-OH-fluindapyr, 1-OH-Met-N-desmethyl-fluindapyr and 1-COOH-fluindapyr, all with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the conjugates would be hydrolysed to their respective aglycones. Acceptance for concurrent recovery control was met with average recoveries ranging from 70 percent to 110 percent and relative standard deviations (RSD) ≤ 20 percent, respectively ≤ 15 percent for higher fortification levels.

The results on wheat grain are summarized in Table 110. The results on wheat forage, hay and straw are summarized in the section on feed commodities (Table 116 to Table 119).

Table 110 Residues of fluindapyr in wheat grain after foliar treatment with a suspension concentrate

Location, year, WHEAT GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [e]	Reference, Trial No.
Weston, GA, United States, 2016 (LOE25) Soil: loamy sand	2 (11) +COC	150 148	80 80	Foliar, 6 May, BBCH77	30	0.010, <0.010 (0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.02, <0.02 (0.02)	2016RES-FNF2456, PSM-16-02-11-01
Richland, IA, United States, 2016 (Soft Red) Soil: silty clay loam	2 (10) +NIS	150 149	99 101	Foliar, 11 June, BBCH77-80	30	0.14, 0.092 (0.12)	0.011, <0.010 (0.010)	0.022, 0.018 (0.020)	0.16, 0.11 (0.14)	2016RES-FNF2456, PSM-16-02-11-02

Location, year, WHEAT GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [e]	Reference, Trial No.
Richland, IA, United States, 2016 (Rollagspring) Soil: silty clay loam	2 (9) +NIS	152 152	88 103	Foliar, 26 June, BBCH85-87	29	0.11, 0.10 (0.10)	0.011, <0.010 (0.010)	0.026, 0.024 (0.025)	0.14, 0.12 (0.13)	2016RES-FNF2456, PSM-16-02-11-11
Bagley, IA, United States, 2016 (Flint Hard Red) Soil: loam	2^ (13)	147 152	120 118	Foliar, June 22, BBCH71	28	0.065, 0.051 (0.058)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.075, 0.061 (0.068)	2016RES-FNF2456, PSM-16-02-11-03
New Providence, IA, United States, 2016 (Glenn) Soil: clay loam	2^ (11)	146 148	88 77	Foliar, 21 June, BBCH54	20	0.086, 0.054 (0.070)	<0.010, <0.010 (<0.010)	0.056, 0.046 (0.051)	0.14, 0.10 (0.12)	2016RES-FNF2456, PSM-16-02-11-12
					27	0.038, 0.016 (0.027)	<0.010, <0.010 (<0.010)	0.026, 0.014 (0.020)	0.064, 0.030 (0.047)	
					30	0.022, 0.027 (0.024)	<0.010, <0.010 (<0.010)	0.018, 0.020 (0.019)	0.040, 0.047 (0.044)	
					35	0.023, 0.027 (0.025)	<0.010, <0.010 (<0.010)	0.020, 0.018 (0.019)	0.043, 0.045 (0.044)	
					38	0.027, 0.020 (0.023)	<0.010, <0.010 (<0.010)	0.038, 0.027 (0.033)	0.065, 0.047 (0.056)	
Bradshaw, NE, United States, 2016 (Cert. Overland) Soil: silt loam	2 (14) +COC	152 146	74 71	Foliar, 14 June, BBCH61	30	0.15, 0.15 (0.15)	0.014, 0.015 (0.014)	0.018, 0.011 (0.015)	0.17, 0.16 (0.16)	2016RES-FNF2456, PSM-16-02-11-04
Lebanon, OK, United States, 2016 (Tam 111) Soil: sandy loam	2 (10) +NIS	151 151	99 99	Foliar, 11 May, BBCH74	30	0.056, 0.051 (0.053)	<0.010, <0.010 (<0.010)	0.019, 0.030 (0.025)	0.075, 0.081 (0.078)	2016RES-FNF2456, PSM-16-02-11-05
Grace City, ND, United States, 2016 (Jerry) Soil: loam	2^ (10)	149 150	80 82	Foliar, 15 July, BBCH83-85	30	0.090, 0.086 (0.088)	0.017, 0.017 (0.017)	0.010, <0.010 (<0.01)	0.10, 0.096 (0.098)	2016RES-FNF2456, PSM-16-02-11-06
Eldrigde, ND, United States, 2016 (Jerry) Soil: sandy loam	2 (7) +COC	156 160	101 80	Foliar, 25 June, BBCH75	30	0.12, 0.12 (0.12)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.13, 0.13 (0.13)	2016RES-FNF2456, PSM-16-02-11-07
Montpelier, ND, United States, 2016 (Jerry)	2 (7) +NIS	155 148	101 81	Foliar, 25 June, BBCH75	30	0.16, 0.15 (0.16)	<0.010, <0.010 (<0.010)	0.011, 0.010 (0.010)	0.17, 0.16 (0.17)	2016RES-FNF2456, PSM-16-02-11-08

Location, year, WHEAT GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [e]	Reference, Trial No.
Soil: loam										
Montpelier, ND, United States, 2016 (Prosper) Soil: sandy loam	2 (10) +COC	149 151	106 106	Foliar, 20 July, BBCH73	30	0.016, 0.018 (0.017)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.026, 0.028 (0.027)	2016RES-FNF2456, PSM-16-02-11-14
Cleveland, ND, United States, 2016 (Prosper) Soil: sandy clay loam	2 (10) +COC	147 150	107 106	Foliar, 20 July, BBCH75	28	0.020, 0.027 (0.023)	<0.010, <0.010 (<0.010)	0.019, 0.014 (0.016)	0.039, 0.041 (0.040)	2016RES-FNF2456, PSM-16-02-11-13
Groom, TX, United States, 2016 (TAM 112) Soil: clay loam	2^ (10)	149 148	70 68	Foliar, 26 May, BBCH83	26	0.088, 0.085 (0.087)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.098, 0.095 (0.097)	2016RES-FNF2456, PSM-16-02-11-09
Claude, TX, United States, 2016 (TAM 112) Soil: clay	2 (10) +COC	150 149	69 70	Foliar, 26 May, BBCH83	20	0.14, 0.16 (0.15)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.15, 0.17 (0.16)	2016RES-FNF2456, PSM-16-02-11-10
					25	0.20, 0.15 (0.17)	0.011, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.21, 0.16 (0.19)	
					32	0.16, 0.19 (0.18)	0.011, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.17, 0.20 (0.19)	
					35	0.19, 0.18 (0.19)	0.013, 0.012 (0.012)	<0.010, <0.010 (<0.010)	0.20, 0.19 (0.20)	
					40	0.10, 0.11 (0.11)	0.010, 0.012 (0.011)	<0.010, <0.010 (<0.010)	0.11, 0.12 (0.12)	
Tulelake, CA, United States, 2016 (Rojo) Soil: silt loam	2^ (10)	149 149	80 116	Foliar, 1 July, BBCH75	31	0.25, 0.27 (0.26)	<0.010, <0.010 (<0.010)	0.012, <0.010 (0.011)	0.26, 0.28 (0.27)	2016RES-FNF2456, PSM-16-02-11-15
Jerome, ID, United States, 2016 (Alturas) Soil: sandy loam	2 (11) +COC	151 150	72 80	Foliar, 11 July, BBCH85	29	0.060, 0.060 (0.060)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.07, 0.07 (0.07)	2016RES-FNF2456, PSM-16-02-11-16
Minto, MB, United States, 2016 (CDC Plentiful) Soil: sandy clay loam	2 (11) +NIS	150 148	94 94	Foliar, 25 July, BBCH77-83	30	0.019, 0.017 (0.018)	<0.010, <0.010 (<0.010)	0.039, 0.042 (0.040)	0.058, 0.059 (0.059)	2016RES-FNF2456, PSM-16-02-11-17
Alvena, SK, United States, 2016 (Carberry)	2^ (11)	155 149	94 94	Foliar, 16 Aug., BBCH85-87	29	0.040, 0.042 (0.041)	<0.010, <0.010 (<0.010)	0.049, 0.032 (0.041)	0.089, 0.074 (0.082)	2016RES-FNF2456, PSM-16-02-11-18

Location, year, WHEAT GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [e]	Reference, Trial No.
Soil: loam										
Fort Saskatchewan, AB, United States, 2016 (Plentiful) Soil: clay loam	2 (11) +COC	151 151	94 94	Foliar, 2 Aug., BBCH69	30	0.025, 0.027 (0.026)	<0.010, <0.010 (<0.010)	0.014, 0.013 (0.013)	0.039, 0.04 (0.040)	2016RES-FNF2456, PSM-16-02-11-19
Lamont, AB, United States, 2016 (Plentiful) Soil: sandy loam	2 (12) +NIS	152 157	94 96	Foliar, 10 Aug, BBCH73	30	0.032, 0.025 (0.029)	<0.010, <0.010 (<0.010)	0.020, 0.011 (0.015)	0.052, 0.036 (0.044)	2016RES-FNF2456, PSM-16-02-11-20
OX12BNJ, South Fawley, United Kingdom, 2015 (winter wheat TRZAW) Soil: clay	2^ (13)	160 162	25 25	Foliar, 25 June, BBCH 69	48	0.015	<0.010	<0.010	0.025	2015RES-IFP1968, SPK-15-20471 GB01
OX12BNJ, South Fawley, United Kingdom, 2015 (winter wheat claire) Soil: clay	2^ (13)	763 721	125 125	Foliar, 25 June, BBCH 69	48	0.13	0.013	0.035	0.17	2015RES-IFP1968, SPK-15-20471 GB01 [P]
OX171AG, Edgecote, United Kingdom, 2015 (winter wheat sky fall) Soil: sand silt loam	2^ (13)	155 138	25 25	Foliar, 24 June, BBCH 69	72	0.024	<0.01	0.018	0.042	2015RES-IFP1968, SPK-15-20471 GB02
74572, Blaufelden-Mittelbach, DE, 2015 (winter wheat: Colonia) Soil: clay loam	2^ (15)	153 153	25 25	Foliar, 25 June, BBCH 69	40	0.044	<0.01	<0.01	0.054	2015RES-IFP1968, SPK-15-20471 DE03
74572, Blaufelden-Mittelbach, DE, 2015 (winter wheat: Colonia) Soil: clay loam	2^ (15)	689 745	125 125	Foliar, 25 June, BBCH 69	40	0.37	0.024	0.054	0.42 [b]	2015RES-IFP1968, SPK-15-20471 DE03 [P]
23847, Kastorf, DE, 2015 (winter wheat:	2^ (14)	140 145	25 25	Foliar, 15 June, BBCH 69	56	0.013	<0.01	<0.01	0.023	2015RES-IFP1968, SPK-15-

Location, year, WHEAT GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [e]	Reference, Trial No.
Tobak) Soil: loamy										20471 DE04
I-44012, Gavello, I, 2015 (winter wheat:50207) Soil: loam	2 (9)	159 159	26 26	Foliar, 20 May, BBCH 69	41	0.011	<0.01	<0.01	0.021	2015RES-IFP1950, RA1508-1H-P
I-44012, Gavello, I, 2015 (winter wheat:50207) Soil: loam	2 (9)	790 794	131 130	Foliar, 20 May, BBCH 69	41	0.26	0.034	0.067	0.33 [c]	2015RES-IFP1950, RA1508-1H-P [P]
F-4590, Marsillargues, F, 2015 (winter wheat:Arezzo) Soil: silty clay loam	2 (15)	152 153	25 25	Foliar, 21 May, BBCH 69	47	0.037	<0.010	0.017	0.054	2015RES-IFP1950, RA1508-2H-P
F-4590, Marsillargues, F, 2015 (winter wheat:Arezzo) Soil: silty clay loam	2 (15)	761 774	123 123	Foliar, 21 May, BBCH 69	47	0.64	0.028	0.12	0.76 [d]	2015RES-IFP1950, RA1508-2H-P [P]
I-44028, Poggio Renatico, I, 2015 (winter wheat: Solehio) Soil: loam	2 (9)	160 159	26 26	Foliar, 20 May, BBCH 69	42	0.037	<0.010	0.019	0.056	2015RES-IFP1950, RA1508-3D
F-33210, Saint Pierre de Mons, F, 2015 (winter wheat Solehio:) Soil: sandy loam	2 (15)	143 152	25 25	Foliar, 21 May, BBCH 73	40	0.041	<0.010	0.031	0.072	2015RES-IFP1950, RA1508-4D

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents.

[b] 0.010 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) was measured, but level was not included in the total calculation.

[c] 0.011 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) was measured, but level was not included in the total calculation.

[d] 0.034 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) was measured, but level was not included in the total calculation.

[e] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

[P] Used for processing study.

Sorghum

Supervised residue trials were conducted in the United States and Canada in 2015 (3) and 2016 (six in the United States only) to measure the magnitude of fluindapyr residues in/on sorghum agricultural commodities following two foliar applications of a fluindapyr SC formulation [Webber, 2018d, 2015RES-FNF1901 and Webber, 2018e, 2016RES-FNF2455]. The two foliar applications were made at an application rate of 148–159 g ai/ha each, with application interval of 9–12 days. At one site (PMS-15-02-04-01) exaggerated doses (747–770 hg ai/ha) were for processing purposes.

The sorghum (forage, grain, stover) samples were harvested at proper times to yield commercially representative samples. At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 12 separate areas within the plots so that each sample yielded a minimum of 1 kilogram of forage and grain and 0.5 kilogram of stover. Bulk samples for processing were at least 250 kg. The sorghum forage samples were harvested at 6–7 days after the last application and the grain and stover samples were harvested 45 days after the last application (2015 trials) and 28–30 days (2016 trials). Decline samples were collected at two trial locations. At these locations samples were collected at 3, 7, 10 and 13/14 days after last application for forage and 30/31, 34/35, 45, 54/55 and 60/61 days for grain and stover.

Sorghum raw agricultural commodity samples were maintained frozen after collection through analysis for up to 819 (2015 trials) and 498 (2016 trials) days. Frozen RAC samples were transferred from the field facility to the analytical laboratory in Norwell, MA, for preparation/homogenization and analysis. Samples were prepared by chopping and homogenizing the entire field sample, then removing a subsample for analysis. Samples were maintained frozen from receipt at the analytical facility until extraction for analysis.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-Met-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone1-OH-Met-fluindapyr.

The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 3-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from the LOQ (0.01 mg/kg) to 16.0 mg/kg for forage, 8.0 mg/kg for grain, and 2.0 mg/kg for stover, 26.0 mg/kg for aspirated grain fractions, and 3.0 mg/kg for flour.

Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum forage were 102 ±16 percent (n = 6), 101 ± 11 percent (n = 5) and 98 ± 9 percent (n = 5), respectively [Webber, 2018d, 2015RES-FNF1901]. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum forage were 99 ± 18 percent (n = 8), 96 ± 17 percent (n = 7) and 100 ± 13 percent (n = 7), respectively [Webber, 2018e, 2016RES-FNF2455].

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum grain were 100 ± 25 percent (n = 6), 88 ± 14 percent (n = 5) and 84 ± 9 percent (n = 5), respectively [Webber, 2018d, 2015RES-FNF1901]. The overall mean

laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum grain were 105 ± 23 percent (n = 8), 92 ±12 percent (n = 7) and 87 ± 11 percent (n = 7), respectively [Webber, 2018e, 2016RES-FNF2455].

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum stover were 109 ± 13 percent (n = 6), 93 ±

10 percent (n = 6) and 80 ± 9 percent (n = 5), respectively [Webber, 2018d, 2015RES-FNF1901]. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum stover were 108 ± 11 percent (n = 8), 99 ± 11 percent (n = 8) and 82 ± 6 percent (n = 7), respectively [Webber, 2018e, 2016RES-FNF2455].

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum aspirated grain fractions were 107 ±

17 percent (n = 4), 104 ± 4 percent (n = 3) and 77 ± 8 percent (n = 3), respectively [Webber, 2018d, 2015RES-FNF1901].

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum flour were 100 ± 31 percent (n = 3), 106 ± 30 percent (n = 3) and 82 ± 13 percent (n = 3), respectively [Webber, 2018d, 2015RES-FNF1901].

The results on sorghum grain are summarized in Table 111.

Table 111 Residues of fluindapyr in sorghum grain after foliar treatment with a suspension concentrate

Location, year, SORGHUM GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
Bradshaw, NE, United States, 2015 (DKS37-07) Soil: silt loam	2 (11) +NIS	159 151	69 70	Foliar, 11 Sept., 2015 BBCH 77	45	0.062, 0.052 (0.057)	<0.01, <0.01 (<0.01)	0.044, 0.022 (0.033)	0.11, 0.074 (0.090)	2015RES-FNF1901, PSM-15-02-04-01
Bradshaw, NE, United States, 2015 (DKS37-07) Soil: silt loam	2 (11) +NIS	770 747	342 347	Foliar, 11 Sept., 2015 BBCH 77	45	4.5, 2.6 (3.5)	0.24, 0.14 (0.19)	0.18, 0.23 (0.20)	4.7, 2.8 (3.8)	2015RES-FNF1901, PSM-15-02-04-01 (P)
Groom, TX, United States, 2015 (H-390W) Soil: silty clay loam	2^ (9)	152 150	68 68	Foliar, 25 Sept., BBCH 83	31	0.25, 0.10 (0.18)	<0.01, <0.01 (<0.01)	0.022, 0.027 (0.025)	0.27, 0.13 (0.20)	2015RES-FNF1901, PSM-15-02-04-02
					34	0.20, 0.30 (0.23)	0.010, 0.014 (0.012)	0.041, 0.043 (0.042)	0.24, 0.34 (0.29)	
					45	0.22, 0.27 (0.24)	0.015, 0.019 (0.017)	0.044, 0.038 (0.041)	0.26, 0.31 (0.29)	
					55	0.18, 0.12 (0.15)	0.013, <0.010 (0.011)	0.024, 0.028 (0.026)	0.20, 0.15 (0.18)	
					60	0.12, 0.11 (0.012)	0.010, <0.010 (0.010)	0.021, 0.031 (0.026)	0.14, 0.14 (0.14)	
Lebanon, OK, United States,	2^ (10)	151 149	63 64	Foliar, 2 Sept.,	30	0.10, 0.29	<0.01, 0.018	0.094, 0.11	0.19, 0.40	2015RES-FNF1901,

Location, year, SORGHUM GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
2015 (H390W) Soil: sandy loam				BBCH 79		(0.20)	(0.013)	(0.10)	(0.30)	PSM-15-02-04-03
					35	0.23, 0.11 (0.17)	0.015, <0.01 (0.011)	0.079, 0.060 (0.069)	0.31, 0.17 (0.24)	
					45	0.18, 0.32 (0.20)	0.013, 0.014 (0.013)	0.052, 0.039 (0.046)	0.23, 0.36 (0.30)	
					54	0.39, 0.24 (0.32)	0.030, 0.019 (0.024)	0.073, 0.078 (0.075)	0.46, 0.32 (0.39)	
					61	0.28, 0.58 (0.43)	0.021, 0.041 (0.031)	0.056, 0.048 (0.052)	0.34, 0.63 (0.48)	
Lebanon, OK, United States, 2016 (H-390W) Soil: sandy loam	2 (10) +COC	148 152	108 97	Foliar, 26 Aug., BBCH 68	29	0.10, 0.095 (0.10)	<0.01, <0.01 (<0.01)	0.049, 0.041 (0.045)	0.15, 0.14 (0.14)	2016RES-FNF2455, PSM-16-02-10-04
Fisk, MO, United States, 2016 (M383C) Soil: sand	2^ (12)	149 153	80 80	Foliar, 16 Aug., BBCH 83	29	0.41, 0.45 (0.43)	0.026, 0.031 (0.029)	0.12, 0.14 (0.13)	0.53, 0.59 (0.56)	2016RES-FNF2455, PSM-16-02-10-01
Richland, IA, United States, 2016 (AG1401) Soil: silty clay loam	2^ (9)	150 150	82 79	Foliar, 2 Sept., BBCH 74-75	28	0.33, 0.34 (0.34)	<0.01, <0.01 (<0.01)	0.038, 0.048 (0.043)	0.37, 0.39 (0.38)	2016RES-FNF2455, PSM-16-02-10-02
York, NE, United States, 2016 (DKS37-07) Soil:silt loam	2 (10) +COC	155 153	93 92	Foliar, 12 Sept., BBCH 87	29	0.34, 0.24 (0.29)	0.015, 0.012 (0.013)	0.067, 0.057 (0.062)	0.41, 0.30 (0.35)	2016RES-FNF2455, PSM-16-02-10-03
Cleveland, ND, United States, 2016 (Sweetie) Soil: sandy clay loam	2 (10) +COC	150 159	108 107	Foliar, 5 Sept., BBCH 83	29	0.29, 0.44 (0.37)	0.011, 0.015 (0.013)	0.050, 0.061 (0.056)	0.34, 0.50 (0.42)	2016RES-FNF2455, PSM-16-02-10-05
Claude, TX, United States, 2016 (Y373) Soil: clay	2 (10) +NIS	150 156	66 67	Foliar, 3 Sept., BBCH 80	30	0.37, 0.37 (0.37)	0.017, 0.017 (0.017)	0.041, 0.040 (0.040)	0.41, 0.41 (0.41)	2016RES-FNF2455, PSM-16-02-10-06

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

[P] Used for processing.

Maize cereals

Two studies with three [Webber, 2018a, 2015RES-FNF1900] and 18 [Webber, 2018b, 2016RES-FNF2453] field trials were conducted in the United States in 2015 and 2016 to measure the magnitude of fluindapyr residues in/on field corn raw agricultural commodities following two foliar application of fluindapyr SC formulation.

The two foliar applications were made at an application rate of 148–169 g ai/ha each, with application interval of 10–11 days. Additional plots [Webber, 2018a, 2015RES-FNF1900] were treated with exaggerated dose levels (5×) for the purpose of processing (see section on processing).

The field corn (forage, grain, stover) samples were harvested at proper times to yield commercially representative samples. At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 12 separate areas within the plots so that each sample yielded a minimum of 1 kilogram of forage, 1 kilogram of grain, and 0.5 kilogram of stover. Bulk grain samples reached the minimum of 240 kg per sample. The field corn raw agricultural commodity of forage was harvested at 6-7 DALA. The field corn raw agricultural commodities of grain and stover were harvested at 45 DALA.

Decline samples were collected at two trial locations (PSM-15-02-03, trial 02 and 03). At these locations samples were collected at a target of 0, 3, 7, 10, and 14 days after last application for forage and 30, 35, 45, 55, and 60 days after last application for grain and stover.

Treated raw crop commodity specimens for the 2015 study were frozen upon collection, shipped frozen, and stored frozen for less than 816 days (27 months) between sampling and analysis (<-20 °C at the analytical facility). In the 2016 study treated raw crop commodity samples were frozen upon collection, shipped frozen, and stored frozen for less than 483 days (16 months) between sampling and analysis (<-20 °C at the analytical facility).

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-Met-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone1-OH-Met-fluindapyr.

The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from the LOQ (0.01 mg/kg) to 15.5 mg/kg for forage, 0.1 mg/kg for grain, 2.0 mg/kg for stover, 1.0 mg/kg for aspirated grain fractions, and 0.1 mg/kg for all processed commodities. Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 3-OH-Met-fluindapyr from field corn grain were 100 ± 4 percent (n = 4), 96 ± 9 percent (n = 4) and 77 ± 7 percent (n = 4), respectively [Webber, 2018a, 2015RES-FNF1900] and 94 ± 7 percent (n = 6), 95 ±3 percent (n = 6) and 85 ± 16 percent (n = 6), respectively in the second study [Webber, 2018b, 2016RES-FNF2453].

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from field corn aspirated grain fractions were 112 ± 12 percent (n = 3), 119 ± 6 percent (n = 3) and 78 ± 8 percent (n = 3), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from field corn starch were 130 percent (n = 2), 117 percent (n = 2) and 77 percent (n = 2), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from field corn refined oil (wet milling) were 93 percent (n = 2), 100 percent (n = 2) and 82 percent (n = 2), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from field corn grits were 89 percent (n = 2), 74 percent (n = 2) and 77 percent (n = 2), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from field corn flour were 98 percent (n = 2), 94 percent (n = 2) and 84 percent (n = 2), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from field corn germ were 94 percent (n = 2), 109 percent (n = 2) and 74 percent (n = 2), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from field corn meal were 98 percent (n = 2), 90 percent (n = 2) and 73 percent (n = 2), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from field corn refined oil (dry milling) were 124 percent (n = 2), 106 percent (n = 2) and 84 percent (n = 2), respectively.

The results on grain are summarized in Table 112.

Table 112 Residues of fluindapyr in field corn grain after foliar treatment with a suspension concentrate

Location, year, MAIZE/ FIELD CORN GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
Farlin, IA, United States, 2015 (P1248) Soil: clay loam	2^ (11)	152 156	63 65	Backpack sprayer, 29 Aug., BBCH75	45	<0.01	<0.01	<0.01	<0.02	2015RES-FNF1900, PSM-15-02-03-01
Perry, IA, United States, 2015 (2F721) Soil: loam	2^ (11)	152 153	76 67	Backpack sprayer, 10 Aug., BBCH74	30	<0.01	<0.01	<0.01	<0.02	2015RES-FNF1900, PSM-15-02-03-02
					35	<0.01	<0.01	<0.01	<0.02	
					45	<0.01	<0.01	<0.01	<0.02	
					56	<0.01	<0.01	<0.01	<0.02	
					60	<0.01	<0.01	<0.01	<0.02	
Hedrick, IA, United States, 2016 (P1311AMXT) Soil: silty clay loam	2^ (10)	152 152	88 92	Backpack Sprayer, 26 Aug., BBCH85-87	29	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-03

Location, year, MAIZE/ FIELD CORN GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
Richland, IA, United States, 2016 (PO937AM) Soil: silty clay loam	2^ (10)	152 152	78 92	Tractor mounted boom, 26 Aug., BBCH85	29	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-04
Bagley, IA, United States, 2016 (9732RR) Soil: clay loam	2 (10) +COC	150 145	93 91	Backpack Sprayer, 19Aug., BBCH85	30	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-08
Lime Springs, IA, United States, 2016 (DKC46-37RIB) Soil: sandy loam	2 (11) +COC	149 148	106 106	Backpack Sprayer, 12 Sept., BBCH87	31	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-12 (corn)
Cresco, IA, United States, 2016 (P9929AM) Soil: silt loam	2 (11) +NIS	149 150	106 107	Backpack Sprayer, 12 Sept., BBCH87	28	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-13 (corn)
Seven Springs, NC, United States, 2015 (DKC68-03) Soil: loamy sand	2 (10) +NIS	167 169	70 71	Tractor mounted sprayer, 24 July, BBCH79	31	<0.01	<0.01	<0.01	<0.02	2015RES-FNF1900, PSM-15-02-03-03
					35	<0.01	<0.01	<0.01	<0.02	
					45	<0.01	<0.01	<0.01	<0.02	
					55	<0.01	<0.01	<0.01	<0.02	
					59	<0.01	<0.01	<0.01	<0.02	
Germansville, PA, United States, 2016 (TA545-33EZ) Soil: loam	2^ (10)	152 159	106 107	Backpack Sprayer, 08 Sept., R5	28	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-01
Wyoming, IL, United States, 2016 (DKC 61-86) Soil: clay loam	2^ (10)	151 148	118 111	Backpack Sprayer, 22 Sept, BBCH87	29	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-02
Carlyle, IL, United States, 2016 (Syngenta N78S-3111) Soil: silt loam	2^ (10)	151 150	86 79	Tractor mounted boom, 12 Sept., BBCH85	30	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-05 (corn)
Geneva, MN, United States, 2016 (NuTech 5D-196AMX) Soil: sandy clay loam	2 (10) +COC	152 151	88 79	Backpack Sprayer, 12 Sept, BBCH85-87	30	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-06

Location, year, MAIZE/ FIELD CORN GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
Ellendale, MN, United States, 2016 (Dekalb DKC44-13RIB) Soil:sandy loam	2 (9) +COC	149 152	86 80	Backpack Sprayer, 15 Sept, BBCH85-87	30	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-07
Paynesville, MN, United States, 2016 (NK 23MGTA) Soil: sandy loam	2 (10) +COC	152 148	97 94	Tractor mounted boom, 30 Aug., BBCH85	30	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-09
York, NE, United States, 2016 (DKC62-77RIB) Soil: silt loam	2 (10) +COC	145 150	93 93	Backpack Sprayer, 12 Sept., BBCH87	30	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-10
Brunswick, NE, United States, 2016 (DKC62-77RIB) Soil: sand	2 (11) +COC	152 151	91 94	Backpack Sprayer, 12 Sept., BBCH87	31	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-11
Brookings, SD, United States, 2016 (DKC 44-13) Soil: loam	2 (10) +NIS	151 151	103 92	Backpack Sprayer, 29 Sept., BBCH85	32	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-14 (corn)
Deerfield, MI, United States, 2016 (AgriGold A6472VT3P RIB) Soil: sandy clay loam	2 (10) +NIS	150 149	80 81	Backpack Sprayer, 20 Aug., BBCH73	30	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-15
Milan, MI, United States, 2016 (Dekalb DKC60-67 Genssrib) Soil: clay loam	2 (10) +NIS	150 152	80 73	Backpack Sprayer, 20Aug., BBCH73	30	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-16
Cleveland, ND, United States, 2016 (01049135) Soil: sandy	2 (10) +COC	151 160	107 107	Tractor mounted boom, 05 Sept., BBCH83	30	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453, PSM-16-02-08-17 (corn)
Claude, TX, United States,	2 (10)	149 153	67 66	Backpack Sprayer,	30	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2453,

Location, year, MAIZE/ FIELD CORN GRAIN (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
2016 (P1234AM) Soil: clay loam	+NIS			03 Sept., BBCH84						PSM-16-02-08-18

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents. Since all results were <0.01 mg/kg in both duplicates, only the mean is included in the table.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Sweet corns

Supervised residue trials were conducted in the United States in 2015 and 2016 to measure the magnitude of fluindapyr residues in/on sweet corn raw agricultural commodities following two foliar applications of a fluindapyr SC formulation [Webber, 2018c, 2016RES-FNF2454]. The two foliar applications were made at an application rate of 148–156 g ai/ha each, with application interval of 10–11 days.

The sweet corn (Kernel + Cobs With Husks Removed = K+CWHR, forage, stover) samples were harvested at proper times to yield commercially representative samples. At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 12 separate areas within the plots so that each sample yielded a minimum of 1 kilogram of K+CWHR, 1 kilogram of forage, and 0.5 kilogram of stover. The sweet corn raw agricultural commodities of K+CWHR, forage, and stover were harvested at 12–16 days after the second application to the treated plot. Decline samples were collected at one trial location (Trial 06). At these locations samples were collected at a target of 3, 7, 14, 21, and 28 days after last application for K+CWHR, forage and stover.

Sweet corn raw agricultural commodity samples were maintained frozen after collection through analysis for up to 564 days. Frozen RAC samples were transferred from the field facility to the analytical laboratory in Norwell, MA, for preparation/homogenization and analysis. Samples were prepared by chopping and homogenizing the entire field sample, then removing a subsample for analysis. Samples were maintained frozen from receipt at the analytical facility until extraction for analysis.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-Met-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone1-OH-Met-fluindapyr.

The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from the LOQ (0.01 mg/kg) to 1.0 mg/kg for K+CWHR, 10.0 mg/kg for forage, and 20.8 mg/kg for stover. Fortified control samples were included in each analysis set for method verification. The

methodology was shown to be robust for the analysis of sweet corn K+CWHR, forage, and stover in this study.

Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sweet corn K+CWHR were 97 ± 11 percent (n = 4), 95 ± 5 percent (n = 4) and 82 ± 7 percent (n = 4), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sweet corn forage were 102 ± 11 percent (n = 5), 104 ±3 percent (n = 4) and 90 ± 12 percent (n = 4), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sweet corn stover were 101 ± 13 percent (n = 5), 102 ± 3 percent (n = 4) and 93 ± 10 percent (n = 4), respectively.

The results on K+CWHR are summarized in Table 113.

Table 113 Residues of fluindapyr in sweet corn K+CWHR after foliar treatment with a suspension concentrate

Location, year, SWEET CORN K+CWHR (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
Alton, NY, United States, 2016, (Precious Gem F1) Soil: sandy loam	2^ (10)	150 150	107 106	Tractor mounted sprayer, 09 Sept., BBCH79	14	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2454, PSM-16-02-09-01
Weston, GA, United States, 2016, (Silver Queen) Soil: loamy sand	2^ (10)	148 150	74 74	Backpack sprayer, 01 Aug., BBCH71	14	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2454, PSM-16-02-09-02
Richland, IA, United States, 2016 (Delectable TRTDF1) Soil: silty clay loam	2^ (9)	150 149	91 65	Tractor mounted sprayer, 09 July, BBCH65-69	16	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2454, PSM-16-02-09-03
Lime Springs, IA, United States, 2016 (Luscious) Soil: sandy loam	2 (11) +COC	153 152	82 84	Backpack sprayer, 18 July, BBCH69	14	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2454, PSM-16-02-09-04
Deerfield, MI, United States, 2016 (Iochief) Soil: sandy clay	2 (9) +COC	150 152	82 73	Backpack sprayer, 20Aug., BBCH71	12	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2454, PSM-16-02-09-05

Location, year, SWEET CORN K+CWHR (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
loam										
York, NE, United States, 2016 (Obession II) Soil: silt loam	2 (11) +COC	151 150	91 91	Backpack sprayer, 07 Aug., BBCH not reported	4	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2454, PSM-16-02-09-06
					8	<0.01	<0.01	<0.01	<0.02	
					13	<0.01	<0.01	<0.01	<0.02	
					22	<0.01	<0.01	<0.01	<0.02	
					26	<0.01	<0.01	<0.01	<0.02	
Payette, ID, United States, 2016 (Ambrosia) Soil: clay loam	2 (10) +NIS	155 156	70 70	Tractor mounted sprayer, 21 July, BBCH67	14	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2454, PSM-16-02-09-07
Oregon City, OR, United States, 2016 (Honey N Pearl L) Soil: silt loam	2 (9) +NIS	152 148	74 84	Backpack sprayer, 17 Aug., BBCH69-71	14	<0.01	<0.01	<0.01	<0.02	2016RES-FNF2454, PSM-16-02-09-08

Notes:

K+CWHR = kernels plus cob with husks removed; RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents. Since all results were <0.01 mg/kg in both duplicates, only the mean is included in the table.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Tree nuts

Almond

Supervised residue trials were conducted in the United States in 2016 to measure the magnitude of fluindapyr residues in/on almond agricultural commodities following three foliar applications of a fluindapyr SC formulation [Webber, 2017a, 2016RES-FNF2450]. At each site two plots received three foliar applications at an application rate of 156–192 g ai/ha, with an interval of 7 to 8 days with the last application made at BBCH75–85. At one plot (T2) 443-528 L water/ha was used and the second plot (T3) 1730–1991 L/ha water was used.

The almond (nutmeat and hulls) samples were harvested at maturity to yield commercially representative samples. At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 4 different trees within the plots so that each sample yielded a minimum of 1 kilogram of nutmeat and hulls. The samples were harvested at 29–31 days after the last application. Decline samples were collected at one trial locations on days 15, 23, 30, 37, and 44 after the last application.

Samples were maintained frozen after collection through analysis for up to 379 (nutmeat) and 384 (hulls) days. Frozen RAC samples were transferred from the field facility to the analytical laboratory in Norwell, MA, for preparation/homogenization and analysis. Samples were prepared by chopping and

homogenizing the entire field sample, then removing a subsample for analysis. Samples were maintained frozen from receipt at the analytical facility until extraction for analysis.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-Met-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone1-OH-Met-fluindapyr.

The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from 1/5 LOQ (0.002 mg/kg) to 10 × LOQ (0.1 mg/kg). The overall mean method validation recoveries and RSDs for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from almond nutmeat were 99 ± 11 percent (n = 10), 82 ± 7 percent (n = 10) and 92 ± 6 percent (n = 10), respectively.

Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from almond nutmeat were 107 ± 18 percent (n = 4), 86 ± 15 (n = 4) and 91 ± 7 percent (n = 4), respectively.

The results on almond nutmeat are summarized in Table 114.

Table 114 Residues of fluindapyr in almond nutmeat after three foliar treatments with a suspension concentrate

Location, year, ALMOND NUTMEAT (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	**Reference, Trial No.**
Yuba City, CA, United States, 2016 (Non-pareil) Soil: clay loam	3^ (8,7)	168 164 156	36 36 34	Foliar, 13 July, BBCH75	29	<0.010, 0.014 (0.011)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.020, 0.024 (0.022)	2016RES-FNF2450, PSM-16-02-05-01
		167 171 167	10 10 9		29	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
Orland, CA, United States, 2016 (Non-pareil) Soil:loam	3^ (7,7)	164 164 164	35 35 35	Foliar, 17 July, BBCH85	30	0.025, 0.019 (0.022)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.035, 0.029 (0.032)	2016RES-FNF2450, PSM-16-02-05-02
		164 164 164	9 9 9		30	0.019, 0.023 (0.021)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.029, 0.033 (0.031)	
Strathmore, CA, United States, 2016 (Fritz) Soil: sandy loam	3 (7,7) +COC	167 168 166	34 34 33	Foliar, 22 July, BBCH77	31	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	2016RES-FNF2450, PSM-16-02-05-03
		168 170 169	9 9 9		31	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
Sanger, CA, United States, 2016 (Non-pareil) Soil: sandy loam	3 (7,7) +COC	188 188 166	36 42 34	Foliar, 10 Aug., BBCH81-85	30	0.019, 0.011 (0.015)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.029, 0.021 (0.025)	(2016RES-FNF2450, PSM-16-02-05-04
		188 192 165	9 11 9		30	0.017, 0.018 (0.018)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.027, 0.028 (0.028)	
Terra Bella,	3 (7,7)	166	35	Foliar,	15	0.026,	<0.010,	<0.010,	0.036,	2016RES-

Location, year, ALMOND NUTMEAT (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
CA, United States, 2016 (Monterey) Soil: sandy loam	+NIS	168 166	36 34	20 July, BBCH79		0.015 (0.021)	<0.010 (<0.010)	<0.010 (<0.010)	0.025 (0.031)	FNF2450, PSM-16-02-05-05
					23	0.027, 0.034 (0.030)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.037, 0.044 (0.041)	
					30	<0.010, <0.010 (<u><0.010</u>)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<u><0.010</u>)	<0.020, <0.020 (<u><0.020</u>)	
					37	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
					44	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
		167 165 166	9 9 9		15	0.011, 0.026 (0.018)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.021, 0.036 (0.029)	
					23	0.047, 0.016 (0.031)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.057, 0.026 (0.042)	
					30	<0.010, <0.010 (<0.010)	0.011, <0.010 (<0.010)	0.010, <0.010 (<0.010)	0.020, <0.020 (0.020)	
					37	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
					44	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Pecan

Supervised residue trials were conducted in the United States in 2016 to measure the magnitude of fluindapyr residues in/on pecan agricultural commodities following three foliar applications of a fluindapyr SC formulation [Webber, 2017b, 2016RES-FNF2451]. At each site two plots received three foliar applications at an application rate of 165–177 g ai/ha, with an interval of 7 days with the last application made at BBCH78–85 (chuck split stage). At one plot (T2) 450–555 L water/ha was used and the second plot (T3) 1583–1994 L/ha water was used.

The pecan nutmeat samples were harvested at maturity to yield commercially representative samples. At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 4 different trees within the plots so that each sample yielded a minimum of 1 kilogram of nutmeat. The

samples were harvested at 30 days after the last application. Decline samples were collected at one trial locations on days 16, 23, 30, 37, and 44 after the last application.

Samples were maintained frozen after collection through analysis for up to 272 days. Frozen RAC samples were transferred from the field facility to the analytical laboratory in Norwell, MA, for preparation/homogenization and analysis. Samples were prepared by chopping and homogenizing the entire field sample, then removing a subsample for analysis. Samples were maintained frozen from receipt at the analytical facility until extraction for analysis.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-Met-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone1-OH-Met-fluindapyr.

The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from 1/5 LOQ (0.002 mg/kg) to 10 × LOQ (0.1 mg/kg). The overall mean method validation recoveries and RSDs for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from pecan nutmeat were 92 ± 4 percent (n = 10), 97 ± 2 percent (n = 10) and 88 ± 5 percent (n = 10), respectively.

Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from pecan nutmeat were 102 percent (n = 4), 102 percent (n = 4) and 74 percent (n = 4), respectively.

The results on pecan nutmeat are summarized in Table 115.

Table 115 Residues of fluindapyr in pecan nutmeat after three foliar treatments with a suspension concentrate

Location, year, PECAN NUTMEAT (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
Weston, GA, United States, 2016 (Elliots) Soil: loamy sand	3^ (7,)	166 167 166	33 33 33	Foliar, 7 Nov, BBCH85	30	0.025, 0.023 (0.024)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.035, 0.033 (0.034)	2016RES-FNF2451, PSM-16-02-06-01
		166 166 166	9 9 9		30	0.014, 0.017 (0.016)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.024, 0.027 (0.026)	
Chula, GA, United States, 2016 (Summer) Soil: loamy sand	3^ (7,7)	167 167 167	31 30 30	Foliar, 2 Nov., BBCH78	30	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	2016RES-FNF2451, PSM-16-02-06-02
		167 167 167	8 8 8		30	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
Port Barre, LA, United States, 2016 (Coupee) Soil: clay loam	3 (7,7) +COC	169 173 174	38 36 36	Foliar, 11 Oct., BBCH n.r.	30	0.011, <0.010 (0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.021, <0.020 (0.021)	2016RES-FNF2451, PSM-16-02-06-03
		171 167 168	11 11 11		30	0.018, 0.013 (0.016)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.028, 0.023 (0.026)	

Location, year, PECAN NUTMEAT (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [b]	Reference, Trial No.
Antioch, OK, United States, 2016 (Choctaw/ pawnee) Soil: clay loam	3 (7,7) +COC	177 165 169	35 34 34	Foliar, 23 Oct., BBCH82	30	<0.010, <0.010 (<u>≤0.010</u>)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<u>≤0.010</u>)	<0.020, <0.020 (<u>≤0.020</u>)	2016RES-FNF2451, PSM-16-02-06-04
		168 176 166	10 10 10		30	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
Chillicothe, TX, United States, 2016 (Pawnee) Soil: sandy loam	3 (7,7) +NIS	168 173 174	35 35 34	Foliar, 12 Oct., Chuck split stage	16	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	2016RES-FNF2451, PSM-16-02-06-05
					23	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
					30	<0.010, <0.010 (<u>≤0.010</u>)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<u>≤0.010</u>)	<0.020, <0.020 (<u>≤0.020</u>)	
					37	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
					44	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
		168 168 169	9 9 9		16	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
					23	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
					30	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
					37	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
					44	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate; n.r. = not reported.

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Animal Feed

Wheat forage, hay and straw–United States trials

Supervised residue trials were conducted in the United States in 2016 to measure the magnitude of fluindapyr residues in/on wheat agricultural commodities following two foliar applications of a fluindapyr SC formulation [Webber, 2018e, 2016RES-FNF2456]. The details of the study are described in the section on food commodities (wheat grain).

The wheat (forage, hay, and straw) samples were harvested at proper times to yield commercially representative samples. At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 12 separate areas within the plots so that each sample yielded a minimum of 1 kilogram of forage and at least 0.5 kilogram of hay or straw. The wheat forage samples were harvested at 7–8 days after the last application and the hay samples were collected from the same plots at 14–15 days after the last treatment. The straw samples were harvested 26-31 days after the last application. Decline samples were collected at two trial locations. At these locations samples were collected at 0, 3, 7±1, 10±1 and 14±1 days after last application for treated forage and 7±1, 10±1, 14±2, 21±2, and 28±2 days after last application for treated hay and 20±2, 25±2, 30±2, 35±2, and 40±2 days after the last application for treated grain and straw.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone1-OH-Met-fluindapyr.

The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from the LOQ (0.01 mg/kg) to 20.0 mg/kg for forage, hay and straw and 1.0 mg/kg for grain. Fortified control samples were included in each analysis set for method verification.

Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from wheat forage were 100 ± 8 percent (n = 9), 101 ± 7 percent (n = 8) and 83 ± 12 percent (n = 9), respectively. Recoveries from wheat hay were 104 ± 11 percent (n = 9), 100 ± 6 percent (n = 8) and 78 ± 12 percent (n = 8), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from wheat grain were 100 ± 8 percent (n = 8), 100 ±

7 percent (n = 8) and 82 ± 18 percent (n = 10), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from wheat straw were 98 ± 12 percent (n = 9), 105 ±

8 percent (n = 8) and 83 ± 22 percent (n = 10), respectively.

Wheat forage, hay and straw–European trials

Supervised residue trials were conducted in Northern (United Kingdom and Germany) and Southern (Italy and France) Europe in 2015 to measure the magnitude of fluindapyr residues in/on wheat agricultural commodities following two foliar applications of a fluindapyr EC formulation [Peterek, 2018, 2015RES-IFP1968 and Ricelli, 2018, 2015RES-IFP1950]. The details of the study are described in the section n food commodities (wheat grain).

For harvest trials, straw samples (at least 0,5 kg) were taken in plots U and T1 at normal commercial harvest. After sampling, the specimens for analysis were all stored in a freezer within 8 hours after collection. Specimen storage was done in the requested frozen conditions. For decline trials, whole plants (≥1 kg) were sampled just before application 2 and also at 0, 7 (±1), 14 (±1) and 28 (±2) days after

application 2; ears and rest of plants (≥1 kg) were taken at 35 (±2) days after application 2; straw (≥ 0.5 kg) and grain (≥1 kg) were sampled at normal commercial harvest (DALA 40–47 days).

Analytical methods used were PTRL Method P3770G for fluindapyr, 3-OH-fluindapyr, and fluindapyr-N-desmethyl-glucoside and method RA.17.01 for determination of 1-OH-fluindapyr, 1-OH-Me-N-desmethyl-fluindapyr and 1-COOH-fluindapyr, all with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the conjugated metabolites would be hydrolysed to their respective aglycones. Acceptance for concurrent recovery control was met with average recoveries ranging from 70 percent to 110 percent and relative standard deviations (RSD) ≤ 20 percent, respectively ≤ 15 percent for higher fortification levels.

The results on wheat forage, hay, whole plants, ears, rest of plants, and straw are summarized in Table 116 to Table 119.

Table 116 Residues of fluindapyr in wheat forage after foliar treatment with a suspension concentrate

Location, year, WHEAT FORAGE (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Weston, GA, United States, 2016 (LOE25) Soil: loamy sand	2 (10) +COC	149 150	80 83	Foliar, 21 Mar., BBCH33	7	6.2, 11 (8.8)	0.071, 0.11 (0.091)	0.36, 0.30 (0.33)	6.6, 12 (9.2)	2016RES-FNF2456, PSM-16-02-11-01
Richland, IA, United States, 2016 (Soft Red) Soil: silty clay loam	2 (9) +NIS	150 153	97 94	Foliar, 25 May, BBCH55	8	0.48, 0.33 (0.41)	0.019, 0.014 (0.017)	0.22, 0.17 (0.19)	0.70, 0.50 (0.60)	2016RES-FNF2456, PSM-16-02-11-02
Richland, IA, United States, 2016 (Rollagspring) Soil: silty clay loam	2 (10) +NIS	150 148	92 97	Foliar, 5 June, BBCH49	7	3.8, 3.2 (3.5)	0.054, 0.045 (0.049)	0.25, 0.22 (0.23)	4.0, 3.4 (3.7)	2016RES-FNF2456, PSM-16-02-11-11
Bagley, IA, United States, 2016 (Flint Hard Red) Soil: loam	2^ (8)	153 155	120 113	Foliar, 11 May, BBCH34	7	1.4, 1.6 (1.5)	0.024, 0.025 (0.024)	0.091, 0.088 (0.090)	1.5, 1.7 (1.6)	2016RES-FNF2456, PSM-16-02-11-03
New Providence, IA, United States, 2016 (Glenn) Soil: clay loam	2^ (11)	148 151	8877	Foliar, 21 June, BBCH54	0	4.8, 4.0 (4.4)	0.038, 0.039 (0.038)	0.27, 0.26 (0.27)	5.1, 4.3 (4.7)	2016RES-FNF2456, PSM-16-02-11-12
					3	2.5, 2.6 (2.6)	0.068, 0.073 (0.071)	0.30, 0.33 (0.31)	2.8, 2.9 (2.9)	
					7	0.93, 0.80 (0.86)	0.047, 0.049 (0.048)	0.32, 0.25 (0.29)	1.3, 1.1 (1.2)	
					10	0.68, 0.88 (0.79)	0.038, 0.047 (0.042)	0.40, 0.43 (0.41)	1.1, 1.3 (1.2)	
					14	0.78,	0.055,	0.45,	1.1, 1.2	

Location, year, WHEAT FORAGE (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
						0.71 (0.74)	0.051 (0.053)	0.44 (0.44)	(1.2)	
Bradshaw, NE, United States, 2016 (Cert. Overland) Soil: silt loam	2 (9) +COC	148 152	69 72	Foliar, 13 April, BBCH29	7	1.4, 1.4 (1.4)	0.036, 0.32 (0.034)	0.21, 0.22 (0.22)	1.6, 1.6 (1.6)	2016RES-FNF2456, PSM-16-02-11-04
Lebanon, OK, United States, 2016 (Tam 111) Soil: sandy loam	2 (10) +NIS	152 152	93 97	Foliar, 20 Mar., BBCH28	7	7.2, 6.2 (6.7)	0.12, 0.095 (0.10)	0.22, 0.25 (0.24)	7.5, 6.5 (6.9)	2016RES-FNF2456, PSM-16-02-11-05
Cleveland, ND, United States, 2016 (Prosper) Soil: sandy clay loam	2 (7) +COC	153 149	106 107	Foliar, 27 June, BBCH36	8	6.4, 6.4 (6.4)	0.11, 0.11 (0.11)	0.58, 0.56 (0.57)	7.0, 6.9 (7.0)	2016RES-FNF2456, PSM-16-02-11-13
Montpelier, ND, United States, 2016 (Prosper) Soil: sandy loam	2 (8) +COC	147 150	107 106	Foliar, 28 June, BBCH36	7	2.5, 2.0 (2.2)	0.072, 0.062 (0.067)	0.26, 0.27 (0.26)	2.7, 2.4 (2.5)	2016RES-FNF2456, PSM-16-02-11-14
Grace City, ND, United States, 2016 (Jerry) Soil: loam	2^ (10)	151 150	80 80	Foliar, 25 May, BBCH45	7	2.6, 2.3 (2.4)	0.063, 0.053 (0.058)	0.11, 0.12 (0.12)	2.7, 2.3 (2.5)	2016RES-FNF2456, PSM-16-02-11-06
Eldrigde, ND, United States, 2016 (Jerry) Soil: sandy loam	2 (10) +COC	152 156	100 101	Foliar, 19 May, BBCH34	7	4.2, 4.2 (4.2)	0.10, 0.10 (0.10)	0.29, 0.24 (0.27)	4.5, 4.5 (4.5)	2016RES-FNF2456, PSM-16-02-11-07
Montpelier, ND, United States, 2016 (Jerry) Soil: loam	2 (10) +NIS	153 148	100 100	Foliar, 19 May, BBCH33	7	2.4, 2.4 (2.4)	0.14, 0.13 (0.14)	0.38, 0.30 (0.34)	2.8, 2.7 (2.8)	2016RES-FNF2456, PSM-16-02-11-08
Groom, TX, United States, 2016 (TAM 112) Soil: clay loam	2^ (6)	151 150	70 69	Foliar, 12 April, BBCH33	8	3.2, 2.8 (3.2)	0.079, 0.083 (0.081)	0.18, 0.18 (0.18)	3.3, 3.0 (3.2)	2016RES-FNF2456, PSM-16-02-11-09
Claude, TX, United States, 2016 (TAM 112) Soil: clay	2 (6) +COC	149 149	70 70	Foliar, 6 April, BBCH35	0	22, 24 (23)	0.17, 0.19 (0.18)	0.21, 0.20 (0.21)	22, 24, (23)	2016RES-FNF2456, PSM-16-02-11-10
					3	15, 13 (14)	0.44, 0.38 (0.41)	0.35, 0.34 (0.34)	15, 13 (14)	

Location, year, WHEAT FORAGE (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
					7	8.4, 7.7 (8.0)	0.35, 0.35 (0.35)	0.36, 0.27 (0.31)	8.7, 8.0 (8.3)	
					11	2.5, 2.2 (2.4)	0.12, 0.12 (0.12)	0.27, 0.28 (0.27)	2.7, 2.5 (2.6)	
					14	1.4, 1.4 (1.4)	0.15, 0.12 (0.14)	0.26, 0.36 (0.26)	1.7, 1.7 (1.7)	
Tulelake, CA, United States, 2016 (Rojo) Soil: silt loam	2^ (8)	150 148	80 80	Foliar, 30 May, BBCH31	7	2.6, 1.9 (2.3)	0.11, 0.080 (0.10)	0.32, 0.18 (0.25)	2.9, 2.0 (2.6)	2016RES-FNF2456, PSM-16-02-11-15
Jerome, ID, United States, 2016 (Alturas) Soil: sandy loam	2 (9) +COC	151 149	82 84	Foliar, 9 June, BBCH 45	7	1.6, 1.4 (1.5)	0.026, 0.022 (0.024)	0.19, 0.24 (0.21)	1.8, 1.7 (1.8)	2016RES-FNF2456, PSM-16-02-11-16
Minto, MB, United States, 2016 (CDC Plentiful) Soil: sandy clay loam	2 (10) +NIS	150 147	94 94	Foliar, 9 June, BBCH12-21	7	0.16, 0.15 (0.16)	0.011, 0.011 (0.011)	0.28, 0.30 (0.29)	0.44, 0.45 (0.44)	2016RES-FNF2456, PSM-16-02-11-17
Alvena, SK, United States, 2016 (Carberry) Soil: loam	2^ (9)	157 151	94 93	Foliar, 7 July, BBCH59-61	7	0.57, 0.51 (0.54)	0.012, 0.012 (0.012)	0.099, 0.10 (0.10)	0.67, 0.61 (0.64)	2016RES-FNF2456, PSM-16-02-11-18
Fort Saskatchewan, AB, United States, 2016 (Plentiful) Soil: clay loam	2 (11) +COC	151 152	93 94	Foliar, 18 July, BBCH57	8	2.3, 2.5 (2.4)	0.036, 0.036 (0.036)	0.19, 0.16 (0.18)	2.5, 2.6 (2.6)	2016RES-FNF2456, PSM-16-02-11-19
Lamont, AB, United States, 2016 (Plentiful) Soil: sandy loam	2 (11) +NIS	150 152	93 94	Foliar, 18 July, BBCH55	7	2.2, 1.7 (1.9)	0.049, 0.037 (0.43)	0.23, 0.21 (0.22)	2.4, 1.9 (2.2)	2016RES-FNF2456, PSM-16-02-11-20

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Table 117 Residues of fluindapyr in wheat hay after foliar treatment with a suspension concentrate

Location, year, WHEAT HAY (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Weston, GA, United States, 2016 (LOE25) Soil: loamy sand	2 (10) +COC	149 150	80 83	Foliar, 21 Mar., BBCH33	14	6.2, 6.6 (6.4)	0.22, 0.23 (0.22)	0.42, 0.49 (0.46)	6.6, 7.1 (6.9)	2016RES-FNF2456, PSM-16-02-11-01
Richland, IA, United States, 2016 (Soft Red) Soil: silty clay loam	2 (9) +NIS	150 153	97 94	Foliar, 25 May, BBCH55	14	0.68, 0.66 (0.67)	0.039, 0.042 (0.040)	0.48, 0.48 (0.48)	1.2, 1.1 (1.2)	2016RES-FNF2456, PSM-16-02-11-02
Bagley, IA, United States, 2016 (Flint Hard Red) Soil: loam	2^ (8)	153 155	120 113	Foliar, 11 May, BBCH34	14	0.95, 0.66 (0.80)	0.025, 0.017 (0.021)	0.18, 0.16 (0.17)	2.4, 3.6 (3.0)	2016RES-FNF2456, PSM-16-02-11-03
Richland, IA, United States, 2016 (Rollagspring) Soil: silty clay loam	2 (10) +NIS	150 148	92 97	Foliar, 5 June, BBCH49	14	1.9, 3.0 (2.4)	0.10, 0.12 (0.11)	0.54, 0.64 (0.59)	1.1, 0.82 (0.98)	2016RES-FNF2456, PSM-16-02-11-11
New Providence, IA, United States, 2016 (Glenn) Soil: clay loam	2^ (11)	148 151	88 77	Foliar, 21 June, BBCH54	7	0.71, 1.3 (1.0)	0.030, 0.067 (0.048)	0.33, 0.46 (0.40)	1.0, 1.8 (1.4)	2016RES-FNF2456, PSM-16-02-11-12
					10	1.5, 1.3 (1.4)	0.078, 0.061 (0.069)	0.66, 0.53 (0.60)	2.1, 1.8 (2.0)	
					14	1.2, 1.1 (1.2)	0.10, 0.082 (0.092)	0.76, 0.75 (0.76)	2.0, 1.8 (1.9)	
					21	0.24, 0.28 (0.26)	0.025, 0.028 (0.026)	0.35, 0.34 (0.34)	0.60, 0.61 (0.60)	
					25	0.31, 0.30 (030)	0.033, 0.025 (0.029)	0.31, 0.22 (0.27)	0.62, 0.52 (0.57)	
Bradshaw, NE, United States, 2016 (Cert. Overland) Soil: silt loam	2 (9) +COC	148 152	69 72	Foliar, 13 April, BBCH29	15	0.70, 0.68 (0.69)	0.028, 0.029 (0.029)	1.2, 1.1 (1.2)	1.9, 1.8 (1.8)	2016RES-FNF2456, PSM-16-02-11-04 [SS]
Lebanon, OK, United States, 2016 (Tam 111) Soil: sandy loam	2 (10) +NIS	152 152	93 97	Foliar, 20 Mar., BBCH28	14	1.9, 1.7 (1.8)	0.089, 0.083, (0.086)	0.76, 0.65 (0.70)	2.7, 2.4 (2.5)	2016RES-FNF2456, PSM-16-02-11-05
Grace City, ND, United States,	2^ (10)	151 150	80 80	Foliar, 25 May,	14	2.3, 2.5 (2.4)	0.10, 0.12 (0.11)	0.24, 0.25	2.5, 2.7 (2.6)	2016RES-FNF2456,

Location, year, WHEAT HAY (variety)	Application					Residues (mean) (mg/kg) [a]				Reference, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	
2016 (Jerry) Soil: loam				BBCH45				(0.25)		PSM-16-02-11-06
Eldrigde, ND, United States, 2016 (Jerry) Soil: sandy loam	2 (10) +COC	152 156	100 101	Foliar, 19 May, BBCH34	14	5.1, 4.6 (4.8)	0.29, 0.28 (0.29)	2.0, 1.5 (1.8)	7.1, 6.1 (6.6)	2016RES-FNF2456, PSM-16-02-11-07
Montpelier, ND, United States, 2016 (Jerry) Soil: loam	2 (10) +NIS	153 148	100 100	Foliar, 19 May, BBCH33	14	1.9, 1.7 (1.8)	0.25, 0.23 (0.24)	0.45, 0.46 (0.46)	2.4, 2.1 (2.3)	2016RES-FNF2456, PSM-16-02-11-08
Cleveland, ND, United States, 2016 (Prosper) Soil: sandy clay loam	2 (7) +COC	153 149	106 107	Foliar, 27 June, BBCH36	15	1.6, 1.7 (1.6)	0.11, 0.11 (0.11)	1.2, 1.1 (1.2)	2.8, 2.8 (2.8)	2016RES-FNF2456, PSM-16-02-11-13
Montpelier, ND, United States, 2016 (Prosper) Soil: sandy loam	2 (8) +COC	147 150	107 106	Foliar, 28 June, BBCH36	14	0.60, 0.66 (0.63)	0.039, 0.039 (0.039)	0.41, 0.48 (0.45)	1.0, 1.1 (1.1)	2016RES-FNF2456, PSM-16-02-11-14
Groom, TX, United States, 2016 (TAM 112) Soil: clay loam	2^ (6)	151 150	70 69	Foliar, 12 April, BBCH33	15	0.97, 1.2 (1.1)	0.046, 0.075 (0.061)	0.16, 0.16 (0.16)	1.1, 1.3 (1.2)	2016RES-FNF2456, PSM-16-02-11-09
Claude, TX, United States, 2016 (TAM 112) Soil: clay	2 (6) +COC	149 149	70 70	Foliar, 6 April, BBCH35	7	16, 15 (15)	0.61, 0.58 (0.60)	2.6, 2.2 (2.4)	18, 17 (18)	2016RES-FNF2456, PSM-16-02-11-10
					11	5.4, 5.6 (5.5)	0.28, 0.30 (0.29)	2.0, 2.0 (2.0)	7.4, 7.6 (7.5)	
					14	2.7, 3.4 (3.1)	0.30, 0.32 (0.31)	1.4, 1.7 (1.5)	4.1, 5.1 (4.6)	
					21	0.78, 0.67 (0.73)	0.098, 0.084 (0.091)	<0.010, 0.20 (0.099)	0.79, 0.87 (0.82)	
					28	0.31, 0.58 (0.44)	0.047, 0.090 (0.069)	0.14, 0.19 (0.16)	0.44, 0.77 (0.61)	
Tulelake, CA, United States, 2016 (Rojo) Soil: silt loam	2^ (8)	150 148	80 80	Foliar, 30 May, BBCH31	14	0.74, 0.90 (0.82)	0.045, 0.063 (0.054)	0.34, 0.49 (0.42)	1.1, 1.4 (1.2)	2016RES-FNF2456, PSM-16-02-11-15
Jerome, ID, United States, 2016 (Alturas)	2 (9) +COC	151 149	82 84	Foliar, 9 June, BBCH 45	14	1.3, 1.3 (1.3)	0.044, 0.044 (0.044)	0.77, 0.74 (0.76)	2.1, 2.1 (2.1)	2016RES-FNF2456, PSM-16-02-11-16

Location, year, WHEAT HAY (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Soil: sandy loam										
Minto, MB, United States, 2016 (CDC Plentiful) Soil: sandy clay loam	2 (10) +NIS	150 147	94 94	Foliar, 9 June, BBCH12-21	14	0.067, 0.077 (0.072)	<0.010, <0.010 (<0.010)	0.89, 0.94 (0.92)	0.96, 1.0 (0.99)	2016RES-FNF2456, PSM-16-02-11-17
Alvena, SK, United States, 2016 (Carberry) Soil: loam	2^ (9)	157 151	94 93	Foliar, 7 July, BBCH59-61	14	0.62, 0.58 (0.60)	0.021, 0.019 (0.020)	1.9, <0.010 (0.97)	2.5, 0.59 (1.6)	2016RES-FNF2456, PSM-16-02-11-18
Fort Saskatchewan, AB, United States, 2016 (Plentiful) Soil: clay loam	2 (11) +COC	151 152	93 94	Foliar, 18 July, BBCH57	15	1.3, 1.3 (1.3)	0.048, 0.046 (0.047)	0.61, 0.60 (0.60)	1.9, 1.9 (1.9)	2016RES-FNF2456, PSM-16-02-11-19
Lamont, AB, United States, 2016 (Plentiful) Soil: sandy loam	2 (11) +NIS	150 152	93 94	Foliar, 18 July, BBCH55	15	0.85, 1.1 (0.98)	0.051, 0.062 (0.057)	0.21, 0.68 (0.44)	1.1, 1.8 (1.4)	2016RES-FNF2456, PSM-16-02-11-20

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[SS] Sample size of one of the duplicate samples was less than 0.5 kg (0.29 kg).

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Table 118 Residues of fluindapyr in wheat whole plants, ears and rest of plants after foliar treatment with an emulsion concentrate in Northern Europe

Location, year, WHEAT WHOLE PLANTS (variety)	Application				DALA (days)	Residues (mean) (mg/kg) [a]				Reference, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS		parent	3-OH-F	1-OH-Met-F	total [a]	
OX171AG, Edgecote, United Kingdom, 2015 (winter wheat sky fall) Soil: sand silt loam	2^ (13)	155 138	25 25	Foliar, 24 June, BBCH 69	0	2.9	0.027	0.32	3.2 [b]	2015RES-IFP1968, SPK-15-20471 GB02
					7	1.5	0.055	0.31	1.8 [c]	
					14	1.5	0.085	0.50	2.0 [d]	
					28	0.50	0.076	0.53	1.0 [e]	
Ears					*35*	*0.34*	*0.074*	*0.53*	*0.87 [f]*	
Rest of plants					*35*	*0.83*	*0.13*	*0.63*	*1.5 [g]*	
23847, Kastorf, DE, 2015	2^ (14)	140 145	25 25	Foliar, 15 June,	0	2.7	0.037	0.16	2.9	2015RES-IFP1968,
					7	1.1	0.033	0.22	1.3	

Location, year, WHEAT WHOLE PLANTS (variety)	Application				DALA (days)	Residues (mean) (mg/kg) [a]				Reference, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS		parent	3-OH-F	1-OH-Met-F	total [a]	
(winter wheat: Tobak) Soil: loamy				BBCH 69	14	0.92	0.049	0.24	1.1	SPK-15-20471 DE04
					28	0.61	0.044	0.23	0.84 [h]	
Ears					*35*	*0.098*	*0.015*	*0.15*	*0.25 [i]*	
Rest of plants					*35*	*0.52*	*0.046*	*0.45*	*0.97 [j]*	
I-44028, Poggio Renatico, I, 2015 (winter wheat: Solehio) Soil: loam	2 (9)	160 159	26 26	Foliar, 20 May, BBCH 69	0	3.448	0.054	0.305	3.8 [k]	2015RES-IFP1950, RA1508-3D
					7	1.370	0.070	0.570	2.0 [l]	
					14	1.959	0.165	0.781	2.8 [m]	
					28	2.726	0.253	2.377	5.1 [n]	
Ears					*35*	*1.040*	*0.204*	*0.435*	*1.4 [o]*	
Rest of plants					*35*	*2.071*	*0.345*	*1.787*	*3.9 [p]*	
F-33210, Saint Pierre de Mons, F, 2015 (winter wheat Solehio:) Soil: sandy loam	2 (15)	143 152	25 25	Foliar, 21 May, BBCH 73	0	5.190	0.059	0.312	5.5 [q]	2015RES-IFP1950, RA1508-4D
					7	3.913	0.302	0.378	4.3 [r]	
					14	3.817	0.302	0.492	4.3 [s]	
					28	3.690	0.237	1.295	5.0 [t]	
Ears					35	1.190	0.224	0.325	1.5 [u]	
Rest of plants					35	2.571	0.355	0.729	3.3 [v]	

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used.

[a] Residues are expressed as parent equivalents and total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

[b] 0.010 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[c] 0.019 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[d] 0.052 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[e] 0.072 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[f] 0.080 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[g] 0.064 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) and 0.011 mg/kg 1-COOH-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[h] 0.014 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[i] 0.011 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[j] 0.049 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) and 0.059 mg/kg 1-COOH-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[k] 0.017 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[l] 0.050 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) and 0.010 mg/kg 1-COOH-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[m] 0.073 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) and 0.023 mg/kg 1-COOH-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[n] 0.30 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) and 0.028 mg/kg 1-COOH-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[o] 0.25 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[p] 0.28 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[q] 0.043 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[r] 0.054 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[s] 0.083 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) and 0.010 mg/kg 1-COOH-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[t] 0.28 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) and 0.022 mg/kg 1-COOH-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[u] 0.15 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

[v] 0.18 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of isomers) and 0.012 mg/kg 1-COOH-fluindapyr (sum of isomers) was measured, but not included in the total calculation.

Table 119 Residues of fluindapyr in wheat straw after foliar treatment with a suspension concentrate (United States trials) or an emulsion concentrate (European trials)

Location, year, WHEAT STRAW (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [a]	Reference, Trial No.
Weston, GA, United States, 2016 (LOE25) Soil: loamy sand	2 (11) +COC	150 148	80 80	Foliar, 6 May, BBCH77	30	0.040, 0.29 (0.16)	0.010, 0.045 (0.027)	0.030, 0.073 (0.052)	0.070, 0.36 (0.22)	2016RES-FNF2456, PSM-16-02-11-01
Richland, IA, United States, 2016 (Soft Red) Soil: silty clay loam	2 (10) +NIS	150 149	99 101	Foliar, 11 June, BBCH77-80	30	2.6, 1.7 (2.1)	0.27, 0.20 (0.23)	0.32, 0.24 (0.28)	2.9, 1.9 (2.4)	2016RES-FNF2456, PSM-16-02-11-02
Bagley, IA, United States, 2016 (Flint Hard Red) Soil: loam	2^ (13)	147 152	120 118	Foliar, June 22, BBCH71	28	2.5, 2.8 (2.7)	0.29, 0.36 (0.32)	0.18, .25 (0.22)	2.7, 3.1 (2.9)	2016RES-FNF2456, PSM-16-02-11-03
Richland, IA, United States, 2016 (Rollagspring) Soil: silty clay loam	2 (9) +NIS	152 152	88 103	Foliar, 26 June, BBCH85-87	29	1.3, 1.5 (1.4)	0.18, 0.22 (0.20)	0.27, 0.32 (0.30)	1.6, 1.8 (1.7)	2016RES-FNF2456, PSM-16-02-11-11
New Providence, IA, United States, 2016 (Glenn) Soil: clay loam	2^ (11)	146 148	88 77	Foliar, 21 June, BBCH54	20	0.62, 0.41 (0.51)	0.068, 0.039 (0.053)	0.21, 0.18 (0.20)	0.82, 060 (0.71)	2016RES-FNF2456, PSM-16-02-11-12
					27	0.32, 0.34 (0.33)	0.32, 0.033 (0.032)	0.16, 0.098 (0.13)	0.48, 0.44 (0.46)	
					30	0.20, 0.35 (0.28)	0.027, 0.049 (0.038)	0.14, 0.18 (0.16)	0.35, 0.53 (0.44)	
					35	0.30, 0.39 (0.34)	0.052, 0.069 (0.060)	0.23, 0.26 (0.24)	0.53, 0.65 (0.59)	
					38	0.31, 0.32 (0.32)	0.052, 0.050 (0.051)	0.27, 0.21 (0.24)	0.58, 0.53 (0.55)	
Bradshaw, NE, United States, 2016 (Cert. Overland)	2 (14) +COC	152 146	74 71	Foliar, 14 June, BBCH61	30	3.1, 3.0 (3.0)	0.28, 0.29 (0.28)	0.18, 0.16 (0.17)	3.3, 3.1 (3.2)	2016RES-FNF2456, PSM-16-02-11-04

Location, year, WHEAT STRAW (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [a]	Reference, Trial No.
Soil: silt loam										
Lebanon, OK, United States, 2016 (Tam 111) Soil: sandy loam	2 (10) +NIS	151 151	99 99	Foliar, 11 May, BBCH74	30	1.3, 1.4 (1.4)	0.24, 0.27 (0.25)	0.45, 0.51 (0.49)	1.7, 1.9 (1.8)	2016RES-FNF2456, PSM-16-02-11-05
Grace City, ND, United States, 2016 (Jerry) Soil: loam	2^ (10)	149 150	80 82	Foliar, 15 July, BBCH83-85	30	1.8, 2.1 (2.0)	0.30, 0.31 (0.31)	0.15, 0.14 (0.14)	1.9, 2.2 (2.1)	2016RES-FNF2456, PSM-16-02-11-06
Eldrigde, ND, United States, 2016 (Jerry) Soil: sandy loam	2 (7) +COC	156 160	101 80	Foliar, 25 June, BBCH75	30	9.7, 10 (9.8)	0.67, 0.69 (0.68)	0.42, 0.38 (0.40)	10, 10 (10)	2016RES-FNF2456, PSM-16-02-11-07
Montpelier, ND, United States, 2016 (Jerry) Soil: loam	2 (7) +NIS	155 148	101 81	Foliar, 25 June, BBCH75	30	9.6, 9.8 (9.7)	1.7, 1.6 (1.6)	0.45, 0.52 (0.49)	10, 10 (10)	2016RES-FNF2456, PSM-16-02-11-08
Cleveland, ND, United States, 2016 (Prosper) Soil: sandy clay loam	2 (10) +COC	147 150	107 106	Foliar, 20 July, BBCH75	28	0.55, 0.54 (0.54)	0.076, 0.073 (0.074)	0.32, 0.25 (0.28)	0.86, 0.79 (0.83)	2016RES-FNF2456, PSM-16-02-11-13
Montpelier, ND, United States, 2016 (Prosper) Soil: sandy loam	2 (10) +COC	149 151	106 106	Foliar, 20 July, BBCH73	30	1.5, 0.97 (1.2)	0.14, 0.093 (0.12)	0.67, 0.50 (0.59)	2.2, 1.5 (1.8)	2016RES-FNF2456, PSM-16-02-11-14
Groom, TX, United States, 2016 (TAM 112) Soil: clay loam	2^ (10)	149 148	70 68	Foliar, 26 May, BBCH83	26	12, 11 (11)	0.89, 0.81 (0.85)	1.0, 0.27 (0.66)	13, 11 (12)	2016RES-FNF2456, PSM-16-02-11-09
Claude, TX, United States, 2016 (TAM 112) Soil: clay	2 (10) +COC	150 149	69 70	Foliar, 26 May, BBCH83	20	10, 11 (11)	1.2, 1.1 (1.2)	0.77, 0.28 (0.52)	11, 11 (11)	2016RES-FNF2456, PSM-16-02-11-10
					25	10, 10 (10)	1.5, 1.6 (1.6)	0.89, 0.26, (0.57)	11, 11 (11)	
					32	10, 9.3 (9.6)	1.6, 1.6 (1.6)	0.34, 0.34 (0.34)	10, 9.6 (10)	
					35	10, 6.2 (8.1)	1.7, 1.4 (1.5)	1.1, 0.82 (0.98)	11, 7.0 (9.0)	
					40	6.4, 9.4 (7.9)	1.4, 1.6 (1.5)	0.35, 0.28 (0.32)	6.7, 9.7 (8.2)	
Tulelake, CA, United States,	2^ (10)	149 149	80 116	Foliar, 1 July,	31	1.8, 1.9 (1.9)	0.049, 0.050	0.052, 0.062	1.9, 2.0 (1.9)	2016RES-FNF2456,

Location, year, WHEAT STRAW (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [a]	Reference, Trial No.
2016 (Rojo) Soil: silt loam				BBCH75			(0.050)	(0.057)		PSM-16-02-11-15
Jerome, ID, United States, 2016 (Alturas) Soil: sandy loam	2 (11) +COC	151 150	72 80	Foliar, 11 July, BBCH85	29	2.7, 2.9 (2.8)	0.28, 0.28 (0.28)	0.21, 0.24 (0.28)	2.9, 3.2 (3.1)	2016RES-FNF2456, PSM-16-02-11-16
Minto, MB, United States, 2016 (CDC Plentiful) Soil: sandy clay loam	2 (11) +NIS	150 148	94 94	Foliar, 25 July, BBCH77-83	30	0.31, 0.33 (0.32)	0.029, 0.035 (0.032)	1.8, 1.7 (1.8)	2.1, 2.0 (2.1)	2016RES-FNF2456, PSM-16-02-11-17
Alvena, SK, United States, 2016 (Carberry) Soil: loam	2^ (11)	155 149	94 94	Foliar, 16 Aug., BBCH85-87	29	0.60, 0.48 (0.54)	0.053, 0.036 (0.044)	0.074, 0.059 (0.067)	0.68, 0.54 (0.61)	2016RES-FNF2456, PSM-16-02-11-18
Fort Saskatchewan, AB, United States, 2016 (Plentiful) Soil: clay loam	2 (11) +COC	151 151	94 94	Foliar, 2 Aug., BBCH69	30	0.41, 0.41 (0.41)	0.027, 0.031 (0.029)	0.24, 0.24 (0.24)	0.64, 0.65 (0.65)	2016RES-FNF2456, PSM-16-02-11-19
Lamont, AB, United States, 2016 (Plentiful) Soil: sandy loam	2 (12) +NIS	152 157	94 96	Foliar, 10 Aug, BBCH73	30	0.81, 0.78 (0.79)	0.11, 0.10 (0.10)	0.35, 0.23 (0.30)	1.1, 1.0 (1.1)	2016RES-FNF2456, PSM-16-02-11-20
OX12BNJ, South Fawley, United Kingdom, 2015 (winter wheat TRZAW) Soil: clay	2^ (13)	160 162	25 25	Foliar, 25 June, BBCH 69	48	2.3	0.40	0.97	3.3 [c]	SPK-15-20471, SPK-15-20471 GB01
OX171AG, Edgecote, United Kingdom, 2015 (winter wheat sky fall) Soil: sand silt loam	2^ (13)	155 138	25 25	Foliar, 24 June, BBCH 69	72	1.2	0.34	0.78	2.0 [c]	SPK-15-20471, SPK-15-20471 GB02
74572, Blaufelden-Mittelbach, DE, 2015 (winter wheat: Colonia) Soil: clay loam	2^ (15)	153 153	25 25	Foliar, 25 June, BBCH 69	40	4.2	0.50	1.4	5.6 [c]	SPK-15-20471, SPK-15-20471 DE03
23847, Kastorf,	2^	140	25	Foliar,	56	0.71	0.12	0.97	1.7 [c]	SPK-15-

Location, year, WHEAT STRAW (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	Total [a]	Reference, Trial No.
DE, 2015 (winter wheat: Tobak) Soil: loamy	(14)	145	25	15 June, BBCH 69						20471, SPK-15-20471 DE04
I-44012, Gavello, I, 2015 (winter wheat:50207) Soil: loam	2 (9)	159 159	26 26	Foliar, 20 May, BBCH 69	41	2.0	0.49	1.4	3.4 [c]	2015RES-IFP1950, RA1508-1H-P
F-4590, Marsillargues, F, 2015 (winter wheat:Arezzo) Soil: silty clay loam	2 (15)	152 153	25 25	Foliar, 21 May, BBCH 69	47	8.1	0.54	3.3	11 [c]	2015RES-IFP1950, RA1508-2H-P
I-44028, Poggio Renatico, I, 2015 (winter wheat: Solehio) Soil: loam	2 (9)	160 159	26 26	Foliar, 20 May, BBCH 69	42	2.5	0.47	3.0	5.5 [c]	2015RES-IFP1950, RA1508-3D
F-33210, Saint Pierre de Mons, F, 2015 (winter wheat Solehio:) Soil: sandy loam	2 (15)	143 152	25 25	Foliar, 21 May, BBCH 73	40	2.2	0.35	0.69	2.9 [c]	2015RES-IFP1950, RA1508-4D

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents and total residues represent the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

[b] Also levels of 0.11 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) and 0.025 mg/kg 1-COOH-fluindapyr (sum of diastereomers) were recorded, but not included in the total residue calculations.

[c] Also levels of 0.16 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) and 0.030 mg/kg 1-COOH-fluindapyr (sum of diastereomers) were recorded, but not included in the total residue calculations.

[d] Also levels of 0.12 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) and 0.023 mg/kg 1-COOH-fluindapyr (sum of diastereomers) were recorded, but not included in the total residue calculations.

[e] Also levels of 0.15 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) and 0.059 mg/kg 1-COOH-fluindapyr (sum of diastereomers) were recorded, but not included in the total residue calculations.

[f] Also levels of 0.13 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) and 0.016 mg/kg 1-COOH-fluindapyr (sum of diastereomers) were recorded, but not included in the total residue calculations.

[g] Also levels of 0.44 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) and 0.043 mg/kg 1-COOH-fluindapyr (sum of diastereomers) were recorded, but not included in the total residue calculations.

[h] Also levels of 0.39 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) and 0.042 mg/kg 1-COOH-fluindapyr (sum of diastereomers) were recorded, but not included in the total residue calculations.

[i] Also levels of 0.24 mg/kg 1-OH-Met-N-desMet-fluindapyr (sum of diastereomers) and 0.018 mg/kg 1-COOH-fluindapyr (sum of diastereomers) were recorded, but not included in the total residue calculations.

Sorghum forage and stover

Supervised residue trials were conducted in the United States and Canada in 2015 (3) and 2016 (6 in United States only) to measure the magnitude of fluindapyr residues in/on sorghum agricultural commodities following two foliar applications of a fluindapyr SC formulation [Webber, 2018d, 2015RES-FNF1901 and Webber, 2018e, 2016RES-FNF2455]. The details of the study are described in the section n food commodities (sorghum grain).

The sorghum forage samples were harvested at 6–7 days after the last application and stover samples were harvested 45 days after the last application (2015 trials) and 28–30 days (2016 trials). At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 12 separate areas within the plots so that each sample yielded a minimum of 1 kilogram of forage and 0.5 kilogram of stover. Decline samples were collected at two trial locations. At these locations samples were collected at 3, 7, 10 and 13/14 days after last application for forage and 30/31, 34/35, 45, 54/55 and 60/61 days for stover.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone1-OH-Met-fluindapyr.

The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from the LOQ (0.01 mg/kg) to 16.0 mg/kg for forage and 2.0 mg/kg for stover.

Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum forage were 102 ±16 percent (n = 6), 101 ± 11 percent (n = 5) and 98 ± 9 percent (n = 5), respectively [Webber, 2018d, 2015RES-FNF1901]. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum forage were 99 ± 18 percent (n = 8), 96 ± 17 percent (n = 7) and 100 ± 13 percent (n = 7), respectively [Webber, 2018e, 2016RES-FNF2455].

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum stover were 109 ± 13 percent (n = 6), 93 ± 10 percent (n = 6) and 80 ± 9 percent (n = 5), respectively [Webber, 2018d, 2015RES-FNF1901]. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum stover were 108 ± 11 percent (n = 8), 99 ± 11 percent (n = 8) and 82 ± 6 percent (n = 7), respectively [Webber, 2018e, 2016RES-FNF2455].

The results on sorghum forage and sorghum stover are summarized in Table 120 and Table 121.

Table 120 Residues of fluindapyr in sorghum forage after foliar treatment with a suspension concentrate

Location,	Application		Residues (mean) (mg/kg) [a]	

year, SORGHUM FORAGE (variety)	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Bradshaw, NE, United States, 2015 (DKS37-07) Soil: silt loam	2 (11) +NIS	159 151	69 70	Foliar, 11 Sept., 2015 BBCH 77	6	0.39, 0.45 (0.42)	0.012, 0.011 (0.012)	0.034, 0.042 (0.038)	0.43, 0.50 (0.46)	2015RES-FNF1901, PSM-15-02-04-01
Groom, TX, United States, 2015 (H-390W) Soil: silty clay loam	2^ (9)	152 150	68 68	Foliar, 25 Sept., BBCH 83	0	3.2, 4.0 (3.6)	0.026, 0.034 (0.030)	0.034, 0.032 (0.033)	3.2, 4.0 (3.6)	2015RES-FNF1901, PSM-15-02-04-02
					3	5.1, 6.1 (5.6)	0.066, 0.064 (0.065)	0.048, 0.038 (0.043)	5.2, 6.1 (5.6)	
					7	5.1, 5.0 (5.0)	0.10, 0.10 (0.010)	0.064, 0.070 (0.067)	5.2, 5.1 (5.1)	
					10	1.7, 2.2 (2.0)	0.041, 0.049 (0.045)	0.076, 0.074 (0.075)	1.8, 2.3 (2.0)	
					14	1.7, 1.1 (1.4)	0.088, 0.046 (0.067)	0.12, 0.090 (0.10)	1.8, 1.2 (1.5)	
Lebanon, OK, United States, 2015 (H390W) Soil: sandy loam	2^ (10)	151 149	63 64	Foliar, 2 Sept., BBCH 79	0	10, 12 (11)	0.15, 0.15 (0.15)	0.13, 0.15 (0.14)	10, 12 (11)	2015RES-FNF1901, PSM-15-02-04-03
					3	4.6, 6.0 (5.3)	0.12, 0.12 (0.12)	0.23, 0.25 (0.24)	4.8, 6.2 (5.5)	
					7	4.0, 5.0 (4.5)	0.15, 0.19 (0.17)	0.15, 0.20 (0.18)	4.2, 5.2 (4.7)	
					10	3.4, 2.9 (3.1)	0.16, 0.15 (0.16)	0.18, 0.18 (0.18)	3.6, 3.0 (3.3)	
					13	0.65, 0.82 (0.74)	0.054, 0.050 (0.052)	0.18, 0.19 (0.19)	0.84, 1.0 (0.93)	
Lebanon, OK, United States, 2016 (H-390W) Soil: sandy loam	2 (10) +COC	148 152	108 97	Foliar, 26 Aug., BBCH 68	7	2.6, 2.2 (2.4)	0.044, 0.036 (0.040)	0.30, 0.36 (0.33)	2.9, 2.6 (2.8)	2016RES-FNF2455, PSM-16-02-10-04
Fisk, MO, United States, 2016 (M383C) Soil: sand	2^ (12)	149 153	80 80	Foliar, 16 Aug., BBCH 83	7	0.25, 0.24 (0.24)	0.010, <0.010 (<0.010)	0.17, 0.21 (0.19)	0.41, 0.44 (0.43)	2016RES-FNF2455, PSM-16-02-10-01
Richland, IA, United States, 2016 (AG1401) Soil: silty clay loam	2^ (9)	150 150	82 79	Foliar, 2 Sept., BBCH 74-75	7	0.37, 0.38 (0.38)	<0.010, <0.010 (<0.01)	0.14, 0.12 (0.13)	0.51, 0.51 (0.51)	2016RES-FNF2455, PSM-16-02-10-02
York, NE, United States, 2016 (DKS37-07) Soil:silt loam	2 (10) +COC	155 153	93 92	Foliar, 12 Sept., BBCH 87	7	0.38, 0.72 (0.55)	0.011, 0.014 (0.012)	0.090, 0.13 (0.11)	0.47, 0.85 (0.66)	2016RES-FNF2455, PSM-16-02-10-03

Location, year, SORGHUM FORAGE (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Cleveland, ND, United States, 2016 (Sweetie) Soil: sandy clay loam	2 (10) +COC	150 159	108 107	Foliar, 5 Sept., BBCH 83	6	1.3, 1.3 (1.3)	0.017, 0.016 (0.016)	0.13, 0.089 (0.11)	1.5, 1.4 (1.4)	2016RES-FNF2455, PSM-16-02-10-05
Claude, TX, United States, 2016 (Y373) Soil: clay	2 (10) +NIS	150 156	66 67	Foliar, 3 Sept., BBCH 80	7	0.38, 0.85 (0.62)	<0.010, 0.013 (0.011)	0.086, 0.11 (0.099)	0.47, 0.96 (0.71)	2016RES-FNF2455, PSM-16-02-10-06

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Table 121 Residues of fluindapyr in sorghum stover after foliar treatment with a suspension concentrate

Location, year, SORGHUM STOVER (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Bradshaw, NE, United States, 2015 (DKS37-07) Soil: silt loam	2 (11) +NIS	159 151	69 70	Foliar, 11 Sept., 2015 BBCH 77	45	0.046, 0.059 (0.052)	<0.010, <0.010 (<0.010)	0.033, 0.023 (0.028)	0.079, 0.082 (0.080)	2015RES-FNF1901, PSM-15-02-04-01
Groom, TX, United States, 2015 (H-390W) Soil: silty clay loam	2^ (9)	152 150	68 68	Foliar, 25 Sept., BBCH 83	31	0.74, 0.38 (0.56)	0.057, 0.041 (0.049)	0.090, 0.088 (0.089)	0.83, 0.46 (0.65)	2015RES-FNF1901, PSM-15-02-04-02
					34	0.75, 0.92 (0.83)	0.064, 0.076 (0.070)	0.11, 0.13 (0.12)	0.85, 1.1 (0.95)	
					45	0.75, 0.61 (0.68)	0.088, 0.078 (0.083)	0.12, 0.12 (0.12)	0.87, 0.73 (0.80)	
					55	0.53, 0.54 (0.53)	0.074, 0.080 (0.077)	0.091, 0.099 (0.095)	0.62, 0.63 (0.63)	
					60	0.62, 0.50 (0.56)	0.098, 0.079 (0.089)	0.10, 0.094 (0.099)	0.72, 0.59 (0.66)	
Lebanon, OK, United States, 2015 (H390W)	2^ (10)	151 149	63 64	Foliar, 2 Sept., BBCH 79	30	1.7, 0.51 (1.1)	0.26, 0.091 (0.18)	0.74, 0.75 (0.75)	2.4, 1.3 (1.8)	2015RES-FNF1901, PSM-15-02-04-03
					35	0.32, 0.085	0.052, 0.010	0.17, 0.048	0.49, 0.13	

Location, year, SORGHUM STOVER (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Soil: sandy loam						(0.20)	(0.031)	(0.11)	(0.31)	
					45	0.21, 0.32 (0.26)	0.038, 0.055 (0.046)	0.21, 0.23 (0.22)	0.42, 0.55 (0.49)	
					54	0.32, 0.38 (0.35)	0.052, 0.066 (0.059)	0.28, 0.27 (0.28)	0.60, 0.65 (0.62)	
					61	0.079, 0.073 (0.076)	0.017, 0.021 (0.019)	0.088, 0.20 (0.15)	0.17, 0.28 (0.22)	
Lebanon, OK, United States, 2016 (H-390W) Soil: sandy loam	2 (10) +COC	148 152	108 97	Foliar, 26 Aug., BBCH 68	29	0.13, 0.14 (0.14)	0.016, 0.019 (0.018)	0.28, 0.35 (0.32)	0.41, 0.49 (0.45)	2016RES-FNF2455, PSM-16-02-10-04
Fisk, MO, United States, 2016 (M383C) Soil: sand	2^ (12)	149 153	80 80	Foliar, 16 Aug., BBCH 83	29	0.22, 0.19 (0.21)	0.017, 0.016 (0.016)	0.11, 0.12 (0.12)	0.33, 0.31 (0.32)	2016RES-FNF2455, PSM-16-02-10-01
Richland, IA, United States, 2016 (AG1401) Soil: silty clay loam	2^ (9)	150 150	82 79	Foliar, 2 Sept., BBCH 74-75	28	0.17, 0.19 (0.18)	0.028, 0.040 (0.034)	0.13, 0.18 (0.16)	0.30, 0.38 (0.34)	2016RES-FNF2455, PSM-16-02-10-02
York, NE, United States, 2016 (DKS37-07) Soil:silt loam	2 (10) +COC	155 153	93 92	Foliar, 12 Sept., BBCH 87	29	0.21, 0.11 (0.16)	<0.010, <0.010 (<0.010)	0.033, 0.029 (0.031)	0.24, 0.14 (0.19)	2016RES-FNF2455, PSM-16-02-10-03
Cleveland, ND, United States, 2016 (Sweetie) Soil: sandy clay loam	2 (10) +COC	150 159	108 107	Foliar, 5 Sept., BBCH 83	29	0.29, 0.18 (0.23)	0.026, 0.017 (0.022)	0.051, 0.046 (0.048)	0.34, 0.22 (0.28)	2016RES-FNF2455, PSM-16-02-10-05
Claude, TX, United States, 2016 (Y373) Soil: clay	2 (10) +NIS	150 156	66 67	Foliar, 3 Sept., BBCH 80	30	0.39, 0.49 (0.44)	0.049, 0.070 (0.060)	0.17, 0.19 (0.18)	0.56, 0.68 (0.62)	2016RES-FNF2455, PSM-16-02-10-06

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Maize forage and stover

Two studies with three [Webber, 2018a, 2015RES-FNF1900] and 18 [Webber, 2018b, 2016RES-FNF2453] field trials were conducted in the United States in 2015 and 2016 to measure the magnitude of fluindapyr residues in/on field corn raw agricultural commodities following two foliar applications of a fluindapyr SC formulation. More details of the study are described in the section on food commodities (maize cereals).

The field corn (forage and stover) samples were harvested at proper times to yield commercially representative samples. At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 12 separate areas within the plots so that each sample yielded a minimum of 1 kilogram of forage and 0.5 kilogram of stover. The field corn raw agricultural commodity of forage was harvested at 6-7 DALA. The field corn raw agricultural commodity stover was harvested at 45 DALA.

Decline samples were collected at two trial locations (PSM-15-02-03, trial 02 and 03). At these locations samples were collected at a target of 0, 3, 7, 10, and 14 days after last application for forage and 30, 35, 45, 55, and 60 days after last application for stover.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone1-OH-Met-fluindapyr.

The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from the LOQ (0.01 mg/kg) to 15.5 mg/kg for forage, and 2.0 mg/kg for stover.

Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance and the overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from field corn forage were 86 ± 11 percent (n = 6), 94 ± 10 percent (n = 4) and 77 ± 5 percent (n = 5), respectively [Webber, 2018a, 2015RES-FNF1900] and 101 ± 5 percent (n = 7), 101 ± 6 percent (n = 6) and 79 ± 8 percent (n = 7), respectively in the second study [Webber, 2018b, 2016RES-FNF2453].

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from field corn stover were 110 ± 8 percent (n = 6), 90 ± 4 percent (n = 6) and 78 ± 6 percent (n = 5), respectively [Webber, 2018a, 2015RES-FNF1900] and 99 ± 13 percent (n = 10), 94 ± 7 percent (n = 10) and 83 ± 12 percent (n = 9), respectively in the second study [Webber, 2018b, 2016RES-FNF2453].

The results on field corn forage and field corn stover are summarized in Table 122 and Table 123.

Table 122 Residues of fluindapyr in field corn forage after foliar treatment with a suspension concentrate

Location, year, MAIZE/ FIELD CORN FORAGE (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Farlin, IA, United States, 2015	2^ (11)	152 156	63 65	Backpack sprayer,	6	1.5, 1.4 (1.5)	0.025, 0.024	0.044, 0.066	1.6, 1.5 (1.5)	2015RES-FNF1900,

Location, year, MAIZE/ FIELD CORN FORAGE (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
(P1248) Soil: clay loam				29 Aug., BBCH75			(0.024)	(0.055)		PSM-15-02-03-01
Perry, IA, United States, 2015 (2F721) Soil: loam	2^ (11)	152 153	76 67	Backpack sprayer, 10 Aug., BBCH74	0	3.0, 2.9 (3.0)	0.020, 0.018 (0.019)	0.092, 0.059 (0.075)	3.2, 3.0 (3.1)	2015RES-FNF1900, PSM-15-02-03-02
					3	1.4, 2.1 (1.8)	0.023, 0.030 (0.027)	0.057, 0.10 (0.081)	1.4, 2.2 (1.8)	
					7	0.34, 0.63 (0.48)	0.013, 0.016 (0.014)	0.073, 0.086 (0.080)	0.41, 0.72 (0.56)	
					9	0.23, 0.33 (0.28)	0.013, 0.020 (0.017)	0.090, 0.13 (0.11)	0.32, 0.45 (0.39)	
					14	0.21, 0.28 (0.25)	0.015, 0.021 (0.018)	0.097, 0.13 (0.11)	0.30, 0.41 (0.36)	
Hedrick, IA, United States, 2016 (P1311AMXT) Soil: silty clay loam	2^ (10)	152 152	88 92	Backpack Sprayer, 26 Aug., BBCH85-87	7	0.60, 0.55 (0.57)	0.019, 0.018 (0.018)	0.067, 0.061 (0.064)	0.67, 0.61 (0.64)	2016RES-FNF2453, PSM-16-02-08-03
Richland, IA, United States, 2016 (PO937AM) Soil: silty clay loam	2^ (10)	152 152	78 92	Tractor mounted boom, 26 Aug., BBCH85	7	0.91, 0.88 (0.90)	0.026, 0.029 (0.028)	0.068, 0.078 (0.073)	0.98, 0.96 (0.97)	2016RES-FNF2453, PSM-16-02-08-04
Lime Springs, IA, United States, 2016 (DKC46-37RIB) Soil: sandy loam	2 (11) +COC	149 148	106 106	Backpack Sprayer, 12 Sept., BBCH87	7	0.36, 0.36 (0.36)	<0.010, <0.010 (<0.010)	0.096, 0.11 (0.10)	0.45, 0.47 (0.46)	2016RES-FNF2453, PSM-16-02-08-12
Cresco, IA, United States, 2016 (P9929AM) Soil: silt loam	2 (11) +NIS	149 150	106 107	Backpack Sprayer, 12 Sept., BBCH87	7	0.16, 0.18 (0.18)	<0.010, <0.010 (<0.010)	0.037, 0.037 (0.037)	0.20, 0.22 (0.21)	2016RES-FNF2453, PSM-16-02-08-13
Bagley, IA, United States, 2016 (9732RR) Soil: clay loam	2 (10) +COC	150 145	93 91	Backpack Sprayer, 19Aug., BBCH85	7	0.24, 0.25 (0.25)	<0.010, 0.011 (0.010)	0.12, 0.16 (0.14)	0.36, 0.41 (0.39)	2016RES-FNF2453, PSM-16-02-08-08
Seven Springs, NC, United States, 2015 (DKC68-03) Soil: loamy sand	2 (10) +NIS	167 169	70 71	Tractor mounted sprayer, 24 July, BBCH79	0	2.8, 2.8 (2.8)	0.015, 0.016 (0.016)	0.18, 0.20 (0.19)	3.0, 3.1 (3.0)	2015RES-FNF1900, PSM-15-02-03-03
					3	3.4, 2.5 (2.9)	0.053, 0.046 (0.050)	0.37, 0.28 (0.33)	3.8, 2.5 (3.3)	
					7	2.6, 2.3	0.068,	0.38, 0.38	3.0, 2.6	

Location, year, MAIZE/ FIELD CORN FORAGE (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
						(2.4)	0.051 (0.059)	(0.38)	(2.8)	
					10	2.9, 2.5 (2.7)	0.099, 0.088 (0.093)	0.28, 0.098 (0.19)	3.2, 2.6 (2.9)	
					13	9.2, 6.1 (7.6)	0.087, 0.071 (0.079)	0.53, 0.54 (0.53)	9.8, 6.6 (8.2)	
Germansville, PA, United States, 2016 (TA545-33EZ) Soil: loam	2^ (10)	152 159	106 107	Backpack Sprayer, 08 Sept., R5	6	1.7, 1.2 (1.4)	0.040, 0.031 (0.036)	0.061, 0.061 (0.061)	1.7, 1.2 (1.5)	2016RES-FNF2453, PSM-16-02-08-01
Wyoming, IL, United States, 2016 (DKC 61-86) Soil: clay loam	2^ (10)	151 148	118 111	Backpack Sprayer, 22 Sept, BBCH87	7	1.8, 1.8 (1.8)	0.030, 0.025 (0.027)	0.062, 0.057 (0.060)	1.9, 1.9 (1.9)	2016RES-FNF2453, PSM-16-02-08-02
Carlyle, IL, United States, 2016 (Syngenta N78S-3111) Soil: silt loam	2^ (10)	151 150	86 79	Tractor mounted boom, 12 Sept., BBCH85	7	1.0, 0.92 (0.96)	0.041, 0.033 (0.037)	0.11, 0.15 (0.13)	1.1, 1.1 (1.1)	2016RES-FNF2453, PSM-16-02-08-05
Geneva, MN, United States, 2016 (NuTech 5D-196AMX) Soil: sandy clay loam	2 (10) +COC	152 151	88 79	Backpack Sprayer, 12 Sept, BBCH85-87	7	0.84, 0.62 (0.73)	0.025, 0.018 (0.021)	0.15, 0.13 (0.14)	0.98, 0.75 (0.86)	2016RES-FNF2453, PSM-16-02-08-06
Ellendale, MN, United States, 2016 (Dekalb DKC44-13RIB) Soil:sandy loam	2 (9) +COC	149 152	86 80	Backpack Sprayer, 15 Sept, BBCH85-87	7	0.24, 0.23 (0.23)	<0.010, <0.010 (<0.010)	0.045, 0.034 (0.039)	0.28, 0.26 (0.27)	2016RES-FNF2453, PSM-16-02-08-07
Paynesville, MN, United States, 2016 (NK 23MGTA) Soil: sandy loam	2 (10) +COC	152 148	97 94	Tractor mounted boom, 30 Aug., BBCH85	7	0.10, 0.050 (0.077)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.11, 0.060 (0.087)	2016RES-FNF2453, PSM-16-02-08-09
York, NE, United States, 2016 (DKC62-77RIB) Soil: silt loam	2 (10) +COC	145 150	93 93	Backpack Sprayer, 12 Sept., BBCH87	7	0.90, 0.69 (0.79)	0.020, 0.016 (0.018)	0.18, 0.11 (0.15)	1.1, 0.80 (0.94)	2016RES-FNF2453, PSM-16-02-08-10
Brunswick, NE, United States, 2016 (DKC62-	2 (11) +COC	152 151	91 94	Backpack Sprayer, 12 Sept., BBCH87	6	0.50, 0.38 (0.44)	0.013, 0.013 (0.013)	0.16, 0.14 (0.15)	0.66, 0.52 (0.59)	2016RES-FNF2453, PSM-16-02-08-11

Location, year, MAIZE/ FIELD CORN FORAGE (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
77RIB) Soil: sand										
Brookings, SD, United States, 2016 (DKC 44-13) Soil: loam	2 (10) +NIS	151 151	103 92	Backpack Sprayer, 29 Sept., BBCH85	8	0.92, 0.63 (0.77)	0.021, 0.016 (0.018)	0.067, 0.050 (0.058)	0.98, 0.68 (0.83)	2016RES-FNF2453, PSM-16-02-08-14
Deerfield, MI, United States, 2016 (AgriGold A6472VT3P RIB) Soil: sandy clay loam	2 (10) +NIS	150 149	80 81	Backpack Sprayer, 20 Aug., BBCH73	7	0.30, 0.35 (0.33)	0.015, 0.020 (0.018)	0.022, 0.026 (0.024)	0.32, 0.37 (0.35)	2016RES-FNF2453, PSM-16-02-08-15
Milan, MI, United States, 2016 (Dekalb DKC60-67 Genssrib) Soil: clay loam	2 (10) +NIS	150 152	80 73	Backpack Sprayer, 20Aug., BBCH73	7	0.36, 0.54 (0.45)	0.020, 0.019 (0.019)	0.058, 0.051 (0.055)	0.42, 0.59 (0.51)	2016RES-FNF2453, PSM-16-02-08-16
Cleveland, ND, United States, 2016 (01049135) Soil: sandy	2 (10) +COC	151 160	107 107	Tractor mounted boom, 05 Sept., BBCH83	6	1.3, 1.3 (1.3)	0.022, 0.020 (0.021)	0.049, 0.050 (0.050)	1.4, 1.4 (1.4)	2016RES-FNF2453, PSM-16-02-08-17
Claude, TX, United States, 2016 (P1234AM) Soil: clay loam	2 (10) +NIS	149 153	67 66	Backpack Sprayer, 03 Sept., BBCH84	7	2. 3, 1.9 (2.1)	0.032, 0.032 (0.032)	0.15, 0.14 (0.15)	2.5, 2.1 (2.3)	2016RES-FNF2453, PSM-16-02-08-18

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Table 123 Residues of fluindapyr in field corn stover after foliar treatment with a suspension concentrate

Location, year, MAIZE/ FIELD CORN STOVER (variety)	Application					Mean residues (individual values) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Farlin, IA, United States, 2015 (P1248) Soil: clay loam	2^ (11)	152 156	63 65	Backpack sprayer, 29 Aug., BBCH75	45	0.14, 0.14 (0.14)	0.013 (0.013, 0.013)	0.13, 0.11 (0.12)	0.27, 0.25 (0.26)	2015RES-FNF1900, PSM-15-02-03-01
Perry, IA,	2^	152	76	Backpack	30	0.36 ,	0.037,	0.30,	0.66,	2015RES-

Location, year, MAIZE/ FIELD CORN STOVER (variety)	Application					Mean residues (individual values) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
United States, 2015 (2F721) Soil: loam	(11)	153	67	sprayer, 10 Aug., BBCH74		0.35 (0.36)	0.037 (0.037)	0.27 (0.28)	0.62 (0.64)	FNF1900, PSM-15-02-03-02 (loam)
					35	0.32, 0.35 (0.34)	0.034, 0.038 (0.036)	0.30, 0.30 (0.30)	0.62, 0.65 (0.64)	
					45	0.29, 0.045 (0.17)	0.030, <0.010 (0.017)	0.18, 0.021 (0.10)	0.48, 0.066 (0.27)	
					56	0.36, 0.25 (0.30)	0.036, 0.023 (0.029)	0.21, 0.20 (0.21)	0.57, 0.45 (0.51)	
					60	0.55, 0.17 (0.36)	0.036, 0.023 (0.024)	0.14, 0.088 (0.11)	0.69, 0.26 (0.48)	
Hedrick, IA, United States, 2016 (P1311AMXT) Soil: silty clay loam	2^ (10)	152 152	88 92	Backpack Sprayer, 26 Aug., BBCH85-87	29	1.1, 1.1 (1.1)	0.065, 0.080 (0.073)	0.27, 0.27 (0.27)	1.4, 1.4 (1.4)	2016RES-FNF2453, PSM-16-02-08-03
Richland, IA, United States, 2016 (PO937AM) Soil: silty clay loam	2^ (10)	152 152	78 92	Tractor mounted boom, 26 Aug., BBCH85	29	1.6, 1.2 (1.4)	0.12, 0.095 (0.11)	0.38, 0.36 (0.37)	2.0, 1.6 (1.8)	2016RES-FNF2453, PSM-16-02-08-04
Bagley, IA, United States, 2016 (9732RR) Soil: clay loam	2 (10) +COC	150 145	93 91	Backpack Sprayer, 19Aug., BBCH85	30	0.40, 0.28 (0.34)	0.034, 0.025 (0.030)	0.56, 0.42 (0.49)	0.96, 0.70 (0.83)	2016RES-FNF2453, PSM-16-02-08-08
Lime Springs, IA, United States, 2016 (DKC46-37RIB) Soil: sandy loam	2 (11) +COC	149 148	106 106	Backpack Sprayer, 12 Sept., BBCH87	31	0.51, 0.58 (0.55)	0.022, 0.028 (0.025)	0.11, 0.12 (0.12)	0.62, 0.71 (0.66)	2016RES-FNF2453, PSM-16-02-08-12 (corn)
Cresco, IA, United States, 2016 (P9929AM) Soil: silt loam	2 (11) +NIS	149 150	106 107	Backpack Sprayer, 12 Sept., BBCH87	28	0.47, 0.61 (0.54)	0.027, 0.032 (0.029)	0.074, 0.063 (0.069)	0.55, 0.67 (0.61)	2016RES-FNF2453, PSM-16-02-08-13 (corn)
Seven Springs, NC, United States, 2015 (DKC68-03) Soil: loamy sand	2 (10) +NIS	167 169	70 71	Tractor mounted sprayer, 24 July, BBCH79	31	1.4, 1.7 (1.5)	0.13, 0.13 (0.13)	1.2, 1.2 (1.2)	2.6, 28 (2.7)	2015RES-FNF1900, PSM-15-02-03-03
					35	1.5, 1.9 (1.7)	0.14, 0.16 (0.15)	1.3, 0.74 (1.0)	2.8, 2.6 (2.7)	
					45	1.1, 0.87 (0.97)	0.11, 0.082 (0.096)	0.77, 0.55 (0.66)	1.8, 1.4 (1.6)	

Location, year, MAIZE/ FIELD CORN STOVER (variety)	Application					Mean residues (individual values) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
					55	0.74, 1.2 (0.97)	0.061, 0.13 (0.094)	0.37, 0.39 (0.38)	1.1, 1.6 (1.3)	
					59	1.3, 1.4 (1.3)	0.15, 0.13 (0.14)	0.46, 0.42 (0.44)	1.7, 1.8 (1.8)	
Germansville, PA, United States, 2016 (TA545-33EZ) Soil: loam	2^ (10)	152 159	106 107	Backpack Sprayer, 08 Sept., R5	28	0.83, 0.95 (0.89)	0.046, 0.051 (0.048)	0.15, 0.14 (0.14)	0.97, 1.1 (1.0)	2016RES-FNF2453, PSM-16-02-08-01
Wyoming, IL, United States, 2016 (DKC 61-86) Soil: clay loam	2^ (10)	151 148	118 111	Backpack Sprayer, 22 Sept, BBCH87	29	2.3, 1.8 (2.0)	0.081, 0.072 (0.077)	0.18, 0.12 (0.15)	2.4, 1.9 (2.2)	2016RES-FNF2453, PSM-16-02-08-02
Carlyle, IL, United States, 2016 (Syngenta N78S-3111) Soil: silt loam	2^ (10)	151 150	86 79	Tractor mounted boom, 12 Sept., BBCH85	30	1.8, 1.6 (1.7)	0.19, 0.17 (0.18)	0.32, 0.28 (0.30)	2.1, 1.9 (2.0)	2016RES-FNF2453, PSM-16-02-08-05 (corn)
Geneva, MN, United States, 2016 (NuTech 5D-196AMX) Soil: sandy clay loam	2 (10) +COC	152 151	88 79	Backpack Sprayer, 12 Sept, BBCH85-87	30	0.43, 0.70 (0.57)	0.023, 0.042 (0.033)	0.14, 0.24 (0.19)	0.57, 0.94 (0.76)	2016RES-FNF2453, PSM-16-02-08-06
Ellendale, MN, United States, 2016 (Dekalb DKC44-13RIB) Soil:sandy loam	2 (9) +COC	149 152	86 80	Backpack Sprayer, 15 Sept, BBCH85-87	30	0.73, 0.80 (0.76)	0.046, 0.045 (0.046)	0.12, 0.14 (0.13)	0.85, 0.95 (0.90)	2016RES-FNF2453, PSM-16-02-08-07
Paynesville, MN, United States, 2016 (NK 23MGTA) Soil: sandy loam	2 (10) +COC	152 148	97 94	Tractor mounted boom, 30 Aug., BBCH85	30	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	2016RES-FNF2453, PSM-16-02-08-09
York, NE, United States, 2016 (DKC62-77RIB) Soil: silt loam	2 (10) +COC	145 150	93 93	Backpack Sprayer, 12 Sept., BBCH87	30	1.0, 0.67 (0.84)	0.046, 0.042 (0.044)	0.36, 0.32 (0.34)	1.4, 0.99 (1.2)	2016RES-FNF2453, PSM-16-02-08-10
Brunswick, NE, United States, 2016 (DKC62-	2 (11) +COC	152 151	91 94	Backpack Sprayer, 12 Sept., BBCH87	31	0.76, 0.43 (0.60)	0.044, 0.028 (0.036)	0.29, 0.16 (0.22)	1.0, 0.59 (0.82)	2016RES-FNF2453, PSM-16-02-08-11

Location, year, MAIZE/ FIELD CORN STOVER (variety)	Application				DALA (days)	Mean residues (individual values) (mg/kg) [a]				Reference, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS		parent	3-OH-F	1-OH-Met-F	total [b]	
77RIB) Soil: sand										
Brookings, SD, United States, 2016 (DKC 44-13) Soil: loam	2 (10) +NIS	151 151	103 92	Backpack Sprayer, 29 Sept., BBCH85	32	2.6, 2.6 (2.6)	0.13, 0.13 (0.13)	0.21, 0.20 (0.21)	2.8, 2.8 (2.8)	2016RES-FNF2453, PSM-16-02-08-14 (corn)
Deerfield, MI, United States, 2016 (AgriGold A6472VT3P RIB) Soil: sandy clay loam	2 (10) +NIS	150 149	80 81	Backpack Sprayer, 20 Aug., BBCH73	30	0.23, 0.22 (0.22)	0.017, 0.017 (0.017)	0.076, 0.077 (0.076)	0.30, 0.29 (0.30)	2016RES-FNF2453, PSM-16-02-08-15
Milan, MI, United States, 2016 (Dekalb DKC60-67 Genssrib) Soil: clay loam	2 (10) +NIS	150 152	80 73	Backpack Sprayer, 20Aug., BBCH73	30	0.35, 0.32 (0.34)	0.026, 0.025 (0.025)	0.11, 0.092 (0.10)	0.47, 0.42 (0.44)	2016RES-FNF2453, PSM-16-02-08-16
Cleveland, ND, United States, 2016 (01049135) Soil: sandy	2 (10) +COC	151 160	107 107	Tractor mounted boom, 05 Sept., BBCH83	30	0.76, 1.0 (0.89)	0.044, 0.058 (0.051)	0.13, 0.14 (0.14)	0.90, 1.2 (1.0)	2016RES-FNF2453, PSM-16-02-08-17 (corn)
Claude, TX, United States, 2016 (P1234AM) Soil: clay loam	2 (10) +NIS	149 153	67 66	Backpack Sprayer, 03 Sept., BBCH84	30	2.0, 2.8 (2.4)	0.16, 0.21 (0.19)	0.22, 0.22 (0.22)	2.2, 3.0 (2.6)	2016RES-FNF2453, PSM-16-02-08-18

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Sweet corn forage and stover

Supervised residue trials were conducted in the United States in 2015 and 2016 to measure the magnitude of fluindapyr residues in/on sweet corn raw agricultural commodities following two foliar application of fluindapyr SC formulation [Webber, 2018c, 2016RES-FNF2454]. More details of the study are described in the section on food commodities (maize cereals).

The sweet corn (Kernel + Cobs With Husks Removed = K+CWHR, forage, stover) samples were harvested at proper times to yield commercially representative samples. At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 12 separate areas within the plots so

that each sample yielded a minimum of 1 kilogram of K+CWHR, 1 kilogram of forage, and 0.5 kilogram of stover. The sweet corn raw agricultural commodities of K+CWHR, forage, and stover were harvested at 12–16 days after the second application to the treated plot. Decline samples were collected at one trial location (Trial 06). At these locations samples were collected at a target of 3, 7, 14, 21, and 28 days after last application for K+CWHR, forage and stover.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone 1-OH-Met-fluindapyr. The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from the LOQ (0.01 mg/kg) to 1.0 mg/kg for K+CWHR, 10.0 mg/kg for forage, and 20.8 mg/kg for stover. Fortified control samples were included in each analysis set for method verification.

Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sweet corn forage were 102 ± 11 percent (n = 5), 104 ±3 percent (n = 4) and 90 ± 12 percent (n = 4), respectively.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sweet corn stover were 101 ± 13 percent (n = 5), 102 ± 3 percent (n = 4) and 93 ± 10 percent (n = 4), respectively.

The results on sweet corn forage and sweet corn stover are summarized in Table 124 and Table 125.

Table 124 Residues of fluindapyr in sweet corn forage after foliar treatment with a suspension concentrate

Location, year, MAIZE/ SWEET CORN FORAGE (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Alton, NY, United States, 2016, (Precious Gem F1) Soil: sandy loam	2^ (10)	150 150	107 106	Tractor mounted sprayer, 09 Sept., BBCH79	14	0.95, 1.0 (0.98)	0.067, 0.071 (0.069)	0.17, 0.15 (0.16)	1.1, 1.2 (1.1)	2016RES-FNF2454, PSM-16-02-09-01
Weston, GA, United States, 2016, (Silver Queen) Soil: loamy sand	2^ (10)	148 150	74 74	Backpack sprayer, 01 Aug., BBCH71	14	0.68, 0.86 (0.77)	0.032, 0.039 (0.036)	0.13, 0.19 (0.16)	0.81, 1.1 (0.93)	2016RES-FNF2454, PSM-16-02-09-02
Richland, IA, United States, 2016	2^ (9)	150 149	91 65	Tractor mounted sprayer,	16	0.028, 0.016 (0.022)	<0.010, <0.010 (<0.010)	0.042, 0.053 (0.047)	0.070, 0.069 (0.069)	2016RES-FNF2454, PSM-16-

Location, year, MAIZE/ SWEET CORN FORAGE (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
(Delectable TRTDF1) Soil: silty clay loam				09 July, BBCH65-69						02-09-03
Lime Springs, IA, United States, 2016 (Luscious) Soil: sandy loam	2 (11) +COC	153 152	82 84	Backpack sprayer, 18 July, BBCH69	14	0.23, 0.28 (0.25)	0.019, 0.021 (0.020)	0.64, 0.64 (0.64)	0.87, 0.92 (0.89)	2016RES-FNF2454, PSM-16-02-09-04
Deerfield, MI, United States, 2016 (Iochief) Soil: sandy clay loam	2 (9) +COC	150 152	82 73	Backpack sprayer, 20Aug., BBCH71	12	0.28, 0.38 (0.33)	0.020, 0.028 (0.024)	0.16, 0.12 (0.14)	0.44, 0.50 (0.47)	2016RES-FNF2454, PSM-16-02-09-05
York, NE, United States, 2016 (Obession II) Soil: silt loam	2 (11) +COC	151 150	91 91	Backpack sprayer, 07 Aug., BBCH not reported	4	0.97, 0.93 (0.95)	0.025, 0.027 (0.026)	0.12, 0.13 (0.12)	1.1, 1.1 (1.1)	2016RES-FNF2454, PSM-16-02-09-06
					8	0.21, 0.30 (0.26)	0.013, 0.019 (0.016)	0.13, 0.10 (0.12)	0.34, 0.40 (0.37)	
					13	0.13, 0.14 (0.14)	0.011, 0.013 (0.012)	0.14, 0.13 (0.14)	0.27, 0.27 (0.27)	
					22	0.093, 0.066 (0.079)	0.014, 0.011 (0.013)	0.16, 0.077 (0.12)	0.25, 0.14 (0.20)	
					26	0.062, 0.054 (0.58)	0.011, 0.010 (0.010)	0.12, 0.087 (0.10)	0.18, 0.14 (0.16)	
Payette, ID, United States, 2016 (Ambrosia) Soil: clay loam	2 (10) +NIS	155 156	70 70	Tractor mounted sprayer, 21 July, BBCH67	14	4.0, 6.8 (5.4)	0.16, 1.5 (0.84)	0.086, 0.11 (0.097)	4.1, 6.9 (5.5)	2016RES-FNF2454, PSM-16-02-09-07
Oregon City, OR, United States, 2016 (Honey N Pearl L) Soil: silt loam	2 (9) +NIS	152 148	74 84	Backpack sprayer, 17 Aug., BBCH69-71	14	0.16, 0.20 (0.18)	<0.010, <0.010 (<0.010)	0.054, 0.059 (0.057)	0.22, 0.26 (0.24)	2016RES-FNF2454, PSM-16-02-09-08

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents. Since all results were <0.01 mg/kg in both duplicates, only the mean is included in the table.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Table 125 Residues of fluindapyr in sweet corn stover after foliar treatment with a suspension concentrate

Location, year, MAIZE/ SWEET CORN STOVER (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Alton, NY, United States, 2016, (Precious Gem F1) Soil: sandy loam	2^ (10)	150 150	107 106	Tractor mounted sprayer, 09 Sept., BBCH79	14	0.70, 0.55 (0.63)	0.085, 0.069 (0.077)	0.28, 0.23 (0.26)	0.98, 0.78 (0.88)	2016RES-FNF2454, PSM-16-02-09-01
Weston, GA, United States, 2016, (Silver Queen) Soil: loamy sand	2^ (10)	148 150	74 74	Backpack sprayer, 01 Aug., BBCH71	14	1.2, 1.3 (1.3)	0.10, 0.10 (0.10)	0.32, 0.48 (0.40)	1.5, 1.8 (1.7)	2016RES-FNF2454, PSM-16-02-09-02
Richland, IA, United States, 2016 (Delectable TRTDF1) Soil: silty clay loam	2^ (9)	150 149	91 65	Tractor mounted sprayer, 09 July, BBCH65-69	16	0.18, 0.19 (0.19)	0.017, 0.022 (0.020)	0.51, 0.43 (0.47)	0.68, 0.62 (0.65)	2016RES-FNF2454, PSM-16-02-09-03
Lime Springs, IA, United States, 2016 (Luscious) Soil: sandy loam	2 (11) +COC	153 152	82 84	Backpack sprayer, 18 July, BBCH69	14	0.17, 0.17 (0.17)	0.021, 0.0190 (0.02)	0.77, 0.66 (0.72)	0.93 0.83 (0.88)	2016RES-FNF2454, PSM-16-02-09-04
Deerfield, MI, United States, 2016 (Iochief) Soil: sandy clay loam	2 (9) +COC	150 152	82 73	Backpack sprayer, 20Aug., BBCH71	12	0.68, 0.61 (0.65)	0.035, 0.030 (0.032)	0.18, 0.18 (0.18)	0.86, 0.80 (0.83)	2016RES-FNF2454, PSM-16-02-09-05
York, NE, United States, 2016 (Obession II) Soil: silt loam	2 (11) +COC	151 150	91 91	Backpack sprayer, 07 Aug., BBCH not reported	4	2.4, 2.4 (2.4)	0.13, 0.12 (0.13)	0.62, 0.61 (0.62)	3.0, 3.0 (3.0)	2016RES-FNF2454, PSM-16-02-09-06
					8	0.36, 0.50 (0.43)	0.059, 0.056 (0.057)	0.35, 0.32 (0.34)	0.71, 0.81 (0.77)	
					13	0.28, 0.24 (0.26)	0.031, 0.027 (0.029)	0.35, 0.32 (0.33)	0.63, 0.56 (0.59)	
					22	0.18, 0.17 (0.18)	0.023, 0.024 (0.024)	0.29, 0.29 (0.29)	0.47, 0.46 (0.46)	
					26	0.20, 0.20 (0.20)	0.032, 0.033 (0.033)	0.49, 0.48 (0.48)	0.69, 0.68 (0.68)	

Location, year, MAIZE/ SWEET CORN STOVER (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
Payette, ID, United States, 2016 (Ambrosia) Soil: clay loam	2 (10) +NIS	155 156	70 70	Tractor mounted sprayer, 21 July, BBCH67	14	12, 13 (13)	0.22, 0.24 (0.23)	0.27, 0.25 (0.26)	13, 13 (13)	2016RES-FNF2454, PSM-16-02-09-07
Oregon City, OR, United States, 2016 (Honey N Pearl L) Soil: silt loam	2 (9) +NIS	152 148	74 84	Backpack sprayer, 17 Aug., BBCH69-71	14	0.33, 0.23 (0.28)	0.026, 0.019 (0.023)	0.19, 0.18 (0.18)	0.52, 0.41 (0.46)	2016RES-FNF2454, PSM-16-02-09-08

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

Almond hulls

Supervised residue trials were conducted in the United States in 2016 to measure the magnitude of fluindapyr residues in/on almond agricultural commodities following three foliar applications of a fluindapyr SC formulation [Webber, 2017a, 2016RES-FNF2450]. More details of the study are described in the section on food commodities (tree nuts).

The almond (nutmeat and hulls) samples were harvested at maturity to yield commercially representative samples. At each of the sampling events, one composite sample from the untreated plot (control) and two independently collected composite samples from the treated plot, were collected randomly from at least 4 different trees within the plots so that each sample yielded a minimum of 1 kilogram of hulls. The samples were harvested at 29–31 days after the last application. Decline samples were collected at one trial locations on days 15, 23, 30, 37, and 44 after the last application.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone 1-OH-Met-fluindapyr. The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from 1/5 LOQ (0.002 mg/kg) to 10 × LOQ (0.1 mg/kg). The overall mean method validation recoveries and RSDs for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from almond hulls were 96 ± 11 percent (n = 10), 77 ± 8 percent (n = 10) and 93 ± 6 percent (n = 10), respectively.

Laboratory fortification samples were analysed concurrently with each analytical set to demonstrate method performance. The overall mean laboratory fortification recoveries for fluindapyr and

the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from almond hulls were 103 ± 6 percent (n = 7), 102 ± 7 percent (n = 5) and 93 ± 10 percent (n = 5), respectively.

The results on almond hulls are summarized in Table 126.

Table 126 Residues of fluindapyr in almond hulls after three foliar treatments with a suspension concentrate

Location, year, ALMOND HULLS (variety)	Application					Residues (mean) (mg/kg) [a]				Reference, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	
Yuba City, CA, United States, 2016 (Non-pareil) Soil: clay loam	3^ (8,7)	168 164 156	36 36 34	Foliar, 13 July, BBCH75	29	5.9, 6.0 (6.0)	0.23, 0.24 (0.23)	0.032, 0.027 (0.029)	5.9, 6.0 (6.0)	2016RES-FNF2450, PSM-16-02-05-01
		167 171 167	10 10 9		29	5.3, 5.9 (5.6)	0.25, 0.30 (0.28)	0.072, 0.037 (0.054)	5.4, 6.0 (5.7)	
Orland, CA, United States, 2016 (Non-pareil) Soil:loam	3^ (7,7)	164 164 164	35 35 35	Foliar, 17 July, BBCH85	30	1.0, 6.6 (3.8)	0.023, 0.14 (0.080)	<0.01, 0.024 (0.015)	1.0, 6.6 (5.9)	2016RES-FNF2450, PSM-16-02-05-02
		164 164 164	9 9 9		30	7.4, 8.9 (8.2)	0.070, 0.10 (0.087)	0.020, 0.012 (0.016)	7.4, 8.9 (8.2)	
Strathmore, CA, United States, 2016 (Fritz) Soil: sandy loam	3 (7,7) +COC	167 168 166	34 34 33	Foliar, 22 July, BBCH77	31	2.5, 2.6 (2.5)	0.22, 0.22 (0.22)	0.43, 0.39 (0.41)	2.9, 3.0 (2.9)	2016RES-FNF2450, PSM-16-02-05-03
		168 170 169	9 9 9		31	2.4, 1.7 (2.0)	0.20, 0.16 (0.18)	0.68, 0.60 (0.64)	3.0, 2.3 (2.7)	
Sanger, CA, United States, 2016 (Non-pareil) Soil: sandy loam	3 (7,7) +COC	188 188 166	36 42 34	Foliar, 10 Aug., BBCH81-85	30	2.6, 2.2 (2.4)	0.15, 0.15 (0.15)	0.010, 0.010 (0.010)	2.6, 2.2 (2.4)	2016RES-FNF2450, PSM-16-02-05-04
		188 192 165	9 11 9		30	3.6, 3.3 (3.4)	0.15, 0.13 (0.14)	0.010, 0.014 (0.012)	3.6, 3.3 (3.4)	
Terra Bella, CA, United States, 2016 (Monterey) Soil: sandy loam	3 (7,7) +NIS	166 168 166	35 36 34	Foliar, 20 July, BBCH79	15	2.3, 2.7 (2.5)	0.13, 0.14 (0.13)	0.18, 0.17 (0.18)	2.5, 2.9 (2.7)	2016RES-FNF2450, PSM-16-02-05-05
					23	4.2, 4.5 (4.3)	0.20, 0.17 (0.19)	0.34, 0.39 (0.36)	4.5, 4.9 (4.7)	
					30	1.7, 1.4 (1.5)	0.14, 0.10 (0.12)	0.27, 0.22 (0.25)	1.9, 1.6 (1.8)	
					37	1.3, 1.5 (1.4)	0.12, 0.13 (0.12)	0.33, 0.26 (0.30)	1.6, 1.7 (1.7)	
					44	0.70, 0.73 (0.72)	0.063, 0.067 (0.065)	0.21, 0.21 (0.21)	0.91, 0.94 (0.92)	
		167 165 166	9 9 9		15	2.4, 2.5 (2.5)	0.13, 0.14 (0.13)	0.17, 0.16 (0.17)	2.6, 2.7 (2.6)	
					23	3.6, 3.9 (3.8)	0.23, 0.22 (0.22)	0.37, 0.32 (0.34)	4.0, 4.2 (4.1)	

Location, year, ALMOND HULLS (variety)	Application					Residues (mean) (mg/kg) [a]				
	No. (RTI)	g ai/ha	g ai/hL	method, timing, GS	DALA (days)	parent	3-OH-F	1-OH-Met-F	total [b]	Reference, Trial No.
					30	1.6, 1.8 (1.7)	0.15, 0.16 (0.15)	0.44, 0.42 (0.43)	2.1, 2.2 (2.1)	
					37	1.4, 1.3 (1.4)	0.16, 0.16 (0.16)	0.44, 0.42 (0.43)	1.9, 1.7 (1.8)	
					44	1.3, 1.3 (1.3)	0.16, 0.17 (0.17)	0.53, 0.48 (0.51)	1.8, 1.8 (1.8)	

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; 3-OH-F = 3-OH-fluindapyr ; 1-OH-Met-F = 1-OH-Met-fluindapyr; ^ no adjuvant used; NIS: Adjuvant Non-Ionic Surfactant; COC: Adjuvant Crop Oil Concentrate.

[a] Residues are expressed as parent equivalents.

[b] Total residues is the sum of fluindapyr and 1-OH-Met-fluindapyr, expressed as parent equivalents.

FATE OF RESIDUES IN PROCESSING

Nature of residues processing study

The behaviour of fluindapyr was studied under conditions simulating pasteurisation, baking/brewing/boiling and sterilisation [Vanini & Zerbinati, 2017, 2016RES-IFP3051]. The phenyl- or pyrazole radiolabelled [^{14}C]-fluindapyr was used for this study at approximately 0.5 µg/mL.

Duplicate solutions were prepared in 0.1 M sterile citrate buffers at pH 4, 5 and 6. The buffer solutions were incubated in the dark for 20 minutes at 90 °C (pH 4), 60 minutes at 100 °C (pH 5) or 20 minutes at 120 °C (pH 6). During the test, the temperature was maintained within ± 2 °C of each required temperatures. At the end of each test the samples were cooled at room temperature and analysed by Liquid Scintillation Counting (LSC) to determine the Total Radioactive Residue (TRR) and by High Performance Liquid Chromatography (HPLC) to establish the amount of unchanged ^{14}C-fluindapyr and its degradation products. The enantiomeric ratio of fluindapyr was also determined using chiral HPLC.

The TRR ranged from 93 percent to 98 percent of applied radioactivity (AR), corresponding to 0.45 and 0.47 mg/L (Table 127). According to the HPLC analyses performed before and after each test, the amount of fluindapyr and its enantiomeric ratio remained constant (50:50) in each test. The results showed that fluindapyr was stable under simulated conditions of processing operations and no additional residues were found.

Table 127 Recovery and identification of radioactivity in 0.5 mg/L ^{14}C-fluindapyr solutions under pasteurisation, baking/brewing/boiling and sterilization simulating conditions

	Pyrazole label		Phenyl label	
Conditions	mg/L	Percent TRR	mg/L	Percent TRR
pH 4 (90 °C, 20 min) (pasteurisation)	0.470/0.450	97.32/93.12	0.453/0.450	95.58/94.97
pH 5 (100 °C, 60 min) (baking/brewing/boiling)	0.474/0.472	97.88/97.53	0.460/0.460	94.94/95.04
pH 6 (120 °C, 20 min) (sterilisation)	0.458/0.458	93.93/93.83	0.463/0.463	95.42/93.97

Supervised processing studies

Wheat

Study 1 + 2

Two processing studies were conducted as part of the residue trial studies to measure the magnitude of fluindapyr residues in/on wheat processed commodities, one at two locations (the United Kingdom and Germany, GB01 and DE03) in Northern Europe [Peterek, 2018, 2015RES-IFP1968] and one at two locations (Italy and France) in Southern Europe [Riccelli, 2017, 2015RES-IFP1950]. Since the study protocols were identical the study results were combined. Two foliar applications of fluindapyr at an exaggerated rate of 689 to 794 g ai/ha and a retreatment interval of 13–19 days for processing purposes. The last application was made at BBCH 69 in both studies.

The wheat grain samples were harvested at proper times to yield commercially representative samples. Bulk samples for processing weighed >50 kg and were harvested 40 to 48 days after the last application.

Grain conditioning

Upon the arrival of the bulk grain specimens from trials, aliquots of grain specimens were taken and cleaned, then the water content of each specimen (10–15 kg) was measured and by adding water for approx. 5 hours the water content was increased to about 17 percent, resulting in final grain weights of 10.00–15 kg. The water content in the treated batch of GB01 did not need any adjustment.

Generation of wheat flour (whole meal)

For bran and flour processing wheat grains (3000–3022 gram) were used for the generation of whole meal flour. The grain was ground through a mill consisting of break rolls and then reduction rolls and screened. After the break stage, coarse bran (543–684 gram) and grinding flour (675–984 gram) were recovered. After the reduction stage, fine bran (1011–1476 gram) and reduction flour (191–295 gram) were recovered. Coarse bran and fine bran together with the grinding flour and reduction flour was combined to whole-meal flour (2957–2993 gram), and samples were taken (sample: *wheat whole meal flour*). The yield from RAC to whole meal was 99–106 percent.

Generation of white flour

Approximately 7 kg of wheat grains (6894–7671 gram) were placed through a mill consisting of break rolls and the reduction rolls and screened. After the break stage, coarse bran (1282–1784 gram) and milling flour (1755–2510gram) were recovered. After the reduction stage, fine bran (2411–3465 gram) and reduction flour (431–968 gram) were recovered. Coarse bran and fine bran were combined to obtain total bran (3914–4941 gram), and a sample was taken (sample: *wheat bran*). Milling flour (1755–2510 gram) and reduction flour (431–968 gram) were combined to obtain white flour (2442–3059 gram) and samples were taken (sample: *wheat white flour*). The yield from RAC to white flour was 33–44 percent.

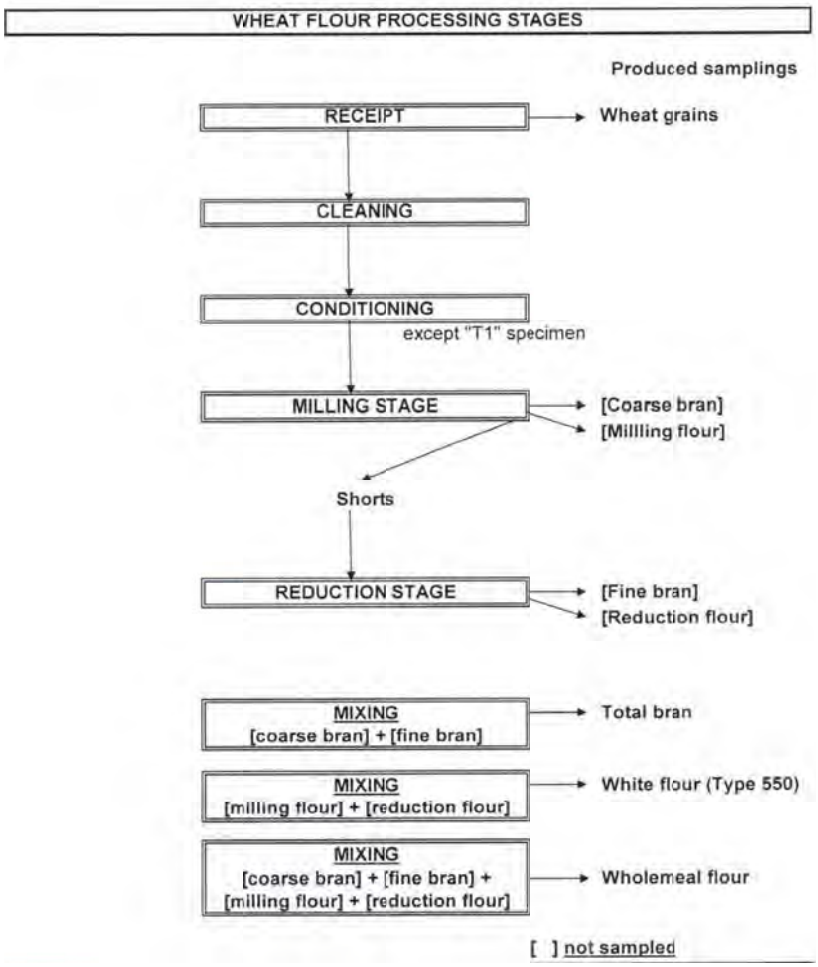


Figure 8 Flow chart of wheat flour processing

Whole meal bread processing

For whole-meal bread processing, whole-meal flour obtained before was kneaded together with prepared yeast and water. Five minutes before the kneading, salt was added. The dough was left to rise for 45 minutes, divided in smaller portions of 350 gram. The divided portions were covered and held at ambient temperature for another 15 minutes. Each portion was then shaped into a baguette, covered and kept at ambient temperature for 2 hours. The baguettes were then notched with a knife and baked in an oven at 250 °C for 30 minutes. Whole-meal bread sub-specimens were taken and stored in a freezer.

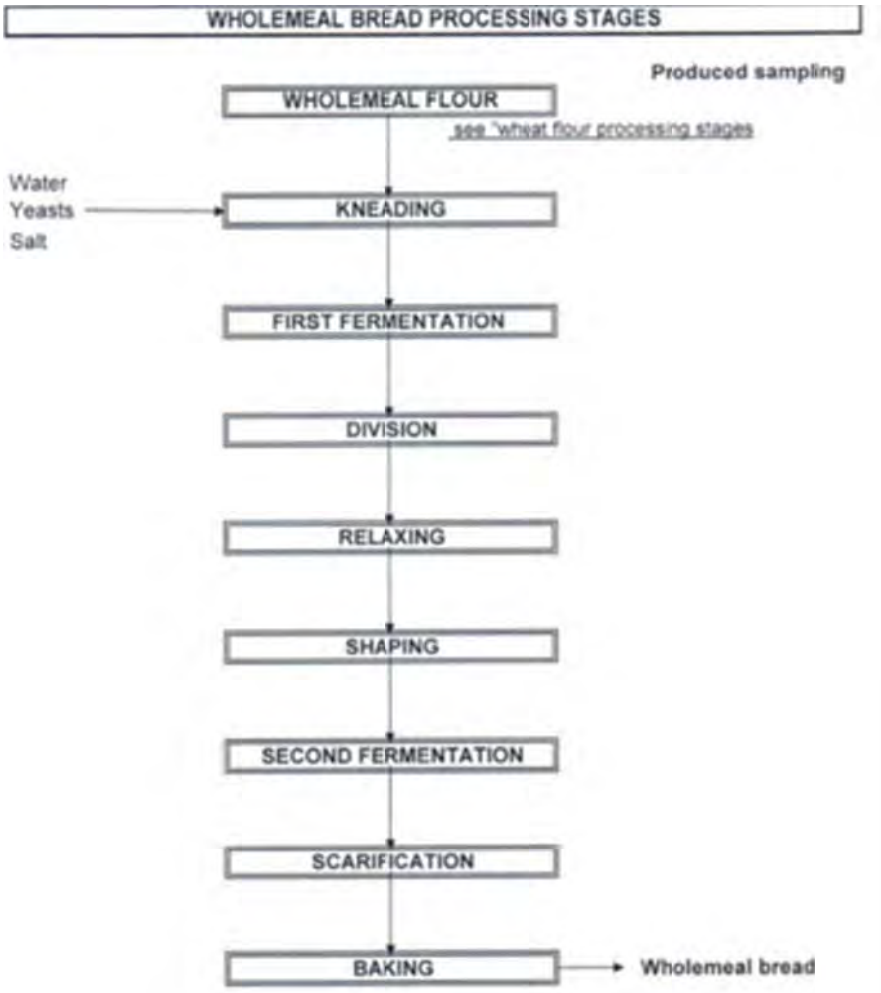


Figure 9 Flow chart of wholemeal bread processing

Gluten and starch of wheat flour separation processing

For gluten and starch of wheat flour separation processing, dough of white flour (2323–3141 gram) was prepared from 2000 gram white flour and 1200 gram (NE study) and 900–904 gram (SE study) water and after a rest, was washed with water to separate milk (30141–30944 gram, NE study; 20910–21332 gram, SE study) and wet gluten (472–809 gram, NE study; 402–517 gram, SE study). Wet gluten was dried in an oven at 50 °C and after settling of starch milk in a cold room, the wet starch (3046–3586 gram, NE study; 1480–2266 gram, SE study) was dried in an oven at 50 °C.

Dry gluten (150–233 gram, NE study; 129–164 gram SE study) and dry starch (1167–1345 gram, NE study; 582–897 gram NE study) were ground separately with a mill and mixed (1/1). Samples of gluten feed meal were taken (sample: *gluten feed meal*) and stored in a freezer.

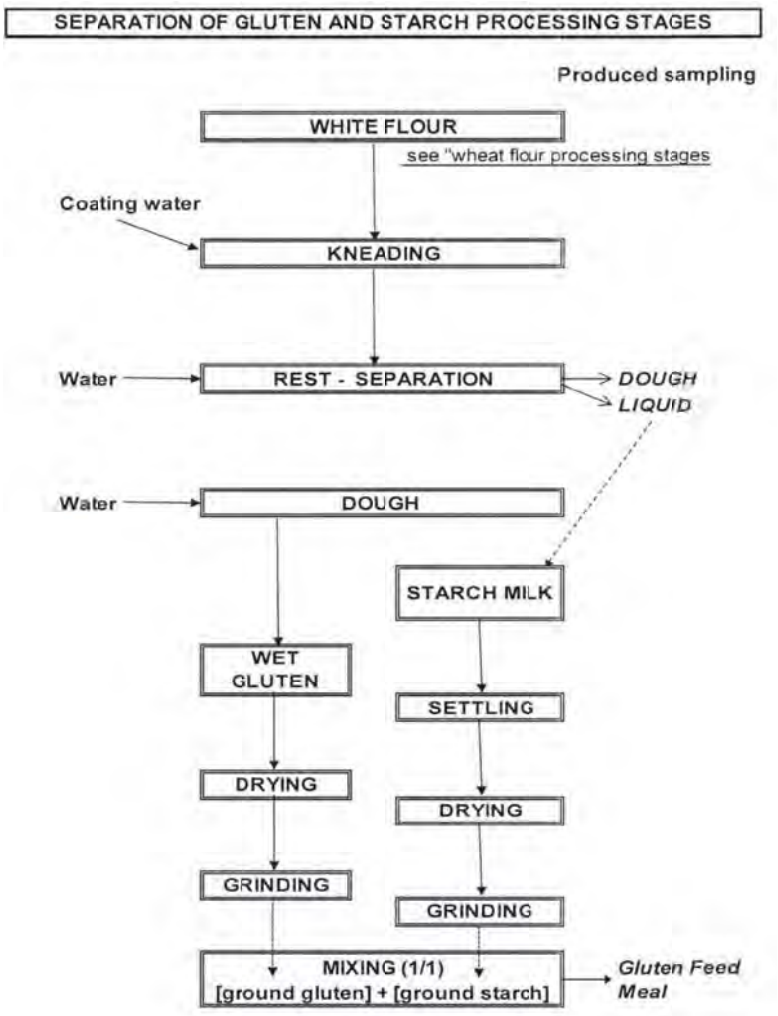


Figure 10 Flow chart of separation of gluten and starch processing

Germ extraction processing

For the germ extraction processing 1.5 kg of cleaned gains were steeped for at least one day in a container with a similar quantity of water. After straining a portion of grains was set down on absorbing paper. The germ was removed from wheat grain with a cutter. Samples of germs were taken (sample: *germ*) and stored in a freezer. The yield from RAC to germ is 5.8–10 percent.

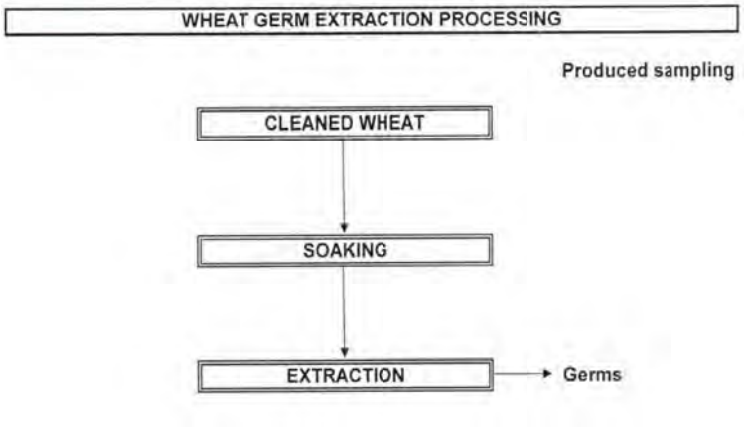


Figure 11 Flow chart of wheat germ extraction processing

Treated raw and processed crop commodity specimens for this study were frozen upon collection, shipped frozen, and stored frozen for less than 814 days (27 months in NE study) and less than 687 days (23 months in SE study) between sampling and analysis (<-18 °C at the analytical facility).

Analytical methods used were PTRL Method P3770G for fluindapyr, 3-OH-fluindapyr, and fluindapyr-N-DesMet-glucoside and method RA.17.01 for determination of 1-OH-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and 1-COOH-fluindapyr, all with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the conjugates would be hydrolysed to their respective aglycones.

Acceptance for concurrent recovery control was met with average recoveries ranging from 70 percent to 110 percent and relative standard deviations (RSD) ≤ 20 percent, respectively ≤ 15 percent for higher fortification levels. Exception: Only for whole meal flour and white flour the individual recoveries (expressed as sum of diastereomers) ranged between 45 percent and 79 percent. However, average recoveries at LOQ and at higher fortification level were always >50 percent. Specific details are still to be included in the section on analytical methods! No residues of F-N-DesMet-glucoside or 1-COOH-fluindapyr were observed in the RAC or either of the processed commodities. Metabolite 1-OH-Met-N-DesMet-fluindapyr was observed in RAC and processed commodities in one trial at levels of 0.012–0.014 mg/kg in the study by Peterek, 2018.

Processing factors were derived for wheat grain processed commodities. A concentration of residues was observed in total bran. Residues were diluted in all other commodities. Trial data, residues and processing factors for each type of commodity are summarised in Table 128.

Table 128 Fluindapyr residues and processing factors in wheat grain and processed commodities [Peterek, 2018, 2015RES-IFP1968 and Riccelli, 2017, 2015RES-IFP1950]

Trial, location, year, (variety), dose rate, interval, DALT [reference]	Crop/Processed commodity	Residues (mg/kg) [a]				PF [b]	
		parent	3-OH-F	1-OH-Met-F	Total [c]	PF_{parent}	PF_{total}
SPK-15-20471-GB01 South Fawley, United Kingdom, 2015 (Claire) 2 × foliar application at 771 & 728 g ai/ha; no adjuvant; BBCH 69, RTI = 13 days, DALA = 48 days.	Grain (bulk)	0.13	0.013	0.035	0.165	-	-
	Whole meal flour	0.13	0.011	0.035	0.165	1.0	1.0
	Total bran	0.18	0.014	0.043	0.223	1.38	1.4
	White flour	0.081	<0.010	<0.010	0.091	0.62	0.55
	Whole meal bread	0.064	<0.010	0.025	0.089	0.49	0.54
	Gluten feed meal	0.12	<0.010	<0.010	0.13	0.92	0.71
	Germs	0.078	<0.010	0.010	0.088	0.60	0.53

Trial, location, year, (variety), dose rate, interval, DALT [reference]	Crop/Processed commodity	Residues (mg/kg) [a]				PF [b]	
		parent	3-OH-F	1-OH-Met-F	Total [c]	PF_{parent}	PF_{total}
[Peterek, 2018, 2015RES-IFP1968]							
SPK-15-20471-DE03 Blaufeld-Mittelbach, DE, 2015 (Colonia) 2 × foliar application at 689 & 745 g ai/ha; no adjuvant BBCH 69, RTI = 15 days, DALA = 40 days. [Peterek, 2018, 2015RES-IFP1968]	Grain (bulk)	0.37	0.024	0.045	0.415	-	-
	Whole meal flour	0.24	0.011	0.054	0.294	0.65	0.71
	Total bran	0.56	0.021	0.060	0.62	1.51	1.49
	White flour	0.11	<0.010	<0.010	0.12	0.29	0.29
	Whole meal bread	0.14	<0.010	0.043	0.183	0.38	0.44
	Gluten feed meal	0.15	<0.010	0.014	0.164	0.41	0.40
	Germs	0.18	<0.010	0.069	0.249	0.49	0.6
RA1508-1H-P Gavello, Italy, 2015 (50207) 2 × foliar application at 790 & 794 g ai/ha; no adjuvant; BBCH 69, RTI = 19 days, DALA = 41 days [Riccelli, 2017, 2015RES-IFP1950]	Grain (bulk)	0.12	0.016	0.042	0.162	-	-
	Whole meal flour	0.10	<0.010	0.031	0.131	0.83	0.81
	Total bran	0.12	0.011	0.034	0.154	1.0	0.95
	White flour	0.038	<0.01	<0.01	0.048	0.32	0.30
	Whole meal bread	0.061	<0.01	0.027	0.088	0.51	0.54
	Gluten feed meal	0.050	<0.010	<0.010	0.060	0.42	0.37
	Germs	0.063	0.012	n.a.	0.075	0.53	0.42
RA1508-1H-P Marsilangues, France, 2015 (Arezzo) 2 × foliar application at 761 & 774 g ai/ha; no adjuvant; BBCH 69, RTI = 15 days, DALA = 47 days. [Riccelli, 2017, 2015RES-IFP1950]	Grain (bulk)	0.58	0.033	0.14	0.75	-	-
	Whole meal flour	0.53	0.011	0.089	0.63	0.91	0.86
	Total bran	0.66	0.022	0.11	0.80	1.14	1.1
	White flour	0.20	<0.010	ND	0.22	0.34	0.28
	Whole meal bread	0.24	<0.010	0.080	0.33	0.41	0.46
	Gluten feed meal	0.19	0.011	0.059	0.26	0.33	0.35
	Germs	0.11	0.011	n.a.	0.12	0.19	0.15

Notes:

DALT = Days After Last Treatment; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval; n.a. = not anlyzed

[a] Expressed as parent fluindapyr.

[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg).

[c] Total is based on parent + 1-OH-Met-fluindapyr, not including 3-OH-fluindapyr.

Sorghum

A processing trial was conducted in the United States as part of the residues trial to measure the magnitude of fluindapyr residues in/on sorghum processed commodities [Webber, 2018d, 2015RES-FNF1901]. Two foliar applications of fluindapyr at an exaggerated rate of 747 and 770 g ai/ha and a retreatment interval of 11 days for processing purposes.

The sorghum grain samples were harvested at proper times to yield commercially representative samples. Bulk samples for processing weighed approximately 250 kg and were harvested 45 days after the last application.

Drying

Samples were weighed and the moisture content of the samples determined with an electronic moisture analyser. Since the moisture content was greater than 13.0 percent, both the control and the treated samples were dried at 43–57 °C in an oven until the moisture content was 10.0–13.0 percent.

Generation of aspirated grain fractions

To generate aspirated grain fractions, each seed sample was placed in a dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. As the samples were moved in the system, aspiration was used to remove light impurities (grain dust). Each batch was moved for 120 minutes. Light impurities were classified using the following sieves: 2360 micron (8 mesh); 2000 micron (10 mesh); 1180 micron (16 mesh); 850 micron (20 mesh); and 425 micron (40 mesh). After classification of each sample, the material through the 2360 micron sieve was recombined to produce one aspirated grain fraction (AGF). For both samples, the material that passed through the 425 micron screen was not greater than half the weight of the total material passing through the 2360 micron screen, so the AGF was recombined in a way that 50 percent of the final AGF fraction was comprised of material passing through the 425 micron screen. A representative sample was removed and the ash content was determined.

Generation of sorghum flour

A representative sample of generated grain sorghum (23 kg) was cleaned by aspiration and screening. Light impurities (907 gram) were removed from the grain sorghum by aspiration in an aspirator. After aspiration, the samples were screened in a screen cleaner to separate large foreign particles (screenings (680 gram)) from the cleaned grain sorghum sample (21 kg). A sample of cleaned grain sorghum (4.5 kg) was ground in a pin mill. Ground material was screened with a rotating sifter equipped with a 62 mesh sieve. Material passing through the screen was grain sorghum flour (3.0 kg). Requested grain sorghum flour fractions were collected and placed into frozen storage. After correction to fractionation, the yield from sorghum grain to flour was 66 percent.

Sorghum raw and processed commodity samples were maintained frozen after collection through analysis for up to 762 days (grain), 572 days (aspirated grain fractions) and 574 days (flour) at the analytical laboratory Samples were maintained frozen from receipt at the analytical facility until extraction for analysis.

Analytical methods used were PTRL Method P3770G for fluindapyr and 3-OH-fluindapyr and method RA.17.01 for determination of 1-OH-fluindapyr, both with an LOQ of 0.010 mg/kg (metabolites expressed as parent equivalents). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone 1-OH-Met-fluindapyr. The efficiency of the analytical method was determined at the time of analysis with each set of samples by fortifying subsamples of the control matrix with fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr. The fortified samples were processed according to the analytical method and analysing them similar to a field-treated sample. Control matrices were fortified at levels ranging from the LOQ (0.01 mg/kg) to 26.0 mg/kg for aspirated grain fractions, and 3.0 mg/kg for flour.

The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum grain were 100 ± 25 percent (n = 6), 88 ± 14 percent (n = 5) and 84 ± 9 percent (n = 5), respectively. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr and 1-OH-Met-fluindapyr from sorghum aspirated grain fractions were 107 ± 17 percent (n = 4), 104 ± 4 percent (n = 3) and 77 ± 8 percent (n = 3), respectively. The overall mean laboratory fortification recoveries for fluindapyr and the metabolites 3-OH-fluindapyr

and 1-OH-Met-fluindapyr from sorghum flour were 100 ± 31 percent (n = 3), 106 ± 30 percent (n = 3) and 82 ± 13 percent (n = 3), respectively.

Processing factors were derived for field corn/maize grain processed commodities. A concentration of residues was observed in aspirated grain fractions and refined bleached deodorized oil, more predominant in the wet milled oil. Residues were diluted in all other commodities. Trial data, residues and processing factors for each type of commodity are summarised in Table 129.

Table 129 Fluindapyr residues and processing factors in field sorghum grain and processed commodities [Webber, 2018d, 2015RES-FNF1901]

Trial, location, year, (variety), dose rate, interval, DALT	Crop/Processed commodity	Residues (mg/kg) [a]				PF [b]	
		parent	3-OH-F	1-OH-Met-F	Total [a] [c]	PF_{parent}	PF_{total}
Bradshaw, NE, United States, 2015 (DKS37-07) 2 × foliar application at 747 & 770 g ai/ha; +NIS; BBCH 75, RTI = 11 days, DALT = 45 days.	Sorghum grain (RAC)	4.5, 2.6 (3.4)	0.24, 0.14 (0.19)	0.18, 0.23 (0.20)	4.7, 2.8 (3.6)	-	-
	Flour	2.2, 0.66 (1.4)	0.095, 0.043 (0.069)	0.13, 0.14 (0.13)	2.3, 0.8 (1.5)	0.41	0.42
	Aspirated grain fraction	22, 21 (22)	0.71, 0.74 (0.72)	2.6, 2.9 (2.7)	25, 24 (25)	6.5	6.9

Notes:

DALT = Days After Last Treatment; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval.

[a] Expressed as parent fluindapyr.

[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg).

[c] Total is based on parent + 1-OH-Met-fluindapyr, not including 3-OH-fluindapyr.

Maize

A processing trial was conducted in the United States as part of the residues trials to measure the magnitude of fluindapyr residues in/on maize processed commodities [Webber, 2018a, 2015RES-FNF1900]. Two foliar applications of fluindapyr at an exaggerated rate of 747 and 728 g ai/ha, respectively, with application intervals of 11 days. Samples of maize grain were collected 45 days after the final application at commercial maturity. Grain was processed according to simulated commercial procedures into grits, meal, flour, starch and oil (wet and dry milled). Throughout the study one representative sample of untreated control and two (duplicate) samples of treated processed commodities were taken.

Generation of Aspirated Grain Fraction (AGF)

To generate aspirated grain fractions, the maize grain sample (241 kg) was dried and the dried maize (192 kg) was placed in a dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. As the samples were moved in the system (120 minutes), aspiration was used to remove light impurities (grain dust) weighing 3.9 kg. Light impurities (501.4 gram) were classified using different sieves and the material through the 2360 micron sieve was recombined to produce one aspirated grain fraction (AGF). A representative sample was removed and the ash content (1.88 percent) was determined.

Dry milling process

After drying, the sample for production of processed fractions (190 kg) was cleaned resulting in batches of cleaned maize grain weighing 167 kg. For the dry milling process, maize grain (92 kg) was moisture conditioned to 21.0 percent and tempered for approximately two hours (steeping with 12 kg water added). The samples were fed into a mill to crack the kernels. Corn stock from the mill was dried and screened to separate bran (4.6 kg), germ (11.8 kg), and large grits (63 kg) from the grits (8.6 kg), meal (8.4 kg), and flour (2.3 kg) and samples were taken (sample: *meal*; sample: *grits*; sample: *flour*). The two germ fractions were combined and dried at 54–71 °C to a final moisture content of 14.0–16.0 percent. Samples of grits, meal, flour, and germ were collected and placed into frozen storage for analysis. Germ material was heated at 71–79 °C for 10 minutes and flaked in a flaking roll. The flaked kernels were placed in stainless steel batch extractors and submerged in hexane at 49–60 °C. After 30 minutes, the miscella (crude oil and hexane) was drained. Hexane was added and the cycle repeated 2 additional times. The miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. The crude oil was heated to 91–96 °C to complete the hexane removal, filtered and collected for refining. The free fatty acid of the crude oil was determined and an appropriate amount of sodium hydroxide was added to neutralize the oil. Neutralized refined oil and soapstock were separated by centrifugation. The refined oil was decanted and heated to 40–50 °C and activated bleaching earth was added. The temperature was increased to 85–100 °C and held for 10–15 minutes. After reducing the temperature to 58–68 °C and breaking the vacuum, the bleached oil was filtered. The bleached oil (405 gram) was steam bathed at 220–230 °C for 28–32 minutes under vacuum. The oil was cooled and citric acid was added. A sample of the refined oil (sample: *refined bleached, deodorized oil, dry milled*) was collected and placed in frozen storage for analysis. The yield of cleaned corn to refined, bleached, deodorized oil via dry milling process was 0.4 percent.

A flowchart of the field corn dry milling process is given below.

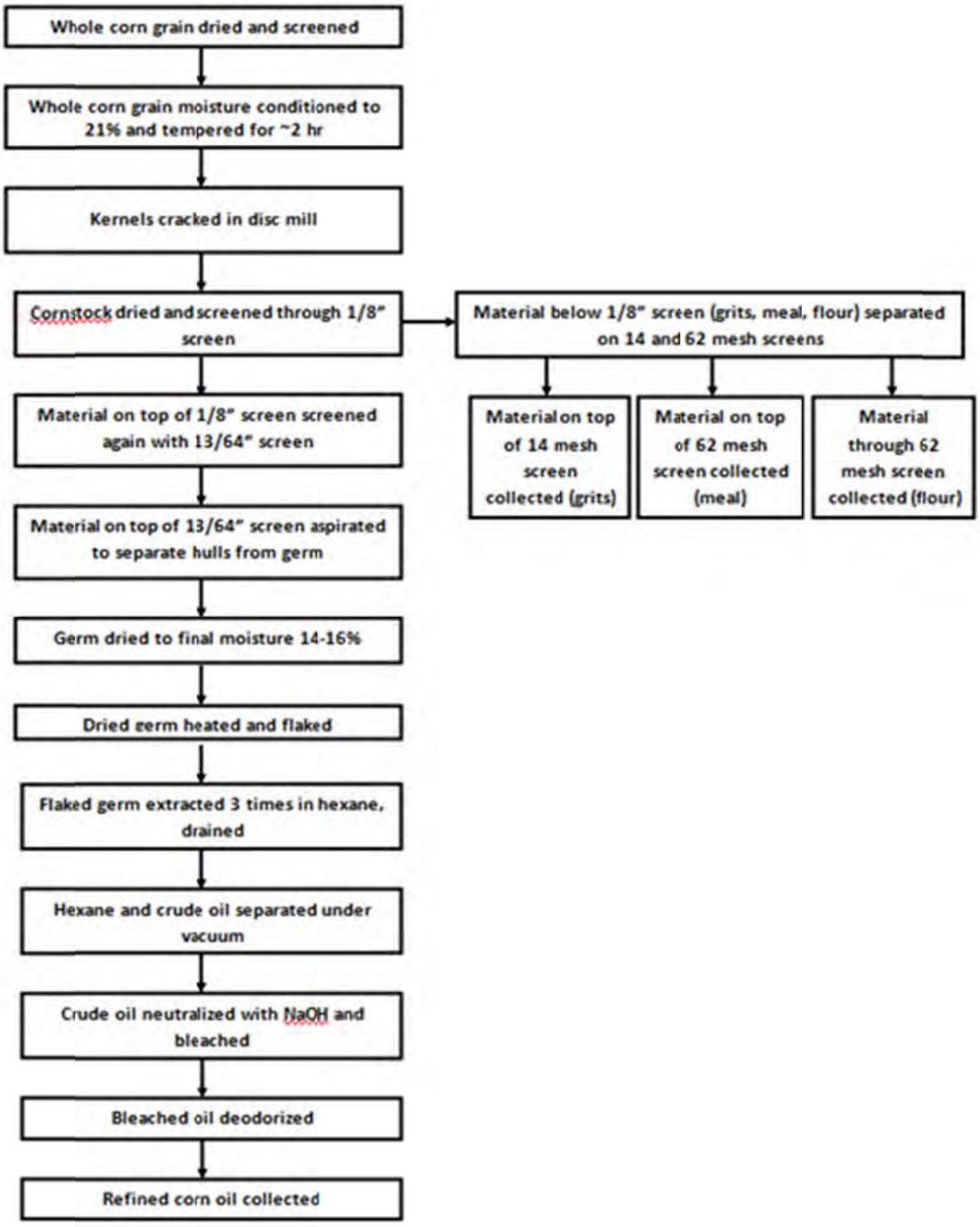


Figure 12 Flow chart of field corn dry milled processing

Wet Milling Process

A sample of cleaned field corn grain (75 kg) was steeped in water (125 kg) containing 0.1–0.2 percent sulfur dioxide for 22–48 hours at 49–54 °C. After the water was drained, steeped whole corn (116 kg) was passed through a disc mill. Most of the germ and hull was separated by a hydroclone. Germ and hulls were dried at 74–91 °C to a final moisture of 5–10 percent. After drying, the germ and hulls were

separated by aspiration and screening, and samples were taken (sample: *germ*). Corn stock (without germ and hulls) was screened with a 50 µm screen. Material on top of the screen (bran) was discarded. Process water passing through the screen was separated into starch and gluten by centrifugation. Starch (50.7 kg) was dried in an oven at 54–71 °C until a moisture content of <15 percent. Starch samples were collected (sample: *starch*) and placed in frozen storage for analysis. Germ samples (3.6 kg) were moisture conditioned to 14–16 percent, heated to 88–104 °C, flaked in a flaking roll, and pressed in an expeller to liberate part of the crude oil (354 gram). The presscake was placed in a stainless steel extractor and submerged in hexane at 49–60 °C. After 30 minutes, the miscella was drained. Hexane was added and the cycle was repeated an additional 2 times. Following the final draining, the miscella was passed through a laboratory evaporator to separate the crude oil from hexane. The crude oil (792 gram) was heated to 91–96 °C to remove the remaining hexane and combined with the crude oil from expelling. The combined crude oil (1087 gram) was neutralized, bleached and deodorized, using the same process as dry milling above. A sample of refined oil (sample: *refined bleached deodorized oil, wet milled*) was collected and placed in frozen storage for analysis. The yield of cleaned corn to refined, bleached, deodorized oil via the wet milling process was *ca* 1.3 percent.

A flowchart of the field corn dry milling process is given below.

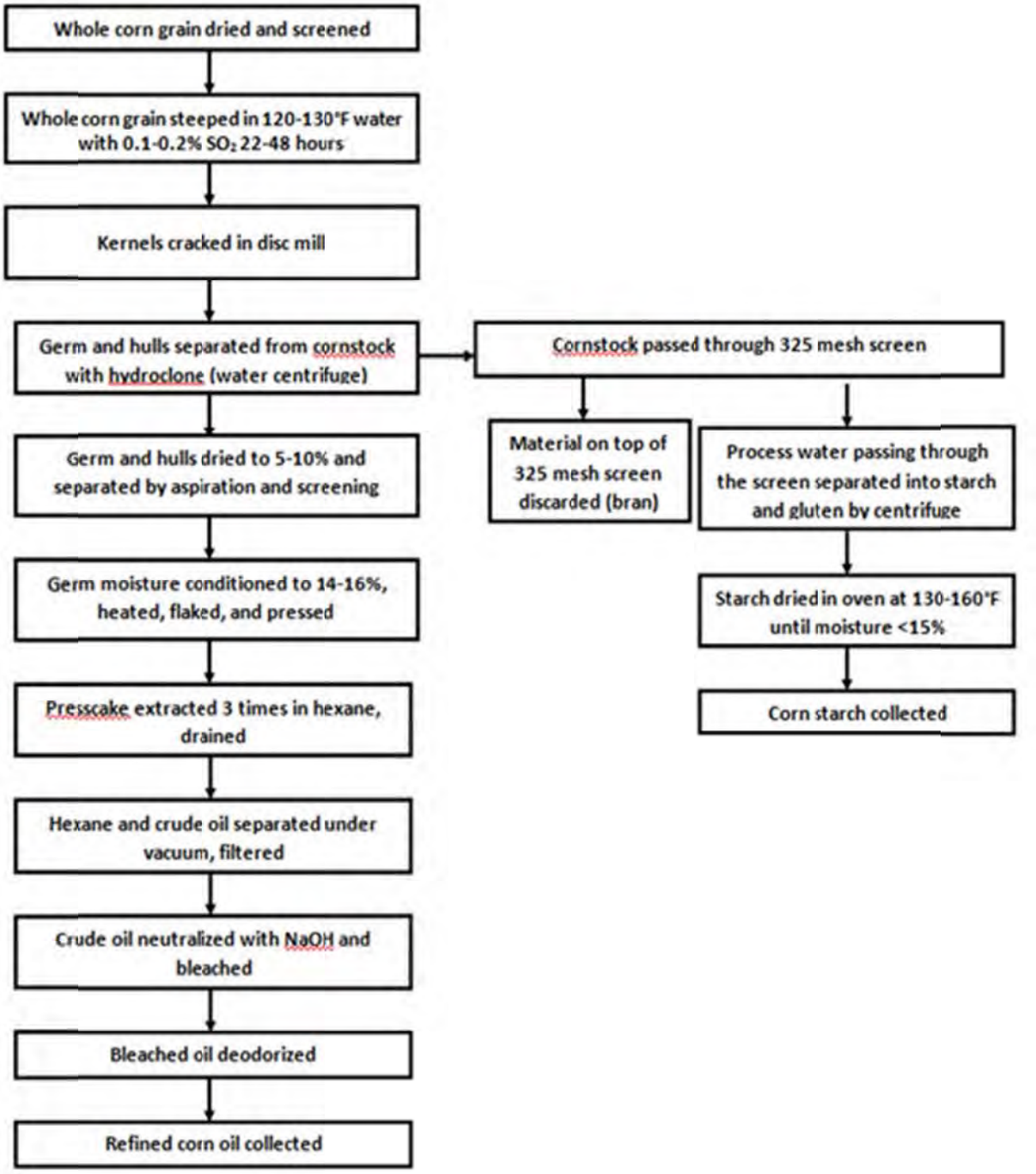


Figure 13 Flow chart of field corn wet milled processing

Samples were stored frozen (<-4 °C) for a maximum of 790 days (*ca* 26 months) prior to analysis. All samples were analysed for residues of fluindapyr, 3-OH-fluindapyr, and 1-OH-fluindapyr using the analytical method P 3770G and RA.17.01 (see section on analytical methods). The analytical method RA.17.01 utilizes acid hydrolysis so that the 1-OH-Met-fluindapyr-glucoside would be hydrolysed to the aglycone 1-OH-Met-fluindapyr. The results are shown in Table 130.

Processing factors were derived for field corn/maize grain processed commodities. A concentration of residues was observed in aspirated grain fractions and refined bleached deodorized oil,

more predominant in the wet milled oil. Residues were diluted in all other commodities. Trial data, residues and processing factors for each type of commodity are summarised in Table 130.

Table 130 Fluindapyr residues and processing factors in field corn/maize grain and processed commodities [Webber, 2018a, 2015RES-FNF1900]

Trial, location, year, (variety), dose rate, interval, DALT	Crop/Processed commodity	Residues (mg/kg)			PF [b]		
		parent	3-OH-F	1-OH-Met-F	Total [c]	PF_{parent}	PF_{total}
PMS-15-02-03-01, Farlin, IA, United States, 2015 () 2 × foliar application at 747 & 728 g ai/ha; +COC; BBCH 75, RTI = 11 days, PHI = 45 days.	Maize grain (RAC)	0.017, 0.012 (0.015)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.027, 0.022 (0.025)	-	-
	Flour	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	<0.59	<0.74
	Grits	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	<0.67	<0.8
	Meal	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	<0.67	<0.8
	Starch	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	<0.67	<0.8
	Refined bleached deodorized oil (dry milled)	0.019, 0.016 (0.018)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.030, <0.030 (<0.030)	1.2	1.1
	Refined bleached deodorized oil (wet milled)	0.035, 0.034 (0.034)	<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	0.055, 0.054 (0.054)	2.3	1.8
	Aspirated grain fraction	0.63, 0.68 (0.66)	0.018, 0.019 (0.019)	0.025, 0.026 (0.026)	0.67, 0.73 (0.70)	44	27

Notes:

DALT = Days After Last Treatment; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval.

[a] Expressed as parent fluindapyr.

[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF.

[c] Total is based on parent + 1-OH-Met-fluindapyr, not including 3-OH-fluindapyr.

Residues in the edible portion of food commodities

No data submitted.

RESIDUES IN ANIMAL COMMODITIES

Direct animal treatments

No data submitted.

Farm animal feeding studies

Animal feeding study in lactating dairy cows

A residue feeding study in dairy cattle was conducted in the United States in 2015/2016 [Brungardt, 2018, 2016RES-IFP2942] to measure the residues of fluindapyr found in milk and tissues. Lactating Holstein (*Bos taurus*) dairy cows (three animals per group) were orally dosed with fluindapyr via capsule for 28 consecutive days at levels of 5.09, 15.25, and 48.06 mg/kg in feed, corresponding to 0.17, 0.50, and 1.55 mg/kg bw/day. One group remained as control group and the highest dose group an additional three animals were dosed for use in a depuration phase. Animals were observed several times daily for any clinical signs of toxicity or ill health. Bodyweights were determined at intervals and concentrate food/hay consumption was monitored daily.

The cows weighed on average 380–614 kg at the beginning of the study and 378–954 kg at the end of the experiment and had an average daily milk production of 19–26 kg/day during the experiment. The milk production was not adversely affected.

Milk samples were collected twice daily (morning and evening) on study days -1, 1, 2, 4, 7, 10, 14, 17, 21, 24 and 28. Additionally, a portion of the study day 2, 14 and 28 milk samples from a single control, and three of the highest dose group animals were separated into skim milk and cream, and each was analysed. On study day 29, within 24 hours of the last dose, one of the control, all of the low

dose group, all of the mid dose group, and three of the high dose group cows were sacrificed and liver, kidney, composite muscle, subcutaneous fat, mesenterial fat and perirenal fat were collected for analysis. The remaining cows entered into the depuration phase of the study where milk and tissues were analysed throughout a 21-day period following the cessation of the last dose. Milk samples from the five cows (two control and three high dose group cows; depuration group) in the depuration phase were collected on study day 35. On study day 36, one cow from the high dose group was sacrificed and the tissues (liver, kidney, composite muscle, mesenterial fat, perirenal fat and subcutaneous fat) were collected. Milk samples from the remaining four cows (two control and two high dose group cows) were collected on study day 42, then on study day 43 one cow from the high dose group was sacrificed and the tissues were collected. Milk was again collected from the remaining three cows (two control and one high dose group cow) on study day 49. On study day 50, one control cow and the remaining cow from the high dose group were sacrificed and the tissues were collected. The remaining control cow was not sacrificed.

After collection and processing, all milk and tissue samples were stored frozen at an average storage temperature of ≤-18 °C during the storage period. The milk samples in this study were extracted within 52 days of collection. Cream and skimmed milk samples were extracted within 35 days of collection. The liver, kidney, muscle and fat samples were extracted within 35, 55, 27 and 50 days of collection, respectively.

Analytical method SRLS133SRUS16R0208 as validated in study 2016RES-IFP2941 (see section on analytical methods) was used. The method uses an additional hydrolysis step before analysis of fluindapyr-1-COOH, 1-OH-Met-fluindapyr, and 1-OH-Met-N-DesMet-fluindapyr to separate the conjugates from the aglycone version of the metabolites. Milk, skim milk, cream and muscle were analysed for parent compound fluindapyr only. Fat samples were analysed for fluindapyr and its metabolite 1-OH-Met-fluindapyr. Liver and kidney samples were analysed for the parent compound fluindapyr and its metabolites 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and fluindapyr-1-COOH. The reported residue values were calculated in parent equivalents and were not corrected for recoveries. Residues were quantitated by high performance liquid chromatography/electrospray ionization/tandem mass spectrometry (ESI LC-MS/MS). The limit of quantitation (LOQ) for fluindapyr in milk, skim milk and cream

was 0.005 mg/kg. The LOQ for all metabolites was 0.010 mg/kg in all relevant matrices. Average concurrent fresh recoveries in liver, kidney, muscle, fat, milk, cream and skim milk were within the range of 70–110 percent for fluindapyr and its metabolites at 0.01–0.4 mg/kg in tissues and 0.005 and 0.05 mg/kg in milk. Control samples had residues below 0.2LOQ.

Analytical results in milk and tissue samples are shown in Table 131 and Table 132.

In milk, the maximum average fluindapyr residue from the high dose group (1.55 mg/kg bw/day) was 0.0093 mg/kg. Milk samples from the medium dose group did not contain quantifiable residues (LOQ=0.005 mg/kg), and no milk samples from the low dose group were therefore analysed. No detectable residues of fluindapyr were found in skim milk samples from the high dose group. Residues were found in all cream samples from the high dose group, with group means ranging from 0.0105 to 0.020 mg/kg. In the six animals of the high dose group, the mean residue levels of fluindapyr in milk reached a plateau at day 4 and declined rapidly during the depuration phase.

For the medium dose group, average residue levels of fluindapyr in milk remained below the LOQ throughout the dosing period.

Table 131 Average (and highest) residues in whole milk of cows (means of 3 cows/dose group) dosed with fluindapyr for 28 days at 48.06 mg/kg in feed

Sample	parent
Feeding level	48.06 mg/kg DM [b]
Whole milk	
Day -1 (n=6)	<LOQ
Day 1 (n=6)	<LOQ (HR 0.0138)
Day 2 (n=6)	0.0066 (HR 0.0157)
Day 4 (n=6)	0.0090 (HR 0.0193)
Day 7 (n=6)	<LOQ (HR 0.0132)
Day 10 (n=6)	0.093 (HR 0.0262)
Day 14 (n=6)	0.070 (HR 0.0188)
Day 17 (n=6)	0.0074 (HR 0.0137)
Day 21 (n=6)	<LOQ (0.0095)
Day 24 (n=6)	0.0086 (HR 0.0175)
Day 28 (n=6)	0.0052 (HR 0.0104)
Dep. 35 (+7) (n=3)	<LOQ
Dep. 42 (+14) (n=2)	<LOQ
Dep. 49 (+21) (n=1)	<LOQ
Mean day 7-28	
Max day 7-28	
Cream – day 2 (n=3)	0.0186
Cream – day 14 (n=3)	0.0200
Cream – day 28 (n=3)	0.0105
Skim milk – day 2 (n=3)	<LOQ
Skim milk – day 14 (n=3)	<LOQ
Skim milk – day 28 (n=3)	<LOQ

Notes:

LOQ = 0.005 mg/kg.

[a] Expressed as fluindapyr.

[b] Mean average of 6 cows, except for the depuration group, which lasted 3, 2 or 1 cows.

Quantifiable residues of fluindapyr were found in liver and fat tissues from the high and medium dose groups. Mean residues of fluindapyr found in fat samples ranged from 0.0324 to 0.0406 mg/kg in

the high dose group; mean residues in the medium dose group were <LOQ (0.010 mg/kg). Mean fluindapyr residues in liver samples from the high and medium dose groups were 0.0454 mg/kg and 0.0165 mg/kg, respectively. No quantifiable residues of fluindapyr were found in any of the low dose group tissues or in the kidney or muscle tissues from the medium and high dose groups, or in the fat samples from the medium dose group.

Quantifiable residues of 1-OH-Met-fluindapyr were found in the high, medium and low dose group samples of tissues. The highest mean residues were found in the perirenal fat at 0.0486, 0.0138 and 0.0118 mg/kg for the high, medium and low dose groups, respectively.

Liver and kidney samples were also analysed for 1-hydroxymethyl-N-desmethyl-fluindapyr and fluindapyr-1-COOH. Quantifiable 1-OH-Met-N-DesMet-fluindapyr residues were detected in all three dose groups; the highest mean residue of 0.259 mg/kg being found in kidney at the high dose group. fluindapyr-1-COOH was not quantifiable in the low dose group samples, but was detected at a maximum of 0.0291 mg/kg in kidney from the high dose group.

Fluindapyr and its metabolite residues were all below the LOQ by the first sampling interval of the depuration phase (day 35). No quantifiable residues of parent (>LOQ) were found in any of the milk, liver, kidney, muscle of fat samples during the depuration phase. No detectable 1-OH-Met-fluindapyr residues were found in any of the samples from the depuration phase (liver, kidney, fat). No quantifiable residues of 1-OH-N-DesMet-fluindapyr or fluindapyr-1-COOH were found in any kidney or liver samples from the depuration phase.

Levels of parent compound in skim milk (not detectable on day 2–28) and cream (0.011–0.020 mg/kg day 2–16) of animals given the highest feeding level indicate that parent compound has a tendency to concentrate in fat. The concentration of parent in fat compared to muscle was >3, also indicating a tendency to concentrate in fat. A similar profile was seen for 1-OH-Met-fluindapyr in muscle (not detectable) and fat tissues (0.015–0.049 mg/kg) at day 28. All residues levels declined rapidly and were <LOQ on day 7 of the depuration period.

Table 132 Fluindapyr related residues in cow tissues for 05.09, 15.25 and 48.06 ppm groups

Sample	Dose rate (ppm feed)	Day of sampling	parent (mg/kg)	F-1-COOH [a]	1-OH-Met-N-DesMet-F [a]	1-OH-Met-F [a]	mean parent (mg/kg) [b]	total parent eq, (mg/kg) [c]	mean total parent eq, (mg/kg) [c]	Parent + 1-OH_Met-F [a,e]
Liver	5.09	28	<0.01	<0.01	0.0294	0.0256	<0.01	0.075	0.0646	0.0356
		28	<0.01	<0.01	0.0131	0.0122		0.0453		0.0222
		28	<0.01	<0.01	0.0245	0.0291		0.0736		0.0391
	15.25	28	0.0126	<0.01	0.0367	0.0333	0.0165	0.0926	0.1265	0.0459
		28	0.0163	<0.01	0.0493	0.0559		0.1315		0.0722
		28	0.0207	<0.01	0.0761	0.0486		0.1554		0.0693
	48.06	28	0.0535	0.0375	0.2538	0.2459	0.0515	0.5907	0.4894	0.2994
		28	0.0602	0.0190	0.2064	0.1761		0.4617		0.2363
		28	0.0408	0.0241	0.1776	0.1733		0.4158		0.2141
	depuration	35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.040	<0.040	<0.020
		42	<0.01	<0.01	<0.01	<0.01		<0.040		<0.020
		49	<0.01	<0.01	<0.01	<0.01		<0.040		<0.020
Kidney	5.09	28	<0.01	<0.01	0.0202	0.0187	<0.01	0.0589	0.0725	0.0287
		28	<0.01	<0.01	0.0213	0.0284		0.0697		0.0384
		28	<0.01	<0.01	0.0400	0.0290		0.089		0.039
	15.25	28	<0.01	0.0138	0.1671	0.1179	<0.01	0.3088	0.2704	0.1279
		28	<0.01	<0.01	0.0883	0.0837		0.192		0.0937

Sample	Dose rate (ppm feed)	Day of sampling	parent (mg/kg)	F-1-COOH [a]	1-OH-Met-N-DesMet-F [a]	1-OH-Met-F [a]	mean parent (mg/kg) [b]	total parent eq, (mg/kg) [c]	mean total parent eq, (mg/kg) [c]	Parent + 1-OH_Met-F [a,e]
		28	<0.01	0.0163	0.1710	0.1131		0.3104		0.1231
	48.06	28	<0.01	0.0309	0.2801	0.2639	<0.01	0.5849	0.5022	0.2739
		28	<0.01	0.0289	0.2393	0.1801		0.4583		0.1901
		28	<0.01	0.0274	0.2587	0.1672		0.4633		0.1772
	depuration	35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.040	<0.040	<0.020
		42	<0.01	<0.01	<0.01	<0.01		<0.040		<0.020
		49	<0.01	<0.01	<0.01	<0.01		<0.040		<0.020
Muscle	5.09	28	<0.01	n.a.	n.a.	n.a.	<0.01	<0.040	<0.040	<0.020
		28	<0.01	n.a.	n.a.	n.a.		<0.040		<0.020
		28	<0.01	n.a.	n.a.	n.a.		<0.040		<0.020
	15.25	28	<0.01	n.a.	n.a.	n.a.	<0.01	<0.040	<0.040	<0.020
		28	<0.01	n.a.	n.a.	n.a.		<0.040		<0.020
		28	<0.01	n.a.	n.a.	n.a.		<0.040		<0.020
	48.06	28	<0.01	n.a.	n.a.	n.a.	<0.01	<0.040	<0.040	<0.020
		28	<0.01	n.a.	n.a.	n.a.		<0.040		<0.020
		28	<0.01	n.a.	n.a.	n.a.		<0.040		<0.020
	depuration	35	<0.01	n.a.	n.a.	n.a.	<0.01	<0.040	<0.040	<0.020
		42	<0.01	n.a.	n.a.	n.a.		<0.040		<0.020
		49	<0.01	n.a.	n.a.	n.a.		<0.040		<0.020
Fat (mesenterial)	5.09	28	<0.01	n.a.	n.a.	<0.01	<0.01	<0.040	<0.040	<0.020
		28	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
		28	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
	15.25	28	0.0111	n.a.	n.a.	<0.01	0.0113	0.0411	0.0414	0.0211
		28	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
		28	0.0130	n.a.	n.a.	<0.01		0.043		0.023
	48.06	28	0.0632	n.a.	n.a.	0.0168	0.0421	0.1	0.0767	0.08
		28	<0.01	n.a.	n.a.	0.0169		0.0469		0.0269
		28	0.0530	n.a.	n.a.	0.0103		0.0833		0.0633
	depuration	35	<0.01	n.a.	n.a.	<0.01	<0.01	<0.040	<0.040	<0.020
		42	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
		49	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
Fat (perirenal)	5.09	28	<0.01	n.a.	n.a.	0.011	<0.01	0.041	0.04406667	0.021
		28	<0.01	n.a.	n.a.	0.0212		0.0512		0.0312
		28	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
	15.25	28	<0.01	n.a.	n.a.	<0.01	<0.01	<0.040	0.04716667	<0.020
		28	<0.01	n.a.	n.a.	0.0238		0.0538		0.0338
		28	0.0177	n.a.	n.a.	<0.01		0.0477		0.0277
	48.06	28	0.0507	n.a.	n.a.	0.0255	0.0338	0.0962	0.10233333	0.0762
		28	<0.01	n.a.	n.a.	0.1037		0.1337		0.1137
		28	0.0406	n.a.	n.a.	0.0165		0.0771		0.0571
	depuration	35	<0.01	n.a.	n.a.	<0.01	<0.01	<0.040	<0.040	<0.020
		42	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
		49	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
Fat (subcut)	5.09	28	<0.01	n.a.	n.a.	<0.01	<0.01	<0.040	<0.040	<0.020
		28	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
		28	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
	15.25	28	<0.01	n.a.	n.a.	<0.01	<0.01	<0.040	0.0401	<0.020
		28	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
		28	0.0103	n.a.	n.a.	<0.01		0.0403		0.0203
	48.06	28	0.049	n.a.	n.a.	0.0226	0.0353	0.0916	0.07113333	0.0716
		28	<0.01	n.a.	n.a.	0.0133		0.0433		0.0233

Sample	Dose rate (ppm feed)	Day of sampling	parent (mg/kg)	F-1-COOH [a]	1-OH-Met-N-DesMet-F [a]	1-OH-Met-F [a]	mean parent (mg/kg) [b]	total parent eq, (mg/kg) [c]	mean total parent eq, (mg/kg) [c]	Parent + 1-OH_Met-F [a,e]
		28	0.0469	n.a.	n.a.	0.0116		0.0785		0.0585
	depuration	35	<0.01	n.a.	n.a.	<0.01	<0.01	<0.040	<0.040	<0.020
		42	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020
		49	<0.01	n.a.	n.a.	<0.01		<0.040		<0.020

Notes:

F-1-COOH = fluindapyr carboxylate; 1-OH-Met-N-DesMet-F = 1-OH-Met-DesMet-fluindapyr; 1-OH-Met-F= 1-OH-Met-fluindapyr

[a] Expressed as parent equivalents.

[b] Mean values are calculated by the reviewer. Where values were <LOQ, 0.01 mg/kg was used for the calculations.

[c] Total residues is the sum of fluindapyr and metabolites F-COOH, 1-OH-Met-DesMet-F, and 1-OH-Met-F expressed as parent equivalents and are used for HR and STMR estimation.

[d] Fluindapyr plus 1-OH-Met-fluindapyr expressed as parent equivalents is used for MRL estimation.

Animal feeding study in laying hens

A residue feeding study in laying hens was conducted in the United States in 2015/2016 [Brungardt & Dixon, 2018, 2016RES-IFP2943] to measure the residues of fluindapyr found in eggs and tissues.

Sixty ISA Brown laying hens (*Gallus gallus domesticus*; 12 hens each for the control, low, and mid dose groups and twenty-four hens for the high dose group) were used in the study. Each group of hens was subdivided into subgroups of four hens each. All hens were dosed orally, via capsule, for 36 consecutive days with fluindapyr at actual dose rates of 2.064, 6.231, and 20.596 ppm in feed, corresponding with 0.108, 0.328, and 1.102 mg ai/kg bw/day.

Animals were observed twice daily for any clinical signs of toxicity or ill health. Bodyweights were determined at intervals and group feed consumption was calculated weekly for each group.

The birds weighed on 1562–2345 gram on study day 1 and between 1585 and 2447 gram on study day 36. Egg production appeared to be consistent throughout the study and did not appear to be affected by treatment with the test substance. Average egg production values were comparable in the test and control groups throughout the study.

Eggs for residue analysis were collected beginning on study day -1 and continuing through the dosing period and depuration phase. Egg samples (pooled AM and PM) were collected from all dose subgroups on Study Days -1, 1, 3, 7, 10, 14, 17, 21, 28, 32, and 35. Additionally, on study days 2, 9, 16 and 27 eggs were collected from the hens in the control and high dose subgroups that were not utilized in the depuration period of the study. Whole eggs were separated for analysis into egg yolk and egg white fractions. On Study Day 36, within approximately 6 hours of the last dose, one of the control subgroups (subgroup A), all of the low dose group (subgroups A, B and C), all of the mid dose group (subgroups A, B and C), and three subgroups of the high dose group hens (subgroups A, B and C) were sacrificed. Liver, muscle and fat were collected from each hen and pooled by subgroup for analysis.

The remaining hens (two control subgroups (B and F) and three high dose subgroups (D, E and F)) entered into the 11-day depuration phase of the study following the last dose. Egg samples (pooled AM and PM) for residue analysis were collected from each subgroup (remaining at that time) on study days 39, 42 and 46 of the depuration period. On study day 40, the hens from subgroup D of the high dose group were sacrificed and the tissues (liver, muscle and fat) were collected. On study day 43, the hens from

subgroup E of the high dose group were sacrificed and the tissues were collected. On study day 47, the hens from subgroup F of the control group and subgroup F of the high dose group were sacrificed and the tissues were collected. The remaining control hens (subgroup B) were not sacrificed.

Samples were stored at ca. -18 °C until they were extracted. Whole egg samples were extracted within 36 days of collection, while egg white and yolk samples were extracted within 40 days of collection. Samples of liver, muscle, and fat were extracted within 76, 51, and 52 days after collection, respectively. Freezer storage stability data demonstrated that residues of fluindapyr and metabolites are stable in fortified samples of eggs at -18 °C for up to 64 days. Freezer storage stability for fortified samples of liver, muscle, and fat were demonstrated for up to 91, 55, and 70 days, respectively.

Analytical method SRLS133SRUS16R0208 as validated in study 2016RES-IFP2941 (see section on analytical methods) was used. The method uses an additional hydrolysis step before analysis of the unconjugated metabolites. Eggs, egg whites, egg yolks, and liver samples were analysed for the parent compound fluindapyr and metabolites N-DesMet-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and 1-OH-Met-fluindapyr. Fat samples were analysed for fluindapyr and 1-OH-Met-fluindapyr, and muscle samples were analysed for fluindapyr alone. The reported residue values were calculated in parent equivalents and were not corrected for recoveries. Residues were quantitated by high performance liquid chromatography/electrospray ionization/tandem mass spectrometry (ESI LC-MS/MS). The LOQ for parent and all metabolites was 0.010 mg/kg in all relevant matrices. Average concurrent fresh recoveries in eggs and relevant poultry tissues were within the range of 70–110 percent for fluindapyr and its metabolites. Control samples had residues below 0.2LOQ.

Analytical results in eggs and tissue samples are shown in Table 133 and Table 134.

Residues of fluindapyr reached a plateau in eggs (group 4, 20.6 mg/kg dose group) after approximately 7 days. Residues of fluindapyr in egg whites were <LOQ at all sampling intervals, while average fluindapyr residues in egg yolks reached a maximum of 0.040 mg/kg (day 9). Residues in eggs were predominately parent fluindapyr with average residues of metabolites N-DesMet-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-OH-Met-fluindapyr at or below the LOQ at all sampling intervals.

Table 133 Average residues in eggs dosed with fluindapyr for 28 days at 48.06 mg/kg in feed

Dose mg/kg	Matrix	Sampling day	Average residues, mg/kg					
			Fluindapyr	N-DesMet-F	1-OH-Met-N-DesMet-F [a]	1-OH-Met-F [a]	Total residue for STMR and HR [b]	Residue for MRL [c]
6.23	whole egg (n=3/day)	-1	<0.01	<0.01	<0.01	<0.01	<0.040	<0.020
		1	<0.01	<0.01	<0.01	<0.01	<0.040	<0.020
		3	<0.01	<0.01	<0.01	<0.010	<0.040	<0.020
		7	<0.010	<0.01	<0.01	<0.010	<0.040	<0.020
		10	<0.010	<0.01	<0.01	<0.010	<0.040	<0.020
		14	<0.010	<0.01	<0.01	<0.010	<0.040	<0.020
		17	<0.010	<0.01	<0.01	0.010	<0.040	<0.020
		21	<0.010	<0.01	<0.01	<0.010	<0.040	<0.020
		28	<0.010	<0.01	<0.01	0.010	<0.040	<0.020
		32	<0.010	<0.01	<0.01	0.012	0.042	0.022
		35	<0.010	<0.01	<0.01	0.012	0.042	0.022
20.6	whole egg (n=6/day)	-1	<0.01	<0.01	<0.01	<0.01	<0.040	<0.020
		1	<0.01	<0.01	<0.01	<0.010	<0.040	<0.020
		3	<0.010	<0.01	<0.01	0.020	0.05	0.03
		7	0.013	<0.01	<0.01	0.032	0.065	0.045
		10	0.012	<0.01	<0.01	0.028	0.06	0.04

Dose mg/kg	Matrix	Sampling day	Average residues, mg/kg					
			Fluindapyr	N-DesMet-F	1-OH-Met-N-DesMet-F [a]	1-OH-Met-F [a]	Total residue for STMR and HR [b]	Residue for MRL [c]
		14	0.011	<0.01	<0.01	0.028	0.059	0.039
		17	0.012	<0.01	<0.01	0.032	0.064	0.044
		21	0.014	<0.01	<0.01	0.033	0.067	0.047
		28	0.014	<0.01	<0.01	0.032	0.066	0.046
		32	0.014	<0.01	<0.01	0.034	0.068	0.048
		35	0.016	<0.01	<0.01	0.034	0.07	0.05
		39 [d]	<0.010	<0.01	<0.01	0.013	0.043	0.023
		42 [d]	<0.010	<0.01	<0.01	<0.010	<0.040	<0.020
		46 [d]	<0.01	<0.01	<0.01	<0.01	<0.040	<0.020
20.6	Egg white (n=3/day)	2	<0.010	<0.01	<0.01	0.016	0.046	0.026
		9	<0.010	<0.01	<0.01	0.015	0.045	0.025
		16	<0.010	<0.01	<0.01	0.016	0.046	0.026
		27	<0.010	<0.01	<0.01	0.012	0.042	0.022
	Egg yolk (n=3/day)	2	<0.010	<0.01	<0.01	0.019	0.049	0.029
		9	0.040	0.011	<0.010	0.066	0.13	0.11
		16	0.032	<0.010	<0.010	0.058	0.11	0.09
		27	0.039	0.010	<0.010	0.058	0.12	0.097

Notes:

N-DesMet-F = N-desmethyl-fluindapyr; 1-OH-Met-N-DesMet-F = 1-hydroxymethyl-N-desmethyl-fluindapyr; 1-OH-Met-fluindapyr = 1-hydroxymethyl-fluindapyr.

[a] Sum of diastereomers.

[b] Total fluindapyr residue is the sum of the residues of fluindapyr and metabolites N-DesMet-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-OH-Met-fluindapyr expressed in parent equivalents and is used for HR and STMR estimation.

[c] Total fluindapyr residues for calculation of MRL consisting of parent fluindapyr + 1-OH-Met-fluindapyr expressed as parents equivalents.

[d] Samples at days 39, 42, and 46 were collected 4, 6, and 10 days after cessation of dosing, respectively, with n = 3, 2 and 1, respectively.

In muscle, average residues of fluindapyr were 0.012 mg/kg in group 4 (20.6 mg/kg dose) while fluindapyr residues were <LOQ in group 3 (6.23 mg/kg dose) and group 2 (2.06 mg/kg dose).

In liver, average residues of fluindapyr were 0.029 mg/kg, 0.012 mg/kg, and <0.010 mg/kg in groups 4, 3, and 2, respectively. Average residues of N-DesMet-fluindapyr were 0.174 mg/kg, 0.070 mg/kg, and 0.022 mg/kg in groups 4, 3, and 2, respectively. Average residues of 1-OH-Met-N-DesMet-fluindapyr were 0.0271 mg/kg in group 4 and <0.010 mg/kg in groups 2 and 3. Average residues of 1-OH-Met-fluindapyr were 0.178 mg/kg, 0.041 mg/kg, and 0.030 mg/kg in groups 4, 3, and 2, respectively. Total average residues (parent plus metabolites) in liver were 0.409 mg/kg, 0.167 mg/kg, and 0.060 mg/kg in groups 4, 3, and 2, respectively.

In fat, average residues of fluindapyr were 0.080 mg/kg, 0.029 mg/kg, and <0.010 mg/kg in groups 4, 3, and 2, respectively. Average residues of 1-OH-Met-fluindapyr were 0.064 mg/kg, 0.025 mg/kg, and <0.010 mg/kg in groups 4, 3, and 2, respectively. Total average residues (parent plus metabolites) in fat were 0.167 mg/kg, 0.054 mg/kg, and 0.015 mg/kg in groups 4, 3, and 2, respectively.

Following cessation of dosing residues rapidly declined. In all matrices, all residues were <LOQ by 4 days after the last dose.

Table 134 Fluindapyr related residues in tissues from poultry fed 2.06, 6.23, or 20.6 ppm fluindapyr

Sample	Dose rate (ppm feed)	Day of sampling	Fluindapyr	N-DesMet-F [a]	1-OH-Met-N-DesMet-F [a,b]	1-OH-Met-F [a,b]	mean parent (mg/kg) [c]	total parent eq, (mg/kg) [d]	mean total parent eq, (mg/kg) [d]	Parent + 1-OH_Met-F [a,e]
Liver	2.06	36	<0.010	0.0269	<0.010	0.0389	0.0302	0.0858	0.0723	0.0489
		36	<0.010	0.0201	<0.010	0.0372		0.0773		0.0472
		36	<0.010	0.0192	<0.010	0.0145		0.0537		0.0245
	6.23	36	0.0165	0.0928	0.0101	0.1106	0.0774	0.23	0.1698	0.1271
		36	<0.010	0.0457	<0.010	0.0460		0.1117		0.056
		36	0.0109	0.0713	<0.010	0.0756		0.1678		0.0865
	20.6	36	0.0364	0.2381	0.0270	0.1299	0.178	0.4314	0.4088	0.1663
		36	0.0226	0.1310	0.0225	0.1740		0.3501		0.1966
		36	0.0276	0.1543	0.0318	0.2312		0.4449		0.2588
	depuration	40	<0.010	<0.010	<0.010	<0.010	<0.010	<0.040	<0.040	0.02
		43	<0.010	<0.010	<0.010	<0.010		<0.040		0.02
		46	<0.010	<0.010	<0.010	<0.010		<0.040		0.02
Muscle	2.06	36	<0.010	n.a.	n.a.	n.a.	<0.010	<0.040	<0.040	0.02
		36	<0.010	n.a.	n.a.	n.a.		<0.040		0.02
		36	<0.010	n.a.	n.a.	n.a.		<0.040		0.02
	6.23	36	<0.010	n.a.	n.a.	n.a.	<0.010	<0.040	<0.040	0.02
		36	<0.010	n.a.	n.a.	n.a.		<0.040		0.02
		36	<0.010	n.a.	n.a.	n.a.		<0.040		0.02
	20.6	36	0.0130	n.a.	n.a.	n.a.	0.01	0.043	0.0421	0.023
		36	<0.010	n.a.	n.a.	n.a.		<0.040		0.02
		36	0.0132	n.a.	n.a.	n.a.		0.0432		0.0232
	depuration	40	<0.010	n.a.	n.a.	n.a.	<0.010	<0.04	<0.040	0.02
		43	<0.010	n.a.	n.a.	n.a.		<0.04		0.02
		46	<0.010	n.a.	n.a.	n.a.		<0.04		0.02
Fat	2.06	36	<0.010	n.a.	n.a.	0.0104	<0.010	0.0404	0.0401	0.0204
		36	<0.010	n.a.	n.a.	<0.010		<0.04		0.02
		36	<0.010	n.a.	n.a.	<0.010		<0.04		0.02
	6.23	36	0.0269	n.a.	n.a.	0.0318	0.0254	0.0787	0.0742	0.0587
		36	0.0297	n.a.	n.a.	0.0182		0.0679		0.0479
		36	0.0297	n.a.	n.a.	0.0262		0.0759		0.0559
	20.6	36	0.1026	n.a.	n.a.	0.0715	0.0639	0.1941	0.1638	0.1741
		36	0.0476	n.a.	n.a.	0.0491		0.1167		0.0967
		36	0.0895	n.a.	n.a.	0.0711		0.1806		0.1606
	depuration	40	<0.010	n.a.	n.a.	<0.010	<0.010	<0.040	<0.040	0.02
		43	<0.010	n.a.	n.a.	<0.010		<0.040		0.02
		46	<0.010	n.a.	n.a.	<0.010		<0.040		0.02

Notes:

N-DesMet-F = N-desmethyl-fluindapyr; 1-OH-Met-DesMet-F = 1-hydroxymethyl-N-desmethyl-fluindapyr; 1-OH-Met-fluindapyr = 1-hydroxymethyl-fluindapyr.

[a] Expressed in fluindapyr equivalents.

[b] Sum of diastereomers.

[b] Mean values are calculated by the reviewer. Where values were <LOQ, 0.01 mg/kg was used for the calculations.

[c] Total fluindapyr residue is the sum of the residues of fluindapyr and metabolites N-DesMet-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-OH-Met-fluindapyr expressed in parent equivalents and is used for HR and STMR estimation.

[d] Fluindapyr plus 1-OH-Met-fluindapyr expressed in parent equivalents is used for MRL estimation.

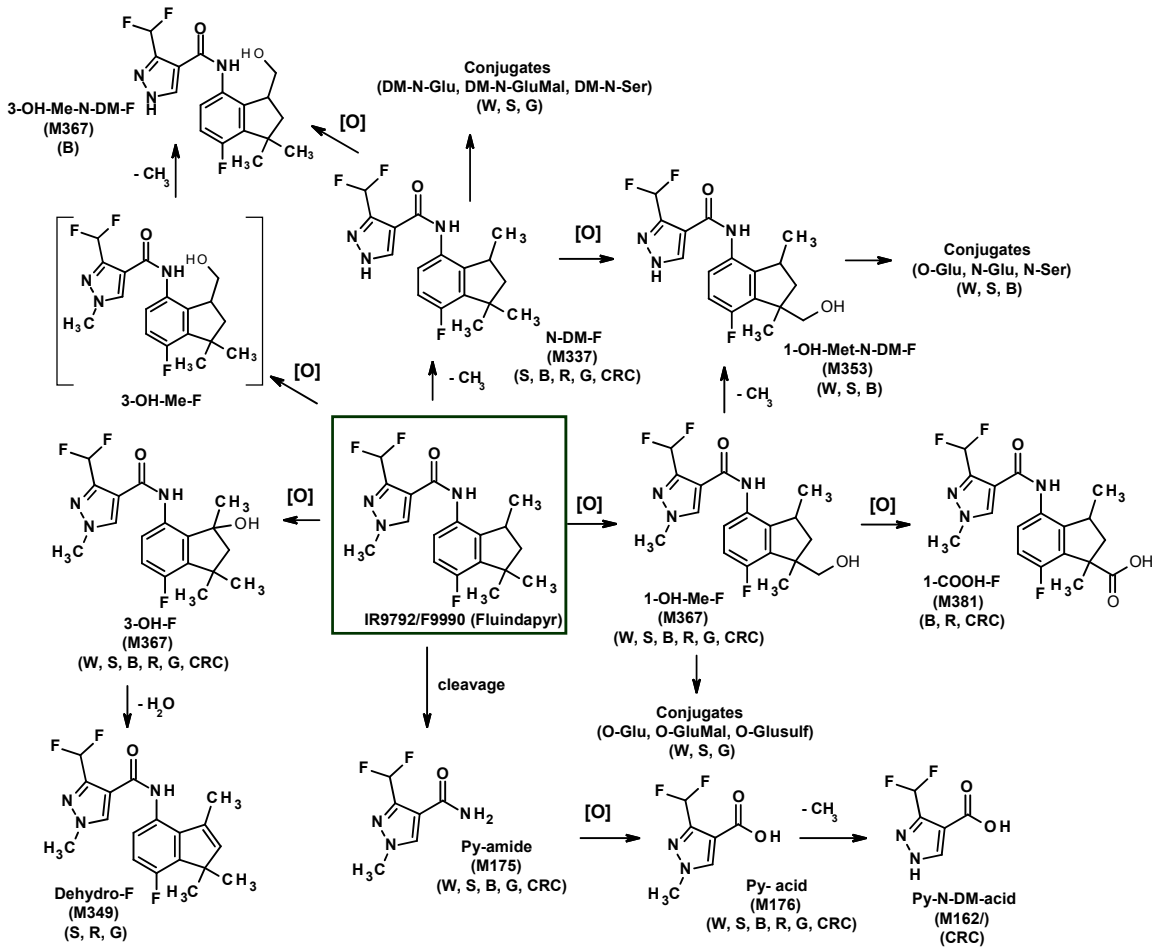
NATIONAL RESIDUE DEFINITION

Fluindapyr is registered in the United States with the following residue definitions:

Commodity	Enforcement (US EPA)	Risk assessment (US EPA)
Primary Crop	Fluindapyr	Sum of fluindapyr (F) plus 3-OH-F, 1-OH-Met-F, 1-OH-Met-F-O-glucoside, DesMet-F-N-glucoside, 1-OH-Met-DesMet-F, and 1-COOH-F
Ruminant meat, fat, milk	Fluindapyr	Sum of fluindapyr plus 1-OH-Met-F, 1-COOH-F, and 1-OH-Met-DM-F
Ruminant edible offal	Sum of fluindapyr and 1-OH-Met-fluindapyr	
Poultry meat, fat, eggs	Fluindapyr	Sum of fluindapyr plus 1-OH-Met-F, 1-OH-Met-DM-F, and DesMet-F
Poultry edible offal	Sum of fluindapyr and 1-OH-Met-fluindapyr	

Notes:

EPA Document ID EPA-HQ-OPP-2018-0551-0020 "Fluindapyr: Human Health Risk Assessment for Section 3 Registration and Tolerance Requests for a New Active Ingredient Proposed for a Use on Cereal Grains Crop Group 15 except Rice; Forage, Fodder and Straw of Cereal Grains Crop Group 16; Nut, Tree, Group 14-12; Soybean; Ornamentals; and Turf."



F: fluindapyr; Me: methyl; DM: desmethyl; py: pyrazole; Glu: glucoside; GluMal: malonylglucoside; Ser: serine; Glusulf: glucosyl sulfate
M: Mol. Wt.
Primary crops: W: wheat, S: soybean; B: sugarbeet; R: rice, G: grape
CRC: confined rotational crops (carrot, lettuce and wheat)

Figure 14 Proposed metabolic pathways of fluindapyr (F9990/IR9792) in primary and rotational crops

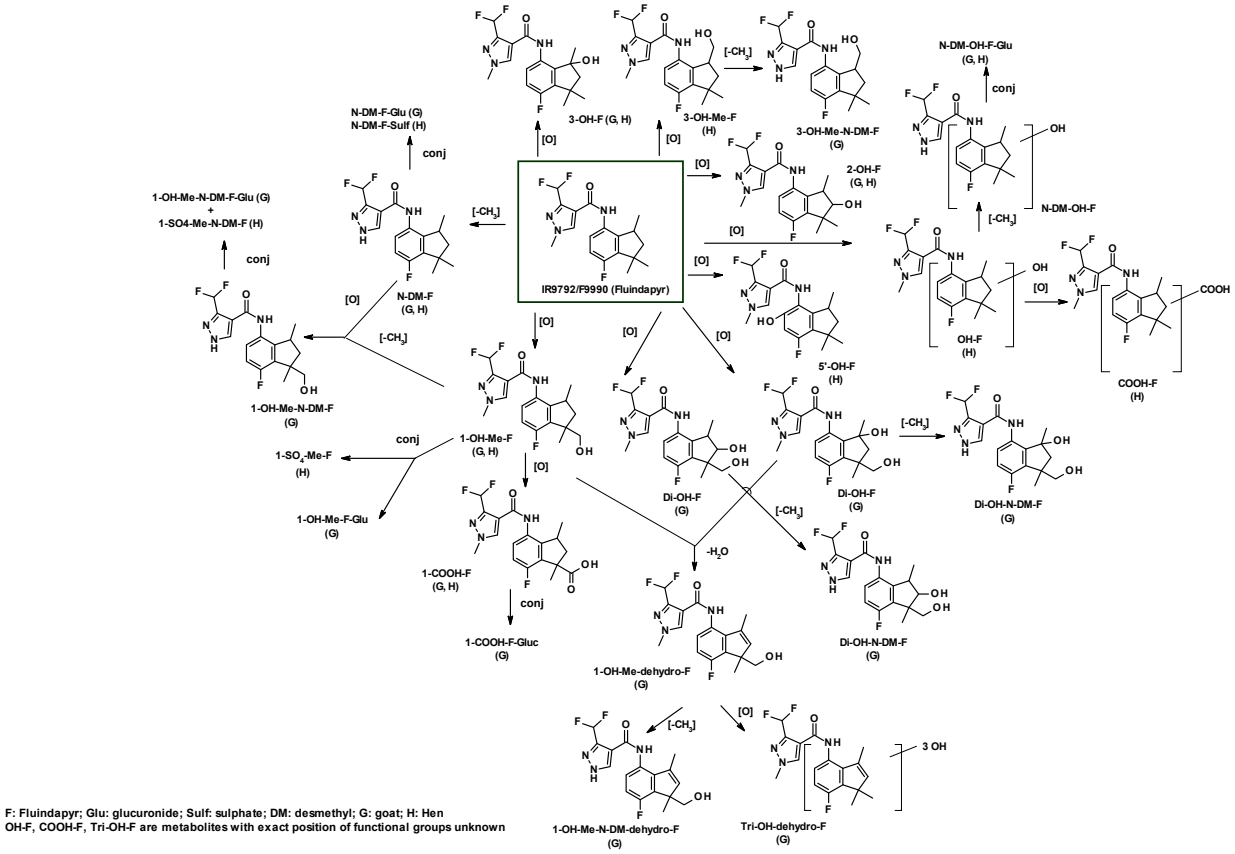


Figure 15 Proposed metabolic pathways of fluindapyr in livestock animals (lactating goat and laying hen)

APPRAISAL

Fluindapyr (ISO common name) is a broad spectrum fungicide, which belongs to the succinate dehydrogenase inhibitors (SDHI) class of compounds. The mode of action is inhibition of the energy production process in pathogenic fungi.

Fluindapyr was scheduled at the Fifty-first Session of the CCPR for evaluation as a new compound by the 2021 JMPR and rescheduled for evaluation by the 2022 JMPR. The Meeting received information on identity, physical chemical properties, plant and animal metabolism, soil degradation, residue analysis, storage stability, use patterns, residues resulting from supervised trials on wheat, sorghum, maize, rice, almonds and pecan nuts, fate of residues during processing, and livestock feeding studies.

The IUPAC name for fluindapyr is 3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-2,3-dihydro-1H-inden-4-yl)-1-methyl-1H-pyrazole-4-carboxamide.

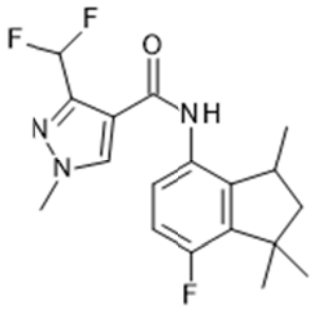
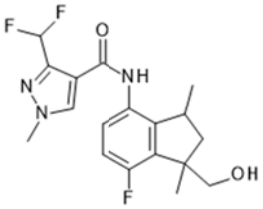
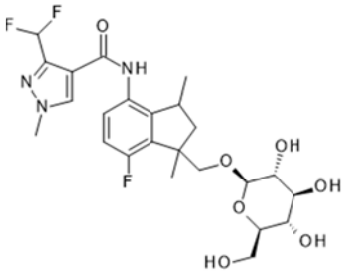
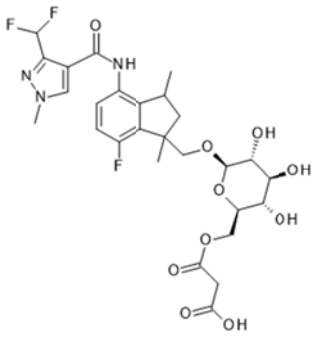
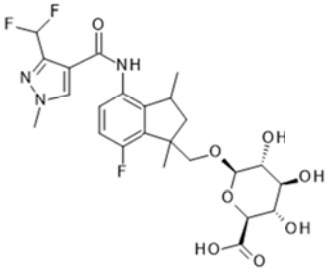
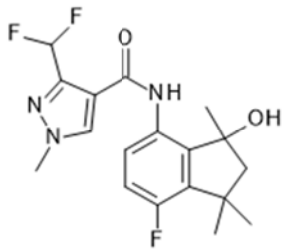
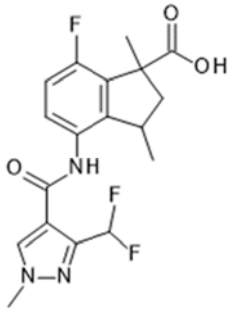
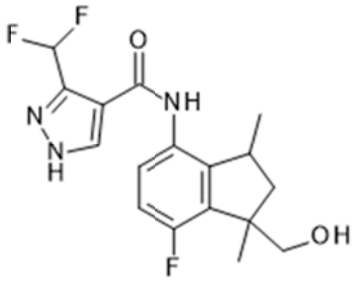
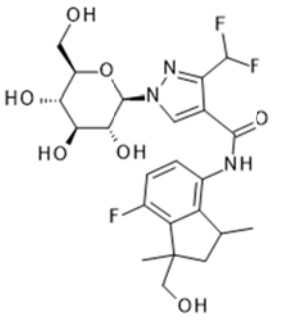
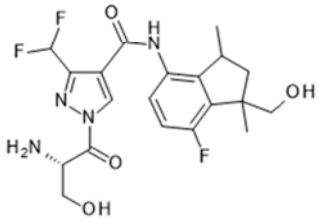
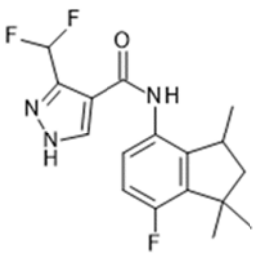
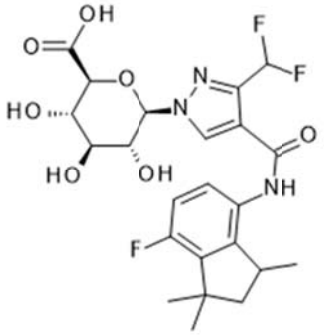
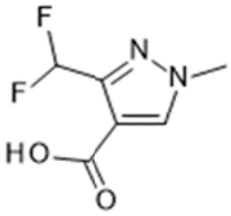
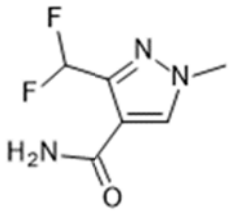
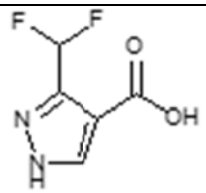
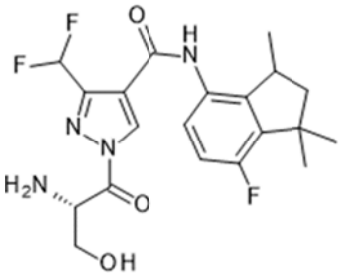
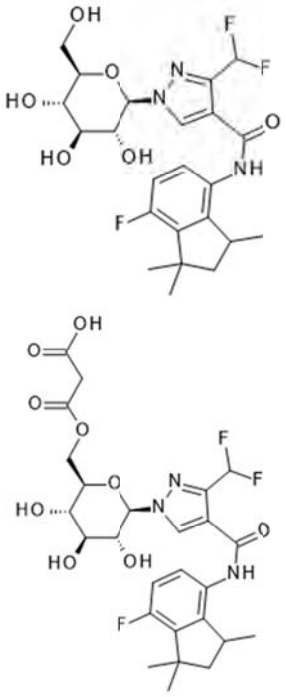
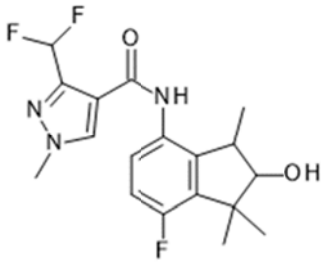
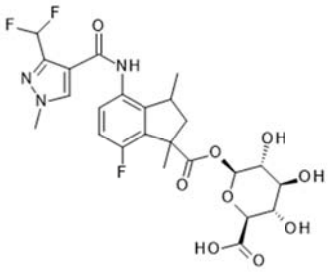
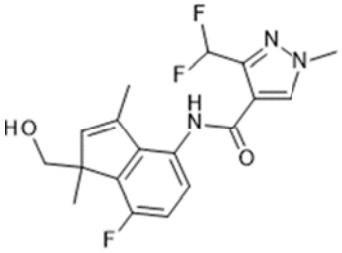


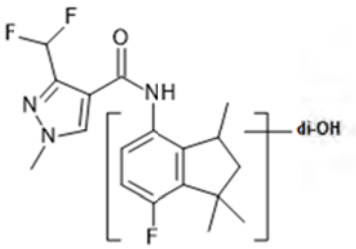
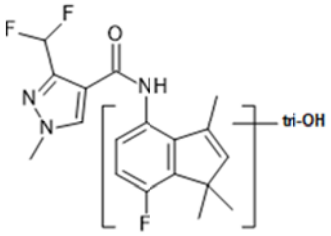
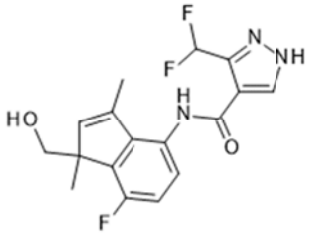
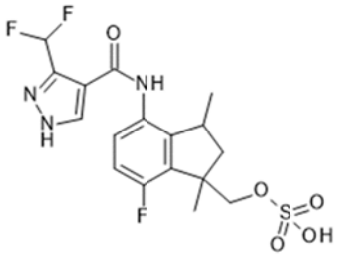
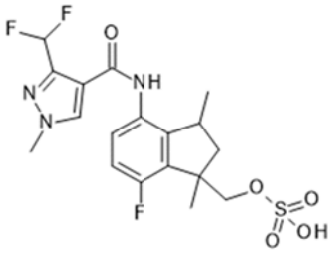
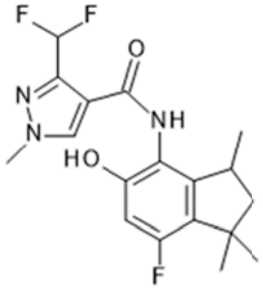
Table 135 Abbreviations used for relevant compounds referred to in the appraisal

Code	Name and Matrix	Structure
1-OH-Met-fluindapyr (Code: 510153) 1-Hydroxymethyl-fluindapyr 1-OH-Met-F M24, M26 in animal studies	3-(difluoromethyl)-N-[7-fluoro-1-(hydroxymethyl)-1,3-dimethyl- 2,3-dihydro-1H-inden-4-yl]-1-methyl-1H-pyrazole-4-carboxamide MW = 367 g/mol Found in: goat (various tissues), hen (all tissues), rat, primary crops (grape, sugar beet, wheat, rice) and rotational (carrot, lettuce, wheat) crops	
1-OH-Met-fluindapyr-Glu 1-Hydroxymethyl-fluindapyr glucoside (plant) 1-Hydroxymethyl-fluindapyr gluc-mal (plant) 1-Hydroxymethyl-fluindapyr glucuronide (animals) M13, M15 in animal studies	3-(difluoromethyl)-N-(7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1H-inden-4-yl)-1-methyl-1H-pyrazole-4-carboxamide gluc MW-Glu= 529 g/mol MW-Glu-Mal = 615 g/mol MW-Glucuronide = 544 g/mol Found in: goat liver, primary crops (grapes fruit and leaves), wheat (forage, hay, straw), soya bean (forage and hay)	  

Code	Name and Matrix	Structure
3-OH-fluindapyr (Code:510152) 3-Hydroxy-fluindapyr M34 in animal studies	3-(difluoromethyl)-N-(7-fluoro-3-hydroxy-1,1,3-trimethyl-2,3-dihydro-1H-inden-4-yl)-1-methyl-1H-pyrazole-4-carboxamide MW = 367 g/mol Found in: some goat tissues, primary crops (grape fruit and leaves, wheat matrices, rice, and limited in sugar beet and rice) and rotational crops	
1-COOH-fluindapyr (Code: 510216) 1-Carboxy-fluindapyr M23, M25 in animal studies Diastereoisomer ratio = 1.72:1	4-(3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamido)-7-fluoro-1,3-dimethyl-2,3-dihydro-1H-indene-1-carboxylic acid MW = 381 g/mol Found in: goat liver and kidney and hen liver and muscle and rat metabolite. Limited amounts in primary crops (sugar beet roots and rice grain and straw) and rotational crops	
1-OH-Met-N-DesMet-fluindapyr (Code: 510215) 1-Hydroxymethyl-N-Desmethyl-fluindapyr M18, M19 in animal studies Diastereoisomer ratio = 2.2:1	3-(difluoromethyl)-N-[7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1H-inden-4-yl]-1H-pyrazole-4-carboxamide MW = 353 g/mol Found in limited amounts in goat muscle and milk and in very limited amounts in primary crops (sugar beet roots and leaves, forage, hay and wheat forage, hay and straw after hydrolysis, indicating that it is the glucosyl conjugate)	
1-OH-Met-N-DesMet-fluindapyr-gluc (Code: N/A) 1-hydroxymethyl-N-desmethyl-fluindapyr glucoside (plant) 1-hydroxymethyl N-desmethyl-fluindapyr-glucuronide (animal) M10 and M11 in animal metabolism studies	3-(difluoromethyl)-N-(7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1H-inden-4-yl)-1H-pyrazole-4-carboxamide gluc MW-N-Ser-conjugate = 440 g/mol MW-N-Glu-conjugate = 515 g/mol Found in: goat liver and kidney and in limited amounts in wheat forage and hay and in soya bean and hay.	 
N-DesMet-fluindapyr (Code: 510220) N-Desmethyl-fluindapyr M33 in animal studies	3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-2,3-dihydro-1H-inden-4-yl)-1H-pyrazole-4-carboxamide MW=337 g/mol Found in: very limited amounts in primary crops, rotational crops, and some goat tissues (fat, muscle, and cream), and all hen matrices.	

Code	Name and Matrix	Structure
N-DesMet-fluindapyr-glu (Code: N/A) N-Desmethyl-fluindapyr-glucuronide M6 and M7 in animal studies	3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-2,3-dihydro-1H-inden-4-yl)-1H-pyrazole-4-carboxamide glucuronide MW = 513 g/mol Found in very limited amounts in goat kidney	
Pyrazole carboxylic acid (Code: 510147)	3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxylic acid MW = 176 g/mol Found in very limited amounts in wheat feed matrices, grape fruit and leaves and rice grain and straw.	
Pyrazole carboxamide (Code: 510151)	3- (difluoromethyl)- 1-methyl-1H-pyrazole-4-carboxamide MW = 175 g/mol Found in very limited amounts in wheat feed matrices, soya bean forage, sugar beet foliage and grape fruit and leaves.	
N-DesMet-pyrazole carboxylic acid (Code: 510219)	3- (difluoromethyl)- 1H-pyrazole-4- carboxylic acid MW = 162 g/mol Found in very limited amounts in sugar beet root and in some rotational crops	
N-DesMet-fluindapyr-N-ser (Code: N/A)	1-(2-amino-3-hydroxy-propanoyl)-3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-indan-4-yl)pyrazole-4-carboxamide MW = 424 g/mol Found in soya bean forage and hay	

Code	Name and Matrix	Structure
N-DesMet-fluindapyr-N1-Glu (Code: 510171) and DesMet-fluindapyr-N1-Glu-Mal (Code: N/A)	3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-2,3-dihydro-1H-inden-4-yl)-1H-pyrazole-4-carboxamide N1-Glu and Glu-Mal MW = 499 g/mol (glu) MW = 585 g/mol (glu-mal) Found in limited amounts in soya bean forage and hay	
2-OH-fluindapyr (Code: 510321) 2-Hydroxy-fluindapyr M27 in animal metabolism studies	3-(difluoromethyl)-N-(7-fluoro-2-hydroxy-1,1,3-trimethyl-2,3-dihydro-1H-inden-4-yl)-1-methyl-1H-pyrazole-4-carboxamide MW = 367 g/mol Found in limited amounts in all goat and hen tissues	
1-COOH-fluindapyr-glu (Code: N/A) 1-Carboxy-fluindapyr-glucuronide M14 in animal metabolism studies	4-(3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamido)-7-fluoro-1,3-dimethyl-2,3-dihydro-1H-indene-1-carboxylic acid glucuronide MW = 557 g/mol Found in goat liver and trace in kidney	
1-OH-Met-dehydro-fluindapyr (Code: N/A) 1-hydroxymethyl-dehydro-fluindapyr M20 in animal metabolism studies	3-(difluoromethyl)-N-(7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-1H-inden-4-yl)-1-methyl-1H-pyrazole-4-carboxamide MW = 365 g/mol Found in skimmed milk	

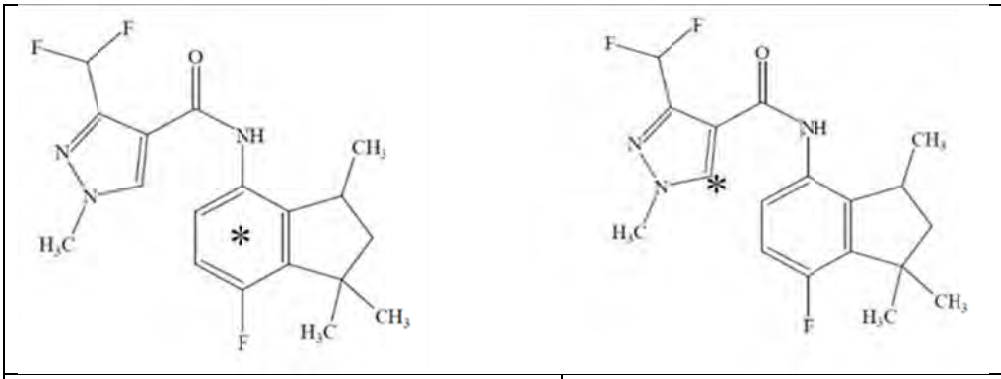
Code	Name and Matrix	Structure
Di-OH-fluindapyr (Code: N/A) Dihydroxy-fluindapyr M4 and M8 in animal metabolism studies	Chemical name: N/A MW = 383 g/mol Found in skimmed milk and goat muscle.	
Tri-OH-dehydro-fluindapyr (Code: N/A) Trihydroxy-dehydro fluindapyr M2 and M3 in animal metabolism studies	Chemical name: N/A MW = 397 g/mol Found in skimmed milk.	
1-OH-Met-N-DesMet-dehydro-fluindapyr (Code: N/A) 1-hydroxymethyl-N-desmethyl-dehydro-fluindapyr M12 in animal metabolism studies	3-(difluoromethyl)-N-(7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-1H-inden-4-yl)-1H-pyrazole-4-carboxamide MW = 351 g/mol Found in skimmed milk and very limited amounts in goat muscle.	
1-SO_4-Met-N-DesMet-fluindapyr (Code: N/A) M39 and M40 in hen metabolism study	[4-[[3-(difluoromethyl)-1H-pyrazole-4-carbonyl]amino]-7-fluoro-1,3-dimethyl-indan-1-yl]methyl hydrogen sulfate MW = 433 g/mol Found in hen liver	
1-SO_4-Met-fluindapyr (Code: N/A) M41 and M42 in hen metabolism study	[4-[[3-(difluoromethyl)-1-methyl-pyrazole-4-carbonyl]amino]-7-fluoro-1,3-dimethyl-indan-1-yl]methyl hydrogen sulfate Found in hen liver	
5′-OH-fluindapyr (Code: 510217) 5′-Hydroxy-fluindapyr M35 in hen metabolism study	3-(difluoromethyl)-N-(7-fluoro-5-hydroxy-1,1,3-trimethyl-2,3-dihydro-1H-inden-4-yl)-1-methyl-1H-pyrazole-4-carboxamide MW = 367 g/mol Found in at limited amounts in various hen tissues and eggs	

Physical and chemical properties

Fluindapyr is not volatile (2.89×10^{-5} Pa at 20 °C), relatively insoluble in water (1.63 mg/L), but appears to be more soluble in organic solvents (up to 325 g/L in acetone). It is hydrolytically stable at environmental conditions. Photolysis is not a significant route of degradation in water. The octanol/water partition coefficient log P_{ow} of 4.12 suggests a potential to partition into fat.

Plant metabolism

The Meeting received outdoor metabolism studies for fluindapyr after foliar applications, conducted with crops representative of four different groups: fruit (grape), root crops (sugar beet), cereal/grass (wheat and rice), pulses and oilseeds (soya bean). Fluindapyr was applied using [phenyl-^{14}C] and [pyrazole-^{14}C] labelled fluindapyr, as shown below. The results for both labels are presented as phenyl/pyrazole, unless indicated otherwise.



Phenyl label	Pyrazole label

Grape

Phenyl or pyrazole labelled fluindapyr was applied as an EC formulation to grapes (variety: Thompson seedless), with two foliar spray applications at fruit development (BBCH stage 55 and 85, corresponding with an RTI of 123 days) at a rate of 237–313 g ai/ha per application. Each outdoor plot contained one plant.

At 14 days after the second application, total radioactive residues (TRR) were 0.089/0.36 mg eq/kg in grapes and 16/26 mg eq/kg in grape leaves. A fruit surface wash with methanol released 79/81 percent TRR, indicating that fluindapyr residues remained mainly on the surface of the grapes. Leaves and grapes were extracted with acetonitrile/water, followed by extraction with methanol/water. Part of the extract was partitioned against dichloromethane and subjected to hydrolysis with HCl, which did not release much more of unconjugated forms. Both conjugated and unconjugated forms could be identified before hydrolysis. The surface wash with methanol and extraction released most of the radioactivity for both labels in fruit (>98.8 percent TRR) and in leaf (>90 percent TRR) samples.

Approximately 99 percent TRR could be identified in grape fruit and 88/92 percent TRR in grape leaveswith both labels. Parent fluindapyr accounted for 63/65 percent TRR (0.056/0.24 mg/kg) in grapes and 38/54 percent TRR (5.9/14 mg/kg) in grape leaves. 1-OH-Met-fluindapyr and its conjugates accounted for 20 percent TRR (0.017/0.07 mg eq/kg) in grapes and 39/25 percent TRR (6.0/6.4 mg eq/kg) in grape leaves. A second metabolite found at significant concentrations was 3-OH-fluindapyr at levels of 15/12 percent TRR (0.013/0.04 mg eq/kg) in grapes and 12–9.3 percent TRR (1.8/2.4 mg eq/kg) in grape

leaves. Minor metabolites were found in fruit and leaves, but none exceeded 0.2 percent TRR (< 0.001–0.064 mg eq/kg).

Chiral analysis of fluindapyr in leaves showed no significant change in the enantiomeric composition, indicating non-selective metabolic biotransformation. However, in grapes, fluindapyr present in the rinsing (79–81 percent of TRR) showed an enantiomeric ratio approximately 50:50 while in the extract this ratio was approximately 70:30.

Sugar beets

Phenyl or pyrazole labelled fluindapyr was applied as an EC formulation to sugar beet plants, with three foliar spray applications at root development (BBCH stage 35/38, 39/49, and 49, corresponding with RTI of 33 and 28 days between the subsequent applications) at a rate of 113–149 g ai/ha per application.

At 30 days after the last application (DALA), total radioactive residues were 0.084/0.122 mg eq/kg in mature sugar beet roots and 1.67/1.64 mg eq/kg in mature sugar beet foliage. Extraction with dichlormethane/water released most of the radioactivity for both labels in roots (90/92 percent TRR) and in foliage (92/93 percent TRR) samples. The aqueous fraction was subjected to acid hydrolysis to release the unconjugated forms of the conjugated metabolites. Exhaustive extraction with enzymes and acid/base released another 4.0–5.7 percent TRR.

Approximately 89/86 percent TRR could be identified in sugar beet root and 90/88 percent TRR in mature foliage with both labels.

Parent fluindapyr accounted for 43/50 percent TRR (0.036/0.062 mg/kg) in sugar beet roots and 18/15 percent TRR (1.5/1.4 mg/kg) in mature foliage. The 1-OH-Met-fluindapyr diastereomer 2 could not be distinguished from 1-COOH-fluindapyr, diastereomer 2, with the phenyl label and together accounted for 26 percent TRR (0.022 mg eq/kg) in mature sugar beet root, where an addition 8.4 percent TRR (0.007 mg eq/kg) accounted for the remaining 1-OH-Met-fluindapyr diastereomers in mature roots. 1-OH-Met-fluindapyr diastereomers in sugar beet foliage accounted for 66/62 percent TRR (1.1/1.0 mg eq/kg). The diastereomer 1 of COOH-fluindapyr accounted for 4.1/2.1 percent TRR (0.003 mg eq/kg) in sugar beet root. Metabolite 1-OH-Met-N-DesMet-fluindapyr accounted for 1.2/0.4 percent TRR and 1.8/1.7 percent TRR in sugar beet root and foliage, respectively, accounting for 0.001 mg eq/kg in roots and 0.029/0.028 mg eq/kg in foliage. Two other metabolites 3-OH-fluindapyr and N-DesMet-fluindapyr, either single or combined, were found, but below 10 percent TRR with only 3-OH-fluindapy observed at levels above 0.01 mg eq/kg(2.0/1.3 percent TRR, 0.034/0.022 mg eq/kg) in sugar beet foliage. Finally, 3-OH-N-DesMet-fluindapyr was observed at low levels (0.24/0.64 percent TRR, 0.004/0.010 mg eq/kg) in sugar beet foliage.

Chiral analysis of fluindapyr showed no significant change in the enantiomeric composition.

Wheat

Phenyl or pyrazole labelled fluindapyr was applied as an EC formulation to wheat plants, with two foliar spray applications at BBCH stage 31–33 and BBCH 65 (RTI 28 days) at a rate of 124–130 g ai/ha per application. Plants were harvested at BBCH47-49 (immature whole plants/forage stage and 3–4 days after the first application), BBCH 83 (21–22 DALA; immature whole plants/hay stage), mature grain and straw (41–42 DALA).

At 41–42 DALA, TRR were 0.020/0.038 mg eq/kg in wheat grain and 15/13 mg eq/kg in wheat straw. Total residues in wheat forage (3–4 days after the first application) and in wheat hay (21–22 DALA) were 1.2/2.2 and 5.5/7.4 mg eq/kg, respectively. Samples were extracted four times with acetone/water. Straw and grain were extracted a fifth time with acetone/HCl. Extracts from forage, hay

and straw were sequentially partioned with n-hexane and ethyl acetate. Most of the radioactivity for both labels was released in samples of grain (66/77 percent TRR), forage (99/97 percent TRR), hay (103/93 percent TRR), and straw (90/84 percent TRR). Exhaustive extraction with enzymes and acid/base released another 22/13 percent TRR in grain and 6.4/7.3 percent TRR in straw.

In wheat grain, 66/78 percent TRR could be identified, whereas 99/97 percent TRR, 103/93 percent TRR, and 90/85 percent TRR was identified in wheat forage, hay and straw, respectively.

Parent fluindapyr accounted for 46/56 percent TRR (0.0093/0.021 mg/kg) in wheat grain and for 37/31 percent TRR (0.46/0.66 mg/kg) in wheat forage, 31/28 percent TRR (1.7/2.1 mg/kg) in wheat hay, and 29/28 percent TRR (4.3/3.7 mg/kg) in wheat straw.

The 3-OH-fluindapyr metabolite accounted for 20/22 percent TRR (0.0042/0.0084 mg eq/kg) in wheat grain, 4.5/5.5 percent TRR (0.056/0.12 mg eq/kg) in wheat forage, 10/11 percent TRR (0.57/0.79 mg eq/kg) in wheat hay, and 12/14 percent TRR (1.8 mg eq/kg) in wheat straw.

Metabolite 1-OH-Met-fluindapyr (free and glucosyl and glucosyl sulpfate conjugates) was not identified in grain, but found to be a major metabolite in wheat forage, hay and straw, ranging from 35-60 percent TRR with both labels (0.64-7.1 mg eq/kg). Metabolite 1-OH-Met-N-DesMet-fluindapyr as glucosyl conjugate was found at low levels in forage, hay and straw (0.38–5.1 percent TRR, 0.029–0.066 mg eq/kg) as were the pyrazole label specific metabolites pyrazole carboxylic acid and carboxamide with 2.9–3.8 percent TRR (0.082–0.42 mg eq/kg) and 0.40–2.9 percent TRR (0.0087–0.38 mg eq/kg), respectively.

Chiral analysis of fluindapyr showed an R:S change of 50:50 in the test formulations to a mean ratio of 66:34 in the forage, hay and straw samples. The radioactivity levels in grain extracts were too low to be analysed by chiral HPLC.

Rice

Phenyl or pyrazole labelled fluindapyr was applied as an EC formulation to rice plants, with two foliar spray applications at BBCH stage 33 and BBCH 75 (corresponding with an RTI of 70 days) at a rate of 114–122 g ai/ha per application. Samples of husked grain and straw were harvested at 58 DALA.

Total radioactive residues were 0.78/0.65 mg eq/kg in rice grain and 1.8/2.2 mg eq/kg in rice straw. Samples were extracted 3 times with acetonitrile/water followed by extraction with methanol/water. Part of the extract was partitioned with dichloromethane and the aqueous fraction subjected to hydrolysis with HCl. Similar identification results were found in the paralel extracts of dichlorometane and the aqueous fraction after acid hydrolysis. Extraction released most of the radioactivity for both labels in samples of rice grain and straw (93–98 percent TRR).

In husked rice grain and rice straw 94/91 percent TRR and 95/96 percent TRR could be identified, respectively. Fluindapyr accounted for 53/57 percent TRR (0.41/0.37 mg/kg) in husked rice grain and 55/56 percent TRR (1.0/1.2 mg/kg) in rice straw.

Metabolite 1-OH-Met-fluindapyr accounted for 22/17 percent TRR (0.17/0.11 mg eq/kg) and 23/19 percent TRR (0.43/0.21 mg eq/kg) in rice grain and rice straw, respectively.

The 3-OH-fluindapyr metabolite accounted for 9.1/8.2 percent TRR (0.072/0.053 mg eq/kg eq) in rice grain and for 11/11 percent TRR (0.20/0.25 mg eq/kg) in rice straw. Metabolite 1-COOH-fluindapyr was found at 4.2/4.0 percent TRR (0.033/0.026 mg eq/kg) in rice grains and 3.7/4.4 percent TRR (0.068/0.099 mg eq/kg) in rice straw. Low levels of N-DesMet-fluindapyr and dehydro-fluindapyr were found in rice grain (0.4–1.0 percent TRR) and rice straw (0.9–1.1 percent TRR), ranging from 0.003–0.006 mg eq/kg in rice grains to 0.016–0.025 mg eq/kg in rice straw.

Chiral analysis of fluindapyr in rice grain and straw indicated that a slight change in the original (50:50) enantiomeric ratio (R:S) took place and was determined to be approximately 60:40.

Soya bean

Phenyl or pyrazole labelled fluindapyr was applied as an EC formulation to soya bean plants, with three foliar spray applications at BBCH stage 15–16, BBCH 55-60, and BBCH 79 (corresponding with RTIs of 21 and 60 days, respectively) at a rate of 117–129 g ai/ha per application. Two additional plots were also treated at a higher rate of 667–676 g ai/ha for generation additional metabolised for identification purposes, if needed. Plants were grown in outdoor pots and samples of immature forage were taken at 21 days after the first application (28 prior to the second application), hay was harvested after two applications, and mature seeds were collected 30 DALA.

Total radioactive residues were 0.013/0.090 mg eq/kg in soya bean seed, and 0.30/0.51 mg eq/kg in soya bean forage and 1.8/1.6 mg eq/kg in soya bean hay. Samples were extracted 3 times with acetonitrile/water followed by extraction with methanol/water. Part of the extract was partitioned with dichloromethane and the aqueous fraction subjected to hydrolysis with HCl. Similar identification results were found in the paralel extracts of dichlorometane and the aqueous fraction after acid hydrolysis, Extraction with dichloromethane/water released most of the radioactivity for both labels in samples of soya bean seed, forage and hay (92–98 percent TRR). Exhaustive (enzyme and acid/base) extraction released another 2–3 percent TRR in soya bean hay.

Insufficient radioactivity was detected for characterisation and identification of metabolites in soya bean seed.

Fluindapyr accounted for 5.7/5.9 percent TRR and 12 percent TRR (0.017/0.031 mg/kg and 0.22/0.19 mg/kg) in soya bean forage and hay, respectively. Free and conjugated 1-OH-Met-fluindapyr accounted for 31–40 percent TRR (0.12/0.47 mg eq/kg) in both RACs. Free and conjugated 1-OH-Met-N-DesMet-fluindapyr represented 9.5–12 percent TRR (0.034–0.17 mg eq/kg) in both matrices. The sum of DesMet-fluindapyr-N1-conjugates ranged from 14 to 18 percent TRR (0.046–0.33 mg eq/kg) in forage and hay. Metabolites found at lower concentrations were N-DesMet-fluindapyr-N-Ser (3.5–4.0 percent TRR, 0.012–0.062 mg eq/kg), 3-OH-fluindapyr (2.4–4 percent TRR, 0.012–0.077 mg eq/kg), N-DesMet-fluindapyr (1.0–4.6 percent TRR, 0.013–0.025 mg eq/kg)), dehydro-fluindapyr (0.046–0.13 percent TRR, 0–0.001 mg eq/kg), and pyrazole carboxamide (1.1 percent TRR (0.006 mg eq/kg) in forage only).

Chiral analysis of fluindapyr in soya bean hay and forage samples indicated that a slight change in enantiomeric ratio (R:S) took place and was determined to be approximately 60:40.

Summary of plant metabolism

Plant metabolism studies have been presented covering foliar treatments in grape, sugar beet, wheat, rice, pulses and soya bean. The application rates used in the metabolism studies with crops covering the current uses on cereals (wheat, sorghum, maize and rice) are slightly lower and RTIs are longer. However, exaggerated application rates were also used, showing similar distribution patterns.

The enantiomeric ratio R:S in some crops remained 50:50 (grape leaves and rinse, sugar beet foliage), however, in other crops a shift could be observed into a ratio ranging from 60:40 to 70:30 (grape, wheat forage/hay/straw, rice grain/straw, soya bean hay/forage).

The metabolic pathways of fluindapyr where similar in the crops investigated, mainly through hydroxylation and oxidative-N-demethylation, both followed by conjugation. Parent fluindapyr was a major residue (43–65 percent TRR) in grapes, sugar beet root, wheat grain, and rice grain. Major identified metabolites were 1-OH-Met-fluindapyr (free and conjugated) accounting for 17 to 66 percent TRR in food

and feed commodities, except wheat grain, 3-OH-fluindapyr accounting for 10 to 22 percent TRR in grapes and leaves and wheat grain, hay and straw, rice straw and 8.2 to 9.1 percent TRR in rice grain, and 1-OH-Met-N-DesMet-fluindapyr (free and conjugated) accounting for 12 percent TRR in soya bean forage only and DesMet-fluindapyr-conjugates accounting for 14–18 percent TRR in soya bean forage and hay only.

Environmental fate

The Meeting received information on hydrolytic stability, photochemical degradation in water and soil, aerobic soil metabolism, and soil degradation field studies for fluindapyr.

Hydrolysis

Radiolabelled fluindapyr, incubated in the dark in sterile aqueous buffered solutions at pH 4, 7, and 9 for 5 days at 50 °C remained stable. No degradation products were detected and the enantiomeric ratio remained unchanged. The results indicate that fluindapyr is hydrolytically stable at environmental conditions.

Photochemical degradation

In an aqueous photolysis study, [^{14}C- phenyl]-fluindapyr and [^{14}C-pyrazole]-fluindapyr was incubated in sterile non-buffered water under simulated sunlight at 25 ± 1.0 ºC, equivalent to summer sunlight (55 °North in June). The DT_{50} of fluindapyr was calculated to be 4.3 and 2.9 years, with only minor degradation products identified. Two of the minor products were confirmed as 3-OH-fluindapyr and the pyrazole-amide. The distribution of the two enantiomers remained 50:50 throughout the entire irradiation duration.

In a soil photodegradation study [^{14}C-pyrazole]- or [^{14}C-phenyl]-labelled fluindapyr slowly degraded in the irradiated samples, with an associated increase of 3-OH-fluindapyr (up to 9.7 percent AR) and pyrazole-carboxamide (up to 7 percent AR after 15 days). The estimated photolysis DT_{50} in clay loam soil was 54–61 experimental days, equivalent to 163–183 natural sunlight days at 50 °N.

In summary, the Meeting concluded that photodegradation contributes to some extent to the overall degradation of fluindapyr in soil, but photolysis of fluindapyr in water is insignificant.

Aerobic soil metabolism (laboratory studies)

The biotransformation of [^{14}C-phenyl]- or [^{14}C-pyrazole]-fluindapyr in soil was investigated in four European and four United States soils under laboratory conditions. The equivalent of 127–128 g fluindapyr/ha was mixed with soil and incubated under aerobic conditions in the dark at 20 °C for 120–151 days.

The estimated DT_{50} for fluindapyr ranged from 141to 353 days in the various soils with both labels, with a geometric mean of 223 days. Three degradation products were identified above 5 percent AR; 3-OH-fluindapyr (max 15 percent AR at DAT-120), and *cis*-1-COOH-fluindapyr (max 13 percent AR at DAT-151) and *trans*-1-COOH-fluindapyr (max 11 percent AR at DAT-151). The enantiomeric ratio remained constant during the studies (ca. 50:50).

The aerobic degradation of the three soil metabolites 3-OH-fluindapyr, *cis*-1-COOH-fluindapyr and *trans*-1-COOH-fluindapyr and the photolytic soil metabolite pyrazole carboxamide was investigated under laboratory conditions for up to approximately 120 days in different soils from Europe orUnited Statesin four studies.

The laboratory DT_{50} values for 3-OH-fluindapyr were >1000 days in three European soils and ranged from 794 to 1302 days in three United States soils. The laboratory DT_{50} values for *cis*- and *trans*-1-

COOH-fluindapyr ranged from 102 to 320 days in three United States soils, and ranged from 1.7 to 4.1 days in two European and two United States soils for pyrazole carboxamide

Soil degradation (field studies)

The field dissipation of fluindapyr has been studied in Europe and the United States. Quantifiable residues of fluindapyr were detected predominantly in the upper 15 cm of the soils, with incidental findings in the following 10–25/15–30 cm layers. The DT_{50} for total residues ranged from 30 to 168 days, with a geometric DT_{50} of 91 days.

Residues in succeeding or rotational crop

The Meeting received information on the metabolism of fluindapyr in wheat, carrot, and lettuce grown as confined rotational crops, and in a range of representative field crops grown in fluindapyr treated soil.

Confined rotational crop studies

In two confined rotational crop studies in Italy, soil was treated with either [^{14}C-phenyl]- or [^{14}C-pyrazole]-labelled fluindapyr at 360/387 g ai/ha (covering the current registered uses at a maximum seasonal rate of 300 g ai/ha) and planted with lettuce, carrots and wheat at plant-back intervals at 30 days, 120, and 300 days. The TRR in the different RACs were highest when using the pyrazole label declining from 0.037 (PBI 30 days) to 0.019 mg eq/kg (PBI 300 days) in carrot root (phenyl label) and increasing from 0.081 (PBI 30) to 0.11 (PBI 300) mg eq/kg (pyrazole label) in first to last rotation. Similar patterns was observed in carrot tops, with a decline from 0.18 to 0.075 mg eq/kg (phenyl) and increase from 1.1 to 1.7 mg eq/kg (pyrazole label); in immature lettuce, from 0.070 mg eq/kg to 0.046 mg eq/kg (phenyl) and 0.22 mg eq/kg to 0.25 mg eq/kg (pyrazole) and mature lettuce 0.081 to 0.044 mg eq/kg (phenyl) and 0.23 to 0.34 mg eq/kg (pyrazole).

Residues in wheat matrices were generally higher, remaining constant over the three rotations and the patterns between the two labels were similar: 1.5 to 0.73 mg eq/kg (phenyl) and 2.9 to 2.8 mg eq/kg (pyrazole) in wheat grain, 0.35 to 0.36 mg eq/kg (phenyl) and 0.54 to 0.37 mg eq/kg (pyrazole) in wheat forage; 0.82 to 0.62 mg eq/kg (phenyl) and 1.5 to1.7 mg eq/kg (pyrazole) in rotated wheat hay; 2.0 to 1.3 mg eq/kg (phenyl) and 3.8 to 3.2 mg eq/kg (pyrazole) in wheat straw.

Extractated radioactivity from all different crop matrices was high and generally ranged from 83 to 99 percent TRR. Only the radioactivity in the PES of wheat straw and grain needed further investigation and was present mainly as cellulose ^{14}C-incorporated natural products, representing 6–10 percent TRR in straw and 3–5 percent TRR in grain.

The identified residues (fluindapyr, 3-OH-fluindapyr, 1-COOH-fluindapyr (and its conjugates), 1-OH-Met-fluindapyr (and its conjugates), N-DesMet-fluindapyr (and its conjugates), N-DesMet-pyrazole carboxylic acid, pyrazole carboxylic acid, pyrazole carboxamide) were common in all crops but their magnitude varied depending on the individual crop, matrix and label.

In commodities relevant for human consumption and considering the phenyl label study, fluindapyr was one of the main components found, accounting for 2.8–20 percent TRR (0.006–0.015 mg eq/kg) in immature lettuce, 6.1–15 percent TRR (0.084–0.39 mg eq/kg) in wheat grain and up to 65–70 percent TRR (0.013–0.026 mg eq/kg) in carrots.

Metabolite 3-OH-fluindapyr accounted for 9.8–25 percent TRR (0.004-0.005 mg eq/kg) in carrot roots, 7.3-8.5 percent TRR (0.003-0.007 mg eq/kg) in (im)mature lettuce, and 11 percent TRR (0.092-0.17 mg eq/kg) in wheat grain.

Free and conjugated 1-OH-Met-fluindapyr accounted for 8.2–9.4 percent TRR (0.002–0.004 mg eq/kg) in first two rotations only in carrot roots, 23–34 percent TRR (0.011–0.027 mg eq/kg) in (im)mature lettuce, and 47–52 percent TRR (0.42–0.78 mg eq/kg) in wheat grain.

Free and conjugated 1-COOH-fluindapyr accounted for 9.5–9.6 percent TRR (0.003–0.004 mg eq/kg) in carrot roots, 16–34 percent TRR (0.012–0.018 mg eq/kg) in (im)mature lettuce, and 5.9–10.0 percent TRR (0.082–0.11 mg eq/kg) in wheat grain.

The pyrazole specific metabolites included (conjugates of) N-DesMet-pyrazole carboxylic acid, pyrazole carboxylic acid and pyrazole carboxamide. Free and conjugated N-DesMet-pyrazole carboxylic acid increased in time and represented the majority of the radioactivity in carrot roots, with 48–65 percent TRR (0.022–0.065 mg eq/kg) and in (im)mature lettuce with 62–82 percent TRR (0.12–0.21 mg eq/kg). In wheat grain, N-DesMet-pyrazole carboxylic acid accounted for 3–10 percent TRR 0.087–0.31 mg eq/kg), pyrazole carboxylic acid for 12–29 percent TRR (0.27–0.69 mg eq/kg) and pyrazole carboxamide for 1.7–16 percent TRR (0.050–0.45 mg eq/kg). Pyrazole carboxylic acid and pyrazole carboxamide were below 10 percent TRR in the other commodities, except in carrot roots at PBI 120 and 300 days (14–18 percent TRR) and in (im)mature lettuce at PBI 120 and 300 days (11–13 percent TRR).

In feed commodities, the levels of parent varied from 0.24–11 percent TRR (0.004–0.013 mg eq/kg) in carrot tops to 13–32 percent TRR (0.050–0.14 mg eq/kg) in wheat forage. The (conjugated) metabolite 1-OH-Met-fluindapyr represented a major part of the radioactive residue in the phenyl-label, ranging from 11–36 percent TRR (0.061–0.13 mg eq/kg) in wheat forage to 17–46 percent TRR (0.49–1.6 mg eq/kg) in wheat straw. In the pyrazole-label, free and conjugated pyrazole carboxylic acid was present in forage (13–24 percent TRR, 0.050–0.12 mg eq/kg) and straw (9.1–28 percent TRR, 0.34–0.89 mg eq/kg), while in carrot tops free and conjugated N-DesMet-pyrazole carboxylic acid was more pronounced (52–58 percent TRR, 0.64–0.99 mg eq/kg). 3-OH-fluindapyr, and free and conjugated pyrazole carboxamide, N-DesMet-fluindapyr, and 1-COOH-fluindapyr also contributed to the total radioactive residues, with levels depending on crop matrix and on PBI.

A third confined rotational crop study was performed using a single bare soil application of 356–360 g ai/ha and wheat as a rotational crop planted at PBI 30 and 120 days. This study confirmed the rather high concentrations TRR found in wheat commodities.

Field rotational crop studies

A series of field rotational crop studies was conducted in Northern Europe (NE), Southern Europe (SE) and the United States. Only the European studies analysed soil samples. In the four European trials, fluindapyr was incorporated into the soil at actual dose rates of 203–232 g ai/ha. This is lower than the anticipated seasonal rates of the currently registered uses of 300 g ai/ha for which crop rotation needs to be taken into account

Carrots/radish, wheat, lettuce, head cabbage, soya beans, and tomato plants were planted at intervals of 30 (±3), 120 (±10), and 270 (± 3) days after the application. In theUnited Statestrials conducted on 2 locations, fluindapyr was applied twice to soya beans as primary crop at 124–128 g ai/ha/application, RTI 12–15 days (21±2 days prior to typical harvest). At one site the seeds were harvested and the plant debris returned to the field and on the other site both seed and straw was removed. The plots were planted with the follow-on rotational crop of mustard, radish, or wheat at target plant back intervals of 30, 60, and 210 days following the last application (DALA).

Samples of mature commodities were analysed for fluindapyr and metabolites 3-OH-fluindapyr, DesMet-N1-fluindapyr-glucoside and free and conjugated 1-COOH-fluindapyr, pyrazole-carboxamide, pyrazole carboxylic acid and N-DesMet-pyrazole carboxylic acid.

In crops for human consumption, residues of fluindapyr and most of its metabolites were found incidentally, with fluindapyr only once at 0.022 mg/kg in radish roots at PBI of 30 days, pyrazole carboxylic acid in one trial at levels of 0.024–0.027 mg/kg on PBI 30, 120 and 300 days, and 1-COOH-fluindapyr once at 0.020 mg eq/kg in immature head cabbage at PHI 30 days and once at 0.018 mg eq/kg in mustard at PBI 30 days.

Quantified residues of fluindapyr were observed in samples of animal feed commodities at plant-back intervals up to 300 days, however, the findings were incidental and the identity of the metabolites varied between crop matrices. Fluindapyr was oberved in wheat straw (0.012–0.022 mg/kg, study 1) and radish tops (< 0.001–0.022 mg/kg, study 3) and metabolite 3-OH-fluindapyr in wheat straw (PBI 30–270 days, 0.012–0.034 mg eq/kg), soya bean hay (PBI 30–270 days, 0.010–0.024 mg eq/kg) and soya bean forage (PBI 120 days, 0.013 mg/kg). Free and conjugated 1-OH-Met-fluindapyr was found in wheat straw (PBI 30–270 days at < 0.01–0.066 mg/k eq), wheat hay (PBI 30 days, 0.010–0.011 mg eq/kg), and radish leaves (< 0.01–0.016 mg eq/kg). Metabolite 1-COOH-fluindapyr (free and conjugated) was a found in soya bean forage (0.014–0.017 mg eq/kg) and hay (< 0.01–0.028 mg eq/kg) at at PBI 30 and 120, as well as free and conjugated N-DesMet-pyrazole carboxylic acid.

The Meeting considered potential residues of 3-OH-fluindapyr, which is persistent in soil. Based on the assumption of a single treatment or subsequent annual applications at the maximum rate per year of 300 g ai/ha, the Meeting estimated soil concentrations of 0.012 and 0.036 mg/kg, respectively, taking into account information from the available European field dissipation studies. In two of the four European studies, the concentrations of 3-OH-fluindapyr in soil corresponded to concentrations expected after applying the single maximum rate per year (~0.012 mg/kg soil). In the two other studies, one soil contained approximately half of the expected soil concentration, whereas in the fourth study 3-OH-fluindapyr remained undetected. However, no clear relationship between soil concentrations for 3-OH-fluindapyr and its uptake into rotational crops was observed based on the plant samples analysed. Residues were found in feed commodities at up to 0.034 mg/kg, but were <LOQ of 0.01 mg/kg in edible commodities.

The Meeting concluded that no systematic occurence of 3-OH-fluindapyr in rotational crops is expected. Even at estimated plateau levels following long-year accumulation, residues in feed commodities are expected at maximum concentrations of 0.1 mg/kg or lower, while quantifiable concentrations are not expected in commodities for human consumption.

None of the other metabolites have a potential to cummulate in soil, . Considering the scattered and low levels found in both food and feed commodities, the Meeting concluded that potential residues found in rational crops will not contribute significantly to the total dietary intake nor to the total dietary burden and need not be further considered.

One exception is the common metabolite N-DesMet-pyrazole carboxylic acid, which was observed at relevant exposure levels in several food crops. This metabolite is also a metabolite formed after use of other active substances, such as bixafen, benzovindiflupyr, and inpyrfluxam. The Meeting considered the residues of N-DesMet-pyrazole carboxylic acid observed in field rotational crop data after use with fluindapyr, bixafen (Report 2021- extra Meeting) and inpyrfluxam or estimated concentrations following direct treatment with benzovindiflupyr. The highest STMRs from each of the compounds were used for the exposure estimation, assuming no combined field treatments since the compounds belong to the same chemical group of fungicidal agents (Table 2).

Table 136 Overview of anticipated N-DesMet-pyrazole carboxylic acid residues in rotational crops found after use of both fluindapyr (F), bixafen (B), and inpyrfluxam (I) in field rotational crop studies or estimated concentrations following direct treatment with benzovindiflupyr (Ben)

Commodity group	Field rotational crop commodity	N-DPCA in mg/kg (highest concentrations per trial from all PBIs)	STMR, mg/kg
Root and tuber vegetables	Carrot and radish roots	F: < 0.01 (3), 0.014	F: 0.01 B: 0.016
	Carrot roots	B: < 0.01, < 0.01, 0.44	
	Potato tuber	B: 0.016, 0.016, 0.061, 0.064	
	Combined	B: < 0.01, < 0.01, 0.016, 0.016, 0.044, 0.061, 0.064	
Leafy crops and brassica (extrapolated to stalk and stem vegetables)	Lettuce (mature and immature)	F: < 0.01 (7), 0.028 B: < 0.01, 0.017, 0.092	F: 0.01 B: 0.01
	Cabbage (mature and immature)	F: < 0.01 (7), 0.025 B: < 0.01 (3), 0.01	
	Radish leaves	F: < 0.01, 0.020 I: 0.015 [a]	
	Carrot foliage	F: < 0.01, < 0.01	
	Combined	F: < 0.01 (15), 0.01 (2), 0.020, 0.025, 0.028 B: < 0.01 (6), 0.01, 0.017, 0.092, 0.18	
Fruiting vegetables	Tomato	F: < 0.01 (4)	F: 0.01 B: 0.01
	Courgettes	B: < 0.01 (2), 0.015, 0.023	
	Strawberries	B: < 0.01 (4)	
	Combined	B: < 0.01 (6), 0.015, 0.023	
Pulses (Extrapolated to legume vegetables and oil seeds)	Soya bean seed (dry)	F: < 0.01 (2), 0.065, 0.073 I: < 0.02 (7), 0.02, 0.023, 0.024, 0.026, 0.028, 0.032, 0.036, 0.037, 0.051, 0.062, 0.095, 0.13, 0.16, 0.19	F: 0.0375 I: 0.026 B: 0.0235
	Peas (dry)	B: < 0.01 (2), 0.037, 0.082	
Cereal grains	Wheat grain	F: < 0.01 (4)	F: 0.01 B: 0.01
	Barley/wheat	B: < 0.01 (3)	
	Maize	B: < 0.01 (3), 0.063	
	Combined	B: < 0.01 (6), 0.063	
Bulb vegetables	Leek	B: < 0.01 (2), 0.016, 0.034	B: 0.013
Oil seeds	Rape seed	B: < 0.01 (4)	B: 0.01
Coffee, green	Direct treatment with benzovindiflupyr using metabolite:parent ratios from metabolism studies	Ben: < 0.01 (6)	Ben: 0.01

Notes:

[a] Single value found in confined rotational crop study

Summary of environmental fate

Fluindapyr is slowly photodegraded on the surface of soil, forming 3-OH-fluindapyr and pyrazole carboxamide. Laboratory soil degradation studies showed the formation of 3-OH-fluindapyr and *cis*- and *trans* 1-COOH-fluindapyr. Field studies showed DT_{50}s for total fluindapyr (1-COOH-fluindapyr, 3-OH-fluindapyr and pyrazole carboxylic acid) ranging from 55 to168 days (geometric mean of 91 days), with 3-OH-fluindapyr being the predominant metabolite.

Confined laboratory studies indicate that, 3-OH-fluindapyr may be persistent in soil and have a potential for residue carry over to the following cropping season if application is performed annually. However, there was sufficient information for the Meeting to conclude that 3-OH-fluindapyr levels in

edible commodities from rotational crops would remain below the LOQ of 0.01 mg/kg. Confined rotational crop studies indicated that in addition to parent and 3-OH-fluindapyr (free and conjugated), 1-COOH-fluindapyr, 1-OH-Met-fluindapyr, N-DesMet-fluindapyr, N-DesMet-pyrazole carboxylic acid, pyrazole carboxylic acid, and pyrazole carboxamide can be formed in both food and feed commodities. However, field rotational crop studies showed that none of the metabolites are expected in rotational crops at levels above 0.01 mg/kg, with exception of N-DesMet-pyrazole carboxylic acid, which is also formed after application of other fungicides within the same chemical class. The Meeting concluded that a TTC approach should be applied considering residues of this metabolite coming from the uses of the different fungicides.

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens, where animals were dosed with fluindapyr radiolabelled in the (phenyl ring) or the (e.g. pyrazole ring).

Rats

The metabolism of fluindapyr in rats was reviewed in the framework of the toxicological evaluation by the WHO Core Assessment Group of the 2022 JMPR.

Lactating goats

Two lactating goats were orally dosed by capsule once daily for 7–8 consecutive days with either pyrazole-labelled fluindapyr or phenyl-labelled fluindapyr at 7.3 or 7.5 ppm feed, corresponding to 0.35 or 0.23 mg/kg bw/day, respectively. The goats were sacrificed approximately 6 hours after the last dose. The majority of the total applied radioactivity (TAR) was recovered in the excreta (65-81 percent TAR), with lower levels in the GI tract (11–13 percent TAR). The radioactivity recovered in tissues (liver, kidney, muscle, and fat) accounted for 0.34 and 0.20 percent TAR, with the respective labels, with the highest amount in liver (0.27 percent and 0.16 percent TAR, respectively). A total of 0.017 and 0.034 percent TAR, respectively, was found in milk. Steady state conditions in milk were reached within 2–3 days of the first dose. TRR levels were higher in cream (0.030–0.088 mg eq/kg) compared to skimmed milk (0.004–0.015 mg eq/kg).

Radioactive residues extracted with either ethyl acetate (skimmed milk) or hexane followed by acetonitrile (fat samples and milk cream), acetonitrile and acetonitrile/water or acetone/water (liver, kidney, and muscle) accounted for at least 78 percent TRR, and liver samples from the acetone/water extractions were further treated with β-glucuronidase. The PES from all matrices was found to contain <10 percent TRR or a low residue level, therefore, no further characterization of unextracted residue was conducted.

Fluindapyr was the predominant compound in cream (75–93 percent TRR, 0.045–0.057 mg/kg), fat (74–75 percent TRR, 0.024–0.042 mg/kg), and muscle (32–39 percent TRR, 0.004–0.006 mg/kg), while it ranged from not detected (kidney) to 8.4 percent TRR in the other samples. Free and conjugated 1-OH-Met-fluindapyr was found in liver (up to 52 percent TRR and 0.13 mg eq/kg), kidney (up to 57 percent TRR and 0.059 mg eq/kg), and muscle (up to 41 percent TRR, 0.006 mg eq/kg). Free and conjugated 1-COOH-fluindapyr was found in liver (up to 27 percent TRR and 0.075 mg eq/kg) and kidney (up to 11 percent TRR and 0.011 mg eq/kg). Free and conjugated 1-OH-Met-N-DesMet-fluindapyr was identified in liver (up to 8.9 percent TRR and 0.025 mg eq/kg) and kidney (up to 24 percent TRR and 0.029 mg eq/kg). No individual metabolite in the remaining edible tissues (skimmed milk, cream, fat, or muscle) exceeded 0.01 mg eq/kg, although 1-OH-Met-fluindapyr and di-hydroxylated species accounted for 16 percent to 43 percent TRR in skimmed milk.

Chiral analysis of fluindapyr isolated from fat and cream, showed that the S/R enantiomeric ratio changed from about 50/50 to 35/65.

Laying hens

A group of laying hens was orally dosed by capsule with [phenyl-^{14}C]-fluindapyr or [^{14}C-pyrazole]-fluindapyr for 9 consecutive days at 10 ppm feed, corresponding with a mean daily dose level of 0.64–0.66 mg/kg bw/day. Hens were sacrificed approximately 6 hours after the last dose. The majority of radioactivity (TAR radioactivity was recovered in the excreta (93–96 percent TAR), and a minor part in tissues (0.15–0.16 percent TAR) and eggs (0.11–0.12 percent TAR). The highest TRR in edible tissues was measured in the liver (0.11–0.12 mg eq/kg), followed by fat (0.079–0.10 mg eq/kg), skin (0.044–0.057 mg eq/kg), and muscle (0.010–0.013 mg eq/kg). Residues in eggs ranged from 0.019 to 0.10 mg eq/kg and reached a steady state at day 6.

Radioactive residues extracted with hexane followed by acetonitrile (fat and skin), hexane, followed by acetone:water and ethyl acetate (eggs), acetone/water and ethyl acetate (liver), acetonitrile and acetonitrile/water (muscle) was at least 89 percent TRR. Liver aqueous extract samples were further treated with acid (HCl) or enzymatic hydrolysis (sulfatase) to release SO_4 conjugates. The PES from all matrices was <10 percent TRR and 0.05 mg eq/kg; therefore, no further characterization of unextracted residue was conducted.

Fluindapyr was the major component identified in skin (88-94 percent TRR, 0.041–0.050 mg/kg), fat (76–95 percent TRR, 0.073–0.090 mg/kg), egg (31–48 percent TRR, 0.018–0.028 mg/kg), and muscle (38 percent TRR, 0.004 mg/kg), but represented only about 5 percent TRR (0.005-0.006 mg/kg) in liver.

Metabolite N-DesMet-fluindapyr represented a large part of the radioactive residue in the liver (62 percent of the TRR, 0.067 mg eq/kg), of which a minor fraction was present as sulfate conjugate (< 2 percent of TRR). This metabolite was detected at even lower levels in the egg (up to 6.8 percent TRR, 0.004 mg eq/kg), fat (up to 2.0 percent TRR, 0.002 mg eq/kg), skin (up to 1.6 percent TRR, 0.001 mg eq/kg), and muscle (4.5 percent of TRR, < 0.001 mg eq/kg).

The 2 diastereomers of 1-OH-Met-fluindapyr and their sulfate conjugates represented 22 percent TRR in liver (0.026 mg eq/kg), 32 percent TRR in eggs (0.019 mg eq/kg), 11 percent TRR in fat (0.010 mg eq/kg), and 14 percent TRR in muscle (0.002 mg eq/kg), but less in skin (9.8 percent TRR, 0.004 mg eq/kg).

The 1-COOH-fluindapyr metabolite reached 12 percent TRR (0.001 mg eq/kg) in muscle and 7.2 percent TRR (0.008 mg eq/kg) in liver. The 2 diastereomers of 1-OH-Met-N-DesMet-fluindapyr and/or the corresponding sulfate conjugates reached 8.5 percent of TRR (0.010 mg eq/kg) in liver and 4 percent of TRR (< 0.001 mg eq/kg) in muscle.

Other metabolites generally represented less than 10 percent TRR and always at < 0.01 mg eq/kg, of which 2-OH-fluindapyr, 5'-OH-fluindapyr, 3-OH-fluindapyr, and 3-OH-Met-fluindapyr were identified in both phenyl and pyrazole extracts. A glycine conjugate of pyrazole carboxylic acid was detected at a trace level of 0.001 mg eq/kg (11.4 percent TRR) in muscle.

Chiral analysis of fluindapyr isolated from fat and skin, showed that the S/R enantiomeric ratio changed from about 50/50 to 18/82 in fat and to 23/77 in the skin.

Conclusions

The Meeting concluded that, in all species investigated (goats, hens and rats), TAR was predominantly eliminated in excreta. Though some qualitative and quantitative differences were observed between the metabolic profiles of rat, goat, and laying hen, in general, the metabolism was considered to be similar.

Metabolism involved mainly demethylation to N-DesMet-fluindapyr and hydroxylation to 1-OH-Met-fluindapyr, with further sulfation being (mainly in hen tissues) and glucuronidation (goat tissues). Hydroxylation leading to 3-OH and 5-OH species in hens and di-OH-fluindapyr species in goats was also observed.

Fluindapyr (parent) is the major component found in the majority of the goat and hen tissues and cream samples (31–95 percent TRR), but with rather low levels in kidney and liver tissues (0.7–8.4 percent TRR).

1-OH-Met-fluindapyr was quantified at major amounts (up to 57 percent TRR) in goat and up to 32 percent TRR in hen tissues. Other major metabolites were 1-COOH-fluindapyr accounting for up to 27 percent TRR, but only in goat liver and kidney and up to 12 percent TRR in hen liver and muscle, N-DesMet-fluindapyr accounting for 36–61 percent TRR, but in hen liver only, 1-OH-Met-N-DesMet-fluindapyr and its glucuronides (12–24 percent TRR) in goat kidney, and 1-SO_4-Met-fluidapyr (17–18 percent TRR) in hen liver.

Methods of analysis

The Meeting received description and validation data for analytical methods for determination of fluindapyr, 3-OH-fluindapyr, DesMet-fluindapyr-1N-glucoside, 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr in plant matrices and for fluindapyr, N-DesMet-fluindapyr, 1-OH-Met-fluindapyr, 1-COOH-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr in animal commodities.

Plant commodities

Analytical methods are provided for the analysis of fluindapyr and/or metabolites in crops. The method selected for enforcement will depend on the compounds that are being analysed.

The QuEChERS (EN 15662:2009-2)-based method P3770G involves extraction with acetonitrile:water for direct analysis of fluindapyr, 3-OH-fluindapyr and DesMet-N- fluindapyr-glucoside by LC-MS/MS. For determination of 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and 1-COOH-fluindapyr, the extract was hydrolysed (1 hour at 60 °C) with HCl, the pH adjusted to 4 to 6 with NaOH, and cleaned-up by dispersive SPE (solid phase extraction) for quantification by LC-MS/MS. The method was fully validated for the determination of fluindapyr, 3-OH-fluindapyr, fluindapyr-DesMet-N-glucoside in the range of 0.01 (LOQ) to0.1 mg/kg in crops with high water content (sugar beet leaves, wheat forage), high starch content (sugar beet root), high acid content (grapes), high oil content (almond, pecan), high protein content (soya bean (dry seeds) and dry beans), and high starch content (wheat grain), and wheat straw and (dry and difficult matrix). An independent laboratory validation (ILV) was conducted to qualify this procedure as an enforcement method.

Corresponding radio-validation experiments demonstrated that Method P3770G was not suitable for the analysis of incurred residues of the metabolites 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr. Therefore, an alteration of the method was developed and validated (Method RA17.01), where the crop samples were extracted with water and acetonitrile, either by subsequent addition or combined addition (wheat straw) or acetontrile followed by water (high protein). The analytes in the extract were hydrolysed with 37 percent HCl (two hours at 80 °C), the pH adjusted (4–5), acetone and water added, followed by clean-up with SPE and quantification by LC-MS/MS. Method

RA.17.01 was fully validated for the determination of the (sum of) diastereomers 1-OH-Met-fluindapyr in crops with high oil content (almond and pecan nutmeat), high protein content (almond hulls, dry beans, soya bean seed), high acid content (grapes), high starch content (sugar beet roots, wheat grain and dry gluten) and high water content (sugar beet leaves and wheat forage), difficult matrices (wheat straw). A ILV was also performed

Both methods were subjected to radiovalidation, where incurred residues of fluindapyr were succesfully recovered from samples of wheat forage, grain and straw. Analytes involved were 3-OH-fluindapyr, 1-OH-Met-fluindapyr, 1-OH-Met-fluindapyr (both only in wheat forage and straw), 1-OH-Met-N-DesMet-fluindapyr, cis- and trans-1-COOH-fluindapyr, N-DesMet-pyr-acid, pyrazole carboxylic acid and pyrazole carboxamide. In addition DesMet-N-fluindapyr N1-Glu was succesfully recoverd from soya bean hay.

The Meeting concluded that the methods were sufficiently validated and are suitable to measure fluindapyr and its metabolites in plant commodities.

Animal commodities

In Method 133SRUS16R0208, samples of muscle, liver, kidney and eggs are blended with acetonitrile (2×) followed by extraction with acetone/water, and milk was extracted with acetonitrile. For analysis of fluindapyr and N-DesMet-fluindapyr, the extract was diluted and analysed by LC-MS/MS. For analysis of 1-COOH-fluindapyr-, 1-OH-Met-fluindapyr, and 1-OH-Met-N-DesMet-fluindapyr, the extract was hydrolysed with 4 mol/L HCl (80°C, 60 min), and cleaned-up by SPE before quantification by LC-MS/MS.

Fat was extracted with acetonitrile/hexane. For analysis of fluindapyr and N-DesMet-fluindapyr, the acetonitrile layer was diluted with water and cleaned-up by SPE before quantification. For analysis of 1-OH-Met-fluindapyr and 1-OH-Met-N-DesMet-fluindapyr, the acetonitrile layer was hydrolysed with 4 mol/L HCL (80 °C, 60 minutes) and cleaned-up before analysis by LC-MS/MS.

Method 133SRUS16R0208 was validated for the determination of fluindapyr, N-DesMet-fluindapyr, 1-OH-Met-fluindapyr (sum of both diastereomers), 1-OH-Met-N-DesMet-fluindapyr (sum of both diastereomers) in bovine muscle, fat, liver and kidney, and in poultry muscle, fat, liver and eggs in the range 0.01 (LOQ) to 0.1 mg/kg, and in the range of 0.005 (LOQ) to 0.05 mg/kg in milk. Validation was also conducted for 1-COOH-fluindapyr (sum of both diastereomers) in the range 0.01–0.1 mg/kg in bovine liver and kidney.

However, in a radiovalidation study, incurred residues of parent fluindapyr could not be succesfully (max 28 percent) recovered from goat (milk, muscle, liver) and hen (egg, fat) matrices. 1-OH-Met-fluindapyr was succesfully recovered from goat liver (110 percent) and egg (116 percent), 1-OH-Met-N-DesMet-fluindapyr from goat liver (90 percent), but the recovery of 1-COOH (both isomers) was questionable (280 percent).

The Meeting concluded that the analytical method for animal commodities is fit for measuring 1-OH-Met-fluindapy and 1-OH-Met-N-DesMet-fluindapyr, but is not fit for measuring residues of parent fluindapyr, 1-COOH-fluindapyr in animal matrices.

Stability of pesticide residues in stored analytical samples

The Meeting received information on storage stability of fluindapyr, 3-OH-fluindapyr, DesMet-fluindapyr-1N-glucoside, 1-OH-Met-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr, and 1-COOH-fluindapyr in wheat grain, wheat straw, grapes, oil seed rape seed, oil seed rape whole plant, and wheat dry gluten and of fluindapyr in wheat forage and hay (0.1 mg/kg fortification level). The data showed that residues of fluindapyr and metabolites (except for wheat forage; not tested for metabolites) are stable for at least 36

months under frozen conditions in crop commodities representative of the high water (oil seed rape whole plant), high acid (grapes), high starch (wheat grain), and high oil (oil seed raped seed) and high protein crops (wheat dry gluten).

The Meeting agreed that the demonstrated storage stability on various representative plant commodities covered the residue sample storage intervals used in the field trials considered by the current Meeting.

In addition, the Meeting received storage information of fluindapyr residues in animal matrices. Noting the uncertainties regarding the extraction efficiencies of the analytical method for fluindapyr and 1-COOH-fluindapyr, the Meeting cannot conclude on the storage stability of these analytes in animal commodities, except for 1-OH-Met-fluindapyr (both diastereomers) in liver and eggs and 1-OH-Met-N-Desmet-fluindapyr (both diastereomers) in liver,

Finally, the Meeting received storage stability information on spiked residue of fluindapyr in soil demonstrating that fluindapyr, 3-OH-fluindapyr, 1-COOH-fluindapyr, and pyrazole carboxylic acid are stable when stored frozen for period of 2 years.

Definition of the residue

Parent fluindapyr is a racemic mixture. In the absence of any indication that there is a difference in toxicology between the isomers of the parent or its metabolites, the Meeting concluded that they could be considered together and are therefore not reported individually.

Plant commodities

In the primary plant metabolism studies involving foliar applications, parent fluindapyr was the predominant residue, accounting for 63–65 percent TRR in grapes, 43–50 percent TRR in sugar beet roots, 46–56 percent TRR in wheat grains and 53–57 percent TRR in rice husked grain. Radioactive residues in soya bean seed were too low to detect any compound. Parent fluindapyr was also found in feed commodities, accounting for 15–18 percent TRR in sugar beet foliage to 31–37 percent TRR in wheat forage, with highest relative levels in rice straw (55–56 percent TRR).

Fluindapyr residues are not expected in rotational crops, and processing studies show that parent compound is also the main analyte in the processed commodities. Furthermore, suitable enforcement analytical methods exist to measure fluindapyr in plant commodities.

The Meeting concluded that fluindapyr is a suitable marker compound and decided to define the residue for compliance with the MRL for plants as fluindapyr.

In deciding which compounds should be included in the residue definition for dietary risk assessment of plant commodities, the Meeting considered the likely occurrence and the toxicological properties for the metabolites 1-OH-Met-fluindapyr (and its conjugates), 3-OH-fluindapyr, 1-COOH-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr (and its conjugate), N-DesMet-fluindapyr (and its conjugates), dehydro-fluindapyr, pyrazole carboxylic acid, pyrazole carboxamide, 3-OH-N-DesMet-fluindapyr, and the rotational crop metabolite N-DesMet-pyrazole carboxylic acid.

The Meeting concluded that metabolites 1-OH-Met-fluindapyr and its conjugate and 3-OH-fluindapyr are covered by the health based reference values for parent.

In metabolism studies, 1-OH-Met-fluindapyr and its conjugates accounted forup to 20 percent TRR (0.017/0.07 mg eq/kg) in grapes, 25 percent TRR (0.031 mg eq/kg) in sugar beet roots, and up to 17/22 percent TRR (0.17/0.11 mgkg eq) in rice grain. The compounds were not found in wheat grain, but contributed significantly to the total residue in wheat forage, hay and straw (35–60 percent TRR). In

contrast, in the wheat field trials, these metabolites were observed in grain, sometimes within the same range as parent fluindapyr. In sorghum grain, these metabolites were quantified in GAP-compliant field trials, but were <LOQ in maize grain, sweet corn, almond and pecan. The Meeting concluded that 1-OH-Met-fluindapyr and its conjugates should be included in the residue definition for dietary risk assessment for plant commodities.

3-OH-fluindapyr accounted for 6.1/1.7 percent TRR (0.002/0.005 mg eq/kg) in sugar beet root, up to 12–15 percent TRR (0.013/0.043 mg eq/kg) in grapes and 20/22 percent TRR (0.0042/0.0084 mg eq/kg) in wheat grain in primary crop metabolism studies. In the GAP-compliant field trials with wheat and sorghum, 3-OH-fluindapyr was found in grains. However, considering that the metabolite generally contributes less than 5 percent (with peaks up to 8.7 percent) to the residue of toxicological concern in GAP compliant field trials with cereals and also more than 80 percent of the residue of concern is covered in grapes without inclusion of 3-OH-fluindapyr, the Meeting decided not to include the metabolite in the residue definition for dietary risk assessment.

1-COOH-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr (and its N-conjugate), N-DesMet-fluindapyr and its conjugates, dehydro-fluindapyr, pyrazole carboxylic acid, pyrazole carboxamide, 3-OH-N-DesMet-fluindapyr were not consistently seen in all crops, contributed less than 6.1 percent TRR (0.005 mg eq/kg) individually, and the sum of them never exceed 10 percent TRR in any of the different primary crop metabolism studies. These metabolites were not further considered for the residue definition for dietary risk assessment for plant commodities.

The rotational crop metabolite N-DesMet-pyrazole carboxylic acid was found in several crops planted in rotation. Since the metabolite is a common metabolite, shared with fluxapyroxad, bixafen, benzovindiflupyr, and inpyrfluxam and is not covered by the health based reference values for fluindapyr, the Meeting concluded that the relevance of this metabolite should be evaluated against the TTC of a Cramer Class III compound (see Dietary Risk Assessment section)

The Meeting decided to define the residue for dietary risk assessment as the sum of fluindapyr and 1-OH-Met-fluindapyr and its conjugates, expressed as fluindapyr.

Animal commodities

Fluindapyr (parent) is a component found in the majority of the goat tissues and cream samples, but with rather low levels in kidney and liver. In poultry tissues the parent was observed in liver, muscle, skin and in eggs.

Noting that the analytical method used in the feeding studies is not fully suitable for quantification of the parent fluindapyr in animal commodities, the results of the dairy feeding studies were considered only qualitatively. In dairy cattle feeding studies, parent fluindapyr was observed in milk, liver and fat, but not muscle and kidney.

Parent fluindapyr is found in almost all goat and hen commodities in metabolism studies and is therefore a suitable marker compound.

The Meeting noted that no suitable analytical method exists to measure fluindapyr in animal commodities.

The Meeting decided to define the residue definition for compliance as fluindapyr.

The Log K_{ow} of fluindapyr is 4.1, indicating a potential to sequester into fatty matrices. In lactating goats, the ratio of fluindapyr in cream to skimmed milk was about 50 fold. It was 4 fold in fat to muscle in lactating goat, and approximately 15–18-fold in fat to muscle in laying hens. The Meeting considered the residue to be fat soluble.

In deciding which additional compounds should be included in the residue definition for risk assessment for animal commodities, the Meeting considered the likely occurrence of the compound at 10 percent TRR and/or absolute concentrations at ≥0.01 mg eq/kg, and their toxicity. Metabolites 1-OH-Met-fluindapyr, 1-COOH-fluindapyr, N-DesMet-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and its glucuronides, 1-SO_4-Met-fluidapyr, and 1-SO_4-Met-N-DesMet-fluidapyr were assessed. The Meeting concluded that all the metabolites, including their conjugates, are covered by the health based reference values for parent.

1-OH-Met-fluindapyr and it conjugates were found in all goat (up to 57 percent TRR) and hen tissues (up to 32 percent TRR) in the animal metabolism studies and were was also observed in the animal feeding studies. The Meeting concluded that 1-OH-Met-fluindapyr and its conjugates should also be included in the residue definition for dietary risk assessment for all animal tissues.

1-COOH-fluindapyr and its conjugates were found in the animal metabolism studies only in goat liver and kidney (up to 21–27 percent TRR; 0.005–0.075 mg eq/kg) and were also observed in the goat feeding study in these matrices. The compound was observed in hen liver and muscle (up to 12 percent TRR), but at levels < 0.01 mg eq/kg. The Meeting concluded that 1-COOH-fluindapyr and its conjugates should be included in the residue definition for dietary risk assessment in mammalian tissues.

N-DesMet-fluindapyr was observed at high relative and absolute levels in hen liver (36–61 percent TRR; 0.042–0.066 mg eq/kg) and at low levels in goat cream, fat and muscle and in hen skin, fat, eggs, and muscle, ranging from 1.3 to 6.8 percent TRR (< 0.001–0.004 mg eq/kg). This finding was confirmed in the laying hen feeding study. The Meeting concluded that N-DesMet-fluindapyr should be included in the residue definition for dietary risk assessment.

1-OH-Met-N-DesMet-fluindapyr accounted for 1.9–2.1 percent TRR (< 0.001 mg eq/kg) in skimmed milk and for 4.7–5.2 percent TRR, but < 0.001 mg eq/kg in goat muscle. The glucuronide conjugates were found in goat liver (4.3–8.9 percent TRR) and goat kidney (13–24 percent TRR), at levels ranging from 0.010 to 0.025 mg eq/kg. The metabolite was found in hen liver (8.5/4.0 percent TRR; 0.010/0.020 mg eq/kg) and muscle (1.9/4.0 percent TRR: < 0.001 mg eq/kg), but not distinguishable from its sulfate conjugate. The metabolites were also found in goat liver and kidney and in hen liver samples in the farm animal feeding studies. The Meeting concluded that 1-OH-Met-N-DesMet-fluindapyr and is conjugates should be included in the residue definition for dietary risk assessment.

1-SO_4-Met-fluidapyr and 1-SO_4-Met-N-DesMet-fluidapyr were observed at 17–18 percent TRR and 4.1–8.6 percent TRR in hen liver, with levels ranging from 0.008–0.022 mg eq/kg. The metabolites are expected to add insignificantly to the dietary intake at realistic dietary burden levels. Therefore, the Meeting concluded that these metabolites need not be included in the residue definition for dietary risk assessment.

The Meeting decided to define the residue for dietary risk assessment for animal commodities as the sum of fluindapyr, 1-OH-Met-fluindapyr, 1-COOH-fluindapyr, 1-OH-Met-N-DesMet-fluindapyr and their conjugates and N-DesMet-fluindapyr, expressed as fluindapyr.

Summary of residue definitions

The Meeting recommended the following residue definitions for fluindapyr:

Definition of the residue for compliance with the MRL assessment for plant commodities: *fluindapyr*

Definition of the residue for dietary risk assessment for plant commodities: *sum of fluindapyr and 3-(difluoromethyl)-N-[7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1H-inden-4-yl]-1-methyl-1H-pyrazole-4-carboxamide (1-OH-Met-fluindapyr) and its conjugates, expressed as parent*

Definition of the residue for compliance with the MRL assessment for animal commodities: *fluindapyr*

Definition of the residue for dietary risk assessment for animal commodities: *sum of fluindapyr, 4-(3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamido)-7-fluoro-1,3-dimethyl-2,3-dihydro-1H-indene-1-carboxylic acid (1-COOH-fluindapyr), 3-(difluoromethyl)-N-[7-fluoro-1-(hydroxymethyl)-1,3-dimethyl- 2,3-dihydro-1H-inden-4-yl]-1-methyl-1H-pyrazole-4-carboxamide (1-OH-Met-fluindapyr), 3-(difluoromethyl)-N-[7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1H-inden-4-yl]-1H-pyrazole-4-carboxamide (1-OH-Met-N-DesMet-fluindapyr) and their conjugates, and 3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-2,3-dihydro-1H-inden-4-yl)-1H-pyrazole-4-carboxamide (N-DesMet-fluindapyr), , expressed as fluindapyr.*

The residue is fat soluble.

Results of supervised residue trials on crops

The Meeting received supervised residue trial data for fluindapyr on wheat, sorghum, maize, sweet corn, tree nuts and almonds. Product labels were available from the United States.

When calculating the sum of fluindapyr and 1-OH-Met-fluindapyr for STMR and HR estimations, values < LOQ were assumed to be at the LOQ. As the metabolite is expressed as parent equivalents, no molecular weight conversion factor was needed.

The highest individual total residue values from the trials was used to derive HRs and the highest residues.

Cereal grains - grasses

The Meeting received supervised residue trials on wheat, sorghum, maize and sweet corn.

Wheat, similar grains, and pseudocereals without husks

In the United States, the critical GAP for cereals grains, except rice, is 2 foliar applications of fluindapyr at 150 g ai/ha, with a retreatment interval (RTI) of 10 days and a pre-harvest interval (PHI) of 30 days for grain.

Seventeen field residue trials conducted in the United States in 2016 matched the GAP. The residue levels for MRL estimation in ranked order were (n=16): 0.010, 0.017, 0.018, 0.023, 0.025, 0.026, 0.029, 0.041, 0.053, 0.060, 0.087, 0.088, 0.10, 0.12, 0.19, and 0.26 mg/kg.

Total residue levels for dietary risk assessment in ranked order were (n=16): 0.020, 0.027, 0.040, 0.040, 0.044, 0.044, 0.059, 0.070, 0.078, 0.082, 0.097, 0.098, 0.13, 0.14, 0.20, and 0.27 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.074 mg/kg for fluindapyr in the Subgroup of Wheat, similar grains, and pseudocereals without husks.

Sorghum grain and millet

In the United States, the critical GAP for sorghum is for 2 foliar applications of fluindapyr at 150 g ai/ha, with an RTI of 10 days and a PHI of 30 days for grain.

Eight field residue trials conducted in the United States in 2015 and 2016 matched the GAP. The residue levels for MRL estimation in ranked order were (n=8): 0.10, 0.24, 0.29, 0.34, 0.37, 0.37, 0.43, and 0.43 mg/kg.

Total residue levels for dietary risk assessment in ranked order were (n=8): 0.14, 0.29, 0.35, 0.38, 0.41, 0.42, 0.48, and 0.56 mg/kg.

Noting that the GAP of United States label for cereal grains, except rice, is similar to the GAP for sorghum grain and includes millet, the Meeting estimated a maximum residue level of 1.0 mg/kg and an STMR of 0.395 mg/kg for fluindapyr in the Subgroup of Sorghum Grain and Millet.

Maize cereals

In the United States, the critical GAP for maize cereals is for 2 foliar applications of fluindapyr at 150 g ai/ha, with an RTI of 10 days and a PHI of 30 days for grain.

Twenty field residue trials conducted in the United States in 2015 and 2016 matched the United States GAP. The residue levels for MRL estimation in ranked order were (n=20): < 0.01 (20) mg/kg.

Total residue levels for dietary risk assessment in ranked order were (n=20): < 0.02 mg/kg.

Since no residues were observed in any of the residue field trials, the Meeting estimated a maximum residue level of 0.01(*) mg/kg and an STMR of 0.02 mg/kg for fluindapyr in the Subgroup of Maize cereals.

Sweet corns

In the United States, the critical GAP for sweet corn is for 2 foliar applications of fluindapyr at 150 g ai/ha, with an RTI of 10 days and a PHI of 14 days for kernel + cobs with husks removed.

Eight field residue trials conducted in the United States in 2016 matched the United States GAP. Residues were measured in kernels + cobs with husks removed. The residue levels for MRL estimation in ranked order were (n=8): < 0.01 (8) mg/kg.

Total residue levels for dietary risk assessment in ranked order were (n=8): < 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and an STMR and an HR of 0.02 mg/kg for fluindapyr in Sweet corn (corn-on-the cob) (kernels plus cob with husk removed).

Tree nuts

In the United States, the critical GAP for tree nuts is for 3 foliar applications of fluindapyr at 168 g ai/ha, with an RTI of 7 days and a PHI of 30 days. The Meeting received data on almonds and on pecan nuts.

Almonds

Five field residue trials conducted in the United States in 2016 matched the United States GAP for tree nuts. The residue levels in almond nutmeat for MRL estimation in ranked order were (n=5): < 0.01 (2), 0.011, 0.018, and 0.022 mg/kg.

Total residue levels for dietary risk assessment in ranked order were (n=5): < 0.020 (2), 0.021, 0.028, and 0.032 mg/kg (highest individual value 0.035 mg/kg).

Pecan

Five field residue trials conducted in the United States in 2016 matched the United States GAP for tree nuts. The residue levels in pecan nutmeat for MRL estimation in ranked order were (n=5): < 0.01 (3), 0.016, and 0.024 mg/kg.

Total residue levels for dietary risk assessment in ranked order were (n=5): < 0.020 (3), 0.025, and 0.034 mg/kg.

Statistical analysis showed that the residue data with almond and pecans were similar. The combined data for maximum residue estimation in ranked order were (n=10): < 0.01 (5), 0.011, 0.016, 0.018, 0.022, and 0.024 mg/kg.

The combined data for dietary risk assessment in ranked order were (n=10): < 0.020 (5), 0.021, 0.025, 0.028, 0.032, and 0.034 mg/kg.

Noting that both almond and pecan are representative commodities for tree nuts, the Meeting estimated a maximum residue level of 0.04 mg/kg and an STMR of 0.0205 mg/kg for fluindapyr for the Group of Tree nuts.

Residues in animal feeds

Forages and fodders

Wheat forage

The critical GAP in the United States for cereals grains, except rice, allows for 2 foliar applications of fluindapyr at 150 g ai/ha, with an RTI of 10 days and a PHI of 7 days for forage.

Field residue trials on wheat forage, conducted in the United States in 2016, matched this GAP. Residue levels (parent only) in ranked order were (n=17): 0.16, 0.41, 0.54, 0.86, 1.4, 1.5, 1.5, 1.9, 2.2, 2.3, 2.4, 2.4, 2.4, 3.5, 4.2, 6.7, and 8.8 mg/kg (highest individual value 11 mg/kg).

Total residue levels (parent + 1-OH-Met-fluindapyr and its conjugates) for dietary intake calculations in ranked order were (n=17): 0.44, 0.60, 0.64, 1.2, 1.6, 1.6, 1.8, 2.2, 2.5, 2.5, 2.5, 2.6, 2.8, 3.7, 4.5, 6.9, and 9.2 mg/kg (highest individual value 12 mg/kg).

The Meeting estimated a median residue of 2.6 mg/kg (as received) and a highest residue of 12 mg/kg (as received) for wheat forage.

Wheat hay and wheat straw (both 88 percent dry matter)

The critical GAP in the United States for cereals grains, except rice, allows for 2 foliar applications of fluindapyr at 150 g ai/ha, with an RTI of 10 days and a PHI of 14 days (hay) and 30 days (straw).

Field residue trials on wheat hay, conducted in the United States in 2016, matched this GAP. The residue levels for MRL estimation in ranked order were (n=17): 0.072, 0.60, 0.63, 0.67, 0.69, 0.80, 0.82, 0.98, 1.2, 1.3, 1.3, 1.8, 1.8, 2.4, 2.4, 4.8, and 6.4 mg/kg (highest individual value 6.6 mg/kg).

Total residue levels (parent + 1-OH-Met-fluindapyr and its conjugates) for dietary burden calculations in ranked order were (n=17): 0.98, 0.99, 1.1, 1.2, 1.2, 1.4, 1.6, 1.8, 1.9, 1.9, 2.1, 2.3, 2.5, 2.6, 3.0, 6.6 and 6.9 mg/kg (highest individual value 7.1 mg/kg)

Field residue trials conducted with wheat straw in the United States in 2016 were performed with two foliar applications of fluindapyr at rates of 146-157 g ai/ha with an RTI of 9-12 days and harvested 30 DALA. The residue levels for MRL estimation in ranked order were (n=16): 0.16, 0.32, 0.34, 0.41, 0.54, 0.54, 0.79, 1.2, 1.4, 1.4, 1.9, 2.0, 2.1, 2.8, 9.6, and 11 mg/kg (highest individual value 12 mg/kg).

Total residue levels (parent + 1-OH-Met-fluindapyr and its conjugates) for dietary burden calculations in ranked order were (n=16): 0.22, 0.59, 0.61, 0.65, 0.83, 1.1, 1.7, 1.8, 1.8, 1.9, 2.1, 2.1, 2.4, 3.1, 10, and 12 mg/kg (highest individual value 13 mg/kg).

Based on the data set for straw, the Meeting estimated a maximum residue level of 15 mg/kg (dw) based on a dry matter content of 88 percent for Wheat, hay and/or straw.

The Meeting estimated a median residue of 1.9 mg/kg (as received) and highest residue of 7.1 mg/kg (as received) for fluindapyr in wheat hay and a median residue of 1.8 mg/kg (as received) and a highest residue of 13 mg/kg (as received) for fluindapyr in wheat straw.

Sorghum forage (35 percent dry matter)

The critical GAP in the United States for sorghum allows for 2 foliar applications of fluindapyr at 150 g ai/ha, with a RTI of 10 days with no livestock feeding restrictions.

Field residue trials on sorghum forage, conducted in the United States in 2015 and 2016, matched this GAP. Residue levels (parent only) in ranked order were (n=9): 0.24, 0.38, 0.42, 0.55, 0.62, 1.3, 2.4, 4.5, and 5.0 mg/kg (highest individual value 5.1 mg/kg).

Total residue levels (parent + 1-OH-Met-fluindapyr and its conjugates) for dietary burden calculations in ranked order were (n=9): 0.43, 0.46, 0.51, 0.66, 0.71, 1.4, 2.8, 4.7, and 5.1 mg/kg (highest individual value of 5.2 mg/kg).

The Meeting estimated a highest residue of 5.2 mg/kg (as received) and a median residue of 0.71 mg/kg (as received) for sorghum forage.

Sorghum stover (88 percent dry matter)

The critical GAP in the United States for sorghum allows for 2 foliar applications of fluindapyr at 150 g ai/ha, with an RTI of 10 days with a PHI of 30 days.

Field residue trials on sorghum stover, conducted in the United States in 2015 and 2016, matched this GAP. The residue levels for MRL estimation in ranked order were (n=8): 0.14, 0.16, 0.18, 0.21, 0.23, 0.44, 0.83, and 1.1 mg/kg (highest individual value of 1.7 mg/kg).

Total residue levels (parent + 1-OH-Met-fluindapyr and its conjugates) for dietary burden calculations in ranked order were (n=8): 0.19, 0.28, 0.32, 0.34, 0.45, 0.62, 0.95, and 1.8 mg/kg (highest individual value 2.4 mg/kg).

The Meeting estimated a maximum residue level of 3 mg/kg (dw), based on a dry matter content of 88 percent) for sorghum stover. The Meeting estimated a median residue of 0.395 mg/kg (as received) and a highest residue of 2.4 mg/kg (as received) for sorghum stover

Maize forage (40 percent dry matter)

The critical GAP in the United States for maize allows for 2 foliar applications of fluindapyr at 150 g ai/ha, with a RTI of 10 days and a PHI of 7 days.

Field residue trials on maize forage, conducted in the United States in 2015 and 2016, matched this GAP. Residue levels (parent only) in ranked order were (n=21): 0.077, 0.18, 0.23, 0.25, 0.33, 0.36, 0.44, 0.45, 0.48, 0.57, 0.73, 0.77, 0.79, 0.90, 0.96, 1.3, 1.4, 1.5, 1.8, 2.1, and 7.6 mg/kg (highest individual value 9.2 mg/kg).

Total residue levels (parent + 1-OH-Met-fluindapyr and its conjugates) for dietary burden calculations in ranked order were (n=21): 0.087, 0.21, 0.27, 0.35, 0.39, 0.46, 0.51, 0.56, 0.59, 0.64, 0.83, 0.86, 0.94, 0.97, 1.1, 1.4, 1.5, 1.5, 1.9, 2.3, and 8.2 mg/kg (highest individual residue 9.8 mg/kg).

The Meeting estimated and a median residue of 0.83 mg/kg (as received) and a highest residue of 9.8 mg/kg (as received) for maize forage.

Maize stover (83 percent dry matter)

The critical GAP in the United States for maize allows for 2 foliar applications of fluindapyr at 150 g ai/ha, with an RTI of 10 days and a PHI of 10 days.

Field residue trials on maize forage, conducted in the United States in 2015 and 2016, matched this GAP. The residue levels for MRL estimation in ranked order were (n=20): < 0.01, 0.22, 0.34, 0.34, 0.36, 0.54, 0.55, 0.57, 0.60, 0.76, 0.84, 0.89, 0.89, 1.1, 1.4, 1.7, 1.7, 2.0, 2.4, and 2.6 mg/kg (highest residue 2.8 mg/kg).

Total residue levels (parent + 1-OH-Met-fluindapyr and its conjugates) for dietary burden calculations in ranked order were (n=20): < 0.02, 0.30, 0.44, 0.61, 0.64, 0.66, 0.76, 0.83, 0.82, 0.90, 1.0, 1.0, 1.2, 1.4, 1.8, 2.0, 2.2, 2.7, 2.6, and 2.8 mg/kg (highest individual value 3.0 mg/kg).

The Meeting estimated a maximum residue level of 5 mg/kg (dw) based on a dry matter content of 83 percent. The Meeting estimated a median residue of 0.95 mg/kg (as received) and a highest residue of 3.0 mg/kg (as received) for maize stover.

Sweet corn forage (48 percent dry matter)

The critical GAP in the United States for sweet corn allows for 2 foliar applications of fluindapyr at 150 g ai/ha, with an RTI of 10 days with no livestock feeding restrictions.

Eight field residue trials on sweet corn forage, conducted in the United States in 2016, matched this GAP. Residue levels (parent only) in ranked order were (n=8): 0.022, 0.14, 0.18, 0.25, 0.33, 0.77, 0.98, and 5.4 mg/kg. (highest individual value 6.8 mg/kg).

Total residue levels (parent + 1-OH-Met-fluindapyr and its conjugates) for dietary burden calculations in ranked order were (n=8): 0.069, 0.24, 0.27, 0.47, 0.89, 0.93, 1.1, and 5.5 mg/kg (highest individual residue 6.9 mg/kg).

The Meeting estimated a median residue of 0.68 mg/kg (as received) and a highest residue of 6.9 mg/kg (as received) for sweet corn forage.

Sweet corn stover (83 percent dry matter)

The critical GAP in the United States for sweet corn allows for 2 foliar applications of fluindapyr at 146 g ai/ha, with an RTI of 10 days and a PHI of 10 days.

Eight field residue trials on sweet corn forage, conducted in the United States in 2016, matched this GAP. The residue levels for MRL estimation in ranked order were (n=8): 0.17, 0.19, 0.26, 0.28, 0.63, 0.65, 1.3, and 13 mg/kg.

Total residue levels (parent + 1-OH-Met-fluindapyr and its conjugates) for dietary burden calculations in ranked order were (n=8): 0.46, 0.59, 0.65, 0.83, 0.88, 0.88, 1.7, and 13 mg/kg.

The Meeting estimated a maximum residue level of 30 mg/kg (dw), based on a dry matter content of 83 percent. The Meeting estimated a median residue of 0.855 mg/kg (as received) and a highest residue of 13 mg/kg (as received) for sweet corn stover.

Miscellaneous animal feed

Almond hulls (90 percent dry matter)

The same trials as for almond (nutmeat) were considered for almond hulls. Five trials on almonds matched the criticalUnited StatesGAP on tree nuts (3 foliar applications each at 168 g ai/ha, an RTI of 7 days and harvested 30 DALA).

Residue levels (parent only) in ranked order were (n=5): 1.7, 2.5, 3.4, 6.0, and 8.2 mg/kg. Total residue levels (parent + 1-OH-Met-fluindapyr and its conjugates) for dietary burden calculations in ranked order were (n=5): 2.1, 2.9, 3.4, 6.0, and 8.2 mg/kg.

The Meeting estimated a maximum residue level of 20 mg/kg (dw), based on a dry matter content of 90 percent and a median residue of 3.4 mg/kg (as received) for fluindapyr in almond hulls.

Fate of residues during processing

High temperature hydrolysis

The Meeting received information on the hydrolysis of fluindapyr simulating typical processing conditions (pH 4, 6 and 6 with 90, 100 and 10 °C for 20, 60 and 20 minutes). No significant hydrolysis of fluindapyr was observed at the conditions studied. The Meeting concluded that fluindapyr is stable under the conditions of pasteurization, boiling, baking and brewing, as well as sterilization.

Residues in processed commodities

The fate of fluindapyr residues during commercial processing has been examined in wheat, sorghum, and maize. Processing factors derived for MRL estimation are based parent only. Processing factors derived for STMR estimation for food and median residue level estimation in feed are based on the residue definition for dietary risk assessment including total residue (parent + 1-OH-Met-fluindapyr). The results are shown in Table 3.

Table 137 Estimation of processing factors for commodities based on parent (MRL estimation) or parent + 1-OH-Met-fluindapyr (STMR and median residue level estimation)

Crop	Residue (mg/kg) in RAC		Processed commodity	Individual processing factors	Mean or best estimate PF	Residue (mg/kg) in processed commodity	
	MRL [a]	STMR [b]				MRL-P [a]	STMR-P or median residue-P [b]
Wheat	0.4	0.074	Whole meal flour	0.71, 0.81, 0.86, 1.0	0.845	-	0.063
			Total bran	0.95, 1.1, 1.4, 1.5	1.24	0.5 [c]	0.92
			White flour	0.28, 0.29, 0.30, 0.55	0.355	-	0.026
			Whole meal bread	0.44, 0.46, 0.54, 0.54	0.495	-	0.037
			Gluten feed meal	0.35, 0.37, 0.40, 0.71	0.458	-	0.034 [d]
			Germ	0.15, 0.42, 0.53, 0.60	0.425	-	0.031
Sorghum	1.0	0.395	Flour	0.42	0.42	-	0.17
			Aspirated grain fraction	6.9	6.9	7 [e]	2.7 [f]

Crop	Residue (mg/kg) in RAC		Processed commodity	Individual processing factors	Mean or best estimate PF	Residue (mg/kg) in processed commodity	
	MRL [a]	STMR [b]				MRL-P [a]	STMR-P or median residue-P [b]
Maize	0.01*	0.02	Flour	< 0.74	< 0.74	-	0.02
			Grits	< 0.8	< 0.8	-	0.02
			Meal	< 0.8	< 0.8	-	0.02
			Starch	< 0.8	< 0.8	-	0.02
			Refined bleached deodorized oil (wet milled)	1.1	1.1	-	0.022
			Refined bleached deodorized oil (dry milled)	1.8	1.8	0.03 [g]	0.36
			Aspirated grain fraction	27	27	0.5 [h]	0.54 [i]

Notes:

[a] parent only

[b] parent + 1-OH-fluindapyr

[c] MRL-P was based on the mean PF of 1.26 (individual values of 1.38, 1.51, 1.0 and 1.14) for parent only.

[d] A median residue-P of 0.025 was also derived for dietary burden estimation for MRL estimation, based on the individual PFs of 0.33, 0.41, 0.42 and 0.92, with a mean PF of 0.52 and a median residue based on parent only of 0.047 mg/kg.

[e] MRL-P was based on the single PF of 6.5 for parent only.

[f] A median residue-P of 2.31 was also derived for dietary burden estimation for MRL estimation, based on the single PF of 6.5 and a median residue based on parent only of 0.355 mg/kg.

[g] MRL-P was based on the single PF of 2.3 for parent only.

[h] MRL-P was based on the single PF of 44 for parent only.

[i] A median residue-P of 0.44 was also derived for dietary burden estimation for MRL estimation, based on the single PF of 44 and a median residue STMR based on parent only of 0.01 mg/kg

Residues in animal commodities

Farm animal feeding studies

The Meeting received farm animal feeding studies in lactating cows and laying hens.

Noting the uncertainties regarding the analytical method used (limited extraction efficiency of parent fluindapyr in a radio validation study) in both the dairy cow and laying hen feeding studies, the Meeting concluded that the results cannot be used for quantitative estimation of maximum residue level, STMR and HR estimation. Therefore, no details of the study were summarized here.

Farm animal dietary burden

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the current JMPR. Some processed and forage commodities do not appear in the Recommendations Table (because no maximum residue level is needed), but they are used in estimating livestock dietary burdens.

Table 138 Processed and forage commodities used in estimating livestock dietary burdens based on parent (P) or parent fluindapyr + 1-OH-Met-fluindapyr and its conjugates (T)

Codex classification	Commodity	Median residue (-P) (mg/kg) [a]	Highest residue (-P) (mg/kg) [a]
AS 0645	Corn, field, forage/silage (40 percent DM)	P: 0.73 T: 0.83	P: 9.2 T: 9.8
AS 3558	Maize stover (83 percent DM)	P: 0.80 T: 0.95	P:2.8 T: 3.0
AS 0656	Pop corn, stover (83 percent DM)	P: 0.455 T: 0.855	P: 13 T: 13
AS 0447	Corn, sweet, forage (48 percent DM)	P: 0.29 T: 0.68	6.9
AS 3563	Sweet Corn, stover (83 percent DM)	P: 0.455 T: 0.855	P: 13 T: 13
AS 0651	Sorghum, forage (green) (35 percent DM)	P: 0.62 T: 0.71	P: 5.1 T: 5.2
AS 3556	Sorghum, grain, stover (88 percent DM)	P: 0.22 T: 0.395	P: 1.7 T: 2.4
AS 3550	Sorghum, silage (21 percent DM)	P: 0.62 T: 0.71	P: 5.1 T: 5.2
AS 3552	Wheat, forage (25 percent DM)	P: 2.2 T: 2.5	P: 11 T: 12
AS 0654	Wheat, hay and/or straw (88 percent DM)	hay: P: 1.2; T: 1.9 straw: P: 1.3; T: 1.8	hay: P: 6.6;T: 7.1 straw: P: 12: T: 13
AS 3553	Wheat, silage (30 percent DM)	P: 2.2 T: 2.5	P: 11 T: 12
GC 0645	Corn, field, grain	0.02	n.a.
GC 0656	Corn, pop, grain	0.02	n.a.
GC 0651	Sorghum, grain	P: 0.355 T: 0.395	n.a.
GC 0654	Wheat, grain	P: 0.047 T: 0.074	n.a.
AM 0660	Almond, hulls	3.4	n.a.
	All corn by-products (asp gr fn, CF 3516; milled by-pdts; hominy meal; cannery waste; feed; meal, CF 3518)	P: 0.44 T: 0.54 for asp gr fn P: 0.01 T: 0.02 all other by products	n.a.
CF 3520	Sorghum, aspirated grain fraction	P: 2.3 T: 2.7	n.a.
CF 3521	Wheat, aspirated grain fraction	P: 0.047 T: 0.074	n.a.
CF 3522	Wheat, gluten meal	P: 0.025 T: 0.031	n.a.
CF 3514	Wheat, middlings (milled by-products)	P: 0.047 T: 0.074	n.a.

Notes:

[a] Levels for cereal straw, hay, and forage are presented on as received basis.

The dietary burdens, estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO manual, are presented in Annex 6 of the 2020 JMPR Report and summarised below.

The Meeting performed two mean and maximum dietary burden calculations; one based on parent only for MRL estimation and one based on parent + 1-OH-Met-fluindapyr (and its conjugates) for STMR and HR estimations (Tables 5 and 6).

Table 139 Estimated maximum and mean dietary burdens of farm animals based on parent only for MRL estimation

	Animal dietary burden: fluindapyr, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	3.772	3.965	25.66	4.202	44❶	12.84	0.176	0.176
Dairy cattle	15.11	10.369	18.17	3.809	35.6❷	12.84	11.64	7.117
Poultry – broiler	0.32	0.32	0.302	0.302	0.301	0.301	0.274	0.274
Poultry – layer	0.323	0.323	4.7❸	1.757	0.301	0.301	0.245	0.245

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

❷ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

❸ Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues and eggs.

Table140 Estimated maximum and mean dietary burdens of farm animals based on total intake

	Animal dietary burden: fluindapyr + 1-OH-Met-fluindapyr and its conjugates, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	4. 051	4.090	27.36	4.445	48❶	13.4	0.212	0.212
Dairy cattle	16.31	10.59	19.58	3.985	38.6	13.4❷	12.4	7.295
Poultry – broiler	0.37	0.37	0.341	0.341	0.341	0.341	0.31	0.31
Poultry – layer	0.365	0.365	5.138❸	1.824❹	0.341	0.341	0.281	0.281

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for HR estimates for mammalian tissues

❷ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues and milk.

❸ Highest maximum poultry dietary burden suitable for HR estimates for poultry tissues and eggs.

❹ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Animal commodity maximum residue levels

In the absence of a suitable analytical method for animal commodities no MRLs for animal commodities were recommended

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL assessment for plant commodities: *fluindapyr*

Definition of the residue for compliance with the MRL assessment for animal commodities: *fluindapyr*

Definition of the residue for dietary risk assessment for plant commodities: *sum of fluindapyr and 3-(difluoromethyl)-N-[7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1H-inden-4-yl]-1-methyl-1H-pyrazole-4-carboxamide (1-OH-Met-fluindapyr) and its conjugates, expressed as parent*

Definition of the residue for dietary risk assessment for animal commodities: sum of fluindapyr, 4-(3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamido)-7-fluoro-1,3-dimethyl-2,3-dihydro-1H-indene-1-carboxylic acid (1-COOH-fluindapyr), 3-(difluoromethyl)-N-[7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1H-inden-4-yl]-1-methyl-1H-pyrazole-4-carboxamide (1-OH-Met-fluindapyr), 3-(difluoromethyl)-N-[7-fluoro-1-(hydroxymethyl)-1,3-dimethyl-2,3-dihydro-1H-inden-4-yl]-1H-pyrazole-4-carboxamide (1-OH-Met-N-DesMet-fluindapyr) and their conjugates, and 3-(difluoromethyl)-N-(7-fluoro-1,1,3-trimethyl-2,3-dihydro-1H-inden-4-yl)-1H-pyrazole-4-carboxamide (N-DesMet-fluindapyr), , expressed as fluindapyr.

The residue is fat-soluble.

Table 141 Residue levels suitable for establishing maximum residue limits and for IEDI and IESTI assessments

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P or median residue mg/kg	HR or HR-P or highest residue mg/kg
		New	Previous		
AM 0660	Almond hulls	20 (dw)	-	3.4	-
GC 2091	Maize cereals, Subgroup of	0.01(*)	-	0.02	0.02
AS 3558	Maize, stover	5 (dw)	-	0.95 (ar)	3.0 (ar)
GC 2089	Sorghum Grain and Millet, Subgroup of	1.0	-	0.41	0.62
AS 3561	Sorghum, stover	3 (dw)	-	0.395	2.4
GC 0447	Sweet corn (corn-on-the cob) (kernels plus cob with husk removed)	0.01(*)	-	0.02	0.02
AS 3563	Sweet corn, stover	30 (dw)	-	0.855	13
TN 0085	Tree nuts, Group of	0.04	-	0.0305	0.045
GC 2086	Wheat, similar grains, and pseudo cereals without husks, Subgroup of	0.4	-	0.092	0.29
AS 0654	Wheat, hay and/or straw	15 (dw)	-	hay: 1.9 (ar) straw: 1.8 (ar)	hay: 7.1 (ar) straw: 13 (ar)
CF 1255	Maize, flour	-	-	0.02	-
-	Maize, grits	-	-	0.02	-
CF 0645	Maize, meal	-	-	0.02	-
-	Maize, starch	-	-	0.02	-
OR 0645	Maize, refined deodorized oil	0.02	-	0.036	-
CF 3520	Sorghum, Grain, flour	-	-	0.17	-
CF 0654	Wheat, bran, processed	0.5	-	0.92	-
CF 3522	Wheat, gluten meal	-	-	0.034	-
CF 1210	Wheat, germ	-	-	0.031	-
CF 1212	Wheat, whole meal	-	-	0.063	-
CF 1211	Wheat, flour	-	-	0.026	-
-	Wheat, wholemeal bread	-	-	0.037	-
AS 3558	Maize, forage	-	-	0.83 (ar)	9.8 (ar)
	Maize, aspirated grain fraction	0.3	-	0.54	-
AS 3561	Sorghum, forage	-	-	0.71 (ar)	5.2 (ar)
	Sorghum, aspirated grain fraction	7	-	2.7	-
AS 3563	Sweet corn, forage	-	-	0.68 (ar)	6.9 (ar)
AS 0654	Wheat, forage	-	-	2.5 (ar)	12 (ar)

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P or median residue mg/kg	HR or HR-P or highest residue mg/kg
		New	Previous		

Notes:

(ar) – as received; (dw) – dry weight

FUTURE WORK

Information to demonstrate the extraction efficiency of fluindapyr and related metabolites in animal commodities.

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for fluindapyr is 0–0.04 mg/kg bw/day. The International Estimated Daily Intakes (IEDIs) for fluindapyr were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 1–5 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of fluindapyr from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for fluindapyr is 0.6 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for fluindapyr were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs varied from 0–1 percent of the ARfD for children and 0–1 percent of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of fluindapyr from uses considered by the present Meeting is unlikely to present a public health concern.

Threshold of toxicological concern (TTC) consideration for metabolites

The Meeting concluded that metabolite N-DesMet-pyrazole carboxylic acid, found in rotational crop studies (root crop and oil seeds), could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day). The Meeting estimated a dietary exposure for metabolite N-DesMet-pyrazole carboxylic acid 0.366 µg/kg bw per day.

The Meeting concluded that the estimated dietary exposure to residues of N-DesMet-pyrazole carboxylic acid from uses considered by the current JMPR is below the TTC for Cramer Class III compounds and is unlikely to present a public health concern. Should further uses be considered in the future, these conclusions may need to be re-evaluated.

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Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
2016RES-IFP2942	Brungardt, J.N.	2018	Magnitude of residue of F9990 in milk, kidney, muscle, fat and liver of lactating dairy cattle following oral administration. SynTech Research Laboratory Services, LLC, United States; Genesis Midwest, LLC, United States. Report Number 133SRUS16R0209, Tracking Number 2016RES-IFP2942. GLP: Yes. Unpublished.
2016RES-IFP2943	Brungardt, J., Dixon, H.	2018	Magnitude of residue of F9990 in eggs, fat, liver and muscle of laying hens following oral administration. SynTech Research Laboratory Services, LLC, United States; Genesis Midwest Laboratories, LLC, United States. Report Number 133SRUS16R0210, Tracking Number 2016RES-IFP2943. GLP: Yes. Unpublished.
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2013MET-IFP0730	Desai, M.	2016	Nature of the residue: Metabolism of [14C]IR9792/F9990 in/on soybean crop. Excel Research Services, Inc.; XenoBiotic Laboratories, Inc., United States. Report Number XBL 13027 - RPT03280, Tracking Number 2013MET-IFP0730. GLP: Yes. Unpublished.
2016MET-IFP2538	Desai, M.	2017a	[14C]IR9792/F9990: Nature of the residue: Metabolism of [14C]IR9792/F9990 in/on grape crop. Lange Research and Consulting, Inc.; XenoBiotic Laboratories, Inc., United States. Report Number XBL 16064, Tracking Number 2016MET-IFP2538. GLP: Yes. Unpublished.
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2015EFT-IFP1999	Gemrot, F.	2018	Soil dissipation study after two applications of IRF205-1 in Northern Europe (Germany and The United Kingdom) and Southern Europe (Southern France and Italy) - 2015-2017 (Amendment No. 1). SGS France - Agricultural & Food, France. Report Number 15SGS089 Amendment No. 1, Tracking Number 2015EFT-IFP1999 Amendment No. 1. GLP: Yes. Unpublished.
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2013MET-IFP0693	Mainolfi, K.	2017	Metabolism of [^{14C}-phenyl]IR9792/F9990 in rotational crops. ISAGRO GLP Test Facility, Italy. Report Number MEF.13.08, Tracking Number 2013MET-IFP0693. GLP: Yes. Unpublished.
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2013EFT-IFP0874	Mainolfi, K., Colombini, A.	2016b	Aerobic metabolism of ^{14C}-IR9792 (F9990) in one US soil. ISAGRO GLP Test Facility, Italy. Report Number MEF.13.19, Tracking Number 2013EFT-IFP0874. GLP: Yes. Unpublished.
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2013PCP-IFP0781	Martinez, M.P.	2014a	IR9792 (F9990) technical product: Determination of the colour, odour and physical state. ChemService S.r.l., Italy. Report Number CH-229/2013, Tracking Number 2013PCP-IFP0781. GLP: Yes. Unpublished.
2013PCP-IFP0782	Martinez, M.P.	2014b	IR9792 (F9990) purified product: Determination of the colour, odour and physical state. ChemService S.r.l., Italy. Report Number CH-228/2013, Tracking Number 2013PCP-IFP0782. GLP: Yes. Unpublished.

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
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2013PCP-IFP0787	Martinez, M.P.	2014d	IR9792 (F9990) purified product: Determination of the melting point. ChemService S.r.l., Italy. Report Number CH-233/2013, Tracking Number 2013PCP-IFP0787. GLP: Yes. Unpublished.
2013PCP-IFP0779	Martinez, M.P.	2014e	IR9792 (F9990) technical product: Determination of the relative density. ChemService S.r.l., Italy. Report Number CH - 232/2013, Tracking Number 2013PCP-IFP0779. GLP: Yes. Unpublished.
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2013PCP-IFP0784	Martinez, M.P.	2014g	IR9792 (F9990) technical product: Determination of the pH value. ChemService S.r.l., Italy. Report Number CH-230/2013, Tracking Number 2013PCP-IFP0784. GLP: Yes. Unpublished.
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Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			Report Number S17-07386, Tracking Number 2017AMT-IFP3873. GLP: Yes. Unpublished.
2017AMT-IFP3870	Sahvorost, N.	2018d	Independent laboratory validation of analytical method for the determination of IR9792/F9990, 3-hydroxy-IR9792/F9990, 1-carboxy-IR9792/F9990 (sum of diastereomers), and pyrazole carboxamide in soil. Eurofins Agroscience Services, Inc. United States. Report Number S17-07372, Tracking Number 2017AMT-IFP3870. GLP: Yes. Unpublished.
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2018RES-FNF4542	Skaggs, C.	2019a	Determination of pyrazole carboxamide residues in almond and pecan nutmeat raw and processed commodities. SGS North America, Inc. Report Number. SGS-18-01-06, Tracking Number 2018RES-FNF4542. GLP: Yes. Unpublished.
2018RES-IFP4183	Skaggs, C.	2019b	Determination of pyrazole carboxamide in or on soybean raw agricultural and processed commodities. SGS North America, Inc. Report Number SGS-18-01-03, Tracking Number 2018RES-IFP4183. GLP: Yes. Unpublished.
2018RES-IFP4200	Skaggs, C., Afedi, P.	2019	Determination of pyrazole carboxamide, pyrazole carboxylic acid and N-desmethyl-pyrazole carboxylic acid in or on rotated crops. SGS North America, Inc. Report number SGS-18-01-04, Tracking Number 2018RES-IFP4200. GLP: Yes. Unpublished.
2014RES-IFP1459	Soddu, R.	2017a	Storage stability of IR9792 (F9990) in wheat matrices (grain, forage, hay and straw) stored in the dark below -20°C. Isagro - Centro di Saggio BPL, Italy. Report Number RA.14.10, Tracking Number 2014RESIFP1459.

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			GLP: Yes. Unpublished.
2014RES-IFP1459	Soddu, R.	2017b	Storage stability of IR9792 (F9990) in wheat matrices (grain, forage, hay and straw) stored in the dark below -20°C. Isagro - Centro di Saggio BPL, Italy. Report Number RA.14.10, Tracking Number 2014RESIFP1459. GLP: Yes. Unpublished.
2016RES-IFP2653	Soddu, R.	2020	Storage stability of IR9792/F9990 and its metabolites in wheat (grain, dry gluten, and straw), oilseed rape (seed and whole plant) and grapes stored in the dark below -20°C. Isagro - Centro di Saggio BPL, Italy. Report Number RA.16.10, Tracking Number 2016RES-IFP2653. GLP: Yes. Unpublished.
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2014RES-IFP1238	Soddu, R. and Sicbaldi, F.	2014b	Matrix effect of IR9792 (F9990) in wheat (grain, forage, hay, and straw). Isagro SpA. Report Number RA.14.03, Tracking Number 2014RES-IFP1238. GLP: Yes. Unpublished.
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2014RES-IFP1240	Soddu, R. and Sicbaldi, F.	2014d	Stability of working (fortification/calibration) solutions of IR9792 (F9990) under controlled storage conditions. Isagro SpA. Report Number RA.14.01, Tracking Number 2014RES-IFP1240. GLP: Yes. Unpublished.
2015RES-IFP2155	Stanislowski, T.	2016a	Development and subsequent validation of a residue analytical method for the determination of F9990, its metabolites and its conjugated metabolites in various plant/crop materials. PTRL Europe, Germany. Report Number P3770G, Tracking Number 2015RES-IFP2155. GLP: Yes. Unpublished
2016RES-IFP2666	Stanislowski, T.	2016b	Development and subsequent validation of a residue analytical method for the enantio-selective (chiral) determination of F9990 in various crop materials. PTRL Europe, Germany. Report Number P 3928 G, Tracking Number 2016RES-IFP2666. GLP: Yes. Unpublished.
2015MET-IFP2176	Thomas, J.A.	2019a	Metabolism of $^{[14C]}$-IR9792/F9990 in the lactating goat. Charles River Laboratories Ashland, LLC, United States. Report Number WIL-236509, Amendment No. 1, Tracking Number 2015MET-IFP2176, Revision No. 1. GLP: Yes. Unpublished.
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2013MET-IFP0758	Tuffnail, W.	2017	Nature of the residue: Metabolism of $^{[14C]}$IR9792/F9990 in/on sugar beet. AgroChemex Limited, Manningtree; Pharmaron United Kingdom Ltd. Report Number FCC/01 and FCC/01 Amendment No. 1, Tracking Number 2013MET-IFP0758, Amendment No. 1.

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			GLP: Yes. Unpublished.
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2013EFT-IFP0763	Vanini, L.	2016b	Aerobic degradation of ^{14C}-IR9792/F9990 in three US soils. Isagro GLP Test Facility, Italy. Report Number MEF.13.17 amended report, Tracking Number 2013EFT-IFP0763. GLP: Yes. Unpublished.
2015EFT-IFP2086	Vanini, L.	2016c	Aerobic degradation of ^{14C}-3-hydroxy-IR9792/F9990 (code 510152) in three U.S. soils. Isagro GLP Test Facility, Italy. Report Number MEF.15.02, Tracking Number 2015EFT-IFP2086. GLP: Yes. Unpublished.
2016EFT-IFP2504	Vanini, L.	2016d	Aerobic degradation of [^{14C}]*trans*-1-carboxy-IR9792/F9990 in three European soils. Isagro GLP Test Facility, Italy. Report Number MEF.16.06, Tracking Number 2016EFT-IFP2504. GLP: Yes. Unpublished.
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2015RES-FNF1900	Webber, T.	2018a	Magnitude and decline of residues of F9944 and metabolites in/on field corn and processed fractions following applications of F9944-6. Precision Study Management, United States; Battelle, United States. Report Number PSM-15-02-03, Tracking Number 2015RES-FNF1900. GLP: Yes. Unpublished.
2016RES-FNF2453	Webber, T.	2018b	Magnitude of the residue of F9944 and metabolites in/on field corn following application of F9944-74. Precision Study Management, United States; Battelle, United States. Report Number PSM-16-02-08, Tracking Number 2016RES-FNF2453. GLP: Yes. Unpublished.

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
2016RES-FNF2454	Webber, T.	2018c	Magnitude and decline of the residues of F9944 and metabolites in/on sweet corn following application of F9944-74. Precision Study Management, United States; Battelle, United States. Report Number PSM-16-02 09, Tracking Number 2016RES-FNF2454. GLP: Yes. Unpublished.
2015RES-FNF1901	Webber, T.	2018d	Magnitude of the residues of F9944 and metabolites in/on sorghum and processed fractions. Precision Study Management, United States; Battelle, United States. Report Number PSM-15-02-04, Tracking Number 2015RES-FNF1901. GLP: Yes. Unpublished.
2016RES-FNF2455	Webber, T.	2018e	Magnitude of the residue of F9944 and metabolites in/on sorghum following application of F9944-74. Precision Study Management, United States; Battelle, United States. Report Number PSM-16-02-10, Tracking Number 2016RES-FNF2455. GLP: Yes. Unpublished.
2016RES-FNF2456	Webber, T.	2018f	Magnitude and decline of the residue of F9944 and metabolites in/on wheat following application of F9944-74. Precision Study Management, United States; Battelle, United States. Report Number PSM 16 02 11, Tracking Number 2016RES-FNF2456. GLP: Yes. Unpublished.

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Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
	Ghiglieri, A.	2016a	Excretory balance (urine, feces, CO2), blood and plasma levels and tissue distribution of radioactivity after administration of [^{14C}-phenyl]-IR9792 (F9990) to rat. Accelera S.r.l. Report Number 2014-0012, Tracking Number 2014MET-IFP1211. GLP: Yes. Unpublished.
	Ghiglieri, A..	2016b	Excretory balance (urine, feces) of radioactivity after administration of [^{14}C-pyrazole]-IR9792 (F9990) to rat. Accelera S.r.l. Report Number 2014-0013, Tracking Number 2014MET-IFP1212. GLP: Yes. Unpublished.
	Ghiglieri, A.	2017	Bile excretion of radioactivity in rat after administration of ^{14}C-pyrazole] IR9792/F9990. Accelera S.r.l. Report Number 2017-0030, Tracking Number 2017MET-IFP3284. GLP: Yes. Unpublished.
	Krebbers, S.F.M.	2017	Interspecies comparison of in vitro metabolism of [^{14}C]-IR9792/F9990 in rat, mouse, dog and human hepatocytes. Charles River Laboratories Den Bosch B.V. Report Number 509750, Tracking Number 2015MET-IFP2071. GLP: Yes. Unpublished.
	Mainolfi, K., Colombini, A.	2014	Adsorption-desorption of ^{14}C-IR9792 in five European soils. Isagro GLP Test Facility, Italy. Report Number MEF.13.11, Tracking Number 2013EFT-IFP0718. GLP: Yes. Unpublished.
	Mainolfi, K., Garau, S.	2017a	Identification/characterization of metabolites in excreta and plasma from ^{14}C-IR9792/F9990 in rat (following single and repeated oral administrations). Isagro GLP Test Facility. Report Number MEF.14.05, Tracking Number 2014MET-IFP1622. GLP: Yes. Unpublished.

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
	Mainolfi, K., Garau, S.	2017b	Anaerobic metabolism of ^{14}C-IR9792/F9990 in one EU soil. Isagro GLP Test Facility, Italy. Report Number MEF.16.02, Tracking Number 2016EFT-IFP2411. GLP: Yes. Unpublished.
	Mainolfi, K., Garau, S.	2017c	Anaerobic degradation of ^{14}C- IR9792/F9990 in three EU soils. Isagro GLP Test Facility, Italy. Report Number MEF.16.01, Tracking Number 2016EFT-IFP2412. GLP: Yes. Unpublished.
	Mainolfi, K., Garau, S.	2018	Identification/characterization of ^{14}C-IR9792/F9990 metabolites in bile duct cannulated rats following repeated oral administration. Isagro GLP Test Facility. Report Number MEF.17.13, Tracking Number 2017MET-IFP3893. GLP: Yes. Unpublished.
	Martinez, M.P.	2014o	IR9792 (F9990) technical product: Determination of the oxidizing properties. ChemService S.r.l., Italy. Report Number CH-239/2013, Tracking Number 2013PCP-IFP0799. GLP: Yes. Unpublished.
	Martinez, M.P.	2014p	IR9792 (F9990) technical product: Determination of the flammability. ChemService S.r.l., Italy. Report Number CH-238/2013, Tracking Number 2013PCP-IFP0785. GLP: Yes. Unpublished.
	Mazzei, N	2014	Explosive properties and relative self-ignition temperature (solid) on the sample IR9792 (F9990) technical product. Innovhub, Italy. Report Number 201302495, Tracking Number 2013PCP-IFP0835. GLP: Yes. Unpublished.
	Noè, F.	2013	IR9792(F9990) technical product: Ready biodegradability in a manometric respirometry test. ChemService S.r.l. Report Number CH-191/2013, Tracking Number 2013EFT-IFP0757. GLP: Yes. Unpublished.
	Riccelli, S.	2018	Method validation for the determination of cis-1-carboxy-IR9792/F9990 (Code # 51070), trans-1-carboxy-IR9792/F9990 (Code # 510169), and 3-hydroxy-IR9792/F9990 (Code # 510152) in drinking water. Isagro SpA. Report Number RA.18.09, Tracking Number 2018RES-IFP4319. GLP: Yes. Unpublished.
	Schmiedt, S.	2018	Independent laboratory validation (ILV) of an analytical method for the determination of fluindapyr and metabolites in drinking water. EAG Laboratories GmbH, Germany. Report Number P 4865 G, Tracking Number 2018AMT-IFP4416. GLP: Yes. Unpublished.
	Simmonds, M., Burgess, M.	2009	SYN524464 - Adsorption desorption of metabolite [^{14}C]-CSCC210616. Battelle United Kingdom Ltd. Report Number NC/08/026, Syngenta file number SYN508272_10886. GLP: Yes. Unpublished.
	Soddu, R., Sicbaldi, F.	2014e	Setup and validation of an analytical method for determination of IR9792 (F9990) residue in drinking water and surface water. Isagro GLP Test Facility, Italy. Report Number RA.14.13, Tracking Number 2014RES-IFP1560. GLP: Yes. Unpublished.
	Vanini, L., Pizzella, S.	2014	Adsorption-desorption of ^{14}C-IR9792 in five U.S. soils. Isagro GLP Test Facility, Italy. Report Number MEF.13.10, Tracking Number 2013EFT-IFP0700. GLP: Yes. Unpublished.
	Vanini, L., Pizzella, S.	2016b	Adsorption-desorption of ^{14}C-3-hydroxy-IR9792/F9990 (code 510152) in three EU and three US soils. Isagro GLP Test Facility, Italy. Report Number MEF.15.01, Tracking Number 2015EFT-IFP2066. GLP: Yes. Unpublished.

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
	Vanini, L., Pizzella, S.	2016c	Adsorption-desorption of ^{14}C-trans-1-carboxy-IR9792/F9990 (code 510169) in three EU soils. Isagro GLP Test Facility, Italy. Report Number MEF.16.08, Tracking Number 2016EFT-IFP2506. GLP: Yes. Unpublished.
	Vanini, L., Pizzella, S.	2016d	Adsorption-desorption of ^{14}C-cis-1-carboxy-IR9792/F9990 (code 510170) in three EU soils. Isagro GLP Test Facility, Italy. Report Number MEF.16.07, Tracking Number 2016EFT-IFP2505. GLP: Yes. Unpublished.
	Vanini, L.	2016e	Aerobic transformation of ^{14}C-IR9792/F9990 in aquatic sediment systems. Isagro GLP Test Facility, Italy. Report Number MEF.14.06; Tracking Number 2014EFT-IFP1765. GLP: Yes. Unpublished.
	Vanini, L.	2017b	Anaerobic transformation of ^{14}C-IR9792/F9990 in aquatic sediment systems. Isagro GLP Test Facility, Italy. Report Number MEF.16.12, Tracking Number 2016EFT-IFP3010. GLP: Yes. Unpublished.
	Vanini, L.	2018	Aerobic mineralization of ^{14}C-IR9792/F9990 in surface water. Isagro GLP Test Facility, Italy. Report Number MEF.17.07, Tracking Number 2017EFT-IFP3410. GLP: Yes. Unpublished.

FLUPYRADIFURONE (285)

First draft prepared by Mr D Lunn, Ministry for Primary Industries, Wellington, New Zealand

EXPLANATION

Flupyradifurone is an insecticide with the structure of butenolides, acting as an agonist of nicotinic acetylcholine receptor. It was first evaluated by the JMPR for toxicology in 2015 and for residues by the 2016, 2017 and 2019 JMPRs.

The 2015 Meeting established an ADI of 0–0.08 mg/kg bw and an ARfD of 0.2 mg/kg bw and the 2016 JMPR established the following residue definitions:-

For compliance with the MRL (plant commodities): *Flupyradifurone*

For estimation of dietary exposure (for plant commodities): *Sum of flupyradifurone, difluoroacetic acid (DFA) and 6-chloronicotinic acid (6-CNA), expressed as parent equivalents*

For compliance with the MRL and for estimation of dietary exposure (animal commodities): *Sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents*

The residue is not fat-soluble.

The Fifty-second Session of the CCPR (2021) listed flupyradifurone for further evaluation by the 2022 JMPR and the current Meeting received revised GAP information and new supporting residue information from the manufacturer for mango, papaya, pineapple, sesame seeds and sunflower seeds.

RESIDUE ANALYTICAL METHODS

Analytical methods

A number of analytical methods (for enforcement and data collection) for plant and animal matrices were evaluated by the 2016 and 2019 Meetings. Method RV-001-P10-02 was shown to be suitable for measuring residues of parent flupyradifurone, difluoroacetic acid (DFA), 6-chloronicotinic acid (6-CNA) and also difluoroethyl-amino-furanone (DFEAF) in a range of plant commodities with a high water content, high acid content, high oil content and high starch/protein content.

A slight modification of this method (RV-001-P10-03) was also evaluated by the 2019 JMPR for measuring residues of parent flupyradifurone, DFA, 6-CNA (and DFEAF) in blackberry, raspberry and avocado.

As summarised by the 2019 JMPR, in these methods, residues are extracted twice from plant material with acetonitrile/water (4/1, v/v) with 2.2 mL/L formic acid and extracts are purified by C-18 SPE (silica or trifunctional amide columns for DFA), with analysis by HPLC-MS/MS.

Validation data for Method RV-001-P10-02 used for determination of flupyradifurone-related residues in mango and papaya are summarized below.

Table 1 Summary of analytical method 01304 (RV-001-P10-02) validation results for plant commodities.

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean percent recovery	RSD (%)
Flupyradifurone	(m/z 289 → 126 for quantification)			**[Ref: RARV0287]**	
Mango (whole fruit)	0.01	5	102, 103, 100, 96, 97	100	3
	0.1	5	98, 96, 99, 99, 99	98	1
	2.0	5	104, 98, 97, 102, 102	101	3

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean percent recovery	RSD (%)
Mango pulp	0.01 0.1	3 3	101, 103, 109 105, 103, 102	104 103	4 1
Mango peel	0.01 0.1 2.0	3 3 3	105, 103, 94 100, 100, 100 95, 95, 93	101 100 94	6 0 1
DFA	(m/z 95 → 51 for quantification)				**[Ref: RARV0287]**
Mango (whole fruit)	0.05 0.5	5 5	96, 98, 97, 96, 96 94, 94, 96, 96, 98	96 96	1 2
Mango pulp	0.05 0.5	3 3	94, 101, 101 100, 98, 98	99 99	4 1
Mango peel	0.05 0.5 1.0	3 3 3	92, 96, 94 94, 98, 92 90, 92, 89	94 95 90	2 3 1
6-CNA	(m/z 156 → 112 for quantification)				**[Ref: RARV0287]**
Mango (whole fruit)	0.01 0.1 0.2	5 5 5	89, 91, 110, 99, 105 94, 97, 103, 104, 105 97, 103, 94, 101, 95	99 100 98	9 5 4
Mango pulp	0.01 0.1 0.2	3 3 3	97, 96, 101 105, 105, 109 97, 99, 103	98 106 99	3 2 3
Mango peel	0.01 0.1 0.2	3 3	93, 103, 110 101, 100, 101 92, 90, 93	102 010 91	8 1 2
DFEAF	(m/z 162 → 98 for quantification)				**[Ref: RARV0287]**
Mango (whole fruit)	0.01 0.1	5 5	93, 92, 104, 110, 82 95, 93, 89, 93, 102	96 94	11 5
Mango pulp	0.01 0.1	3 3	94, 110, 99 103, 110, 102	101 105	8 4
Mango peel	0.01 0.1 0.25	3 3 3	102, 107, 94 104, 110, 101 97, 90, 93	101 105 93	6 4 3

Concurrent recovery data for the methods used for determination of flupyradifurone residues in plant commodities for which supervised trial data were submitted to the current Meeting are summarized below.

Table 2 Summary of analytical method 01304 (RV-001-P10-02) concurrent recovery results for plant commodities

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean percent recovery	RSD (%)	Reference
Flupyradifurone						
Mango (whole fruit)	0.01 0.1	3 2	103, 98, 110 102, 106	104 104	6 -	RARV0287
Mango pulp	0.01 0.1	1 1	95 99	95 99	- -	RARV0287

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean percent recovery	RSD (%)	Reference
Mango peel	0.01 0.1	2 1	93, 93 93	93 93	- -	RARV0287
Papaya fruit	0.01 10	5 5	103 ;92; 98; 93; 99 99; 80; 93; 85; 105	97 92	4.6 11	I17-009
DFA						
Mango (whole fruit)	0.05 0.5	1 1	81 97	81 97	- -	RARV0287
Mango pulp	0.05 0.5	1 1	96 95	96 95	- -	RARV0287
Mango peel	0.05 0.5	2 1	83, 96 89	90 89	- -	RARV0287
Papaya fruit	0.05 10	5 5	89; 90; 87; 90; 95 103; 78; 85; 79; 98	90 89	3.2 12.7	I17-009
6-CNA						
Mango (whole fruit)	0.01 0.1	2 1	88, 106 106	97 106	- -	RARV0287
Mango pulp	0.01 0.1	2 1	89, 101 96	95 96	- -	RARV0287
Mango peel	0.01 0.1	2 1	110, 82 90	96 90	- -	RARV0287
Papaya fruit	0.01 1.0	5 5	93; 85; 86; 95; 85 99; 76; 86; 81; 95	89 87	5.4 11	I17-009
DFEAF						
Mango (whole fruit)	0.01 0.1	1 1	90 102	90 102	- -	RARV0287
Mango pulp	0.01 0.1	1 1	95 83	95 83	- -	RARV0287
Mango peel	0.01 0.1	2 1	103, 101 100	102 100	- -	RARV0287

Table 3 Summary of analytical method 01304 (RV-001-P10-03) concurrent recovery results for plant commodities

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean percent recovery	RSD (%)	Reference
Flupyradifurone						
Pineapple	0.01 0.5	6 5	88; 91; 85; 88; 102; 86 96; 108; 91; 91; 97	90 97	7 7	IR4-11711
Pineapple juice	0.01 0.5	1 1	95 111	95 111	- -	IR4-11711
Pineapple wet bran	0.01 0.5	1 1	79 105	79 105	- -	IR4-11711

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean percent recovery	RSD (%)	Reference
Sesame seed	0.01	7	88; 85; 77; 85; 86; 87; 86	85	4	IR4-11725
	0.03	3	99; 101; 94	98	4	
	0.1	3	95; 94; 95	95	1	
	2.5	3	98; 97; 97	97	1	
Sesame oil	0.01	3	94, 99, 92	95	4	IR4-11725
	0.5	3	102, 101, 97	100	3	
Sunflower seed	0.01	6	109; 99; 96; 96; 82; 104	98	9.4	IR4-11674
	0.03	7	101; 106; 84; 91; 102; 94; 104	97	8.2	
	0.1	3	112; 98; 104	105	6.7	
	0.5	3	95; 96; 89	93	4.1	
Sunflower oil (refined)	0.01	3	80, 86, 92	86	7	IR4-11674
	0.5	3	103, 103, 99	102	2.3	
Sunflower meal (extracted)	0.1	3	85, 101, 98	95	9	IR4-11674
	0.5	3	94, 99, 98	97	2.7	
DFA						
Pineapple	0.02	6	109; 77; 92; 92; 112; 92	96	13	IR4-11711
	0.5	5	113; 109; 99; 103; 116	108	6	
Pineapple juice	0.01	1	86	86	-	IR4-11711
	0.5	1	104	104	-	
Pineapple wet bran	0.01	1	84	84	-	IR4-11711
	0.5	1	106	106	-	
Sesame seed	0.05	7	72; 72; 61; 66; 79; 68; 69	70	8	IR4-11725
	0.15	3	79; 73; 79	77	4	
	0.5	3	85; 85; 82	84	2	
	1.2	3	78; 80; 76	78	3	
Sesame oil	0.05	3	102, 104, 95	100	5	IR4-11725
	0.5	3	99, 95, 99	98	2	
Sunflower seed	0.05	6	108; 103; 110; 93; 102; 98	102	6.1	IR4-11674
	0.06	1	93	93	-	
	0.15	6	93; 88; 73; 97; 85; 83	87	9.7	
	0.5	6	92; 94; 100; 99; 91; 86	94	5.6	
Sunflower oil (refined)	0.05	3	83, 82, 89	85	4.5	IR4-11674
	0.5	3	93, 92, 94	93	1.1	
Sunflower meal (extracted)	0.1	3	67, 74, 75	72	6.1	IR4-11674
	0.5	3	71, 76, 74	74	3.4	
6-CNA						
Pineapple	0.01	6	93; 93; 91; 89; 97; 87	92	4	IR4-11711
	0.5	5	97; 100; 100; 102; 100	100	2	
Pineapple juice	0.01	1	89	89	-	IR4-11711
	0.5	1	99	99	-	
Pineapple wet bran	0.01	1	83	83	-	IR4-11711
	0.5	1	102	102	-	
Sesame seed	0.01	7	72; 88; 70; 85; 96; 71; 82	81	12	IR4-11725
	0.03	3	91; 78; 81	83	8	
	0.1	3	86; 83; 84	85	1	
	0.5	3	92; 93; 99	95	4	
Sesame oil	0.01	3	75, 98, 93	89	14	IR4-11725
	0.5	3	102, 100, 106	103	3	

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean percent recovery	RSD (%)	Reference
Sunflower seed	0.01 0.03 0.1 0.5	6 7 3 3	93; 94; 102; 112; 82; 111 84; 89; 82; 91; 81; 91; 94 84; 85; 85 94; 105; 97	99 87 85 99	11.7 5.8 0.7 5.8	IR4-11674
Sunflower oil (refined)	0.01 0.5	3 3	96, 97, 92 92, 100, 108	95 100	2.8 8	IR4-11674
Sunflower meal (extracted)	0.1 0.5	3 3	74, 77, 68 78, 81, 82	73 80	6.3 2.6	IR4-11674

USE PATTERN

Flupyradifurone has been registered in a number of countries for use as a foliar spray to a range of fruit, vegetable and field crops. Information on new uses were provided to the Meeting and those relevant to the supervised trials submitted to the current Meeting are summarized below.

Table 4 Registered uses of flupyradifurone (200 SL formulation) for the crops for which supervised trials were submitted

Crop	Country	Application						Minimum PHI, days (notes)
		Method	max rate (kg ai/ha)	Interval days	Water L/ha (min-max)	max seasonal rate (kg ai/ha)	max seasonal number	
Assorted tropical and sub-tropical fruits – smooth inedible peel								
Mango	United States[1]	foliar	0.2	14	min 234 (ground) min 93 (air)	0.41		1
	Brazil	foliar	0.2	7	300-1000		2	3
	Australia	foliar	0.2 (0.02 kg ai/hL)	14			2	3
Papaya	United States[1]	foliar	0.2	14	min 234 (ground) min 93 (air)	0.41		1
	Brazil	foliar	0.2	7	300-1000		2	3
	Australia	foliar	0.2 (0.02 kg ai/hL)	14			2	3
Pineapple	United States	foliar	0.2	7	min 94 (ground) min 28 (air)	0.41		0
	Brazil	foliar	0.2	7	300-1000		2	3
Oilseeds								
Sesame	United States	foliar	0.2	10	min 94 (ground) min 28 (air)	0.41		14
Sunflower Subgroup 20B)	United States[2]	foliar	0.2	10	min 94 (ground) min 28 (air)	0.41		14

Notes:

[1] Included in the US Subgroup 24B: Abiu; Akee apple; Avocado; Avocado, Guatemalan; Avocado, Mexican; Avocado, West Indian; Bacury; Banana; Banana, dwarf; Binjai; Canistel; Cupuacú; Etambe; Jatobá; Kei apple; Langsat; Lanjut; Lucuma; Mabolo; **Mango**; Mango, horse; Mango, Saipan; Mangosteen; Paho; **Papaya**; Pawpaw, common; Pelipisan; Pequi; Pequia;

Persimmon, American; Plantain; Pomegranate; Poshte; Quandong; Sapote, black; Sapote, green; Sapote, white; Sataw; Screw-pine; Star apple; Tamarind-of-the-Indies; Wild loquat; cultivars, varieties, and hybrids of these commodities.

(2) Included in the US Subgroup 20B: Calendula; Castor oil plant; Chinese tallowtree; Euphorbia; Evening primrose; Jojoba; Niger seed; Rose hip; Safflower; Stokes aster; **Sunflower**; Tallowwood; Tee oil plant; Vernonia; cultivars, varieties, and/or hybrids of these

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials involving foliar applications of flupyradifurone on the following crops.

Group	Crop	Countries	Table
Assorted tropical and sub-tropical fruits-inedible peel (FI)			
Assorted tropical and sub-tropical fruits – smooth inedible peel – large	Mango	Brazil	5
	Papaya	Brazil	6
Assorted tropical and sub-tropical fruits – inedible rough or hairy peel - large	Pineapple	United States	7
Oilseeds and oilfruits (SO)			
Small seed oilseeds	Sesame seed	United States	8
Sunflower seeds	Sunflower seed	United States	9

The new supervised trials were well documented with laboratory and field reports. Laboratory reports included method validation and/or procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ.

Intervals of freezer storage between sampling and analysis were recorded for all trials and were covered by the conditions of the freezer storage stability studies reviewed by previous JMPR meetings.

Residues and application rates have generally been rounded to two significant digits and residue concentrations are presented without correction for concurrent method recoveries. Where duplicate samples were analysed, mean values have been calculated from unrounded individual values and reported in brackets.

The results from trials conducted according to the maximum GAP and used for the estimation of maximum residue levels have been (underlined).

When residues were not quantifiable, they are shown as below the LOQ of the relevant analytical method (e. g. < 0.01 mg/kg).

Residue results are all expressed as flupyradifurone equivalents, using molecular weight conversion factors of 3.01 (DFA), 1.83 (6-CNA) and 1.77 (DFEAF).

For the calculation of sum of flupyradifurone, DFA and 6-CNA, expressed as parent equivalents (total residues), the Meeting used the approach adopted by the 2016 JMPR:

"Where parent or DFA residues were not detected or were less than the LOQ (*i.e.* < 0.01 mg/kg for parent or 0.05 mg/kg for DFA) the LOQ value was utilized for maximum residue estimation and dietary intake assessment. For 6-CNA, values less than the LOQ were not added for calculation of total residues of flupyradifurone."

Parent	DFA	6-CNA	Total
<0.01	0.05	0.01	0.07
0.01	<0.05	0.01	0.07
<0.01	<0.05	<0.01	<0.06
0.01	0.05	<0.01	0.06
0.01	0.05	0.01	0.07

In some trials, residue concentrations of DFEAF were also reported. While DFEAF is neither included in the residue definition for compliance with MRL nor the one for estimation of dietary exposure for plant commodities, DFEAF concentrations are shown in the following tables for consistency with the previous JMPR Evaluations.

Assorted tropical and sub-tropical fruits – smooth inedible peel – large

Mango

In field trials on mango, conducted in Brazil (2019), two foliar sprays of flupyradifurone (200 SL formulation) were applied to maturing fruit 10 days before harvest (BBCH 76–78) and 7 days later (BBCH 79–80). All applications were made as simulated commercial applications using a knapsack mist-blower with spray volumes of 509–524 L/ha. No adjuvant was used in the applications. Treated plots ranged from 150–315 m^2 (six trees).

Duplicate samples of ripe fruit (12 units, minimum of 6 kg) were processed (weighed, separated into pulp and peel, with stones weighed and discarded) and placed in frozen storage (< -20 °C) within 5 hours. The frozen samples were shipped to the analytical laboratory where they were homogenized with dry ice and then stored frozen for up to 8 months before extraction and analysis.

Residues of flupyradifurone and its metabolites DFA, 6-CNA and DFEAF were determined according to method RV-001-P10-02 by HPLC-MS/MS, using external standards. Overall mean concurrent recovery rates ranged from 83-104 percent in samples spiked with 0.01–0.5 mg/kg (See Table 2 above). The LOQs were 0.01 mg/kg for all analytes and matrices except DFA (0.05 mg/kg in whole fruit, pulp, peel).

Table 5 Residues in mango from trials conducted in Brazil, involving two foliar applications of flupyradifurone (200 SL formulation), retreatment interval of 7 days. [Ref: RARV0287]

Trial No., Location, Country, Year (Variety)	Application			Matrix	DALA	Residues as parent (mg/kg)				
	No (RTI)	Rate (kg ai/ha)	Water (L/ha)			Parent	DFEAF	DFA	6-CNA	Parent + DFA + 6-CNA
GAP: Brazil	2 (7d)	0.2 max	300-1000		3	Min 7d RTI				
GAP: United States		0.2	234 min		1	Max 0.41 kg ai/ha/season, min 14d RTI				

Trial No., Location, Country, Year (Variety)	Application			Matrix	DALA	Residues as parent (mg/kg)				
	No (RTI)	Rate (kg ai/ha)	Water (L/ha)			Parent	DFEAF	DFA	6-CNA	Parent + DFA + 6-CNA
007SRBR1804-01 Petrolina Brazil, 2019 (Palmer)	2 (7d) 0.21 0.21		520 520	Whole fruit (calc)[1]	3	0.13, 0.15 (0.14)	0.024, 0.026 (0.025)	<0.05, <0.05 (<0.05)	0.03, 0.03 (0.03)	0.21, 0.23 (0.22)
					7	0.11, 0.063 (0.087)	0.034, 0.02 (0.027)	0.062, <0.05 (0.056)	0.055, 0.036 (0.0455)	0.23, 0.15 (0.19)
					14	0.12, 0.079 (0.1)	0.03, 0.025 (0.0275)	0.15, 0.15 (0.15)	0.086, 0.073 (0.0795)	0.36, 0.3 (0.33)
					21	0.046, 0.038 (0.042)	0.011, <0.01 (0.01)	0.18, 0.19 (0.185)	0.085, 0.073 (0.079)	0.31, 0.3 (0.31)
					28	0.035, 0.039 (0.037)	<0.01, <0.01 (<0.01)	0.21, 0.23 (0.22)	0.077, 0.07 (0.074)	0.32, 0.34 (0.33)
				Peel	3	0.76	0.1	0.14	0.053	0.95
					7	0.59	0.18	0.33	0.11	1.0
					14	0.19, 0.17 (0.18)	<0.01, 0.051 (0.0305)	0.36, 0.39 (0.375)	0.066, 0.072 (0.069)	0.62, 0.63 (0.62)
					21	0.13	0.036	0.62	0.092	0.84
					28	0.067	0.016	0.89	0.083	1.0
				Flesh	3	0.02	<0.01	<0.05	0.019	0.089
					7	0.035	0.015	0.066	0.047	0.15
					14	0.034, 0.033 (0.034)	<0.01, <0.01 (<0.01)	0.11, 0.11 (0.11)	0.057, 0.063 (0.06)	0.2, 0.21 (0.2)
					21	0.035	<0.01	0.19	0.084	0.31
					28	0.032	<0.01	0.27	0.077	0.38
				Fruit without stone	3	0.15, 0.18 (0.165)	0.028, 0.031 (0.029)	<0.05, <0.05 (<0.05)	0.034, 0.035 (0.035)	0.23, 0.265 (0.25)
					7	0.12, 0.072 (0.096)	0.039, 0.023 (0.031)	0.072, 0.051 (0.062)	0.064, 0.041 (0.053)	0.26, 0.16 (0.21)
					14	0.14, 0.092 (0.116)	0.034, 0.028 (0.031)	0.17, 0.18 (0.175)	0.098, 0.095 (0.097)	0.41, 0.37 (0.39)
					21	0.054, 0.044 (0.049)	0.013, 0.011 (0.012)	0.22, 0.21 (0.215)	0.098, 0.085 (0.092)	0.37, 0.34 (0.36)
					28	0.04, 0.045 (0.0425)	<0.01, <0.01 (<0.01)	0.26, 0.24 (0.25)	0.089, 0.081 (0.085)	0.39, 0.37 (0.38)
007SRBR1804-02 Petrolina Brazil, 2019 (Palmer)	2 (7d) 0.21 0.21		520 520	Whole fruit (calc)[1]	3	0.21, 0.19 (0.2)	0.047, 0.043 (0.045)	<0.05, <0.05 (<0.05)	0.039, 0.037 (0.038)	0.3, 0.28 (0.29)
					7	0.1, 0.099 (0.1)	0.031, 0.042 (0.037)	0.058, 0.066 (0.062)	0.049, 0.056 (0.053)	0.21, 0.22 (0.21)
					14	0.047, 0.074 (0.061)	0.018, 0.023 (0.021)	0.094, 0.13 (0.11)	0.051, 0.062 (0.057)	0.19, 0.27 (0.23)
					21	0.046, 0.04 (0.043)	0.011, <0.01 (0.01)	0.17, 0.13 (0.15)	0.07, 0.045 (0.058)	0.29, 0.215 (0.25)
					28	0.026, 0.031 (0.029)	<0.01, <0.01 (<0.01)	0.15, 0.15 (0.15)	0.048, 0.045 (0.047)	0.22, 0.23 (0.225)
				Peel	3	0.7	0.12	0.14	0.044	0.88
					7	0.71	0.2	0.32	0.1	1.1
					14	0.17, 0.14 (0.155)	0.063, 0.056 (0.06)	0.38, 0.37 (0.375)	0.069, 0.061 (0.065)	0.62, 0.57 (0.595)
					21	0.1	0.035	0.61	0.063	0.77
					28	0.039	0.012	0.6	0.046	0.685
				Flesh	3	0.018	<0.01	<0.05	0.011	0.079
					7	0.047	0.019	0.064	0.045	0.16
					14	0.029, 0.027 (0.028)	<0.01, <0.01 (<0.01)	0.097, 0.11 (0.1)	0.053, 0.051 (0.052)	0.18, 0.19 (0.18)
					21	0.032	<0.01	0.18	0.074	0.29
					28	0.016	<0.01	0.18	0.051	0.25

Trial No., Location, Country, Year (Variety)	Application			Matrix	DALA	Residues as parent (mg/kg)				
	No (RTI)	Rate (kg ai/ha)	Water (L/ha)			Parent	DFEAF	DFA	6-CNA	Parent + DFA + 6-CNA
				Fruit without stone	3	0.24, 0.22 (0.23)	0.055, 0.049 (0.052)	0.056, <0.05 (0.053)	0.045, 0.042 (0.044)	0.34, 0.31 (0.33)
					7	0.12, 0.12 (0.12)	0.036, 0.049 (0.052)	0.067, 0.077 (0.072)	0.057, 0.066 (0.062)	0.24, 0.26 (0.25)
					14	0.054, 0.085 (0.07)	0.021, 0.027 (0.024)	0.11, 0.14 (0.125)	0.058, 0.071 (0.065)	0.22, 0.3 (0.26)
					21	0.052, 0.046 (0.049)	0.012, <0.01 (0.01)	0.19, 0.15 (0.17)	0.08, 0.052 (0.066)	0.32, 0.25 (0.285)
					28	0.029, 0.035 (0.032)	<0.01, <0.01 (<0.01)	0.17, 0.17 (0.17)	0.055, 0.052 (0.054)	0.25, 0.26 (0.26)
007SRBR1804-03 Juazeiro Brazil, 2019 (Tommy)	2 (7d)	0.21 0.21	520 520	Whole fruit (calc)[1]	3	0.32, 0.28 (0.3)	<0.01, <0.01 (<0.01)	<0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01)	0.37, 0.33 (0.35)
					7	0.3, 0.29 (0.295)	<0.01, <0.01 (<0.01)	0.073, 0.097 (0.085)	<0.01, <0.01 (<0.01)	0.37, 0.39 (0.38)
					14	0.21, 0.2 (0.205)	<0.01, <0.01 (<0.01)	0.16, 0.12 (0.14)	0.011, <0.01 (0.011)	0.38, 0.32 (0.35)
					21	0.17, 0.12 (0.145)	<0.01, <0.01 (<0.01)	0.21, 0.16 (0.185)	0.012, <0.01 (0.011)	0.39, 0.28 (0.34)
					28	0.18, 0.096 (0.14)	<0.01, <0.01 (<0.01)	0.3, 0.19 (0.245)	0.014, <0.01 (0.012)	0.49, 0.29 (0.39)
				Peel	3	1.5	<0.01	0.16	<0.01	1.7
					7	1.2	<0.01	0.24	<0.01	1.4
					14	1.1, 0.77 (0.935)	0.01, <0.01 (0.01)	0.59, 0.47 (0.53)	0.014, <0.01 (0.012)	1.7, 1.2 (1.5)
					21	0.52	<0.01	0.56	0.013	1.1
					28	0.31	<0.01	0.88	0.015	1.2
				Flesh	3	0.058	<0.01	0.056	<0.01	0.11
					7	0.079	<0.01	0.089	<0.01	0.17
					14	0.091, 0.071 (0.081)	<0.01, <0.01 (<0.01)	0.22, 0.17 (0.195)	<0.01, <0.01 (<0.01)	0.31, 0.24 (0.28)
					21	0.073	<0.01	0.21	<0.01	0.28
					28	0.045	<0.01	0.31	0.01	0.365
				Fruit without stone	3	0.38, 0.33 (0.355)	<0.01, <0.01 (<0.01)	0.058, 0.055 (0.057)	<0.01, <0.01 (<0.01)	0.44, 0.385 (0.41)
					7	0.36, 0.33 (0.345)	<0.01, <0.01 (<0.01)	0.088, 0.11 (0.099)	<0.01, <0.01 (<0.01)	0.45, 0.44 (0.44)
					14	0.23, 0.23 (0.23)	<0.01, <0.01 (<0.01)	0.18, 0.14 (0.16)	0.013, <0.01 (0.012)	0.42, 0.37 (0.4)
					21	0.2, 0.15 (0.175)	<0.01, <0.01 (<0.01)	0.25, 0.19 (0.22)	0.014, <0.01 (0.012)	0.46, 0.34 (0.4)
					28	0.21, 0.11 (0.16)	<0.01, <0.01 (<0.01)	0.33, 0.22 (0.275)	0.016, 0.011 (0.014)	0.56, 0.34 (0.45)
007SRBR1804-04 Curaca Brazil, 2019 (Tommy)	2 (7d)	0.2 0.2	510 510	Whole fruit (calc)[1]	3	0.21, 0.25 (0.23)	<0.01, <0.01 (<0.01)	<0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01)	0.26, 0.3 (0.28)
					7	0.13, 0.14 (0.135)	<0.01, <0.01 (<0.01)	<0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01)	0.18, 0.19 (0.185)
					14	0.15, 0.12 (0.135)	<0.01, <0.01 (<0.01)	0.12, 0.1 (0.11)	0.018, 0.015 (0.017)	0.29, 0.235 (0.26)
					21	0.12, 0.1 (0.11)	<0.01, <0.01 (<0.01)	0.17, 0.15 (0.16)	0.021, 0.014 (0.018)	0.31, 0.26 (0.29)
					28	0.11, 0.14 (0.125)	<0.01, <0.01 (<0.01)	0.15, 0.2 (0.175)	0.016, 0.021 (0.019)	0.28, 0.36 (0.32)
				Peel	3	1.1	<0.01	0.095	0.016	1.2
					7	0.82	<0.01	0.17	0.015	1.0
					14	0.57, 0.58 (0.575)	0.012, 0.015 (0.014)	0.39, 0.41 (0.4)	0.024, 0.029 (0.027)	0.98, 1.0 (1.0)
					21	0.39	<0.01	0.54	0.018	0.95
					28	0.28	<0.01	0.56	0.018	0.86

Trial No., Location, Country, Year (Variety)	Application			Matrix	DALA	Residues as parent (mg/kg)				
	No (RTI)	Rate (kg ai/ha)	Water (L/ha)			Parent	DFEAF	DFA	6-CNA	Parent + DFA + 6-CNA
				Flesh	3	0.017	<0.01	<0.05	<0.01	0.067
					7	0.038	<0.01	<0.05	<0.01	0.088
					14	0.057, 0.06 (0.059)	<0.01, <0.01 (<0.01)	0.13, 0.12 (0.125)	0.012, 0.016 (0.014)	0.2, 0.2 (0.2)
					21	0.062	<0.01	0.19	0.015	0.27
					28	0.063	<0.01	0.19	0.018	0.27
				Fruit without stone	3	0.24, 0.29 (0.265)	<0.01, <0.01 (<0.01)	<0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01)	0.29, 0.34 (0.315)
					7	0.15, 0.16 (0.155)	<0.01, <0.01 (<0.01)	<0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01)	0.2, 0.21 (0.205)
					14	0.17, 0.14 (0.155)	<0.01, <0.01 (<0.01)	0.13, 0.12 (0.125)	0.02, 0.018 (0.019)	0.32, 0.28 (0.3)
					21	0.14, 0.11 (0.125)	<0.01, <0.01 (<0.01)	0.2, 0.16 (0.18)	0.024, 0.016 (0.02)	0.36, 0.29 (0.325)
					28	0.12, 0.15 (0.135)	<0.01, <0.01 (<0.01)	0.17, 0.22 (0.195)	0.018, 0.023 (0.021)	0.31, 0.39 (0.35)
007SRBR1804-05 Casa Nova Brazil, 2019 (Palmer)	2 (7d)	0.21 0.21	520 520	Whole fruit (calc)[1]	3	0.23, 0.21 (0.22)	0.047, 0.039 (0.043)	<0.05, 0.06 (0.055)	0.053, 0.062 (0.058)	0.33, 0.33 (0.33)
					7	0.14, 0.15 (0.145)	0.035, 0.06 (0.047)	0.094, 0.1 (0.097)	0.076, 0.093 (0.085)	0.31, 0.34 (0.33)
					14	0.041, 0.061 (0.051)	0.018, 0.024 (0.021)	0.16, 0.18 (0.17)	0.086, 0.11 (0.098)	0.29, 0.35 (0.32)
					21	0.036, 0.034 (0.035)	0.01, <0.01 (0.01)	0.21, 0.16 (0.185)	0.093, 0.074 (0.084)	0.34, 0.27 (0.3)
					28	<0.01, 0.011 (0.011)	<0.01, <0.01 (<0.01)	0.12, 0.12 (0.12)	0.027, 0.034 (0.031)	0.16, 0.165 (0.16)
				Peel	3	0.91	0.2	0.4	0.15	1.5
					7	0.3	0.21	0.42	0.15	0.87
					14	0.46, 0.16 (0.31)	0.093, 0.06 (0.076)	0.59, 0.39 (0.49)	0.11, 0.079 (0.095)	1.2, 0.63 (0.895)
					21	0.1	0.028	0.88	0.11	1.1
					28	0.015	<0.01	0.46	0.042	0.52
				Flesh	3	0.019	0.01	0.055	0.043	0.12
					7	0.039	0.016	0.09	0.072	0.2
					14	0.038, 0.028 (0.033)	<0.01, <0.01 (<0.01)	0.17, 0.14 (0.155)	0.1, 0.081 (0.091)	0.31, 0.25 (0.28)
					21	0.025	<0.01	0.26	0.11	0.395
					28	<0.01	<0.01	0.16	0.042)	0.21
				Fruit without stone	3	0.26, 0.25 (0.255)	0.052, 0.046 (0.049)	0.05, 0.07 (0.06)	0.059, 0.073 (0.066)	0.37, 0.39 (0.38)
					7	0.16, 0.17 (0.165)	0.04, 0.068 (0.054)	0.11, 0.11 (0.11)	0.087, 0.11 (0.099)	0.36, 0.39 (0.37)
					14	0.046, 0.069 (0.058)	0.021, 0.027 (0.024)	0.19, 0.21 (0.2)	0.099, 0.13 (0.115)	0.335, 0.41 (0.37)
					21	0.041, 0.038 (0.04)	0.011, <0.01 (0.01)	0.23, 0.18 (0.205)	0.1, 0.083 (0.092)	0.37, 0.3 (0.34)
					28	<0.01, 0.012 (0.011)	<0.01, <0.01 (<0.01)	0.13, 0.14 (0.135)	0.03, 0.038 (0.034)	0.17, 0.19 (0.18)

Notes:

[1] Calculated residues in whole fruit based on a flesh+peel content of 80-90 percent w/w (median 87 percent w/w).

Papaya

In field trials on papaya, conducted in Brazil (2019), two foliar sprays of flupyradifurone (200 SL formulation) were applied to maturing fruit 10 days before harvest (BBCH 75–81) and 7 days later (BBCH

79-81). All applications were made as simulated commercial applications using a motorised pump and single cone hand-gun to apply spray volumes of 660–1092 L/ha. No adjuvant was used in the applications. Treated plots ranged from 50–140 m^2 (minimum of four trees).

Samples of ripe fruit (12 units, min 5 kg) were placed in frozen storage (< -20 °C) within 24 hours. The frozen samples were shipped to the analytical laboratory where they were homogenized with dry ice and then stored frozen for up to 6 months before extraction and analysis.

Residues of flupyradifurone and its metabolites DFA and 6-CNA were determined according to method RV-001-P10-02 by HPLC-MS/MS, using internal standards. Overall mean concurrent recovery rates ranged from 88–95 percent in samples spiked with 0.01–10 mg/kg (See table 2 above). The LOQs were 0.01 mg/kg for flupyradifurone and 6-CAN and 0.05 mg/kg for DFA.

Table 6 Residues in papaya from trials conducted in Brazil, involving two foliar applications of flupyradifurone (200 SL formulation), with a retreatment interval of 7 days. [Ref: I17-009]

Trial No., Location, Country, Year (Variety)	Application			Matrix	DALA	Residues as parent (mg/kg)			
	No (RTI)	Rate (kg ai/ha)	Water (L/ha)			Parent	DFA	6-CNA	Parent + DFA + 6-CNA
GAP: United States		0.2	234 min		1	Min 7d RTI, max 0.41 kg ai/ha/season			
GAP: Brazil	2 (7d)	0.2 max	300-1000		3	Min 7d RTI			
I17-009-01 Paulinia Brazil, 2018 (Havai)	2 (7d)	0.21 0.21	1070 1050	Whole fruit	3 7 14 21 28	0.1 0.038 0.016 0.01 <0.01	0.053 <0.05 0.16 0.22 0.24	0.023 0.026 0.032 0.034 0.033	0.18 0.11 0.21 0.26 0.28
I17-009-02 Ariranha Brazil, 2018 (Tainung n° 1)	2 (7d)	0.22 0.22	1090 1100	Whole fruit	3 7 14 21 28	0.11 0.043 0.023 0.011 <0.01	<0.05 <0.05 0.071 0.098 0.12	0.011 0.013 0.017 0.015 0.012	0.17 0.11 0.11 0.12 0.14
I17-009-03 Prata Brazil, 2018 (Not specified)	2 (7d)	0.2 0.21	660 700	Whole fruit	3 7 13 21 28	0.068 0.059 0.046 0.03 0.015	0.097 0.075 0.1 0.15 0.14	<0.01 <0.01 <0.01 <0.01 <0.01	0.165 0.13 0.15 0.18 0.155
I17-009-04 Monte Alto Brazil, 2018 (Tainung n° 1)	2 (7d)	0.21 0.21	1020 1030	Whole fruit	3 7 14 21 28	0.02 0.02 <0.01 <0.01 <0.01	<0.05 <0.05 0.1 0.14 0.17	<0.01 0.12 <0.01 0.019 0.023	0.07 0.19 0.11 0.17 0.2
I17-009-05 Piracaiba Brazil, 2018 (Not specified)	2 (7d)	0.19 0.2	960 1010	Whole fruit	3 7 14 21 28	0.2 0.18 0.13 0.1 0.042	<0.05 <0.05 0.073 0.096 0.11	<0.01 <0.01 0.01 0.012 <0.01	0.25 0.23 0.21 0.21 0.15

Assorted tropical and sub-tropical fruits – inedible rough or hairy peel - large

Pineapple

In field trials on pineapple, conducted in Hawaii and Puerto Rico, two foliar sprays of flupyradifurone (200 SL formulation) were applied 7–8 days apart using pressurised backpack sprayers (with 2 or 3 flat fan

nozzles) to apply spray volumes of 560–2440 L/ha. No adjuvant was used in the applications except in trials HI157 and HI160, where a non-ionic surfactant was added. Treated plots ranged from 37–74 m^2.

Samples of ripe fruit (12 units) were trimmed to remove the crowns, quartered longitudinally and single quarters were placed in frozen storage (< -18 °C) within 3 hours. The frozen samples were shipped to the analytical laboratory where they were homogenized with dry ice and then stored frozen for up to 17 months before extraction and analysis.

Residues of flupyradifurone and its metabolites DFA and 6-CNA were determined according to method RV-001-P10-03 by HPLC-MS/MS using internal standards. Overall mean concurrent recovery rates ranged from 93–101 percent in samples spiked with 0.01–0.5 mg/kg (See Table 3 above). The LOQs were 0.01 mg/kg for flupyradifurone, 6-CNA and 0.02 mg/kg for DFA.

Table 7 Residues in pineapple from trials in Hawaii and Puerto Rico, involving two foliar applications of flupyradifurone (200 SL formulation), with retreatment intervals of 7–8 days. [Ref: IR4-11711]

Trial No., Location, Country, Year (Variety)	Application			Matrix	DALA	Residues as parent (mg/kg)			
	No (RTI)	Rate (kg ai/ha)	Water (L/ha)			Parent	DFA	6-CNA	Parent + DFA + 6-CNA
GAP: United States		0.2	234 min		0	Min 7d RTI, max 0.41 kg ai/ha/season			
GAP: Brazil	2	0.2 max	300-1000		3	Min 7d RTI			
11711.16-HI157 Wahiawa, HI United States, 2016 (Tropical Gold 73-50)	2 (8d)	0.2 0.21	1440 1470	Fruit without crown	0	0.11, 0.11 (0.11)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)	0.13, 0.13 (0.13)
11711.16-HI158 Wahiawa, HI United States, 2016 (Tropical Gold 73-50)	2 (7d)	0.21 0.21	1920 1940	Fruit without crown	0	0.047, 0.046 (0.046)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)	0.067, 0.066 (0.066)
11711.16-HI160 Wahiawa, HI United States, 2016 (Tropical Gold 73-50)	2 (7d)	0.21 0.21	2440 2410	Fruit without crown	0	0.0575, 0.0665 (0.062)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)	0.0775, 0.0865 (0.082)
11711.16-PR335 Manatí, PR United States, 2016 (MD2)	2 (7d)	0.2 0.21	560 570	Fruit without crown	0 4 7 14 21	0.13, 0.12 (0.12) 0.094, 0.098 (0.096) 0.05, 0.1 (0.075) 0.042, 0.034 (0.038) 0.026, 0.05 (0.038)	<0.02, <0.02 (<0.02) <0.02, <0.02 (<0.02) <0.02, <0.02 (<0.02) <0.02, <0.02 (<0.02) <0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	0.15, 0.14 (0.14) 0.11, 0.12 (0.12) 0.07, 0.12 (0.095) 0.062, 0.054 (0.058) 0.046, 0.07 (0.058)
11711.16-PR509 Manatí, PR United States, 2016 (MD2)	2 (7d)	0.21 0.21	570 580	Fruit without crown	0	0.14, 0.17 (0.155)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)	0.16, **0.19** (0.175)

Notes:

Mean values calculated from unrounded individual values. For flupyradifurone and DFA, values below the respective LOQs were set at LOQ for calculation.

The three Hawaiian trials and the two Puerto Rican trials were conducted in the same locations (in Wahiawa and Manati, respectively) but since the application dates differed by more than 30 days in all cases, they are all considered independent.

Small seed oilseeds

Sesame seed

In field trials on sesame, conducted in United States, two foliar sprays of flupyradifurone (200 SL formulation) were applied 9–11 days apart using a tractor-mounted boom sprayer (8 nozzles) or pressurised backpack mini-boom sprayers (2–6 nozzles) to apply spray volumes of 234–280 L/ha. A non-ionic adjuvant was added to the spray mixtures. Treated plots ranged from 140–548 m^2.

Duplicate composite samples (two separate runs through the plot) of sesame stalks or pods were harvested and allowed to dry in the field (if necessary) before threshing and cleaning. Seed samples (minimum of 0.9 kg) were frozen within 1 hour and shipped to the analytical laboratory where they were homogenized with dry ice and stored frozen for up to 13 months before extraction and analysis.

Residues of flupyradifurone and its metabolites DFA and 6-CNA were determined according to method RV-001-P10-03 by HPLC-MS/MS, using external standards. Overall mean concurrent recovery rates ranged from 75–92 percent in samples spiked with 0.01–2.5 mg/kg (See Table 3 above). The LOQs were 0.01 mg/kg for flupyradifurone, 6-CNA and 0.05 mg/kg for DFA.

Table 8 Residues in sesame seed from trials conducted in United States, involving two foliar applications of flupyradifurone (200 SL formulation), with retreatment intervals of 9–11 days. [Ref: IR4-11725]

Trial No., Location, Country, Year (Variety)	Application			Matrix	DALA	Residues as parent (mg/kg)			
	No (RTI)	Rate (kg ai/ha)	Water (L/ha)			Parent	DFA	6-CNA	Parent + DFA + 6-CNA
GAP: United States		0.2 max			14	Min 10d RTI, max 0.41 kg ai/ha/season			
11725.16-FL132 Citra, FL United States, 2016 (S39)	2 (9d)	0.2 0.2	270 270	Seed[1]	15	0.11, 0.093 (0.1)	0.13, 0.14 (0.13)	0.28, 0.29 (0.29) (c=0.066)	0.52, 0.53 (0.52)
11725.16-TX373 Weslaco, TX United States, 2016 (S36)	2 (9d)	0.2 0.2	230 240	Seed[2]	5 10 14 21 27	0.65, 0.36 (0.51) 0.34, 0.38 (0.36) 0.4, 0.35 (0.38) 0.18, 0.23 (0.2) 0.15, 0.3 (0.23)	0.84, 0.56 (0.7) 0.57, 0.55 (0.56) 0.53, 0.72 (0.62) 1.1, 1.1 (1.1) 0.75, 0.96 (0.85)	0.48, 0.4 (0.44) 0.35, 0.3 (0.33) 0.32, 0.42 (0.37) 0.77, 0.64 (0.7) 0.5, 0.64 (0.57)	2.0, 1.3 (1.7) 1.3, 1.2 (1.2) 1.3, 1.5 1.4 2.0, 1.9 (2.0) 1.4, 1.9 (1.7)
11725.16-NM263 Las Cruces NM United States, 2016 (S32)	2 (9d)	0.21 0.21	280 280	Seed[3]	19	0.96, 1.2 (1.1)	0.092, 0.099 (0.096)	0.067, 0.076 (0.0715)	1.1, 1.4 (1.2)
11725.16-CA519 Davis, CA United States, 2016 (Sesaco S36)	2 (11d)	0.2 0.21	230 230	Seed[4]	14	0.12, 0.11 (0.12)	0.11, 0.11 (0.11)	0.14, 0.15 (0.15)	0.38, 0.38 (0.38)

Notes:

(1) Pods harvested and forced-air dried (24 hours at 32 °C) before threshing and seed sampling.

(2) Stalks air-dried in the field and/or greenhouse for 4-5 days before threshing and seed sampling.

(3) Plots harvested using a small-plot harvester.

(4) Stalks harvested and dried in the field for 25 days before threshing and seed sampling.

Sunflower seeds

In field trials on sunflower, conducted in United States, two foliar sprays of flupyradifurone (200 SL formulation) were applied during the seed ripening stage using vehicle-mounted, wheeled or backpack pressurised boom sprayers (3–9 nozzles) to apply spray volumes of 47–430 L/ha. A non-ionic adjuvant was added to the spray mixtures except in trials ND 238 and SD 358. Treated plots ranged from 21-186 m^2.

Duplicate samples of 12–24 flowerheads were clipped, manually or mechanically threshed and the seeds were screened to remove the debris, frozen within 1.5 hours and shipped to the analytical laboratory where they were homogenized with dry ice and stored frozen for up to 21 months before extraction and analysis.

Residues of flupyradifurone and its metabolites DFA and 6-CNA were determined according to method RV-001-P10-03 by HPLC-MS/MS, using external standards. Overall mean concurrent recovery rates ranged from 92–98 percent in samples spiked with 0.01–0.5 mg/kg (See Table 3 above). The LOQs were 0.01 mg/kg for flupyradifurone, 6-CNA and 0.05 mg/kg for DFA.

Table 9 Residues in sunflower seed from trials conducted in United States, involving two foliar applications of flupyradifurone (200 SL formulation), with retreatment intervals of 9–15 days. [Ref: IR4-11674]

Trial No., Location, Country, Year (Variety)	Application			Matrix	DALA	Residues as parent (mg/kg)			
	No (RTI)	Rate (kg ai/ha)	Water (L/ha)			Parent	DFA	6-CNA	Parent + DFA + 6-CNA
GAP: United States		204			14	Min 10d RTI, max 0.41 kg ai/ha/season			
11674.16-CA39 Davis, CA United States, 2016 (Panther DMR)	2 (11d)	0.21 0.21	47 47	Seed	14	0.035, 0.02 (0.028)	<0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01)	0.085, 0.07 (0.078)
11674.16-ND238 Fargo, ND United States, 2016 (Mycogen 8N270 CLDM)	2 (9d)	0.21 0.2 [no surfactant]	110 110	Seed	15	0.03, 0.0255 (0.028)	<0.05, <0.05 (<0.05)	<0.01, 0.025 (0.017)	0.08, 0.1 (0.09)
11674.16-ND239 Fargo, ND United States, 2016 (Mycogen 8N358 CLDM)	2 (11d)	0.21 0.2	220 220	Seed	5 9 13 19 23	0.19, 0.12 (0.15) 0.21, 0.16 (0.18) 0.26, 0.25 (0.25) 0.22, 0.17 (0.19) 0.16, 0.28 (0.22)	<0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05) <<0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	0.24, 0.17 (0.205) 0.26, 0.21 (0.23) 0.31, 0.3 (0.3) 0.265, 0.22 (0.24) 0.21, 0.33 (0.27)
11674.16-ND240 Minot, ND United States, 2016 (Jaguar DMR)	2 (11d)	0.215 0.21	94 94	Seed	13	0.23, 0.14 (0.18)	<0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01)	0.28, 0.19 (0.235)

Trial No., Location, Country, Year (Variety)	Application			Matrix	DALA	Residues as parent (mg/kg)			
	No (RTI)	Rate (kg ai/ha)	Water (L/ha)			Parent	DFA	6-CNA	Parent + DFA + 6-CNA
11674.16-ND241 Minot, ND United States, 2016 (8H288CLMD)	2 (11d)	0.21 0.21	94 94	Seed	13	0.41, 0.48 (0.44)	<0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01)	0.46, 0.53 (0.49)
11674.16-NM259 Las Cruces, NM United States, 2016 (8N668S)	2 (15d)	0.21 0.2	47 47	Seed	7 15	0.32, 0.2 (0.26) 0.14	<0.05, <0.05 (<0.05) <0.05	0.021, <0.01 (0.015) <0.01	0.39, 0.25 (0.32) 0.19
11674.16-OH514 Fremont, OH United States, 2016 (Giant, striped) [21 m^2 plot]	2 (9d)	0.2 0.2	420 430	Seed	14	0.042, 0.039 (0.04)	<0.05, <0.05 (<0.05)	0.01, 0.01 (0.01)	0.1, 0.089 (0.096)
11674.16-SD358 Ree Heights, SD United States, 2016 (RRC 2215 - confectionary)	2 (12d)	0.22 0.22 [no surfactant]	160 160	Seed	14	0.18, 0.14 (0.16)	<0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01)	0.23, 0.19 (0.21)
11674.16-SD360 Ree Heights, SD United States, 2016 (RRC 2215) confectionary	2 (12d)	0.22 0.21	290 280	Seed	14	0.18, 0.16 (0.17)	<0.05, <0.05 (<0.05)	<0.01, <0.01 (<0.01)	0.23, 0.21 (0.22)
11674.16-SD359 Aurora, SD United States, 2016 (Cobalt II)	2 (11d)	0.21 0.21	110 110	Seed	13	0.012, 0.015 (0.0135)	<0.05, <0.05 (<0.05)	0.012, 0.012 (0.012)	0.073, 0.077 (0.075)

Notes:

Trials ND240 and ND 241 not considered independent (differing only in the variety used).

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Information and Data from Residues in Processed Commodities

A study on the effects of heating at different pH and temperature on the flupyradifurone residues was evaluated by the 2016 JMPR which concluded that flupyradifurone was not degraded during the simulation of pasteurization (pH 4, 90 °C, 20 minutes), baking, boiling or brewing (pH 5, 100 °C, 60 minutes) or during sterilization (pH 6, 120 °C, 20 minutes).

The 2016 JMPR also evaluated the effects of processing on the concentrations of flupyradifurone residues in orange, apples, grapes, tomato, soybean, potato, barley, maize, wheat, cotton and peanut.

Subsequently, processing studies on peaches, plums and cherries were evaluated by the 2017 JMPR and cocoa, coffee and hop processing studies were evaluated by the 2019 JMPR.

The current Meeting received information on the processing of pineapple, sesame seed and sunflower seed.

Pineapple

In one field trial on pineapple, conducted in Hawaii, two foliar sprays of flupyradifurone (200 SL formulation) with added non-ionic surfactant were applied at an exaggerated (5×) rate of 1.05 kg ai/ha, 8 days apart using a pressurised backpack sprayer (with 2 flat fan nozzles) to apply spray volumes of 1490 L/ha. The treated plot size was 45 m^2.

Samples (24 units) of treated and untreated ripe fruit were trimmed to remove the crowns, transported fresh to the processing facility and processed into juice and wet bran the same day.

Juice was prepared by trimming both ends of each fruit, hand peeling and scraping the remaining flesh from the peel. The peeled fruit, scraped flesh and end cuts were processed in a commercial juicer and the pulp and the juice were collected in separate containers. The juice, after the addition of an antifoam agent, was heated in a water bath to 88 °C for 30 seconds, then cooled to 8 °C in ice water bath and subsamples were taken, frozen and shipped to the analytical laboratory where they were stored frozen for up to 14 months before extraction and analysis.

Wet bran (process residue) was obtained by chopping the retained peel to a coarse consistency and combining it with the pulp left over from juicing. After thorough mixing, subsamples were taken, frozen and shipped to the analytical laboratory where they homogenized with dry ice and stored frozen for up to 14 months before extraction and analysis.

Residues of flupyradifurone and its metabolites DFA and 6-CNA were determined according to method RV-001-P10-03 by HPLC-MS/MS using internal standards. Overall mean concurrent recovery rates ranged from 92–103 percent in samples spiked with 0.01–0.5 mg/kg (See table 3 above). The LOQs were 0.01 mg/kg for flupyradifurone, 6-CNA and 0.02 mg/kg for DFA.

Table 10 Residues in pineapple processing fractions from a study conducted in Hawaii, involving two foliar applications of flupyradifurone (200 SL formulation). [Ref: IR4-11711]

Trial No., Location, Country, Year (Variety)	Application			Matrix	DALA	Residues as parent (mg/kg)			
	No (RTI)	Rate (kg ai/ha)	Water (L/ha)			Parent	DFA	6-CNA	Parent + DFA + 6-CNA
11711.16-HI157 Wahiawa, HI United States, 2016 (Tropical Gold 73-50)	2 (8d)	1.1 1.1	1497 1487	Fruit[(1)]	0	0.54	<0.02	<0.01	0.56
				Juice		0.17	<0.02	<0.01	0.19
				Wet bran		0.55	<0.02	<0.01	0.57

Notes:

(1) Without crown

Sesame seed

In one field trial on sesame, conducted in United States, two foliar sprays of flupyradifurone (200 SL formulation) were applied at an exaggerated (5×) rate of about 1.0 kg ai/ha with added non-ionic surfactant, 9 days apart using a pressurised backpack mini-boom sprayer (4 nozzles) to apply spray

volumes of 280 L/ha. A non-ionic adjuvant was added to the spray mixtures. The treated plot size was 223 m^2.

Plots were harvested with a small plot harvester, the seed was sifted to remove debris and frozen within 1 hour and shipped and stored frozen at the processing facility for 3.6 months before being processed into oil.

Three subsamples (minimum of 11 kg) were screened to remove field debris, straw, etc and expelled for crude oil and presscake recovery using an electric screw press expeller with an attached heating element. The recovered oil was centrifuged and decanted to separate the crude oil from emulsion and particulate and the crude oil was subsampled, frozen and stored for shipment to the analytical laboratory where they were stored frozen for up to 8 months before extraction and analysis.

Residues of flupyradifurone and its metabolites DFA and 6-CNA were determined according to method RV-001-P10-03 by HPLC-MS/MS, using internal standards. Overall mean concurrent recovery rates ranged from 96–99 percent in samples spiked with 0.01–0.5 mg/kg (See Table 3 above). The LOQs were 0.01 mg/kg for flupyradifurone, 6-CNA and 0.05 mg/kg for DFA.

Table 11 Residues in sesame seed and crude oil from a trial involving two foliar applications of flupyradifurone (200 SL formulation), retreatment interval of 9 days. [Ref: IR4-11725]

Trial No., Location, Country, Year (Variety)	Application		Matrix	DALA	Residues as parent (mg/kg)			
	Rate (kg ai/ha)	Water (L/ha)			Parent	DFA	6-CNA	Parent + DFA + 6-CNA
11725.16-NM263 Las Cruces NM United States, 2016 (S32)	1.04 1.01	280 280	Seed	19	2.1, 2.1, 2.4 (2.2)	0.3, 0.28, 0.3 (0.29)	0.17, 0.15, 0.16 (0.16)	2.6, 2.5, 2.8 (2.6)
			Crude oil		0.3, 0.3, 0.29 (0.29)	<0.05, <0.05, <0.05 (<0.05)	<0.01, <0.01, <0.01 (<0.01)	0.35, 0.35, 0.34 (0.34)

Sunflower seed

In one field trial on sunflower, conducted in United States, two foliar sprays of flupyradifurone (200 SL formulation) were applied, 11 days apart, at an exaggerated (5×) rate of 1.03 kg ai/ha with added non-ionic surfactant during the seed ripening stage using a pressurised boom sprayer (5 nozzles) to apply spray volumes of 103 L/ha. The treated plot size was 535 m^2.

Triplicate samples of 120 flowerheads were clipped, mechanically threshed and the seeds frozen within 1 hour and shipped to the processing facility where they were stored frozen for 4 months before processing into meal and refined oil.

Seed samples were tempered (~60 °C for 90 minutes) before screening to remove field debris and partially dehulled using a seed scarifier. After further screening and aspiration, the partially dehulled seeds were heated on a bin air tray dryer (70–83 °C for 20 minutes) and flaked on a rotary drum dryer (2.5 mm drum spacing). The flaked seed was processed through an oil expeller to obtain crude oil and presscake.

The crude oil was centrifuged, heated to ~60 °C, mixed with phosphoric acid (0.2 percent v/v) then distilled water (5.0 percent *v/v)* and centrifuged to separate the oil and gum. The degummed oil was heated to about 40 °C before the addition of sodium hydroxide (0.3 percent v/v) and then heated to about 75 °C to separate the soapstock from the neutralized oil. The oil was separated from the soapstock via centrifugation and decanted.

The neutralized oil was washed with soft water (15 percent v/v) at 90 °C to remove traces of non-precipitated soap in the oil. The oil was separated from the water/soapstock via centrifugation, decanted and heated to ~ 105 °C to remove traces of moisture. The refined oil was subsampled, frozen and stored for shipment to the analytical laboratory where they were stored frozen for up to 8 months before extraction and analysis.

The presscake was mixed with hexane and the miscella (oil/hexane) was separated from the meal by vacuum filter. The meal was dried in a fluid bed dryer (70–83 °C) and then heated with a steam attachment to about 90 °C to remove any remaining solvent. The final moisture content of the toasted meal was 2.24 percent (UTC) and 3.36 percent (TRT). The toasted meal was milled and subsamples were frozen and stored for shipment to the analytical laboratory where they were stored frozen for up to 7 months before extraction and analysis.

Residues of flupyradifurone and its metabolites DFA and 6-CNA were determined according to method RV-001-P10-03 by HPLC-MS/MS, using external standards. Overall mean concurrent recovery rates ranged from 73–98 percent in samples spiked with 0.01–0.5 mg/kg (See table 3 above). The LOQs were 0.01 mg/kg for flupyradifurone, 6-CNA and 0.05 mg/kg for DFA.

Table 12 Residues in sunflower seed, refined oil and meal from a trial involving two foliar applications of flupyradifurone (200 SL formulation), retreatment interval of 11 days. [Ref: IR4-11674]

Trial No., Location, Country, Year (Variety)	Application		Matrix	DALA	Residues as parent (mg/kg)			
	Rate (kg ai/ha)	Water (L/ha)			Parent	DFA	6-CNA[1]	Parent + DFA + 6-CNA
11674.16-SD359 Aurora, SD United States, 2016 (Cobalt II)	1.04 1.03	112 112	Seed	13	0.33, 0.3, 0.27 (0.3)	<0.05 (3) (<0.05)	0.027, 0.021, 0.022 (0.023)	0.4, 0.38, 0.34 (0.37)
			Oil (refined)		0.33, 0.43, 0.32 (0.36)	<0.05 (3) (<0.05)	0.054, 0.063, 0.053 (0.057)	0.43, 0.54, 0.42 (0.46)
			Meal		0.012, 0.013, 0.012 (0.012)	<0.05 (3) (<0.05)	<0.01, <0.01, <0.01 (<0.01)	0.062, 0.063, 0.062 (0.062)

Notes:

(1) Residues of 6-CNA were measured in all control samples of seeds and oil, ranging from 0.022 mg/kg to 0.024 mg/kg (mean values were 0.023 mg/kg in both matrices)

Processing factors

In processing studies conducted on pineapples, sesame seed and sunflower seed, flupyradifurone residues decreased in pineapple juice and sesame seed oil but concentrated in sunflower seed oil. In non-food commodities, residues decreased in sunflower meal but not in pineapple meal.

Table 13 Summary of calculated processing factors for flupyradifurone

RAC Commodity	Flupyradifurone		Total residue[1]	
	Residues (mean)	Processing Factor[2]	Residues (mean)	Processing Factor[2]
Pineapple without crown (RAC)	0.54	-	0.56 (0.56)[3]	-
Juice	0.17	0.32	0.19	0.34
Wet bran	0.55	1.0	0.57 (0.57[3]	1.0[3]
Sesame seed (RAC)	2.2	-	2.6	-
Oil (crude)	0.29	0.13	0.34	0.13

RAC Commodity	Flupyradifurone		Total residue[1]	
	Residues (mean)	Processing Factor[2]	Residues (mean)	Processing Factor[2]
Sunflower seed (RAC)	0.3	-	0.37 (0.35)[3]	-
Oil (refined)	0.36	1.2	-[4]	nc[4]
Meal	0.012	0.04	0.062 (0.062)[3]	0.17 (0.18)[3]

Notes:

[1] Total residue = flupyradifurone + DFA + 6-CNA, all expressed as flupyradifurone equivalents

[2] Each value represents a separate study where residues were above the LOQ in the RAC. The processing factors (PFs) are the ratios of the residue in the processed item divided by the residue in the Raw Agricultural Commodity.

[3] Residue values in brackets are for the sum of flupyradifurone and DFA (as parent equivalents), relevant for calculating processing factors for animal feed commodities.

[4] Total residue value compromised because 6-CNA residues measured in control samples. Processing factor not calculated

APPRAISAL

Flupyradifurone, is a butenolide insecticide acting as an agonist of nicotinic acetylcholine receptor. It was first evaluated by the JMPR for toxicology in 2015 and for residues by the 2016, 2017 and 2019 JMPRs.

The 2015 Meeting established an ADI of 0–0.08 mg/kg bw and an ARfD of 0.2 mg/kg bw and the 2016 JMPR established the following residue definitions:-

- For compliance with the MRL (plant commodities): *Flupyradifurone*
- For estimation of dietary exposure (for plant commodities): Sum of flupyradifurone, difluoroacetic acid (DFA) and 6-chloronicotinic acid (6-CNA), expressed as parent equivalents
- For compliance with the MRL and for estimation of dietary exposure (animal commodities): *Sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents*

The residue is not fat-soluble.

The Fifty-second Session of the CCPR (2021) listed flupyradifurone for further evaluation by the 2022 JMPR and the current Meeting received revised GAP information and new supporting residue information from the manufacturer for mango, papaya, pineapple, sesame seeds and sunflower seeds.

METHODS OF ANALYSIS

A number of analytical methods (for enforcement and data collection) for plant and animal matrices were evaluated by the 2016 and 2019 Meeting, including the HPLC-MS/MS Method RV-001-P10-02 and RV-001-P10-03 and shown to be suitable for measuring residues of parent flupyradifurone, difluoroacetic acid (DFA), 6-chloronicotinic acid (6-CNA) and also difluoroethyl-amino-furanone (DFEAF) in a range of plant commodities with a high water content, high acid content, high oil content and high starch/protein content.

The current Meeting received validation and concurrent recovery data supporting the use of Method RV-001-P10-02 for mango and Method RV-001-P10-03 for pineapple fruit, juice and wet bran, sesame seed and oil and for sunflower seed, oil and meal.

Conclusions

The Meeting concluded that the analytical methods used in the supervised trials and processing studies provided to this Meeting were suitable for measuring residues of flupyradifurone and its DFA and 6-CNA metabolites, with LOQs of 0.01 mg/kg for all analytes and matrices except DFA (0.02–0.05 mg/kg). The Meeting also noted that the frozen sample storage periods in the trials were all within the acceptable (52 month) storage stability interval for high water, high acid, high oil, high protein, and high starch content matrices.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Supervised trials were available for the use of flupyradifurone on mango, papaya, pineapple, sesame seed and sunflower seed. Product labels were available from Australia, Brazil and the United States of America (United States).

Residue results are all expressed as flupyradifurone equivalents, using molecular weight conversion factors of 3.01 (DFA) and 1.83 (6-CNA). For dietary exposure estimation, total residues (the

sum of flupyradifurone, DFA and 6-CNA, expressed as parent equivalents) were calculated using the approach adopted by the 2016 JMPR:

"Where parent or DFA residues were not detected or were less than the LOQ (i.e. < 0.01 mg/kg for parent or 0.05 mg/kg for DFA) the LOQ value was utilized for maximum residue estimation and dietary intake assessment. For 6-CNA, values less than the LOQ were not added for calculation of total residues of flupyradifurone."

Table 14 Approach followed for the summing of residues

Parent	DFA	6-CNA	Total
< 0.01	0.05	0.01	0.07
0.01	< 0.05	0.01	0.07
< 0.01	< 0.05	< 0.01	< 0.06
0.01	0.05	< 0.01	0.06
0.01	0.05	0.01	0.07

Mango

The critical GAP for flupyradifurone on mango in the United States is for foliar applications of 0.2 kg ai/ha, with a minimum retreatment interval of 14 days, a PHI of 1 day and a maximum seasonal application rate of 0.41 kg ai/ha. No trials matched this GAP.

The GAP for flupyradifurone on mango in Brazil is for up to 2 foliar applications of 0.2 kg ai/ha, with a minimum retreatment interval of 7 days and a PHI of 3 days.

In five independent trials on mangos, conducted in Brazil and matching the Brazilian GAP, flupyradifurone residues (for maximum residue level estimation) in whole fruit were: 0.14, 0.2, 0.22, 0.23 and 0.3 mg/kg.

For dietary exposure estimation, since the total residues (sum of flupyradifurone, DFA and 6-CNA, expressed as parent equivalents) did not appear reach a plateau in three of the five decline trials, the Meeting agreed it was not possible to estimate an STMR and HR for mango.

The Meeting estimated a maximum residue level of 0.7 mg/kg for flupyradifurone in Mango.

Papaya

The critical GAP for flupyradifurone on papaya in the United States is for foliar applications of 0.2 kg ai/ha, with a minimum retreatment interval of 14 days, a PHI of 1 day and a maximum seasonal application rate of 0.41 kg ai/ha. No trials matched this GAP.

The GAP for flupyradifurone on papaya in Brazil is for up to 2 foliar applications of 0.2 kg ai/ha, with a minimum retreatment interval of 7 days and a PHI of 3 days.

In five independent trials on papaya, conducted in Brazil and matching the Brazilian GAP, flupyradifurone residues in whole fruit (for maximum residue level estimation) were: 0.02, 0.068, 0.1, 0.11 and 0.2 mg/kg.

For dietary exposure estimation, since the total residue concentrations (sum of flupyradifurone, DFA and 6-CNA, expressed as parent equivalents) did not appear to reach a plateau in three of the five decline trials, the Meeting agreed it was not possible to estimate an STMR and HR for mango.

The Meeting estimated a maximum residue level of 0.4 mg/kg for flupyradifurone in Papaya.

Pineapple

The critical GAP for flupyradifurone on pineapple in the United States is for foliar applications of 0.2 kg ai/ha, with a minimum retreatment interval of 7 days, a PHI of 0 days and a maximum seasonal application rate of 0.41 kg ai/ha.

In five independent trials on pineapple, conducted in the United States and matching theUnited StatesGAP, flupyradifurone residues in trimmed whole fruit (for maximum residue level estimation) were: 0.046, 0.062, 0.11, 0.12 and 0.155 mg/kg.

For dietary exposure estimation, total residues (sum of flupyradifurone, DFA and 6-CNA, expressed as parent equivalents) in trimmed whole fruit were (n = 5): 0.066, 0.082, 0.13, 0.14 and 0.175 mg/kg and the highest individual value was 0.19 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for flupyradifurone, an STMR of 0.13 mg/kg and an HR of 0.19 mg/kg for total residues in Pineapple.

Sesame seed

The GAP for flupyradifurone on sesame in the United States is for foliar applications of 0.2 kg ai/ha with a minimum retreatment interval of 10 days, a PHI of 14 days and a maximum seasonal application rate of 0.41 kg ai/ha.

In four independent trials on sesame (including one decline trial), conducted in the United States and matching theUnited StatesGAP application rate and timing, flupyradifurone residues in sesame seed samples taken 14–19 DALA were: 0.1, 0.12, 0.38 and 1.1 mg/kg.

Based on the residue decline rate shown in the decline trial, the Meeting considered that residues in samples taken 19 DALA would be within 25 percent of the expected residues in samples taken 14 DALA (GAP), and agreed that the data set was sufficient to estimate a maximum residue level.

For dietary exposure estimation, total residues (sum of flupyradifurone, DFA and 6-CNA, expressed as parent equivalents) in sesame seed were (n=4): 0.38, 0.52, 1.2 and 2.0 mg/kg and the highest individual value was 2.0 mg/kg. The medan residue was 0.86 mg/kg.

In field studies on succeeding crops evaluated by the 2016 JMPR, the overall mean and highest total residues in rape seed as a rotational crop were 0.16 mg/kg. The Meeting decided to add the mean residue found in rape seed as a rotational crop (0.16 mg/kg) to the median residue obtained from the sesame seed residue trials (0.86 mg/kg) to estimate an overall STMR of 1 mg/kg for total flupyradifurone residues in the sesame seed.

The Meeting estimated a maximum residue level of 3 mg/kg for flupyradifurone and an STMR of 1 mg/kg for total residues in Sesame seed.

Sunflower seed

The GAP for flupyradifurone in the United States on sunflowers (US sub-group 20B) is for foliar applications of 0.2 kg ai/ha with a minimum retreatment interval of 10 days, a PHI of 14 days and a maximum seasonal application rate of 0.41 kg ai/ha.

In eight independent trials on sunflower, conducted in the United States and matching theUnited StatesGAP, flupyradifurone residues in sunflower seed (for maximum residue level estimation) were: 0.0135, 0.028, 0.028, 0.04, 0.16, 0.17, 0.25 and 0.44 mg/kg.

For dietary exposure estimation, total residues (sum of flupyradifurone, DFA and 6-CNA, expressed as parent equivalents) in sunflower seed were (n=8): 0.075, 0.078, 0.09, 0.096, 0.21, 0.22, 0.3 and 0.49 mg/kg. The median residue was 0.15 mg/kg.

The Meeting decided to add the mean residue found in rape seed as a rotational crop (0.16 mg/kg) to the median residue obtained from the sunflower seed residue trials (0.15 mg/kg) to estimate an overall STMR of 0.31 mg/kg for total flupyradifurone residues in the sunflower seeds subgroup.

The Meeting estimated a maximum residue level of 0.8 mg/kg for flupyradifurone and an overall STMR of 0.31 mg/kg for total residues in the subgroup of Sunflower seeds.

Fate of residues during processing

Residues in processed commodities

The current Meeting received information on the processing of pineapple, sesame seeds and sunflower seeds. Residues decreased in pineapple juice and and sesame oil, but increased in sunflower oil.

Processing factors were calculated for total residues (dietary risk assessment) and for flupyradifurone+DFA (livestock dietary burden estimation).

For processed food commodities, STMR-Ps were calculated using the STMRs for the raw commodities and applying the calculated mean processing factors for total residues.

For sunflower oil, no processing factor for total residues could be estimated because significant residues of 6-CNA were found in the control samples.

Table 15 Calculated STMR-Ps and median-Ps for processed food and feed commodities

RAC	Processing factors		Flupyradifurone+DFA+6-CNA	Flupyradifurone + DFA
	Calculated Processing factors [a]	Best Estimate	STMR-P [b] (mg/kg)	median-P [c] (mg/kg)
Pineapple			STMR=0.13	median=0.13
Juice	0.34	0.34 [b]	0.044	
Wet bran	1.0	1.0 [c]		0.13
Sunflower seed			STMR=0.31	median=0.31
Meal	0.18	0.18 [c)]		0.056
Sesame seed			STMR=1.0	
Oil (crude)	0.13	0.13 [b]	0.13	

Notes:

[a] The ratios of the residue in the processed item divided by the residue in the Raw Agricultural Commodity

[b] Flupyradifurone + DFA + 6-CNA, expressed as parent equivalents

[c] Flupyradifurone + DFA, expressed as parent equivalents

Residues in animal commodities

Farm animal dietary burden

The maximum dietary burdens estimated by the 2016 JMPR were 72 ppm for beef, dairy cattle and 15 ppm for poultry.

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the current and previous JMPRs and using the most recent version of the OECD livestock dietary burden calculator. The results are presented in Annex 6 and summarised below.

Table 16 Estimated maximum and mean dietary burdens of farm animals

Animal dietary burden: Sum of flupyradifurone+DFA residues (as parent), ppm dry matter diet								
	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	23	7.7	77❶	17	77	23❸	8.2	5.4
Dairy cattle	47	11	67	13	77❷	21❹	50	8.8
Poultry – broiler	2.9	2.9	4.8	3.5	4.0	4.0	2.7	2.7
Poultry – layer	2.9	2.9	17❺	6.2❻	4.0	4.0	2.4	2.4

Notes:

❶ Highest maximum cattle dietary burden suitable for HR and MRL estimates for mammalian tissues.

❷ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk.

❸Highest mean cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

❺ Highest maximum poultry dietary burden suitable for HR and MRL estimates for poultry tissues and eggs.

❻ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Animal commodity maximum residue levels

Noting that the additional feed commodities considered by the Meeting increased the maximum dietary burdens estimated by the 2016 JMPR by less than 7 percent (cattle) and less than 10 percent (poultry), the Meeting agreed that the maximum residue levels, HRs and STMRs for cattle and poultry commodities need not be re-estimated.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL for plant commodities: *Flupyradifurone*

Definition of the residue for dietary risk assessment for plant commodities: *Sum of flupyradifurone, difluoroacetic acid (DFA) and 6-chloronicotinic acid (6-CNA), expressed as parent equivalents*.

Definition of the residue for compliance with the MRL for animal commodities: *Sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents*.

Definition of the residue for dietary risk assessment for animal commodities: *Sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents.*

The residue is not fat-soluble.

Table 4 Recommendations for residues of flupyradifurone from the 2022 JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FI 0353	Pineapple	0.3		0.13	0.19
SO 2091	Sunflower seeds (subgroup)	0.8		0.31	
SO 0700	Sesame seed	3		1.0	
OC 7000	Sesame seed oil (crude)			0.13	
JF 0341	Pineapple juice			0.044	
AM 3591	Pineapple process residue (wet bran)			0.13	
AM 0702	Sunflower seed meal			0.056	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for flupyradifurone is 0–0.08 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for flupyradifurone were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 6–20 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of flupyradifurone from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for flupyradifurone is 0.2 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for flupyradifurone were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs varied from 0–8 percent of the ARfD for children and 0–5 percent of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of flupyradifurone from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Reference	Author(s)	Year	Title
I17-009	Alves, F. M.	2019	Determination of the residues of BYI 02960 in/on papaya (fruit) after spraying of BYI 02960 SL 200 in the field in Brazil. Bayer S.A. – CropScience Division, SP, Brazil. Bayer AG, Report No. I17-009, Edition Number: M-649316-01-1. Unpublished.
RARV0287	Oliviera, R. C.	2020	Sivanto 200 SL - Magnitude of the residues of BYI02960 in/on mango (fruit, peel and pulp) after spraying of Sivanto 200 SL in Brazil. Bayer S.A. – CropScience Division, SP, Brazil. Bayer AG, Report No. RARV0287, Edition Number: M-678045-01-1. Unpublished.

Reference	Author(s)	Year	Title
IR4-11674	Samoil, K. S.	2018	Flupyradifurone: Magnitude of the residue on sunflower. Bayer CropScience LP, RTP, NC, United States. Bayer AG, Report No. IR-4 PR No. 11674, Edition Number: M-658103-01-1. Unpublished.
IR4-11725	Samoil, K. S.	2019	Flupyradifurone: Magnitude of the residue on sesame. Bayer CropScience LP, RTP, NC, United States. Bayer AG, Report No. IR-4 PR No. 11725, Edition Number: M-658107-01-1. Unpublished.
IR4-11711	Samoil, K. S.	2019	Flupyradifurone: Magnitude of the residue on pineapple. Bayer CropScience LP, RTP, NC, United States. Bayer AG, Report No. IR-4 PR No. 11711, Edition Number: M-658098-01-1. Unpublished.

FLUTRIAFOL (248)

First draft prepared by Hidetaka Kobayashi, Agricultural Chemicals Office, Ministry of Agriculture, Forestry and Fisheries, Japan; and Makoto Irie, Food and Agricultural Materials Inspection Center, Japan

EXPLANATION

Flutriafol, a triazole fungicide, was first evaluated for toxicology and residues by the 2011 JMPR, which established the ADI of 0–0.01 mg/kg bw and the ARfD of 0.05 mg/kg bw. The compound was evaluated by the 2015 JMPR for additional MRLs.

The following residue definitions for flutriafol were recommended by the 2011 JMPR and confirmed by the 2015 JMPR.

Definition of the residue for both compliance with MRL and estimation of dietary intake (for plant and animal commodities): flutriafol

The residue is fat soluble.

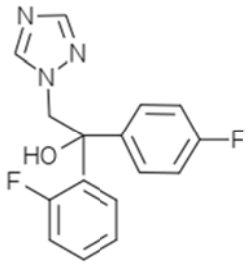
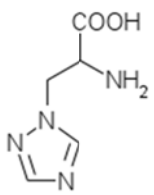
At the Fifty-First Session of CCPR (2019), flutriafol was scheduled for evaluation of additional use on hops by the 2020 JMPR. At the Forty-third Session of the Codex Alimentarius Commission (2020), flutriafol was scheduled for evaluation of additional use on barley, rice, tree nuts and sweet corn by the 2021 JMPR. The current Meeting received new information on methods of analysis, use patterns, supervised field trials and storage and processing studies on hops, barley, rice, sweet corn, almond and pecan.

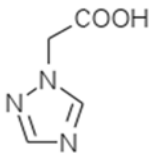
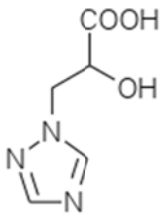
RESIDUE ANALYSIS

Analytical methods

The current JMPR received three methods of analysis for flutriafol and its metabolites. The IUPAC name and structure of flutriafol and its metabolites are shown in Table 1.

Table 1 Name and structure of flutriafol and its metabolites

Common name IUPAC name	Structure
Flutriafol (*RS*)-2,4′-difluoro-alfa-(1*H*-1,2,4-triazol-1-ylmethyl)benzhydryl alcohol	
1,2,4-triazole (T) 1*H*-1,2,4-triazole	
Triazole alanine (TA) 2-amino-3-(1,2,4-triazol-1-yl)propanoic acid	

Common name IUPAC name	Structure
Triazole acetic acid (TAA) 2-(1,2,4-triazol-1-yl)acetic acid	
Triazole lactic acid (TLA) 2-hydroxy-3-(1,2,4-triazol-1-yl)propanoic acid	

Method #1 (Carringer (2015), TCI-14-393; Carringer (2015), TCI-14-392; Chadwick (2019), S17-02483; and Rice (2011), 65573)

The method of analysis is for flutriafol in barley (hay, grain and flour), sweet corn (forage, corn-on-the-cob and stover), rice (whole plant, straw, grain with husk, husked rice, husks, polished rice and bran) and almond (nutmeat and hulls). Samples were extracted with acetonitrile-water (7:3, v/v) and centrifuged. The supernatant was made up to 150 mL with acetonitrile-water (7:3, v/v) and 1 mL of the solution was added to 9 mL of acetonitrile-water (5:5, v/v). The solution was introduced to LC-MS/MS (turbo ion spray in positive mode) with a C_{18} column. The gradient solvent system was the mixture of solution A and solution B of 6:4→1:9 (v/v) (solution A: water containing 0.1 percent (v/v) of formic acid, solution B: methanol containing 0.1 percent (v/v) of formic acid). The monitored transitions were 302→70 (quantitation) and 302→123 (confirmation). Determination was using by external matrix-matched standard. The calibration curves for flutriafol were linear (Table 2).

The LOQs for all matrices were 0.01 mg/kg. Mean recoveries of flutriafol were 80-82 percent (barley, hay), 82-87 percent (barley, grain), 93 percent (barley, flour), 101–109 percent (rice, whole plant), 93–106 percent (rice, straw), 101–105 percent (rice grain with husk), 102–110 percent (risk, husked), 106–107 percent (rice husks), 101–109 percent (rice, polished), 109–110 percent (rice bran), 84–105 percent (sweet corn, forage), 83–89 percent (sweet corn, corn-on-the-cob), 80–81 percent (sweet corn, stover), 101–109 percent (almond nutmeat), and 104–108 percent (almond hulls)(Table 3).

Table 2 Linearity of calibration curve for Method #1

Analyte	Matrix	Linear range (mg/kg)	R
Flutriafol	Barley (all matrices)	0.012–0.099	>0.998
	Rice (all matrices)	0.0030–0.75	>0.980
	Tree nuts (all matrices)	0.0050–0.10	>0.998
	Sweet corn (all matrices)	0.012–0.10	>0.998

Table 3 Method validation data for Method #1

Analyte	Matrix	Fortification mg/kg	n	Recovery range percent (mean)	RSD percent
flutriafol	Barley, hay	0.01	5	75-94 (82)	9.6
		1.0	5	72-84 (80)	5.8
	Barley, grain	0.01	5	81-91 (87)	4.6

Analyte	Matrix	Fortification mg/kg	n	Recovery range percent (mean)	RSD percent
		1.0	5	72-80 (82)	9.4
	Barley, flour	0.01	5	89-97 (93)	3.4
		1.0	5	87-105 (93)	7.6
	Rice, whole plant	0.010	5	97-106 (101)	3.5
		0.10	5	106-112 (109)	2.5
	Rice straw	0.010	5	82-105 (93)	11
		0.10	5	102-109 (106)	9.7
	Rice grain (with husk)	0.010	5	103-110 (105)	3.3
		0.10	5	88-105 (101)	6.8
	Rice, husked	0.010	5	100-105 (102)	2.1
		0.10	5	106-113 (110)	2.5
	Rice, husks	0.010	5	103-110 (106)	3.3
		0.10	5	104-109 (107)	2.5
	Rice, polished	0.010	5	99-103 (101)	1.5
		0.10	5	106-111 (109)	2.5
	Rice, bran	0.010	5	107-112 (110)	1.7
		0.10	5	107-110 (109)	1.2
	Sweet corn, forage	0.01	5	73-91 (84)	9.3
		1.0	5	88-110 (105)	9.1
	Sweet corn (corn-on-the-cob)	0.01	5	74-94 (83)	9.7
		1.0	5	79-93 (89)	6.6
	Sweet corn, stover	0.01	5	73-86 (80)	6.1
		1.0	5	77-84 (81)	3.4
	Almond nutmeat	0.01	5	102-119 (109)	6.9
		0.1	5	83-108 (101)	10
	Almond hulls	0.01	5	105-111 (108)	2.2
		0.1	5	99-111 (104)	4.6

Method #2 (Carringer (2015), TCI-14-393; Carringer (2015), TCI-14-392; and Rice (2011), 65573)

The method was for 1,2,4-triazole (T), triazole alanine (TA) and triazole acetic acid (TAA) in barley (hay, grain and flour), sweet corn (forage, corn-on-the-cob and stover) and almond (nutmeat and hulls). Samples were extracted with methanol-water (8:2, v/v). The extract, to which celite was added, was filtered and methanol-water (8:2, v/v) was added to the filtrate. It was processed through solid phase extraction (SPE). Then, aliquots were taken for different derivation processes for T, TA and TAA.

For T, the aliquot was derivatized with dansyl chloride and partitioned into ethyl acetate. For TA, the aliquot was derivatized by reaction with HCl (3 mol/L) in n-butanol followed by heptafluorobutyric anhydride. For TAA, the aliquot was derivatized by reaction with HCl (3 mol/L) in n-butanol. Each of derivative mixtures was evaporated to dryness and then dissolved in 2.5 mL of acetonitrile-water (3:7, v/v). They were individually introduced to LC/MS/MS (turbo ion spray in positive mode) with C18 column. The solvent system was 0.1 percent (v/v) formic acid (aq) – acetonitrile (8:2→1:9, v/v). Mass transitions monitored were shown in Table 4. Determination was using by derivatized stable isotope internal standards.

The LOQs were 0.01–0.16 mg/kg (Table 5). For barley (hay, grain and flour), sweet corn (forage, corn-on-the-cob and stover) and almond (nutmeat and hulls), the mean recovery ranges for T, TA and TAA were 78–100 percent, 87–109 percent and 93–111 percent, respectively (Table 6).

The method for T, TA and TAA in hops was Method Meth-160 with a LOQ of 0.01 mg/kg.

Table 4 Mass transition monitored in Method #2

Analyte	Mass transition	
	Quantitation	Confirmation
1,2,4-triazole (T)	303 →181	303→195
Triazole alanine (TA)	409→70	409→284 409→210
Triazole acetic acid (TAA)	184→70	184→128

Table 5 Reported LOQs of Method #2 for analyte/matrix combination

Analyte	Matrix	Reported LOQ	Linearity of calibration curve	
			Range (mg/kg)	R
1,2,4-triazole (T)	Barley, hay	0.01	0.0015-0.99	>0.998
	Barley, grain	0.01	0.0015-0.99	>0.998
	Barley, flour	0.01	0.0015-0.99	>0.998
	Almond nutmeat	0.01	0.015-1.0	>0.998
	Almond hulls	0.01	0.015-1.0	>0.998
	Sweet corn (corn-on-the-cob)	0.01	0.0015-1.0	>0.998
	Sweet corn forage	0.01	0.0015-1.0	>0.998
	Sweet corn stover	0.01	0.0015-1.0	>0.998
Triazole alanine (TA)	Barley, hay	0.03	0.0015-0.99	>0.998
	Barley, grain	0.06	0.0015-0.99	>0.998
	Barley, flour	0.07	0.0015-0.99	>0.998
	Almond nutmeat	0.01	0.015-1.0	>0.998
	Almond hulls	0.01	0.015-1.0	>0.998
	Sweet corn (corn-on-the-cob)	0.16	0.0015-1.0	>0.998
	Sweet corn forage	0.01	0.0015-1.0	>0.998
	Sweet corn stover	0.01	0.0015-1.0	>0.998
Triazole acetic acid (TAA)	Barley, hay	0.04	0.0025-0.99	>0.998
	Barley, grain	0.05	0.0025-0.99	>0.998
	Barley, flour	0.02	0.0025-0.99	>0.998
	Almond nutmeat	0.01	0.0025-1.0	>0.998
	Almond hulls	0.01	0.0025-1.0	>0.998
	Sweet corn (corn-on-the-cob)	0.01	0.0025-1.0	>0.998
	Sweet corn forage	0.01	0.0025-1.0	>0.998
	Sweet corn stover	0.01	0.0025-1.0	>0.998

Table 6 Method validation data for Method #2

Analyte	Matrix	Fortification mg/kg	n	Recovery range percent (mean)	RSD percent
1,2,4-triazole (T)	Barley, hay	0.01	5	87-110 (100)	9.2
		1.0	5	95-97 (96)	1.1
	Barley, grain	0.01	5	97-103 (100)	2.7
		1.0	5	94-97 (96)	1.3
	Barley, flour	0.01	5	91-104 (97)	4.9
		1.0	5	91-98 (94)	3.1
	Sweet corn, forage	0.01	5	87-93 (90)	2.4
		1.0	5	85-94 (89)	3.8
	Sweet corn (corn-on-the-cob)	0.01	5	94-102 (99)	3.0
		1.0	5	89-96 (93)	2.9

Analyte	Matrix	Fortification mg/kg	n	Recovery range percent (mean)	RSD percent
	Sweet corn, stover	0.01	5	89-106 (98)	7.6
		1.0	5	92-101 (97)	3.8
	Almond nutmeat	0.01	5	84-106 (100)	9.1
		0.1	5	82-96 (87)	7.0
	Almond hulls	0.01	5	70-99 (78)	16
		0.1	5	81-94 (86)	5.6
Triazole alanine (TA)	Barley, hay	0.03	5	86-93 (89)	3.5
		1.0	5	96-101 (98)	2.2
	Barley, grain	0.06	5	82-98 (88)	7.5
		1.0	5	96-101 (98)	2.2
	Barley, flour	0.07	5	96-99 (98)	1.4
		1.0	5	92-97 (95)	2.4
	Sweet corn, forage	0.01	5	101-105 (102)	1.9
		1.0	5	106-110 (109)	1.5
	Sweet corn (corn-on-the-cob)	0.16	5	98-106 (91)	14
		1.0	5	96-106 (101)	3.6
	Sweet corn, stover	0.01	5	71-101 (87)	13
		1.0	5	94-102 (98)	3.4
	Almond nutmeat	0.01	5	98-114 (104)	6.4
		0.1	5	88-97 (93)	4.4
	Almond hulls	0.01	5	80-91 (83)	5.4
		0.1	5	82-90 (87)	3.9
Triazole acetic acid (TAA)	Barley, hay	0.04	5	104-113 (108)	3.4
		1.0	5	108-112 (111)	1.5
	Barley, grain	0.05	5	92-104 (98)	5.6
		1.0	5	97-102 (100)	2.1
	Barley, flour	0.02	5	89-98 (93)	3.8
		1.0	5	104-110 (106)	2.4
	Sweet corn, forage	0.01	5	91-106 (99)	5.8
		1.0	5	100-110 (103)	4.0
	Sweet corn (corn-on-the-cob)	0.01	5	91-110 (101)	9.1
		1.0	5	110-117 (113)	2.3
	Sweet corn, stover	0.01	5	86-106 (96)	8.0
		1.0	5	100-110 (107)	3.7
	Almond nutmeat	0.01	5	99-115 (108)	5.4
		0.1	5	100-109 (104)	3.7
	Almond hulls	0.01	5	90-101 (95)	4.3
		0.1	5	92-98 (95)	2.3

Method #3 (Chadwick (2019), S17-02483)

The method is for T, TA, TAA and triazole lactic acid (TLA) in rice (whole plant, straw, grain with husk, husked rice, husks, polished rice and bran). Samples were extracted with methanol-water (8:2, v/v), centrifuged and filtered through glass wool. The supernatant was dried under N_2 gas and the volume adjusted to 5 mL with water, and then introduced to LC-MS/MS (turbo ion spray in positive mode), using a polar end capped C_{18} column. Gradient solvent system was 0.5 percent (v/v) acetic acid in methanol - 0.5 percent (v/v) acetic acid (aq) (2:8->7:3). Mass transitions monitored and HPLC column used are shown in Table 7. Determination was using by derivatized stable isotope internal standards. The calibration curves were linear (R>0.980) between 0.0020–2.0 mg/kg for all analytes/matrices.

The LOQs were 0.010 mg/kg for T, TA, TAA and TLA. Mean recoveries of T, TA, TAA and TLA were 90–110 percent, 72–116 percent, 81–112 percent and 74–110 percent, respectively (Table 8).

Table 7 Mass transition and column used in Method #3

Analyte	Quantitation/confirmation	Column	Mass transition
1,2,4-triazole (T)	Quantitation	Polar-reversed phase (RP)	70->43
	Confirmation	C18	70->43
Triazole alanine (TA)	Quantitation	Polar-RP	157->70
	Confirmation	C18	157->88
Triazole acetic acid (TAA)	Quantitation	Polar-RP	128->70
	Confirmation	C18	128->70
Triazole lactic acid (TLA)	Quantitation	Polar-RP	158->70
	Confirmation	C18	158->70

Table 8 Method validation data for Method #3

Analyte	Matrix	Fortification mg/kg	n	Recovery range percent (mean)	RSD percent
1,2,4-triazole (T)	Rice, whole plant	0.010	5	93-116 (106)	9.1
		0.10	5	91-120 (110)	9.1
	Rice straw	0.010	5	81-116 (98)	14
		0.10	5	89-107 (98)	8.1
	Rice grain (with husk)	0.010	5	96-118 (110)	8.5
		0.10	5	95-119 (104)	9.6
	Rice, husked	0.010	5	79-95 (90)	6.9
		0.10	5	71-106 (84)	16
	Rice, husks	0.010	5	95-115 (106)	7.3
		0.10	5	89-119 (108)	8.3
	Rice, polished	0.010	5	87-110 (99)	10
		0.10	5	86-104 (96)	8.5
	Rice, bran	0.010	5	84-116 (100)	14
		0.10	5	90-118 (104)	13
Triazole alanine (TA)	Rice, whole plant	0.0081	5	78-112 (95)	13
		0.081	5	81-106 (91)	11
	Rice straw	0.0081	5	96-117 (107)	8.4
		0.081	5	84-119 (101)	16
	Rice grain (with husk)	0.0081	5	77-120 (93)	20
		0.081	5	80-99 (89)	7.8
	Rice, husked	0.0081	5	72-112 (96)	18
		0.081	5	80-90 (84)	4.6
	Rice, husks	0.0081	5	88-112 (99)	10
		0.081	5	107-139 (116)	11
	Rice, polished	0.0081	5	69-93 (82)	14
		0.081	5	61-77 (72)	8.9
	Rice, bran	0.0081	5	62-102 (80)	20
		0.081	5	81-114 (96)	16
Triazole acetic acid (TAA)	Rice, whole plant	0.010	5	91-114 (103)	9.0
		0.10	5	96-119 (106)	8.0
	Rice straw	0.010	5	98-119 (108)	7.9
		0.10	5	90-102 (98)	4.9
	Rice grain (with husk)	0.010	5	80-105 (94)	9.7
		0.10	5	70-88 (81)	8.8
	Rice, husked	0.010	5	71-90 (80)	10

Analyte	Matrix	Fortification mg/kg	n	Recovery range percent (mean)	RSD percent
		0.10	5	80-98 (85)	8.7
	Rice, husks	0.010	5	91-115 (97)	6.8
		0.10	5	96-116 (112)	3.5
	Rice, polished	0.010	5	66-114 (88)	20
		0.10	5	76-88 (82)	5.3
	Rice, bran	0.010	5	76-113 (88)	15
		0.10	5	78-100 (94)	6.8
Triazole lactic acid (TLA)	Rice, whole plant	0.010	5	84-120 (105)	14
		0.10	5	99-107 (103)	3.3
	Rice straw	0.010	5	83-112 (95)	12
		0.10	5	97-105 (100)	3.4
	Rice grain (with husk)	0.010	5	82-93 (87)	5.8
		0.10	5	78-89 (84)	5.6
	Rice, husked	0.010	5	77-80 (78)	1.7
		0.10	5	72-81 (77)	4.6
	Rice, husks	0.010	5	96-116 (105)	8.8
		0.10	5	107-113 (110)	2.4
	Rice, polished	0.010	5	79-88 (82)	4.6
		0.10	5	72-76 (74)	2.3
	Rice, bran	0.010	5	71-87 (81)	8.6
		0.10	5	86-96 (91)	4.8

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

The 2011 JMPR concluded that flutriafol residues were stable for at least 4 months in animal commodities, for at least 5 months in soya bean seed[4a], for at least 12 months in apple[1], barley grains[5] and coffee beans[6], for at least 23 months in grapes[2], for at least 24 months in cabbage[1] and oilseed rape[4a], and for at least 25 months in wheat[5] (grains and straw), pea seed[5] and sugar beet root[1]. The 2011 JMPR also concluded that triazole metabolite residues were stable for at least 4 months in apple fruits and juice[1] and for at least 5 months in animal commodities (1/high water content, 2/high acid content and high water content, 4a/high oil content and very low water content, 5/high starch and/or protein content and low water and fat content, 6/difficult or unique commodities).

For hops, the Meeting received data on the storage stability of triazole metabolites stored frozen (Rodgers (2016), 82662).

The stability study of triazole metabolites was conducted on hops (dried cones) stored frozen at approximately -20 °C. Samples of untreated homogenized hops were fortified at 0.1 mg/kg for T and TAA, and at 1.2 mg/kg for TA then placed in storage at approximately -20 °C, except for the 0 day analysis set. These samples were analysed after 0, 3, 6 and 9 months frozen storage. All samples were analysed in duplicate using Method Meth-160. The residues of triazole metabolites were determined using LC-MS/MS. The LOQ was 0.01 mg/kg (Table 9).

Table 9 Recovery from stored fortified samples of hops (separately fortified with T, TA and TAA)

Time stored (days/months)		Fortification level (mg/kg)	percent Recovery		
			Procedural recovery	percent remaining	Mean of percent remaining
1,2,4-Triazole (T)	0	0.1	-	92, 94	93

Time stored (days/months)		Fortification level (mg/kg)	percent Recovery		
			Procedural recovery	percent remaining	Mean of percent remaining
	104 / 3		91, 94	56, 59	58
	182 / 6		97, 98	46, 47	47
	274 / 9		108, 109	49, 60	55
Triazole alanine (TA)	0	1.2	-	101, 102	102
	91 / 3		96, 102	99, 104	102
	182 / 6		96, 104	100, 100	100
	274 / 9		97, 98	91, 96	94
Triazole acetic acid (TAA)	0	0.1	-	113, 118	116
	182 / 3		114, 117	114, 116	115
	274 / 6		117, 121	122, 122	122

USE PATTERN

The Meeting received the GAP for barley; rice; sweet corn; almond and walnut; pecan and other tree nuts; and hops as shown in Table 10. While the labels provided cover a broader spectrum of uses, only those relevant to the current evaluation are reported.

Table 10 Use pattern of flutriafol

Crop	Country	Formulation	Application					
			Type	kg ai/ha	Growth stage	No	minimum RTI (days)	PHI (days)
Barley	United States	SC	Foliar spray	0.128	Footnote 4	2	7	30 (grain) 15 (hay) 0 (forage)
Barley	United States	SC	Foliar spray	0.128	Anthesis (Feekes growth stage 10.51)	2	7	30 (grain) 15 (hay) 0 (forage)
Rice	Italy	SC	Foliar spray	0.125	From end of rising	1	-	28
Almond	United States	SC	Foliar spray	0.128	Footnote 4	6	7	14
Walnut	United States	SC	Foliar spray	0.128	Footnote 4	4	7	14
Pecan and other tree nuts[(1)]	United States	SC	Foliar spray	0.128	Footnote 4	4	7	14
Corn[(2)]	United States	SC	T-band application or in-furrow application to soil, fFoliar sprays	0.128	No later than stage R4 (early dough stage)[(4)]	2[(3)]	7	7 (grain and stover) 0 (forage)
Hops	United States	SC	Ground or aerial	0.128		4	14	7

Notes:

[1] Other tree nuts include African tree nut, brazil nut, burr oak, butternut, cajou, cashew, castanha-do-maranhao, coconut, coquito nut, dika nut, Guiana chestnut, hazelnut, heartnut, hickory nut, Japanese horse-chestnut, macadamia nut, monogongo nut, monkey-pot, pachira nut, pecan and sapucaia nut.

[2] Corn includes field corn, field corn grown for seed, sweet corn, and popcorn

[3] Maximum two foliar applications, or one at planting and one foliar application.

[4] When conditions are favourable for disease

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue levels are reported as measured, without correction for recovery. When residue concentrations were less than LOQ, they are shown as below the LOQ, e.g., < 0.01 mg/kg. Residues for metabolites were expressed as the compounds. Residue values from the trials conducted according to the maximum GAP were used for the estimation of maximum residue levels, STMR and HR. These results are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analysis or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. The results of field trials are shown in tables indicated in Table 11.

Table 11 Results of supervised field trials for flutriafol

Commodity	Result
Cereal grains	
Barley	Table 12
Sweet corn (corn-on-the-cob)	Table 13
Rice	Table 14
Tree nuts	
Almonds	Table 15
Pecan	Table 16
Dried herbs	
Hops, dry	Table 17
Animal feed commodities	
Barley, straw	Table 18
Barley, hay	Table 19
Sweet corn forage	Table 20
Sweet corn stover	Table 21
Rice straw	Table 22
Rice, whole plant	Table 23
Almond hulls	Table 24

Cereal grains

Barley (Carringer (2015), TCI-14-393)

The Meeting received 12 supervised trials conducted in 2014 on barley in United States. In these trials, barley received two foliar applications of flutriafol (125 g/L SC) at 0.127–0.130 kg ai/ha with intervals of 6–8 days. In 11 trials, plants were harvested at 28–37 days after the last application (DALA). One trial was a decline study (harvested at 17–45 DALA).

The residues of flutriafol and its metabolites in barley (grain) were analysed by Method #1 and Method #2, respectively. The LOQs were 0.01 mg/kg for flutriafol and T, 0.06 mg/kg for TA, and 0.05 mg/kg for TAA. Samples were stored at ≤-20 °C for ≤ 329 days before analysis. Procedural recoveries in barley (hay, grain and straw) of flutriafol, T, TA and TAA were 69–102 percent, 79–107 percent, 87–113 percent and 98–119 percent, respectively.

In some trials, TA and/or TAA were detected in the control sample. Since TA and TAA in control samples were considered to be derived from previous treatment with other pesticides, they should not be attributed to flutriafol treatment. In the table, analytical values for control sample were shown if they were higher than LOQ. The results are shown in Table 12.

Table 12 Residues of flutriafol and its metabolites in barley grain after foliar application of flutriafol 125 g/L SC

Location, year (variety)	No	RTI (day)	kg ai/ha	DALA	Residue [/a] (mg/kg)			
					Flutriafol [a]	T	TA	TAA
GAP (United States)	2	7	2 × 0.128	30				
Baptistown, NJ United States 2014 (AC Minoa)	2	7	0.129 0.128	28	<u>0.11</u>	<0.01	0.37	0.17
York, NE, United States 2014 (Haybet)	2	7	0.128 0.128	17 24 31 38 45	0.91 0.89 0.65 <u>0.77</u> 0.28	<0.01 <0.01 <0.01 <0.01 <0.01	0.30 0.32 0.43 c 0.13 0.29 0.24	<0.05 0.056 0.053 0.050 <0.05
Geneva, MN, United States 2014 (Rasmusson)	2	8	0.129 0.128	30	<u>0.84</u>	<0.01	0.24 c 0.073	<0.05
Richland, IA, United States 2014 (Robust)	2	7	0.128 0.128	30	<u>0.15</u>	<0.01	0.27 c 0.14	<0.05
Grand Island, NE, United States 2014 (Haybet)	2	7	0.129 0.128	30	<u>0.29</u>	<0.01	0.30	0.057
Jamestown, ID, United States 2014 (Tradition)	2	6	0.129 0.130	31	<u>0.18</u>	<0.01	0.15	<0.05
Velva, ND, United States 2014 (Tradition)	2	7	0.127 0.127	29	<u>0.17</u>	<0.01	0.80 c 0.30	0.20 c 0.092

Location, year (variety)	No	RTI (day)	kg ai/ha	DALA	Residue [/a] (mg/kg)			
					Flutriafol [a]	T	TA	TAA
Carrington, ND, United States 2014 (Rasmusson)	2	8	0.126 0.130	29	0.12	<0.01	0.53 c 0.20	0.14 c 0.051
Jerome, ID, United States 2014 (Moravian 69)	2	7	0.128 0.128	37	0.19	<0.01	<0.06	<0.05
Porterville, CA, United States 2014 (Lockwood Chowford Beardless)	2	7	0.127 0.127	29	0.23	<0.01	0.078	0.060
Payette, ID, United States 2014 (Millenium)	2	7	0.132 0.129	29	0.20	<0.01	0.21 c 0.097	0.11 c 0.060
Ephrata, WA, United States 2014 (Champion)	2	7	0.129 0.128	30	0.34	<0.01	0.22	<0.05

Notes:

[a] Residues in untreated samples are denoted by (c XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg for all matrices except TA (0.06 mg/kg) and TAA (0.05 mg/kg)). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples

Sweet corn (Carringer (2015), TCI-14-392)

The Meeting received 16 supervised trials conducted in 2014 on sweet corn in United States. In 12 trials, sweet corn received an in-furrow application of flutriafol (125 g/L SC) at 0.289–0.305 kg ai/ha when planting and two foliar applications of flutriafol (125 g/L SC) at 0.126–0.131 kg ai/ha with an interval of 6–8 days. In four trials, sweet corn received two foliar applications of flutriafol (125 g/L SC) at 0.128–0.136 kg ai/ha with an interval of 7–8 days. Corn-on-the-cob was harvested 0 DALA (on the day of the last application). Two trials were decline study (0–14 DALA for corn-on-the-cob and forage, and 0–21 DALA for stover).

The residues of flutriafol and its metabolites (T, TA and TAA) in sweet corn (corn-on-the-cob) were analysed by Method #1 and Method #2, respectively. The LOQs were 0.01 mg/kg for flutriafol, T and TAA and 0.16 mg/kg for TA. Samples were stored at ≤-20°C for ≤287 days before analysis. Procedural recoveries for sweet corn (corn-on-the-cob, forage and stover) of flutriafol, T, TA and TAA ranged 71–102 percent, 84–107 percent, 71–114 percent, and 91–120 percent, respectively.

The results are shown in Table 13.

Table 13 Residues of flutriafol and its metabolites in sweet corn (corn-on-the-cob) after foliar application of flutriafol 125 g/L SC

Location, year (variety)	Application					Residue (mg/kg) [e]			
	Method/ Timing	No	RTI [d]	kg ai/ha	DALA	Flutriafol	T	TA	TAA
GAP (United	Foliar (2)	2	7	0.128+0.128	7				

Location, year (variety)	Application Method/ Timing	No	RTI[d]	kg ai/ha	DALA	Residue (mg/kg)[e] Flutriafol	T	TA	TAA
States)	or In-furrow (1)+ foliar (1)	or 1+1[a]		or 2x0.128					
Germanville, PA, United States 2014 (MIrai 421 W F1)	In-furrow[b] 8 DBH[c] 0 DBH	1+2	8	0.305 0.130 0.130	0	<0.01	<0.01	0.25	<0.01
Germanville, PA, United States 2014 (MIrai 421 W F1)	8 DBH 0 DBH	2	8	0.136 0.133	0	<0.01	<0.01	<0.16	<0.01
Alton, NY, United States 2014 (Precious Gem)	In-furrow 7 DBH 0 DBH	1+2	7	0.295 0.128 0.129	0	<0.01	<0.01	0.23 c 0.17	<0.01
Seven springs, NC, United States 2014 (Sweet G90)	In-furrow 7 DBH 0 DBH	1+2	7	0.294 0.131 0.128	0	0.019	<0.01	0.51	0.016
Oviedo, FL, United States 2014 (Mirai 308 BC F1)	In-furrow 6 DBH 0 DBH	1+2	6	0.289 0.126 0.127	0	<0.01	<0.01	0.17	<0.01
Conklin, MI, United States 2014 (Luscious)	In-furrow 6 DBH 0 DBH	1+2	6	0.290 0.128 0.128	0	<0.01	<0.01	<0.16	<0.01
Carlyle, IL, United States 2014 (Providence)	In-furrow 7 DBH 0 DBH	1+2	7	0.289 0.130 0.129	0	0.010	<0.01	0.16	<0.01
Carlyle, IL, United States 2014 (Providence)	7 DBH 0 DBH	2	7	0.130 0.130	0	<0.01	<0.01	<0.16	<0.01
Richland, IA, United States 2014 (Xtra-tender 2573 F1 (sh2))	In-furrow 7 DBH 0 DBH	1+2	7	0.293 0.129 0.128	0 1 7 14	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.16 <0.16 <0.16 <0.16	<0.01 <0.01 <0.01 <0.01
Richland, IA, United States 2014 (Xtra-tender 2573 F1	7 DBH 0 DBH	2	7	0.129 0.128	0 1 7 14	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.16 <0.16 <0.16 <0.16	<0.01 <0.01 <0.01 <0.01

Location, year (variety)	Application Method/ Timing	No	RTI[d]	kg ai/ha	DALA	Residue (mg/kg)[e] Flutriafol	T	TA	TAA
(sh2))									
Delavan, WI, United States 2014 (NK 199)	In-furrow 7 DBH 0 DBH	1+2	7	0.298 0.129 0.129	0	<0.01	<0.01	<0.16	<0.01
York, NE, United States 2014 (Obsession II)	In-furrow 7 DBH 0 DBH	1+2	7	0.291 0.129 0.127	0	<0.01	<0.01	<0.16	<0.01
Porterville, CA, United States 2014 (Bodacious)	In-furrow 6 DBH 0 DBH	1+2	6	0.290 0.131 0.128	0	<0.01	<0.01	<0.16	<0.01
Ephrata, WA, United States 2014 (Serendipity)	In-furrow 7 DBH 0 DBH	1+2	7	0.291 0.129 0.128	0	0.016	<0.01	0.33	<0.01
Ephrata, WA, United States 2014 (Serendipity)	7 DBH 0 DBH	2	7	0.128 0.128	0	<0.01	<0.01	<0.16	<0.01
Hillsboro, OR, United States 2014 (Hony 'N Pearl)	In-furrow 7 DBH 0 DBH	1+2	7	0.290 0.126 0.128	0	0.012	<0.01	<0.16	<0.01

Notes:

[a] 2 foliar applications with the interval of 7 days or more, or 1 application at planting (T-band application or in-furrow application) + 1 foliar application.

[b] In-furrow application at planting.

[c] DBH – Days before harvest of sweet corn forage/corn-on-the-cob.

[d] RTI between 2 foliar applications.

[e] Residues in untreated samples are denoted by (c XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg for all matrices except for TA in K+CWHR for which the LOQ is 0.16 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

Rice (Chadwick (2018), S17-02483 and Chadwick (2019), S18-04372)

The Meeting received 12 supervised trials conducted in 2017–2018 on rice in Bulgaria, France, Italy, Portugal and Spain. In these trials, rice received one foliar application of flutriafol (250 g/L SC) at 0.119–0.134 kg ai/ha. Straw (12 trials) and grains with husk (8 trials) were harvested at 27–28 days after treatment (DAT). In addition, for decline study, whole plants were collected and analysed at 0–21 DAT in six trials.

The residues of flutriafol and its metabolites (T, TA, TAA and TLA) in rice (whole plant, straw and grains with husk) were analysed by Method #1 and Method #3, respectively. LOQs were 0.01 mg/kg. Samples were stored at ≤-20 °C for ≤ 88 days before analysis. Procedural recoveries for rice (whole plant,

straw and grains with husk) of flutriafol, T, TA, TAA and TLA ranged 82–112 percent, 81–120 percent, 72–120 percent, 70–119 percent, and 70–120 percent, respectively.

The results are shown in Table 14.

Table 14 Residues of flutriafol and its metabolites in rice grain (with husk) after foliar application of flutriafol 250 g/L SC

Location, year (Variety)			Residue (mg/kg) [a]				
	kg ai/ha	DALA	Flutriafol	T	TA	TAA	TLA
GAP (Italy)	0.125	28					
Arles, France 2017 (Gajeron)	0.122	28	1.6	<0.01	0.07 c 0.06	0.04	<0.01
Casale Monferrato, Italy 2017 (Sagitanio)	0.134	28	1.1	<0.01	0.02	0.01	<0.01
Alfarelos, Portugal 2017 (Ariete)	0.125	28	0.87	<0.01	0.02 c 0.02	<0.01	<0.01
Kostievo, Bulgaria 2017 (Lince)	0.132	28	1.4	<0.01	0.16 c 0.06	0.08	<0.01
Arle, France 2018 (Gageron)	0.129	28	1.1	<0.01	0.06 c 0.06	0.05 c 0.05	<0.01
Plovdiv, Bulgaria 2018 (Lince)	0.130	28	1.1	<0.01	0.15 c 0.06	0.07 c 0.04	<0.01
Malalbergo, Italy 2018 (Volano)	0.133	28	0.57	<0.01	0.07 c 0.08	0.09 c 0.11	<0.01
Isla Mayor, Spain 2018 (J-Sendra)	0.119	28	0.82	<0.01	<0.01 c 0.01	0.03 c 0.02	0.02

Notes:

[a/] Residues in untreated samples are denoted by (c XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

Tree nuts

Almond (Rice (2011), 65573)

The Meeting received five supervised trials conducted in 2010 on almond in United States. In these trials, almond received six foliar applications of flutriafol (125 g/L SC) at 0.127–0.134 kg ai/ha with intervals of 6–8 days. Almonds were harvested at 14 DALA. One trial was a decline study in which samples were harvested at 1–28 DALA.

The residues of flutriafol and its metabolites (T, TA and TAA) in nutmeat and hulls of almonds were analysed by Method #1 and Method #2, respectively. The LOQs were 0.01 mg/kg for flutriafol, T and TAA; 0.2 mg/kg for TA in nutmeat; and 0.15 mg/kg for TA in hulls. Samples were stored at ≤-20 °C for ≤ 230 days (almond nutmeat) or ≤92 days (almond hulls) before analysis. Procedural recoveries for almond (nutmeat and hulls) of flutriafol, T, TA and TAA ranged 77–112 percent, 73–111 percent, 73–113 percent and 96–119 percent, respectively.

The results are shown in Table 15.

Table 15 Residues of flutriafol and its metabolites in almond (nutmeat) after foliar application of flutriafol 125 g/L SC

Location Year (Variety)	Application Timing [a]	No	kg ai/ha	DALA	Residue [b] Flutriafol	T	TA	TAA
GAP (United States)		6	6 × 0.128	14				
Dinuba, CA, United States 2010 (Sonora)	54 46 38 30 22 14	6	0.128 0.129 0.128 0.129 0.128 0.128	14	0.064	<0.01	<0.2	<0.01
Strathmore, CA, United States 2010 (fritz)	48 42 35 28 21 14	6	0.128 0.128 0.129 0.128 0.128 0.128	14	0.012	0.021 c 0.11	0.913 c 2.7	0.011 c 0.026
Wasco, CA, United States 2010 (price)	50 42 36 29 22 14	6	0.128 0.128 0.128 0.128 0.128 0.128	14	0.066	<0.01	0.55 c 0.29	<0.01
Buttonwillow, CA, United States 2010 (Monterey)	49 42 35 28 21 14	6	0.128 0.127 0.134 0.128 0.128 0.128	14	<0.01	<0.01	0.69 c 0.49	<0.01
Terra Bella, CA 2010 (non-pareil)	41 32 25 16 9 1	6	0.127 0.128 0.127 0.128 0.129 0.128	1 7 14 21 28	0.41 0.27 0.30 0.42 0.24	<0.01 <0.01 <0.01 0.010 <0.01	0.64 0.58 0.70 c 2.1 0.90 0.68	<0.01 <0.01 <0.01 <0.01 <0.01

Notes:

[a] Days before the earliest harvest.

[b] Residues in untreated samples are denoted by (c XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg for all analytes except TA for which the LOQ is 0.2 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

Pecan (Rice (2011), 65573)

The Meeting received five supervised trials conducted in 2010 on pecan in United States. In these trials, pecan received six foliar applications of flutriafol (125 g/L SC) at 0.126–0.132 kg ai/ha with intervals of 6–8 days. Pecans were harvested at 11–14 DALA.

The residues of flutriafol and its metabolites (T, TA and TAA) in pecan nutmeat were analysed by Method #1 and Method #2, respectively. The LOQs were 0.01 mg/kg for flutriafol, T and TAA and 0.2 mg/kg for TA. Samples were stored at ≤-20 °C for ≤ 162 days before analysis. Procedural recoveries for pecan nutmeat of flutriafol, T, TA and TAA ranged 80–112 percent, 88–109 percent, 73–104 percent, and 98–114 percent, respectively.

The results are shown in Table 16.

Table 16 Residues of flutriafol and its metabolites in pecan (nutmeat) after foliar application of flutriafol 250 g/L SC

Location Year (Variety)	Application Timing [a]					Residue [b]			
		No	RTI	kg ai/ha	DALA	Flutriafol	T	TA	TAA
GAP (United States)		4	7	4 × 0.128	14				
Chula, GA, United States 2010 (summer)	49 43 35 28 21 14	6	6 8 7 7 7	0.129 0.129 0.129 0.129 0.129 0.129	14	<0.01	<0.01	0.47 c 0.24	0.042 c 0.010
Chula, GA, United States 2010 (summer steward)	49 42 35 28 21 14	6	7 7 7 7 7	0.129 0.130 0.128 0.130 0.129 0.129	14	<0.01	<0.01	0.40 c 0.31	0.049 c 0.012
Bertrand, MO, United States 2010 (pawnee)	54 41 34 28 21 14	6	13 7 6 7 7	0.126 0.127 0.128 0.127 0.127 0.127	12	<0.01	<0.01	0.02 c 0.017	<0.01
D'Hanis, TX, United States 2010 (Cheyenne)	49 43 35 28 21 14	6	6 8 7 7 7	0.129 0.126 0.128 0.128 0.127 0.127	14	0.011	<0.01	0.017	<0.01
Anton, TX, United States 2010 (hockley)	50 43 36 30 22 14	6	7 7 6 8 8	0.132 0.127 0.126 0.126 0.131 0.128	11	<0.01	<0.01	<0.01	<0.01

Notes:

[a] Days before harvest.

[b] Residues in untreated samples are denoted by (c XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

Hops (Carringer (2014), TCI-13-365)

Four field trials on hops were conducted in the United States during the 2013 growing season. The treated plots received four foliar airblast applications of the SC formulation (125 g ai/L), normally at 0.128 kg ai/ha with intervals of 9–11 days. At all test sites, one untreated control and duplicate treated green hops cone samples were harvested at maturity 7 DALA. The green hops cones were collected and dried at 32–53 ºC for ~3–8.5 hours to a moisture content of ~8–10 percent prior to the collection of the dried cone samples. The samples were placed in frozen storage within 0.5 hours after collection.

The analytical method RAM 219/04 was used for analysis of flutriafol residues on hops, dried cone samples with HPLC-MS/MS. The LOQ was 0.01 mg/kg for flutriafol.

Triazole metabolite residues in/on hops, dried cones, were also determined using HPLC-MS/MS. The analytical method was Method Meth-160. The LOQs were 0.01 mg/kg for T and TAA triazole metabolites and 0.05 mg/kg for TA.

Both analytical methods were validated on hops dried cones prior to sample analysis. Additionally, concurrent procedural recovery samples were analysed in conjunction with each analytical set for quality control purposes. These samples were extracted and analysed according to the same procedure as the study samples. Procedural recoveries for flutriafol, T, TA and TAA in hops ranged from 89–132 percent, 77–101 percent, 69–91 percent and 107–121 percent, respectively.

The results were shown in Table 17. No residues of T were found above the LOQ in any of the samples analysed.

Table 17 Residues of flutriafol and its metabolites in hops (dried cones) after foliar application of flutriafol of 128 g ai/ha SC

Barley country, year (variety)	Timing (BBCH)	No.	RTI	kg ai/ha	DALA	Residue (mg/kg)[a]		
						Flutriafol	TA	TAA
GAP, United States		*4*	*14*	4 × 0.128	*7*			
Ephrata/ WA[bd] United States, 2013 (Cascade)	73 75 82 85	4	 10 10 10	0.128 0.128 0.129 0.128	0 7 14 21 28	16 8.0 7.4 5.6 5.2	0.14 0.12 0.067 0.08 0.068	0.021 0.018 0.018 0.018 0.020
Ephrata/ WA[cd] United States, 2013 (Cascade)	64 71 81 87	4	 10 11 9	0.128 0.128 0.127 0.129	7	4.1	0.11	<0.01
Hillsboro/ OR United States, 2013 (Glacier)	68-71 75 78-80 82-84	4	 11 10 9	0.129 0.127 0.128 0.129	7	7.3	0.16 c 0.16	0.048 c 0.043
Woodbum/ OR United States, 2013 (Nugget)	71-73 75 77-80 80-84	4	 11 10 10	0.128 0.127 0.128 0.126	7	4.6	0.092	0.024

Notes:

[1)] 6119 Dodson Road; application date: 12-Aug, 22-Aug, 1-Sep, 11-Sep.

[2)] 3975 Dodson Road N; application date: 15-Jul, 25-Jul, 5-Aug, 14-Aug.

Sampling to extraction interval: 125–230 days.

[a/] Residues in untreated samples are denoted by (c XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

Animal feed commodities

Barley, straw and hay (Carringer (2015), TCI-14-393)

The Meeting received 12 supervised trials conducted in 2014 on barley in United States. In these trials, barley received two foliar applications of flutriafol (125 g/L SC) at 0.127–0.130 kg ai/ha with intervals of 6–8 days. In 11 trials, plants were harvested at 28–37 days after the last application (DALA) for straw and 14–16 DALA for hay. One trial was a decline study (harvested at 17–45 DALA for straw and 1–28 DALA for hay). Samples as received were analysed.

The residues of flutriafol and its metabolites in barley (straw and hay) were analysed by Method #1 and Method #2, respectively. The LOQs were 0.01 mg/kg for flutriafol and T, 0.03 mg/kg for TA, and 0.04 mg/kg for TAA. Samples were stored at ≤-20°C for ≤329 days before analysis. Procedural recoveries in barley (hay, grain and straw) of flutriafol, T, TA and TAA were 69–102 percent, 79–107 percent, 87–113 percent and 98–119 percent, respectively.

In some trials, TA and/or TAA were detected in the control sample. As TA and TAA were considered natural origin, the increase of TA and TAA from the control should be attributed to flutriafol treatment. In the table, analytical values for control sample were shown if they were higher than LOQ. The results (as received) are shown in Table 18 (straw) and Table 19 (hay).

Table 18 Residues of flutriafol and its metabolites in barley straw after foliar application of flutriafol 125 g/L SC (as received)

Location, year (variety)					Residue (mg/kg) [a]			
	No Interval	RTI (day)	kg ai/ha	DALA	Flutriafol	T	TA	TAA
GAP (United States)	2	7	2 × 0.128	30				
Baptistown, NJ United States 2014 (AC Minoa)	2	7	0.129 0.128	28	0.49	<0.01	0.10 c 0.012	0.057 c 0.011
York, NE, United States 2014 (Haybet)	2	7	0.128 0.128	17 24 31 38 45	1.4 1.3 1.0 1.2 0.63	<0.01 <0.01 <0.01 <0.01 <0.01	0.035 0.049 0.069 0.061 0.051	0.015 0.020 0.035 c 0.011 0.039 0.023
Geneva, MN, United States 2014 (Rasmusson)	2	8	0.129 0.128	30	3.4	<0.01	<0.01	0.017
Richland, IA, United States 2014 (Robust)	2	7	0.128 0.128	30	0.57	<0.01	0.013	0.014 c 0.011
Grand Island, NE, United	2	7	0.129 0.128	30	0.28	<0.01	0.030	0.024 c 0.010

Location, year (variety)					Residue (mg/kg) [a]			
	No Interval	RTI (day)	kg ai/ha	DALA	Flutriafol	T	TA	TAA
States 2014 (Haybet)								
Jamestown, ID, United States 2014 (Tradition)	2	6	0.129 0.130	31	0.98	<0.01	<0.01	<0.01
Velva, ND, United States 2014 (Tradition)	2	7	0.127 0.127	29	0.64	<0.01	0.045 c 0.011	0.021 c 0.018
Carrington, ND, United States 2014 (Rasmusson)	2	8	0.126 0.130	29	1.8	<0.01	0.368 c 0.035	0.048 c 0.036
Jerome, ID, United States 2014 (Moravian 69)	2	7	0.128 0.128	37	0.32	<0.01	<0.01	<0.01
Porterville, CA, United States 2014 (Lockwood Chowford Beardless)	2	7	0.127 0.127	29	1.2	<0.01	0.01	<0.01
Payette, ID, United States 2014 (Millenium)	2	7	0.132 0.129	29	5.9	<0.01	0.02	0.024 c 0.017
Ephrata, WA, United States 2014 (Champion)	2	7	0.129 0.128	30	1.0	<0.01	0.039	0.018

Notes:

[a] Residues in untreated samples are denoted by (c=XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg for all analytes). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

Table 19 Residues of flutriafol and its metabolites in barley hay after foliar application of flutriafol 125 g/L SC (as received)

Location, year (variety)					Residue (mg/kg)[a]			
	No	RTI (day)	kg ai/ha	DALA	Flutriafol	T	TA	TAA
GAP (United States)	2	7	2 × 0.128	14				
Baptistown, NJ United States 2014 (AC Minoa)	2	7	0.128 0.129	15	0.93	<0.01	0.05	<0.04
York, NE, United States	2	7	0.128	1	9.8	<0.01	0.055	<0.04
2014			0.128	8	2.7	<0.01	0.096	<0.04

Location, year (variety)					Residue (mg/kg)[a]			
	No	RTI (day)	kg ai/ha	DALA	Flutriafol	T	TA	TAA
(Haybet)				14	1.7	<0.01	0.14	0.042
				21			c 0.031	
				28	1.1	<0.01	0.12	0.041
					0.87	<0.01	0.13	0.041
Geneva, MN, United States	2	7	0.126	16	0.25	<0.01	0.21	<0.04
2014			0.129				c 0.030	
(Rasmusson)								
Richland, IA, United States	2	6	0.128	15	0.39	<0.01	0.26	0.044
2014			0.128				c 0.079	
(Robust)								
Grand Island, NE, United States	2	7	0.129	14	0.54	<0.01	0.094	<0.04
2014			0.128					
(Haybet)								
Jamestown, ID, United States	2	8	0.132	14	3.2	<0.01	0.078	<0.04
2014			0.13					
(Tradition)								
Velva, ND, United States	2	8	0.127	15	0.32	<0.01	0.2	0.042
2014			0.127				c 0.079	
(Tradition)								
Carrington, ND, United States	2	7	0.13	14	1.1	<0.01	0.24	0.083
2014			0.129				c 0.14	c 0.053
(Rasmusson)								
Jerome, ID, United States	2	9	0.129	14	1.7	<0.01	0.037	<0.04
2014			0.128					
(Moravian 69)								
Porterville, CA, United States	2	7	0.126	14	1.9	<0.01	0.053	<0.04
2014			0.126					
(Lockwood Chowford Beardless)								
Payette, ID, United States	2	8	0.132	14	4.9	<0.01	0.088	0.043
2014			0.131				c 0.057	
(Millenium)								
Ephrata, WA, United States	2	7	0.128	15	0.92	<0.01	0.11	<0.04
2014			0.129					
(Champion)								

Notes:

[a] Residues in untreated samples are denoted by (c=XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg for T, 0.03 mg/kg for TA, and 0.04 mg/kg for TAA). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples

Sweet corn, forage and stover (Carringer (2015), TCI-14-392)

The Meeting received 16 supervised trials conducted in 2014 on sweet corn in United States. In 12 trials, sweet corn received an in-furrow application of flutriafol (125 g/L SC) at 0.289–0.305 kg ai/ha when planting and two foliar applications of flutriafol (125 g/L SC) at 0.126–0.131 kg ai/ha with an interval of 6-8 days. In four trials, sweet corn received two foliar applications of flutriafol (125 g/L SC) at 0.128–0.136 kg ai/ha with an interval of 7–8 days. Corn-on-the-cob and forage were harvested 0 DALA (on the day of the last application) and stover was harvested 6–7 DALA. Two trials were decline studies (0–14 DALA for corn-on-the-cob and forage, and 0–21 DALA for stover). Samples as received were analysed.

The residues of flutriafol and its metabolites (T, TA and TAA) in sweet corn (forage and stover) were analysed by Method #1 and Method #2, respectively. The LOQs were 0.01 mg/kg for flutriafol, T, TA and TAA. Samples were stored at ≤-20 °C for ≤ 287 days before analysis. Procedural recoveries for sweet corn (corn-on-the-cob, forage and stover) of flutriafol, T, TA and TAA ranged 71–102 percent, 84–107 percent, 71–114 percent, and 91–120 percent, respectively.

The results (as received) are shown in Table 20 (forage) and Table 21 (stover).

Table 20 Residues of flutriafol and its metabolites in sweet corn forage after foliar application of flutriafol 125 g/L SC (as received)

Location, year (variety)	Application Method/ Timing	No	RTI[d]	Kg ai/ha	DALA	Residue (mg/kg)[e]			
						Flutriafol	T	TA	TAA
GAP (United States)		2 or 1+1[a]		2x0.128	7				
Germanville, PA, United States 2014 (Mirai 421 W F1)	In-furrow[b] 8 DBH[c] 0 DBH	1+2	8	0.305 0.130 0.130	0	2.62	<0.01	0.040	0.021
Germanville, PA, United States 2014 (Mirai 421 W F1)	8 DBH 0 DBH	2	8	0.136 0.133	0	2.80	<0.01	<0.01	<0.01
Alton, NY, United States 2014 (Precious Gem)	In-furrow 7 DBH 0 DBH	1+2	7	0.295 0.128 0.129	0	2.41	<0.01	0.048 c 0.029	0.010
Seven springs, NC, United States 2014 (Sweet G90)	In-furrow 7 DBH 0 DBH	1+2	7	0.294 0.131 0.128	0	3.89	<0.01	0.099	0.036
Oviedo, FL, United States 2014 (Mirai 308 BC F1)	In-furrow 6 DBH 0 DBH	1+2	6	0.289 0.126 0.127	0	3.78	<0.01	0.018	0.010

Location, year (variety)	Application Method/ Timing	No	RTI[d]	Kg ai/ha	DALA	Residue (mg/kg)[e]			
						Flutriafol	T	TA	TAA
Conklin, MI, United States 2014 (Luscious)	In-furrow 6 DBH 0 DBH	1+2	6	0.290 0.128 0.128	0	2.79	<0.01	0.013	<0.01
Carlyle, IL, United States 2014 (Providence)	In-furrow 7 DBH 0 DBH	1+2	7	0.289 0.130 0.129	0	2.07	<0.01	0.018 c 0.031	0.011
Carlyle, IL, United States 2014 (Providence)	7 DBH 0 DBH	2	7	0.130 0.130	0	1.59	<0.01	<0.01 c 0.031	<0.01
Richland, IA, United States 2014 (Xtra-tender 2573 F1 (sh2))	In-furrow 7 DBH 0 DBH	1+2	7	0.293 0.129 0.128	0 1 7 14	3.06 1.88 1.44 0.883	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 0.010 0.015	<0.01 <0.01 <0.01 0.010
Richland, IA, United States 2014 (Xtra-tender 2573 F1 (sh2))	7 DBH 0 DBH	2	7	0.129 0.128	0 1 7 14	2.87 2.35 1.20 0.519	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01
Delavan, WI, United States 2014 (NK 199)	In-furrow 7 DBH 0 DBH	1+2	7	0.298 0.129 0.129	0	1.08	<0.01	0.012	<0.01
York, NE, United States 2014 (Obsession II)	In-furrow 7 DBH 0 DBH	1+2	7	0.291 0.129 0.127	0	3.24	<0.01	0.016	<0.01
Porterville, CA, United States 2014 (Bodacious)	In-furrow 6 DBH 0 DBH	1+2	6	0.290 0.131 0.128	0	6.44	<0.01	<0.01	<0.01
Ephrata, WA, United States 2014 (Serendipity)	In-furrow 7 DBH 0 DBH	1+2	7	0.291 0.129 0.128	0	1.64	<0.01	0.027	<0.01
Ephrata, WA, United States 2014 (Serendipity)	7 DBH 0 DBH	2	7	0.128 0.128	0	10.2	<0.01	<0.01	<0.01
Hillsboro, OR, United States 2014 (Hony 'N Pearl)	In-furrow 7 DBH 0 DBH	1+2	7	0.290 0.126 0.128	0	3.07 c 0.023	<0.01	<0.01	<0.01

Notes:

[a] 2 foliar applications with the interval of 7 days or more, or 1 foliar application + 1 application at planting (T-band application or in-furrow application).

[b] In-furrow application at planting.

[c] DBH – Days before harvest of sweet corn forage/corn-on-the-cob.

[d] RTI between 2 foliar applications.

[e] Residues in untreated samples are denoted by (c=XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

Table 21 Residues of flutriafol and its metabolites in sweet corn stover after foliar application of flutriafol 125 g/L SC (as received)

Location, year (variety)	Application					Residue (mg/kg) [e]			
	Method/ Timing	No	RTI [d]	kg ai/ha	DALA	Flutriafol	T	TA	TAA
GAP (United States)		2 or 1+1 [a]		2x0.128	7				
Germanville, PA, United States 2014 (Mirai 421 W F1)	In-furrow [b] 8 DBH [c] 0 DBH	1+2	8	0.305 0.130 0.130	6	2.41	<0.01	0.049	0.055
Germanville, PA, United States 2014 (Mirai 421 W F1)	8 DBH 0 DBH	2	8	0.136 0.133	6	2.11	<0.01	0.014	<0.01
Alton, NY, United States 2014 (Precious Gem)	In-furrow 7 DBH 0 DBH	1+2	7	0.295 0.128 0.129	7	1.71	<0.01	0.056 c 0.052	0.016
Seven springs, NC, United States 2014 (Sweet G90)	In-furrow 7 DBH 0 DBH	1+2	7	0.294 0.131 0.128	7	1.87	<0.01	0.110	0.052
Oviedo, FL, United States 2014 (Mirai 308 BC F1)	In-furrow 6 DBH 0 DBH	1+2	6	0.289 0.126 0.127	7	2.14	<0.01	0.061	0.024
Conklin, MI, United States 2014 (Luscious)	In-furrow 6 DBH 0 DBH	1+2	6	0.290 0.128 0.128	7	1.38	<0.01	0.013	<0.01
Carlyle, IL, United States 2014 (Providence)	In-furrow 7 DBH 0 DBH	1+2	7	0.289 0.130 0.129	7	1.25	<0.01	0.032	0.023
Carlyle, IL, United States 2014 (Providence)	7 DBH 0 DBH	2	7	0.130 0.130	7	0.834	<0.01	0.014	<0.01
Richland, IA, United States	In-furrow 7 DBH	1+2	7	0.293 0.129	0 1	6.52 5.15	<0.01 <0.01	0.025 0.023	0.011 <0.01

Location, year (variety)	Application Method/ Timing	No	RTI[d]	kg ai/ha	DALA	Residue (mg/kg)[e] Flutriafol	T	TA	TAA
2014 (Xtra-tender 2573 F1 (sh2))	0 DBH			0.128	7 14 21	3.70 2.01 1.76	<0.01 <0.01 <0.01	0.024 0.033 0.026	0.020 0.014 0.017
Richland, IA, United States 2014 (Xtra-tender 2573 F1 (sh2))	7 DBH 0 DBH	2	7	0.129 0.128	0 1 7 14 21	7.70 4.97 <u>4.14</u> 1.72 1.30	<0.01 <0.01 <0.01 <0.01 <0.01	0.012 0.015 0.026 0.022 0.022	<0.01 <0.01 0.010 0.010 <0.01
Delavan, WI, United States 2014 (NK 199)	In-furrow 7 DBH 0 DBH	1+2	7	0.298 0.129 0.129	7	0.418	<0.01	<0.01	<0.01
York, NE, United States 2014 (Obsession II)	In-furrow 7 DBH 0 DBH	1+2	7	0.291 0.129 0.127	7	5.17	<0.01	0.010	<0.01
Porterville, CA, United States 2014 (Bodacious)	In-furrow 6 DBH 0 DBH	1+2	6	0.290 0.131 0.128	7	5.17	<0.01	0.039 c 0.012	<0.01
Ephrata, WA, United States 2014 (Serendipity)	In-furrow 7 DBH 0 DBH	1+2	7	0.291 0.129 0.128	7	1.17	<0.01	0.037	0.012
Ephrata, WA, United States 2014 (Serendipity)	7 DBH 0 DBH	2	7	0.128 0.128	7	<u>0.499</u>	<0.01	0.011	<0.01
Hillsboro, OR, United States 2014 (Hony 'N Pearl)	In-furrow 7 DBH 0 DBH	1+2	7	0.290 0.126 0.128	7	0.683	<0.01	0.013 c 0.013	<0.01

Notes:

[a] 2 foliar applications with the interval of 7 days or more, or 1 foliar application + 1 application at planting (T-band application or in-furrow application).

[b] In-furrow application at planting.

[c] DBH – Days before harvest of sweet corn forage/corn-on-the-cob.

[d] RTI between 2 foliar applications.

[e] Residues in untreated samples are denoted by (c=XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

<u>Rice, straw and whole plant</u> (Chadwick (2018), S17-02483 and Chadwick (2019), S18-04372)

The Meeting received 12 supervised trials conducted in 2017–2018 on rice in Bulgaria, France, Italy, Portugal and Spain. In these trials, rice received one foliar application of flutriafol (250 g/L SC) at 0.119–0.134 kg ai/ha. Straw (12 trials) was harvested at 27–28 days after treatment (DAT). In addition, for

decline study, whole plants were collected and analysed at 0–21 DAT in six trials. Samples as received were analysed.

The residues of flutriafol and its metabolites (T, TA, TAA and TLA) in rice (whole plant, straw and grains with husk) were analysed by Method #1 and Method #3, respectively. LOQs were 0.01 mg/kg. Samples were stored at ≤-20 °C for ≤ 88 days before analysis. Procedural recoveries for rice (whole plant, straw and grains with husk) of flutriafol, T, TA, TAA and TLA ranged 82–112 percent, 81–120 percent, 72–120 percent, 70–119 percent, and 70–120 percent, respectively.

The results (as received) are shown in Table 22 (straw) and Table 23 (whole plant).

Table 22 Residues of flutriafol and its metabolites in rice straw after foliar application of flutriafol 250 g/L SC (as received)

Location, year (Variety)	Application		Residue (mg/kg) [b]				
	kg ai/ha	DALA	Flutriafol	T	TA	TAA	TLA
GAP (Italy)	0.125	28					
Santa Anastasia, Spain 2017 (Guadiamar)	0.128	28	2.4	<0.01	<0.01	0.01 c 0.01	0.01 c 0.01
Alcolea de Cinca, Spain 2017 (Guadiamar)	0.129	28	4.0 c 0.03	<0.01	0.10 c 0.02	0.32 c 0.24	0.10 c 0.09
Isla Mayor, Spain 2017 (J-Sendra)	0.119	28	1.4	<0.01	<0.01	0.06 c 0.05	0.04 c 0.02
Pegola di Malalbergo, Italy 2017 (Ducato)	0.129	28	0.45	<0.01	0.06 c 0.08	0.20 c 0.18	0.23 c 0.22
Arles, France 2017 (Gajeron)	0.122	28	1.9	<0.01	<0.01	0.05 c 0.06	0.04 c 0.03
Casale Monferrato, Italy 2017 (Sagitanio)	0.134	28	1.1	<0.01	0.02	0.03	0.01
Alfarelos, Portugal 2017 (Ariete)	0.125	28	1.9	<0.01	<0.01	<0.01	<0.01
Kostievo, Bulgaria 2017 (Lince)	0.132	28	1.3	<0.01	<0.01	0.08 c 0.05	0.17 c 0.06
Arle, France 2018 (Gageron)	0.129	28	2.1	<0.01	<0.01	0.04 c 0.06	0.02 c 0.03
Plovdiv, Bulgaria	0.130	28	0.92	<0.01	<0.01	0.08 c 0.09	0.11 c 0.10

Location, year (Variety)	Application		Residue (mg/kg) [b]				
	kg ai/ha	DALA	Flutriafol	T	TA	TAA	TLA
2018 (Lince)							
Malalbergo, Italy 2018 (Volano)	0.133	28	1.1	<0.01	0.02	0.26 c 0.15	0.18 c 0.15
Isla Mayor, Spain 2018 (J-Sendra)	0.119	28	0.76	<0.01	<0.01	0.03 c 0.04	0.02 c 0.02

Notes:

[a] Days before harvest.

[b] Residues in untreated samples are denoted by (c=XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

Table 23 Residues of flutriafol and its metabolites in rice (whole plant) after foliar application of flutriafol 250 g/L SC (as received)

Location, year (Variety)	Application		Residue (mg/kg) [a]				
	kg ai/ha	DALA	Flutriafol	T	TA	TAA	TLA
GAP (Italy)	0.125	28					
Santa Anastasia, Spain 2017 (Guadiamar)	0.128	0 7 14 21	3.3 2.6 1.3 1.1	<0.01 <0.01 <0.01 <0.01	0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01
Alcolea de Cinca, Spain 2017 (Guadiamar)	0.129	0 7 14 21	3.7 3.3 2.4 2.1	<0.01 <0.01 <0.01 <0.01	0.08 c 0.07 0.08 0.08 0.06	0.18 c 0.17 0.20 0.19 0.17	0.09 c 0.08 0.10 0.07 0.07
Isla Mayor, Spain 2017 (J-Sendra)	0.119	0 7 14 21	2.0 1.6 1.5 1.2	<0.01 <0.01 <0.01 <0.01	0.01 c 0.02 0.02 0.01 0.02	0.05 c 0.05 0.05 0.05 0.05	0.03 c 0.13 0.03 0.02 0.02
Pegola di Malalbergo, Italy 2017 (Ducato)	0.129	0 7 14 21	3.7 1.2 0.65 0.66	<0.01 <0.01 <0.01 <0.01	0.11 c 0.09 0.14 0.09 0.10	0.21 c 0.18 0.24 0.22 0.26	0.14 c 0.13 0.11 0.13 0.14
Malalbergo, Italy 2018 (Volano)	0.133	0 8 14 22	3.0 0.96 1.0 0.75	<0.01 <0.01 <0.01 <0.01	0.04 c 0.04 0.06 0.04 0.04	0.09 c 0.05 0.08 0.10 0.14	0.12 c 0.07 0.09 0.11 0.10
Isla Mayor, Spain 2018 (J-Sendra)	0.119	0 8 13	1.4 0.89 1.5	<0.01 <0.01 <0.01	0.01 c 0.01 0.01 0.02	0.03 c 0.02 0.03 0.03	0.02 c 0.02 0.02 0.02

Location, year (Variety)	Application		Residue (mg/kg) [a]				
	kg ai/ha	DALA	Flutriafol	T	TA	TAA	TLA
		20	0.93	<0.01	0.01	0.03	0.01

Notes:

[a] Residues in untreated samples are denoted by (c=XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

Almond hulls (Rice (2011), 65573)

The Meeting received five supervised trials conducted in 2010 on almond in United States. In these trials, almond received six foliar applications of flutriafol (125 g/L SC) at 0.127–0.134 kg ai/ha with intervals of 6–8 days. Almonds were harvested at 14 DALA. One trial was a decline study in which samples were harvested at 1–28 DALA. Samples as received were analysed.

The residues of flutriafol and its metabolites (T, TA and TAA) were analysed by Method #1 and Method #2, respectively. The LOQs were 0.01 mg/kg for flutriafol, T and TAA and 0.15 mg/kg for TA. Samples were stored at ≤-20°C for ≤230 days (almond nutmeat) or ≤92 days (almond hulls) before analysis. Procedural recoveries for almond (nutmeat and hulls) of flutriafol, T, TA and TAA ranged 77–112 percent, 73–111 percent, 73–113 percent and 96–119 percent, respectively.

The results (as received) are shown in Table 24.

Table 24 Residues of flutriafol and its metabolites in almond hulls after foliar application of flutriafol 125 g/L SC (as received)

Location Year (Variety)					Residue [a]			
	Timing [a]	No	kg ai/ha	DALA	Flutriafol	T	TA	TAA
GAP (United States)		6	6 × 0.128	14				
Dinuba, CA, United States 2010 (Sonora)	54 46 38 30 22 14	6	0.128 0.129 0.128 0.129 0.128 0.128	14	2.0	<0.01	0.017 c 0.019	<0.01
Strathmore, CA, United States 2010 (fritz)	48 42 35 28 21 14	6	0.128 0.128 0.129 0.128 0.128 0.128	14	6.7	<0.01	0.10 c 0.165	0.020 c 0.041
Wasco, CA, United States 2010 (price)	50 42 36 29 22 14	6	0.128 0.128 0.128 0.128 0.128 0.128	14	1.8	<0.01	0.017 c 0.016	<0.01
Buttonwillow, CA, United States 2010 (Monterey)	49 42 35 28 21	6	0.128 0.127 0.134 0.128 0.128	14	4.0	<0.01	0.052 c 0.025	0.017 c 0.015

	14		0.128					
Terra Bella, CA 2010 (non-pareil)	41 32 25 16 9 1	6	0.127 0.128 0.127 0.128 0.129 0.128	1 7 14 21 28	2.6 1.1 1.1 1.3 0.76	<0.01 <0.01 <0.01 <0.01 <0.01	0.050 0.044 0.046 c 0.11 0.052 0.036	<0.01 <0.01 <0.01 c 0.017 <0.01 <0.01

Notes:

[a] Residues in untreated samples are denoted by (c=XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples.

FATE OF RESIDUES IN STORAGE AND PROCESSING

Barley (Carringer (2015), TCI-14-393)

One field trial applied flutriafol as a foliar spray at an exaggerated (5 × GAP) rate of 2 × 0.640 kg ai/ha with an application interval of 7 days. Barley grain was harvested at 30 DALA.

The barley grain was processed as follows (Figure 1). Cleaned barley was hulled and resulted in blocked barley and husks. For production of pearled barley, blocked barley was processed in an abrasive testing mill. After milling, the material was separated with a 24-mesh sieve. Material on top of the sieve was pearled barley.

Conditioned blocked barley was fed through the break side of a Chopin mill. Breaking of the barley was accomplished by three break rolls. After passing through the break rolls, the material was fed onto the break sifter screens (140 and 800 micron). Material exiting the break rolls passed over the number 120 screen first. Material passing through the 120-screen was break flour. Material not passing through was conveyed over the number 25 screen. Material passing through the 25-screen was middlings. Material not passing through was conveyed to the end of the sifter. Material exiting the end was coarse bran.

Middlings were then fed into the reduction side of the Chopin mill. Reduction was achieved through two reduction rolls. After passing through the reduction rolls, the material was passed over a 160-micron screen. Material passing through the screen was reduction flour. Material remaining on top of the screen was shorts. Shorts were passed through the reduction roll two additional times. Break and reduction flours were combined and mixed for 13-17 minutes, resulting barley flour.

The coarse bran was conveyed by beater bars over a number-140 (128 micron) screen. Material passing through the screen was shorts and was added to shorts from the reduction mill. Material passing over the screen and exiting the end was bran.

Pearled barley, barley flour and bran were placed into frozen storage (≤-12 °C) for ≤ 64 days before analysis.

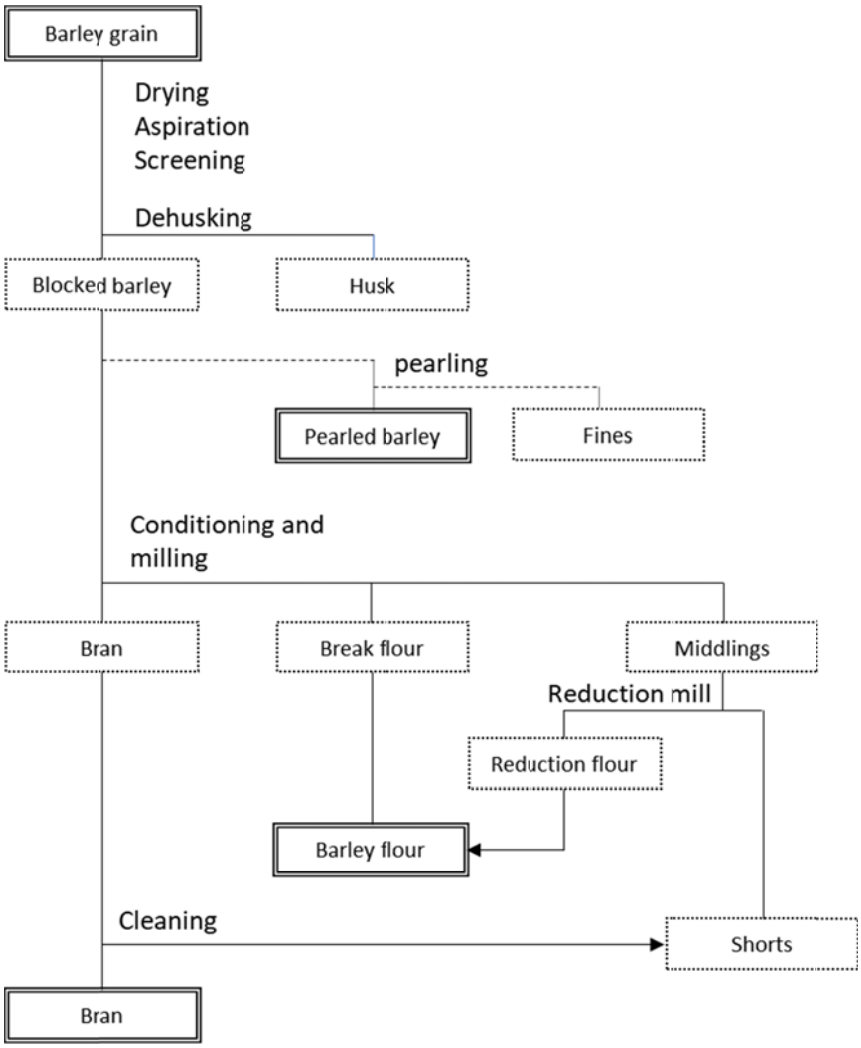


Figure 1 Barley processing to pearled barley, barley flour and barley bran

Residues of flutriafol and metabolites (T, TA and TAA) in barley and its processed commodities were analysed with Method #1 and Method #2, respectively. LOQs were 0.01 mg/kg for flutriafol and T; 0.06 mg/kg and 0.07 mg/kg for TA in barley grain and barley flour, respectively; and 0.05 mg/kg and 0.02 mg/kg for TAA in barley grain and barley flour, respectively. Concurrent recoveries of flutriafol at fortification levels of 0.01–4.0 mg/kg in barley grain, pearled barley, bran and flour were 79–120 percent with RSD≤16 percent. Concurrent recoveries of T (fortification levels of 0.01–0.8 mg/kg), TA (fortification levels of 0.06 and 0.8 mg/kg in grain and pearled barley; 0.09 and 0.8 mg/kg for bran; and 0.07 and 0.8 mg/kg for flour), and TAA (fortification levels of 0.05 and 0.8 mg/kg for grain and pearled barley; 0.03 and 0.8 mg/kg for bran; and 0.02 and 0.8 mg/kg for flour) were 88–107 percent, 87–110 percent and 92–115 percent, respectively.

Residue levels of T and TAA in all matrices were <LOQ. The processing factors for flutriafol were 0.48–0.92 (Table 25).

Table 25 Residues of flutriafol in barley (RAC and processed commodities) after foliar application at exaggerated rate

Location, year (variety)	Application No	RTI (days)	kg ai/ha	DALA	Products	Residue (mg/kg)[a]			
						Flutriafol (processing factor)	T	TA	TAA
Ephrata, WA, United States 2014 (Champion)	2	7	0.639 0.640	30	Barley grain (RAC)	2.1	<0.01	0.55 c 0.20	<0.05
					Pearled barley	1.1 (PF 0.52)	<0.01	0.22	<0.02
					Bran	1.9 (PF 0.92)	<0.01	0.39	0.035
					Flour	1.0 (PF 0.48)	<0.01	0.29	<0.02

Notes:

[a] Residues in untreated samples are denoted by (c XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg for all analytes except TA in bran (0.09 mg/kg) and flour (0.07 mg/kg) and TAA in flour (0.02 mg/kg)). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples. Processing factors are denoted as (PF XXX).

Rice (Chadwick (2018), S17-02483 and Chadwick (2019), S18-04372)

Twelve field trials applied as a foliar spray of flutriafol (125 g/L SC) at a rate of 0.119-0.134 kg ai/ha were conducted in Bulgaria, France, Italy, Spain and Portugal in 2017 and 2018. Rice was harvested at 27-28 DAT.

Processing procedure is shown in Figure 2. Rice grain (with husk) harvested were stored for 0–4 days at 7 °C, and dried if the moisture content was >15 percent. Using a rice mill, rice husks were removed from the cleaned rice grain by rubber rolls rotating in opposite directions at different speeds. Husks were then separated from the remaining husked rice by aspiration. Husked rice was processed through a decorticator to obtain an abrasion of between 25.0–29.0 percent and the specimens polished rice and bran were sampled. Husked rice, husks, polished rice and bran were placed into frozen storage (≤-18 °C) for ≤ 30 days before analysis.

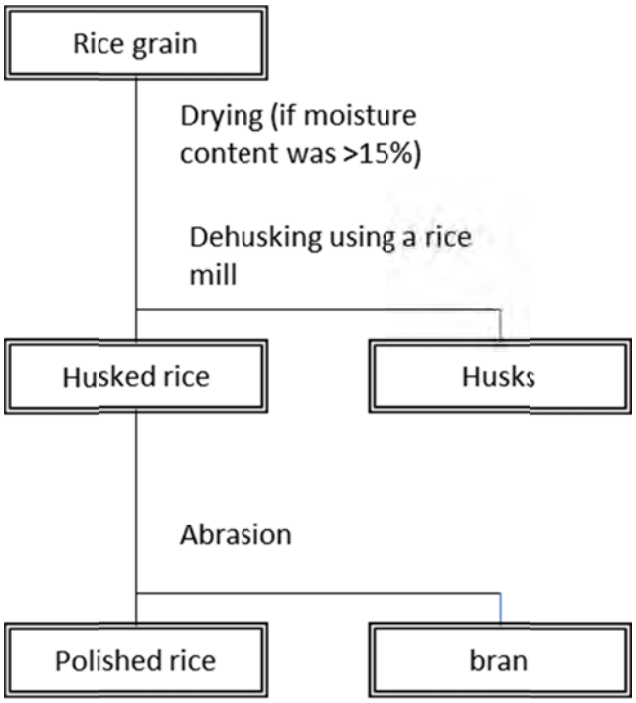


Figure 2 Rice grain (with husk) processing to husked rice and polished rice

Residues of flutriafol and its metabolites (T, TA, TAA and TLA) in rice and its processed commodities were analysed with Method #1 and Method #3, respectively. LOQs were 0.01 mg/kg for all analytes and all matrices. Procedural recoveries for rice (husked rice, polished rice, husks and bran) of flutriafol, T, TA, TAA and TLA ranged 99–119 percent, 71–118 percent, 72–139 percent, 71–116 percent and 71–113 percent, respectively.

The processing factors of flutriafol and its metabolites in rice grain processing were shown in Table 27.

Table 26 Residues of flutriafol in rice and its processed commodities after foliar application of flutriafol 125 g/L SC

Location, year (Variety)			Matrix	Residue (mg/kg) [a]				
	kg ai/ha	DALA		Flutriafol	T	TA	TAA	TLA
GAP (Italy)	0.125	28						
Arles, France 2017 (Gajeron)	0.122	28	Rice grain (with husk)	1.6	<0.01	0.07 c 0.06	0.04 c 0.03	<0.01
			Polished rice	0.26 (PF 0.16)	<0.01 (PF <0.062)	0.05 c 0.022	0.03 c 0.02	<0.01
			Bran	0.71 (c 0.03) (PF 0.44)	<0.01 (PF <0.0062)	<0.01 c 0.15	<0.01 c 0.06	<0.01
Casale Monferrato, Italy 2017 (Sagitanio)	0.134	28	Rice grain (with husk)	1.1	<0.01	0.02	0.01	<0.01
			Polished rice	0.42	<0.01	0.02	0.01	<0.01

Location, year (Variety)			Matrix	Residue (mg/kg) [a]				
	kg ai/ha	DALA		Flutriafol	T	TA	TAA	TLA
				(PF 0.38)	(PF <0.0090)		c 0.01	
			Bran	4.1 (PF 3.7)	<0.01 (PF <0.0090)	0.09 c 0.03	0.05 c 0.03	<0.01
Alfarelos, Portugal 2017 (Ariete)	0.125	28	Rice grain (with husk)	0.87 (c 0.02)	<0.01	0.02 c 0.02	<0.01	<0.01
			Polished rice	0.38 (PF 0.44)	<0.01 (PF <0.011)	0.02	<0.01	<0.01
			Bran	0.58 (c 0.02) (PF 0.67)	<0.01 (PF <0.011)	0.06 c 0.02	0.03 c 0.01	<0.01
Kostievo, Bulgaria 2017 (Lince)	0.132	28	Rice grain (with husk)	1.4	<0.01	0.16 c 0.06	0.08 c 0.03	<0.01
			Polished rice	0.47 (PF 0.34)	<0.01 (PF <0.0067)	0.12 c 0.05	0.06 c 0.03	<0.01
			Bran	0.80 (PF 0.57)	<0.01 (PF <0.0067)	0.41 c 0.17	0.14 c 0.08	<0.01
Arle, France 2018 (Gageron)	0.129	28	Rice grain (with husk)	1.1	<0.01	0.06 c 0.06	0.05 c 0.05	<0.01
			Husked rice	0.39 (PF 0.35)	<0.01 (PF <0.0090)	0.08 c 0.08	0.07 c 0.07	<0.01
			husks	8.7 (PF 7.9)	<0.01 (PF <0.0090)	0.02 c 0.01	0.07 c 0.07	<0.01
Plovdiv, Bulgaria 2018 (Lince)	0.130	28	Rice grain (with husk)	1.1	<0.01	0.15 c 0.06	0.07 c 0.04	<0.01
			Husked rice	0.44 (PF 0.40)	<0.01 (PF <0.0083)	0.18 c 0.06	0.09 c 0.04	<0.01
			husks	5.9 (PF 5.4)	<0.01 (PF <0.0083)	0.03 c 0.02	0.06 c 0.04	<0.01
Malalbergo, Italy 2018 (Volano)	0.133	28	Rice grain (with husk)	0.57	<0.01	0.07 c 0.08	0.09 c 0.11	<0.01
			Husked rice	0.29 (PF 0.53)	<0.01 (PF <0.017)	0.05	0.10 c 0.14	<0.01
			husks	3.7 (PF 6.5)	<0.01 (PF <0.017)	0.01	0.06 c 0.07	<0.01
Isla Mayor,	0.119	28	Rice grain	0.82	<0.01	0.03	0.03	0.02

Location, year (Variety)			Matrix	Residue (mg/kg) [a]				
	kg ai/ha	DALA		Flutriafol	T	TA	TAA	TLA
Spain 2018 (J-Sendra)			(with husk)			c 0.01	c 0.02	
			Husked rice	0.49 (PF 0.61)	<0.01 (PF <0.012)	0.03 c 0.02	0.03 c 0.03	<0.01
			husks	4.8 (PF 5.9)	<0.01 (PF <0.012)	<0.01	0.02 c 0.01	<0.01
Santa Anastasia, Spain 2017 (Guadiamar)	0..128	28	Husked rice	0.13	<0.01	0.03	<0.01	<0.01
			Husks	3.5	<0.01	<0.01	0.01	<0.01
Alcolea de Cinca, Spain 2017 (Guadiamar)	0.129	28	Husked rice	0.54	<0.01	0.19	0.16	<0.01
			Husks	11	<0.01	0.04	0.19	<0.01
Isla Mayor, Spain 2017 (J-Sendra)	0.119	28	Husked rice	0.34	<0.01	0.04	0.06	<0.01
			Husks	6.5	<0.01	<0.01	0.04	<0.01
Pegola di Malalbergo, Italy 2017 (Ducato)	0.129	28	Husked rice	0.14	<0.01	0.22	0.22	<0.01
			Husks	2.8	<0.01	0.02	0.06	<0.01

Notes:

[a] Residues in untreated samples are denoted by (c XXX). For trials in which no value is listed, residues were not observed >LOQ (0.01 mg/kg). Residues in treated samples were not corrected for background levels observed in corresponding untreated samples. Processing factors are denoted as (PF XXX).

Table 27 Processing factors for flutriafol in rice (summary)

Portion	Flutriafol
	Calculated processing factors[a] (Mean or best estimate)
Husked rice	0.35, 0.40, 0.53, 0.61 (0.46)
Husk	5.4, 5.9, 6.5, 7.9 (6.2)
Polished rice	0.16, 0.34, 0.38, 0.44 (0.36)
Bran	0.44, 0.57, 0.67, 3.7 (0.62)

Notes:

[a/] Each value represents a separate trial. The factor is the ratio of the residue in inedible portion divided by the residue in the edible portion.

Hops (Block (2016), S14-00843)

The Meeting received a processing study for hops which were conducted in Germany. The trials received 4 applications of flutriafol 125g/L SC at the application rate of 0.125 kg ai/ha at 45–48 days before harvest (DBH), 36–37 DBH and 21–22 DBH and at 0.625 kg ai/ha (5 × GAP) at 7 DBH. Samples of hops from the untreated and treated plots of all trials were taken at 7 days after last application and kiln-dried afterwards.

The processing was done according to brewery standards and the standard operating procedures. The processing phase simulated the processing of hops to malt, wort, young beer, brewer´s yeast and beer. Process products samples from hop were analysed for residues of flutriafol and its triazole metabolites (T, TA, TAA and TLA).

Brewing procedure was shown in Figure 3.

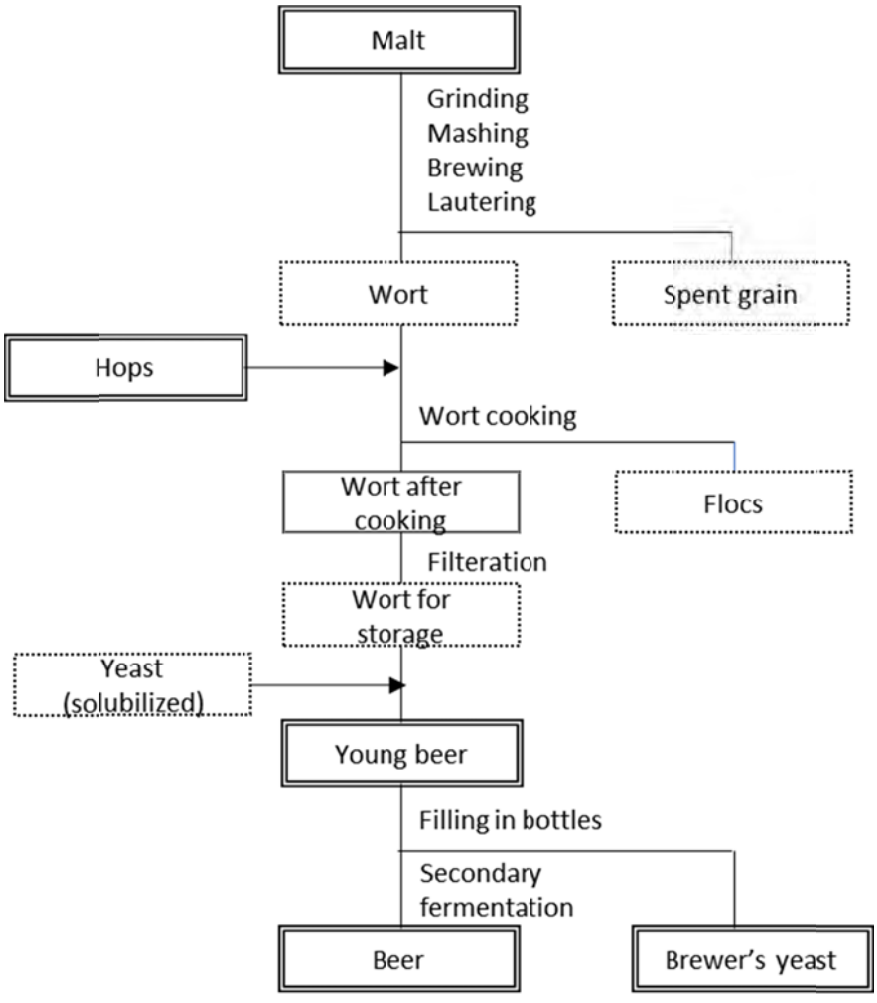


Figure 3 Hops processing to beer

The analytical method referenced AGR/MOA/FLUTRI-1 was validated for determination of flutriafol in hop processed products (hops and beer). This method was already validated for flutriafol in wheat. A confirmation for the validation was performed in brewer's yeast.

The analytical method referenced AGR/MOA/TRZ-1 was validated for determination of T, TA, TAA and TLA in hop processed products (hops and beer). This method was already validated for T, TA and TAA in wheat and for TLA in wheat and barley. The method was also confirmed for the analysis of triazole metabolites in brewer's yeast.

For the validation in hops and beers, for each compound, the analysis of two control sample, one reagent blank, five samples fortified at 0.01 mg/kg and five samples fortified at 0.1 mg/kg were performed. For the confirmation of validation on brewer's yeast and for each compound, the analysis of one control sample, three samples fortified at 0.01 mg/kg and three samples fortified at 0.1 mg/kg were performed.

The recoveries of analytes from hops were 75–87 percentfor flutriafol, 78–101 percent for T, 65–100 percentfor TA, 78–108 percent for TAA, and 77–102 percent for TLA respectively. The recoveries from beer were 88–109 percent for flutriafol, 74–114 percent for T, 81–103 percent for TA, 94–110 percent for TAA, and 76–89 percent for TLA. The LOQ was 0.01 mg/kg for all analytes.

The recoveries of analytes from brewer's yeast were 90–95 percent for flutriafol, 79–116 percent for T, 62–94 percent for TA, 73–107 percent for TAA, and 92–104 percent for TLA respectively.

The samples were stored at a target temperature of -20°C in the freezers. Samples were stored frozen for less than 245 days from frozen storage to analyses, and 109 days from ambient storage to analyses.

The results were shown in Table 28 and Table 29.

Table 28 Flutriafol and triazole metabolites residues in processed commodities of hops after foliar application at exaggerated rate

Hops country, year (variety)	Application			DALA	Commodity	Residues (mg/kg)				
	kg ai/ha	water, L/ha	no.			Flutriafol	T	TA	TAA	TLA
Bayern, Germany 2014 (Herkules)	0.12 0.12 0.13 0.59	1971 1889 2130 1871	4	7	Hops Wort after cooking Young beer Brewer's yeast Beer	5.8 0.11 (0.019) 0.03 (0.0052) 0.16 (0.028) 0.05 (0.0086)	<0.01 <0.01 0.01 <0.01 <0.01	0.12 <0.01 0.02 0.04 0.02	0.02 <0.01 0.02 0.01 0.02	0.35 <0.01 <0.01 <0.01 <0.01
	0.12 0.12 0.12 0.59	1976 1959 1882 1901	4	7	Hops Beer	3.3 0.07 (0.021)	<0.01 <0.01	0.11 0.01	0.02 0.01	0.31 <0.01
Baden-Württem berg, Germany 2014 (Hallertauer Tradition)	0.13 0.13 0.13 0.64	2151 2095 2008 2058	4	7	Hops Beer	6.2 0.02 (0.0032)	0.17 <0.01	0.06 <0.01	<0.01 <0.01	0.04 <0.01
	0.13 0.12 0.13 0.66	2068 2079 2065 2103	4	7	Hops Beer	9.3 0.03 (0.0032)	0.25 <0.01	0.09 <0.01	0.02 0.02	0.10 <0.01

Table 29 Flutriafol residues in processed commodities of hops (summary)

Commodity	Flutriafol
	Calculated processing factors[b] (Mean or best estimate)
Beer	0.0032(2),0.0086,0.021 (0.0059)
Wort after cooking	0.019
Young beer	0.005
Brewer's yeast	0.028

APPRAISAL

Flutriafol, whose IUPAC name is (*RS*)-2,4'-difluoro-alfa-(1*H*-1,2,4-triazol-1-ylmethyl)benzhydryl alcohol, is a triazole fungicide. It was first evaluated for toxicology and residues by the 2011 JMPR. The ADI of flutriafol is 0–0.01 mg/kg bw and the ARfD is 0.05 mg/kg bw. The compound was evaluated by the 2015 JMPR for additional MRLs.

The following residue definitions for flutriafol were recommended by the 2011 JMPR and confirmed by the 2015 JMPR :

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *flutriafol*

The residue is fat soluble.

At the Fifty-first Session of the CCPR (2019), flutriafol was scheduled for evaluation of the additional uses for hops by the 2020 JMPR and postponed to the current JMPR. At the Forty-third Session of CAC (2020), flutriafol was scheduled for evaluation of additional use on almond, pecan, barley, sweet corn and rice by the 2021 JMPR. The current Meeting received new information on methods of analysis, storage studies, use patterns, supervised field trials and processing studies on hops, barley, rice, sweet corn, almond and pecan.

Methods of analysis

The Meeting received information on a method of analysis for supervised field trials.

Method #1 was for analysis of flutriafol in barley, sweet corn, rice and almond. Flutriafol was extracted with acetonitrile/water (7:3, v/v) and quantified by LC-MS/MS. The method was validated for flutriafol in barley (hay, grain and flour), sweet corn (forage, corn-on-the-cob and stover), rice (whole plant, straw, grain with husk, husked rice, husks, polished rice and bran) and almond (nutmeat and hulls) with an LOQ of 0.01 mg/kg.

Stability of pesticide residues in stored analytical samples

The 2011 JMPR concluded that when stored frozen flutriafol residues were stable for at least 5 months in soya bean seed; at least 12 months in apple, barley grains and coffee beans; for at least 23 months in grapes; for at least 24 months in cabbage and oilseed rape; and for at least 25 months in wheat (grains and straw), pea seed, and sugar beet root. The periods of demonstrated storage stability for flutriafol residues cover the frozen storage intervals used in the field trials.

Results of supervised residue trials on crops

Cereal grains

Barley

The critical GAP for flutriafol on barley in the United States is two foliar applications each at 0.128 kg ai/ha with a minimum interval between sprays of 7 days and a PHI of 30 days. In trials conducted in the United States matching theUnited StatesGAP, residues of flutriafol in barley were (n=11): 0.11, 0.12, 0.15, 0.17, 0.18, 0.20, 0.23, 0.29, 0.34, 0.77 and 0.84 mg/kg.

The Meeting estimated a maximum residue level and STMR of 1.5 and 0.20 mg/kg, respectively.

Rice

The critical GAP for flutriafol on rice in Italy is one foliar application at 0.125 kg ai/ha and a PHI of 28 days. In trials conducted in Bulgaria, France, Italy, Portugal and Spain matching the Italian GAP, residues of flutriafol in rice grain(with husk; n=8) were: 0.57, 0.82, 0.87, 1.1 (3), 1.4 and 1.6 mg/kg.

The Meeting estimated a maximum residue level and STMR for rice grain of 4 and 1.1 mg/kg, respectively.

Residues of flutriafol in husked rice were (n=8): 0.13, 0.14, 0.29, 0.34, 0.39, 0.44, 0.49 and 0.54 mg/kg.

The Meeting estimated a maximum residue level and STMR for husked rice of 1 and 0.365 mg/kg, respectively.

In trials matching the Italian GAP, residues of flutriafol in polished rice were (n=4): 0.26, 0.38, 0.42 and 0.47 mg/kg. Corresponding processing factors for rice with rice grain to polished rice were 0.16-0.44 (median 0.36, See Processing section). The Meeting agreed to utilise the residue data on rice grain together with the median processing factor from rice grain to polished rice to estimate a maximum residue level and an STMR for polished rice of 1.5 [4×0.36] and 0.40 mg/kg [1.1×0.36], respectively.

Sweet corn (Corn-on-the-cob)

The critical GAP for flutriafol on sweet corn in the United States is for two foliar applications each at 0.128 kg ai/ha with a minimum interval between sprays of 7 days with a PHI of 7 days. The Meeting received 16 trials conducted in the United States. Of these trials only four matched the United States cGAP, i.e., the majority consisted of an in-furrow application followed by foliar applications.

The Meeting noted that a rotational crop study provided to the 2011 JMPR indicated that uptake from soil may be significant, and could not use 12 of the provided trials to estimate the potential contribution from the in-furrow application to the final residue.

The Meeting could therefore not estimate a maximum residue level, STMR and HR of corn-on-the-cob due to an insuffient number of trials.

Tree nuts

Almonds

The critical GAP for flutriafol on almond in the United States is six foliar applications each at 0.128 kg ai/ha with a minimum interval between sprays of 7 days and a PHI of 14 days. In trials conducted in the

United States matching the GAP, residues in flutriafol in almonds were (n=5): < 0.01, 0.012, 0.064, 0.066 and 0.41 mg/kg (highest individual value: 0.42 mg/kg).

The Meeting estimated a maximum residue level, STMR and HR for almonds of 0.8, 0.064 and 0.42 mg/kg, respectively.

Pecan

The critical GAP for flutriafol on tree nuts in the United States is four foliar applications each at 0.128 kg ai/ha with a minimum interval between sprays of 14 days and a PHI of 14 days.

Since no trials matched the GAP, the Meeting could not estimate a maximum residue level and STMR of pecans.

Dried herbs

Hops, dry

The critical GAP for hops in the United States allows four applications each at 0.128 kg ai/ha with a maximum seasonal rate of 0.51 kg ai/ha at a minimum interval of 14 days and a PHI of 7 day.

Data were available from supervised trials on hops (dried cones) in United States.

Residues in hops from independent trials in the United States with four applications of 0.13 kg ai/ha at intervals of 9–11 days at a total application rate of 0.51 kg ai/ha with a PHI of 7 days were (n=3): 4.6, 7.3 and 8.0 mg/kg.

The Meeting could not estimate maximum residue level and STMR for hops due to insufficient trial numbers.

Animal feed commodities

Barley, hay and/or straw

The critical GAP for flutriafol on barley in the United States is two foliar applications each at 0.128 kg ai/ha with a minimum interval between sprays of 7 days and a PHI of 30 days (for straw) or 14 days (for hay).

In trials conducted in the United States matching the United States GAP, residues of flutriafol in barley, straw (as received) were (n=11): 0.28, 0.49, 0.57, 0.64, 0.98, 1.0, 1.2 (2), 1.8, 3.4 and 5.9 mg/kg (highest individual value: 6.4 mg/kg).

In trials conducted in the United States matching the United States GAP, residues of flutriafol in barley, hay (as received) were (n=12): 0.25, 0.32, 0.39, 0.54, 0.92, 0.93, 1.1, 1.7 (2), 1.9, 3.2 and 4.9 mg/kg (highest individual value: 5.0 mg/kg).

Based on data for straw which lead to a higher maximum residue level than data for hay, the Meeting estimated the maximum residue level of flutriafol in barley hay and/or straw of 10 mg/kg (dw, based on 89 percent DM content).

The Meeting estimated median and highest residue of flutriafol in barley, straw of 1.0 and 6.4 mg/kg, respectively (as received), and median and highest residue in barley, hay of 1.0, 5.0 mg/kg, respectively (as received).

Rice straw

The critical GAP for flutriafol on rice in Italy is one foliar application at 0.125 kg ai/ha and a PHI of 28 days.

In trials conducted in Bulgaria, France, Italy, Portugal and Spain matching the Italian GAP, residues of flutriafol in rice straw (as received) were (n=12): 0.45, 0.76, 0.92, 1.1 (2), 1.3, 1.4, 1.9 (2), 2.1, 2.4 and 4.0 mg/kg. The Meeting estimated the maximum residue level for flutriafol in rice straw of 6 mg/kg (dw, based on 90 percent DM content) and median and highest residue of 1.4 and 4.0 mg/kg, respectively (as received).

Sweet corn, stover

The critical GAP for flutriafol on sweet corn in the United States is two foliar applications each at 0.128 kg ai/ha with a minimum interval between sprays of 7 days and a PHI of 7 days.

In trials matching critical GAP, residues of flutriafol in sweet corn stover were (n=4): 0.50, 0.83, 2.1 and 4.1 mg/kg.

The Meeting noted that rotational crop study provided to 2011 JMPR indicated that uptake from soil may be significant and could not assume the impact from in-furrow application.

The Meeting considered four trials insufficient to estimate a maximum residue level for corn stover.

Almond hulls

The critical GAP for flutriafol on almond in the United States is six foliar applications each at 0.128 kg ai/ha with a minimum interval between sprays of 7 days and a PHI of 14 days.

In trials conducted in the United States matching theUnited StatesGAP, residues of flutriafol in almond hulls (as received) were (n=5): 1.3, 1.8, 2.0, 4.0 and 6.7 mg/kg. The Meeting estimated the maximum residue level for flutriafol in almond hulls of 15 mg/kg (dw, based on 90 percent DM content) and median of 2.0 mg/kg, respectively (as received).

Fates of residues during processing

Processing

The Meeting estimated processing factors for flutriafol as follows. STMR-P and maximum residue levels for polished rice were derived from processing factors.

Table 30 Calculated STMR-Ps for processed food and feed commodities

RAC	Processed commodity	Processing factor	RAC STMR (mg/kg)	STMR-P or median (mg/kg)	Maximum residue level for RAC (mg/kg)	Maximum residue level for processed commodity (mg/kg)
Barley	Pealed barley	0.52	0.20	0.099	1.5	-
	Barley bran	0.92	0.20	0.17		-
	Barley flour	0.48	0.20	0.091		-
Rice grain with husk	Polished rice	0.36	1.1	0.40	4	1.5
	Husks	6.2	1.1	6.8 (as received)	4 (dw)	20 (dw)
	Rice bran	0.62	1.1	0.68		

				(as received)		

Residues in animal commodities

Farm animal dietary burden

The OECD diets include barley (straw and hay) and rice (straw, grain, husks and bran). Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the Meeting. The dietary burdens, estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO manual, are presented in Annex 6 and summarised below.

Table 31 Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden of flutriafol, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	Max	Mean	max	mean	max	mean	Max	mean
Beef cattle	2.3	1.4	4.8	2.1	11❶	4.6❷	2.6	0.97
Dairy cattle	4.2	2.0	5.2	2.0	11❸	4.6❹	1.2	0.52
Poultry – broiler	0.44	0.44	0.24	0.24	0.78	0.78	0.23	0.23
Poultry – layer	0.44	0.44	1.2❺	0.49	0.78	0.78 ❻	0.21	0.21

Notes:

❶ Highest maximum beef cattle dietary burden suitable for MRL estimates for mammalian tissues.

❷ Highest mean beef cattle dietary burden suitable for STMR estimates for mammalian tissues.

❸ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

❺ Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues and eggs.

❻ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Animal commodity maximum residue levels

The calculations used to estimate highest total residues for use in estimating maximum residue levels, STMR and HR values are shown below.

Table 32 Residues in milk and tissues from cattle dosed with flutriafol in the diet

Cattle	Feed Level (ppm) for milk residues	Residues (mg /kg) in milk	Feed Level (ppm) for tissue residues	Residues (mg /kg)			
				Muscle	Liver	Kidney	Fat
HR Determination (beef or dairy cattle)							
Feeding Study [a]	16	< 0.01	16	< 0.01	0.77	0.02	0.02
Dietary burden and estimate of highest residue	11	< 0.0069	11	< 0.0069	0.53	0.014	0.014
STMR determination (beef or dairy cattle)							
Feeding Study [b]	5	< 0.01	5	< 0.01	0.33	< 0.01	< 0.01
Dietary burden and estimate of highest residue	4.6	< 0.0029 [c]	4.6	< 0.0029 [c]	0.30	< 0.0092	< 0.0092

Notes:

[a] Highest residues for tissues and mean residues for milk

[b] Mean residues for tissues and mean residues for milk

[c] Calculated based on the feeding study of feed level at 16 ppm

Table 33 Residues in eggs and tissues from poultry dosed with flutriafol in the diet

Poultry	Feed Level (ppm) for egg residues	Residues (mg /kg) in egg	Feed Level (ppm) for tissue residues	Residues (mg /kg)		
				Muscle	Liver	Fat
HR Determination (poultry – broiler or layer)						
Feeding Study [a]	5	0.03	5	< 0.01	0.10	0.07
Dietary burden and estimate of highest residue	1.2	0.0072	1.2	< 0.0024	0.024	0.017
STMR determination (poultry – broiler or layer)						
Feeding Study [b]	5	0.03	5	< 0.01	0.07	0.06
Dietary burden and estimate of highest residue	0.78	0.0047	0.78	< 0.0016	0.011	0.0094

Notes:

[a] Highest residues for tissues and mean residues for egg.

[b] Mean residues for tissues and mean residues for egg.

The Meeting confirmed its decision in 2015 of maximum residue levels of 0.02 (fat) mg/kg for meat (from mammals other than marine mammals), 0.01 (*) mg/kg for milks, 0.02 mg/kg for mammalian fats (except milk fats) and 1 mg/kg for edible offal (mammalian).

The Meeting estimated maximum residue levels of 0.03 (fat) mg/kg for poultry meat and 0.03 mg/kg for poultry fats to replace previous recommendations, and confirmed its decision in 2015 of maximum residue levels of 0.03 mg/kg for poultry, edible offal of, and 0.01(*) mg/kg for eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with MRL and dietary intake for plant and animal commodities: *flutriafol*

The residue is fat soluble.

Table 34 Residue levels suitable for establishing maximum residue limits and for IEDI and IESTI assessments

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
TN0660	Almonds	0.8		0.064	0.42
GC0640	Barley	1.5		0.20	
MO0105	Edible offal, mammalian	1	1	0.30	0.53
PE0112	Eggs	0.01(*)	0.01(*)	0.0047	0.0072
MF0100	Mammalian fats (except milk fat)	0.02	0.02	0.0092	0.014
MM0095	Meat (from mammals other than marine mammals)	0.02(fat)	0.02(fat)	0.0042	0.0083
ML0106	Milks	0.01(*)	0.01(*)	0.0047	0.0066

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
TN0660	Almonds	0.8		0.064	0.42
PO0111	Poultry, edible offal of	0.03	0.03	0.011	0.024
AM0660	Almond hulls	15 (dw)		2.00 (ar)	
GC0640	Barley	1.5		0.2	
AS0640	Barley hay and/or straw	10 (dw)		Median: 1.0 (ar)	Highest: Straw: 6.4 (ar) Hay: 5.0 (ar)
MO0105	Edible offal, mammalian	1	1	0.3	0.53
PE0112	Eggs	0.01(*)	0.01(*)	0.0047	0.0072
MF0100	Mammalian fats (except milk fat)	0.02	0.02	0.0092	0.014
MM0095	Meat (from mammals other than marine mammals)	0.02(fat)	0.02(fat)	0.0042	0.0083
ML0106	Milks	0.01(*)	0.01(*)	0.0047	0.0066
PO0111	Poultry, edible offal of	0.03	0.03	0.011	0.024
PF0111	Poultry fats	0.03	0.02	0.0094	0.017
PM0110	Poultry meat	0.03(fat)	0.01(*)	0.0043	0.0048
GC0649	Rice	4 (dw)		1.1	
AS0649	Rice, hay and/or straw	6 (dw)		Median: 1.40 (ar)	Highest: 4.0 (ar)
AS 3570	Rice, hulls (husks)	20 (dw)		Median: 6.8 (ar)	
For dietary risk assessment and/or dietary burden calculations				(median)	
CM0640	Barley, pearled			0.099	
CF0640	Barley bran, processed			0.17	
CM1206	Rice bran, unprocessed			0.068 (ar)	

Notes:

(ar) As received

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The International Estimated Daily Intakes for the 17 GEMS/Food cluster diets, based on the recommendations of the current JMPR, were in the range 9–30 percent of the maximum ADI of 0.01 mg/kg bw for flutriafol. The results are shown in Annex 3 to the report.

The Meeting concluded that the long-term dietary exposure from residues of flutriafol, from uses that have been considered by the JMPR, is unlikely to present a public health concern.

Acute dietary exposure

The International Estimated Short Term Intake (IESTI) for flutriafol was calculated. The results are shown in Annex 4 to the Report.

The IESTIs for flutriafol from the intake of the residue evaluated by the Meeting were 0–30 percent for general population and 0–70 percent for children of the ARfD (0.05 mg/kg bw). The Meeting concluded that acute dietary exposure from the residues of flutriafol, from uses that have been considered by the JMPR, is unlikely to present a public health concern.

REFERENCES

Code	Author(s)	Year	Title
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82662	Rodgers, C.	2016	Evaluation of frozen stability of 1,2,4-Triazole, Triazole Alanine, and Triazole Acetic Acid residues in or on hops raw agricultural commodities. ABC Labs, Inc (now Eurofins Agroscience Services). Report Number: 82662. Sponsor study number: 2016SST FLT2973. GLP, Unpublished
TCI-13-365	Carringer, S.	2014	Magnitude and decline of flutriafol and metabolite residues in/on hops raw agricultural commodities following four foliar applications of Flutriafol 125 g/L SC (2013). The Carringers, Inc. Report Number TCI-13-365. Sponsor study number: 2696 FLU. GLP, Unpublished.
TCI-14-393	Carringer, S.	2015	Magnitude and decline of flutriafol and metabolite residues in/on barley raw agricultural commodities and processed commodities following application of flutriafol 125 g/l SC (2014). The Carringers, Inc. Report Number TCI-14-393 (CHA Doc. No. 2750 FLU). GLP unpublished
TCI-14-392	Carringer, S.	2015	Magnitude and decline of flutriafol and metabolite residues in/on sweet corn raw agricultural commodities following one in-furrow application and two foliar applications of flutriafol 125 g/l SC (2014). The Carringers, Inc. Report Number TCI-14-392 (CHA Doc. No. 2749 FLU) GLP unpublished
S14-00843	Block, H.	2016	Determination of residues of flutriafol in the processed fractions of hop after four applications of Flutriafol 125 g/L SC at two sites in Germany in 2014. Eurofins Agroscience Services. Report Number S14-00843. Sponsor study number: 2753 FLU. GLP, Unpublished.
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S18-04372	Chadwick, G.	2019	Determination of residues if flutriafol and its triazole metabolites after a single foliar application Flutriafol 125 g/L SC (CHA 1310-05) in rice at 4 sites (2 × MOR and 2 × DEC) in Southern Europe 2018. Eurofins Agroscience Services, Ltd. Report number: S18-04372 (FMC No. 2018RES-FLT4410). GLP unpublished

INDOXACARB (216)

First draft prepared by Dr Julian Cudmore, Chemicals Regulation Division of the Health and Safety Executive, United Kingdom

EXPLANATION

Indoxacarb is an indeno-oxadiazine insecticide that is used for the control of lepidopteran and other insect pests. It was first evaluated by the 2005 JMPR when an ADI of 0–0.01 mg/kg bw and an ARfD of 0.1 mg/kg bw were established. The residue definition for compliance with the MRL for plant and animal commodities and dietary risk assessment for plant commodities is the sum of indoxacarb and its R enantiomer. The residue definition for dietary risk assessment for animal commodities is sum of indoxacarb, its R enantiomer and methyl 7-chloro-2,5-dihydro-2-[[[4-(trifluoromethoxy)phenyl]amino]carbonyl]indeno[1,2-e][1,3,4]oxadiazine-4a(3H)- carboxylate (IN-JT333), expressed as indoxacarb. The residue is fat soluble.

Indoxacarb has been evaluated for additional uses in 2007, 2009 and 2013. At the Fifty-first Session of the CCPR, indoxacarb was scheduled for the evaluation by the current Meeting for additional uses on bush berries, okra, beans with pods, pulses, beetroot, maize and tree nuts. The current Meeting received information on residue analysis, storage stability, use pattern, supervised field trials and processing.

RESIDUE ANALYSIS

Analytical Methods

One new analytical method (DuPont 36189) was received by the current Meeting. All other analytical methods outlined below have been considered previously by the JMPR. However, additional validation data were received by the current Meeting.

In all the analytical methods, indoxacarb and its R enantiomer are determined and reported together; the methods are not enantioselective and the LOQ refers to the sum of indoxacarb (S isomer) and its R enantiomer.

Method AMR 4271-96

The method was considered by the 2005 JMPR. The method is based on multi-residue method DFG S19 with a modified extraction solvent. Samples are extracted with water/acetone/ethyl acetate/cyclohexane. Extracts are cleaned up by gel permeation chromatography (GPC) and by adsorption chromatography on silica gel. Analytes are determined by capillary GC-ECD on a non-polar stationary phase. The method was validated with an LOQ of 0.01 mg/kg for apple, cabbage, grape and tomato and an LOQ of 0.02 mg/kg for cotton seed.

The Meeting received additional validation data for blueberries which is summarized in Table 1.

AMR 3493-95

The method was considered by the 2005 JMPR. Analytes are extracted from crop samples into ethyl acetate after the addition of water. An aliquot from the extraction solution is concentrated by evaporation under nitrogen and cleaned up by solid phase extraction with silica and carbon. The cleaned-up extract is then analysed by GC-MSD. The method was validated with an LOQ of 0.02 mg/kg for apple.

The Meeting received additional validation data for maize grain which is summarized in Table 1.

AMR 2712 93

The method was considered by the 2005 JMPR with additional recovery data considered by the 2009 JMPR for a range of commodities. Residues are extracted from crop matrices with hexane-acetonitrile and the acetonitrile extract is concentrated and cleaned up by solid-phase extraction with a combination of silica and strong anion exchanger. The analytes are measured by reversed-phase HPLC (2-column system with switching) with UV detection at 310 nm. The method was validated with an LOQ of 0.01 mg/kg for high water crops.

The Meeting received additional validation data for snap beans, dry beans and beetroot which is summarised in Table 1.

DuPont 36189

This method was used in the residue trials for maize and tree nuts. Residues are extracted twice with hexane-acetonitrile. The acetonitrile layer is collected and a portion is evaporated to dryness under nitrogen. The residue is reconstituted in acetonitrile and 0.01 M formic acid. Final analysis is by LC-MS/MS using gradient elution on a C18 column. The ion mass transition m/z 528 → 249 was used for quantification. Validation data are summarized in Table 1.

Table 1 Method validation for the determination of indoxacarb (and its R enantiomer)

Method	Matrix	Fortification level (mg//kg)	Recoveries (%)	Mean Recovery (%)	RSD (%)
AMR 4271-96	Blueberries	0.02	79, 90, 96	89	9
		1	106, 109, 110	108	2
		10	101, 102, 105	103	2
AMR 3493-95	Maize grain	0.01	73, 77, 81, 85, 86, 87, 89, 89, 92, 98, 104	87	10
		0.1	77, 78, 82, 83, 87, 87, 88, 89, 90, 106	87	9
		0.5	73	-	-
AMR 2712 93	Snap beans – pods with seeds	0.01	96, 98, 99,104, 107, 111	103	6
		0.03	78, 89, 90	86	8
		0.1	69, 82, 85	79	11
		1	95, 98, 98	97	2
	Snap beans – whole plant	0.03	70, 72, 79, 83,83, 86	79	7
		0.5	103, 105, 108	105	2
		10	97, 103, 103	101	3
		32	90, 92, 97	93	3
	Dry beans	0.01	92, 95, 100	96	4
		0.3	87, 87, 93	89	4
		3	97, 100, 103	100	3
	Beetroot	0.01	86, 93, 96	92	6
		0.1	86, 91, 99	92	7
		1	96, 101, 102	100	3

Method	Matrix	Fortification level (mg//kg)	Recoveries (%)	Mean Recovery (%)	RSD (%)
	Beet tops	0.01	62, 74, 88	75	17
		0.1	106, 115, 115	112	5
		1	97, 101, 102	100	3
		10	98, 98, 103	100	3
DuPont 36189	Maize forage	0.01	104, 107, 109, 112, 113	109	4
		0.1	104, 107, 110, 110, 112	112	3
	Maize grain	0.01	104, 106, 106, 109, 111	107	3
		0.1	101, 103, 103, 104, 105	103	1
	Maize flour	0.01	85, 87, 89, 91, 96	90	5
		0.1	89, 89, 90, 96, 96	92	4
	Maize dry milling meal	0.01	84, 85, 86, 88, 90	87	3
		0.1	83, 85, 86, 89, 92	87	4
	Maize dry milling oil	0.01	96, 100, 101, 103, 105	101	3
		0.1	98, 99, 99, 99, 99	99	0.5
	Almond hulls	0.01	91, 94, 98, 99, 99	96	4
		0.1	90, 94, 95, 96, 100	95	4
	Almond Nutmeats	0.01	89, 94, 96, 98, 103	96	5
		0.1	92, 94, 99, 99, 100	97	4

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

New storage stability data for indoxacarb and its R enantiomer on blueberries, peppers, snap beans, beetroots and maize were submitted to the Meeting. Samples were ground, homogenized with dry ice and fortified with indoxacarb at 1 mg/kg for blueberries, 0.1 mg/kg for peppers, 0.03 mg/kg for snap beans, 0.3 mg/kg for dry beans, 0.1 mg/kg for beetroots and 0.1 mg/kg for maize grain. Samples were stored at ≤ -18 ° C for the duration of the storage stability studies. The sample preparation and storage conditions reflect those employed in the residue trials.

For maize and pepper only, the initial fortified samples were analysed prior to storage and served as the time zero samples. At subsequent time periods, one - three stored samples were taken and analysed along with at least one stored control sample and one stored control sample freshly fortified with indoxacarb to serve as a procedural recovery sample.

Residues were determined in the stored samples using analytical method AMR 4271-96 for blueberries, analytical method AMR 3493-95 for peppers, analytical method AMR 2712-93 for snap beans, dry beans and beetroots, and analytical method AMR 3493-95 for maize grain.

Results from the storage stability samples are summarized in Table 2.

Table 2 Stability of indoxacarb residues in various commodities when stored at ≤ -18 ºC

Commodity	Storage period (days)	Residue level (sum of indoxacarb and its R enantiomer) in stored sample (mg/kg)	Percentage recovery (%)	Procedural recovery sample of freshly prepared sample (%)
Fortified storage stability samples				
Blueberries (high acid)	0	-	-	-
	505	0.934, 0.981, 0.975	93, 98, 98	98
Peppers (high water)	0	0.094, 0.098	94, 98	105, 95
	330	0.087, 0.11	87, 110	87, 94
Snap beans - pods with seeds (high water)	0	-	-	-
	209	0.03, 0.027, 0.027	101, 91, 90	109
Snap bean – whole plant (high water)	0	-	-	-
	209	0.028, 0.03, 0.032	93, 99, 106	94
Dry beans (high protein)	0	-		
	210	0.31, 0.31, 0.31	103, 103, 103	93
Beet roots (high starch)	0	-	-	-
	530	0.097, 0.1, 0.1	97, 100, 100	95
Beet tops (high water)	0	-	-	-
	530	0.087, 0.088, 0.089	87, 88, 89	90
Maize grain (high starch)	0	0.097	97	99
	60	0.099	99	99
	90	0.094	94	100
	180†	0.035	35	76
	330	0.090	90	90
	390	0.096	96	98

Notes:

† Considered an outlier based on the stability observed at subsequent time points

USE PATTERN

Information on the registered uses of indoxacarb was provided to the Meeting and is summarised in Table 3.

In some trials the formulation was based on racemic indoxacarb and in others indoxacarb 3S+1R was used. In all situations, the application rate and spray concentration are expressed in terms of the active ingredient, indoxacarb.

Table 3 Registered use of indoxacarb

Crop	Country	Indoor/ outdoor	Type	Rate (g ai/ha)	No. of applications	RTI (days)	PHI (days)
Bush berries	United States	Outdoor	Foliar	123	4	7	7
Okra	United States	Outdoor	Foliar	123	4	5	3
Beans with pods (except soybean)	United States	Outdoor	Foliar	123	4	7	3

Crop	Country	Indoor/ outdoor	Type	Rate (g ai/ha)	No. of applications	RTI (days)	PHI (days)
Beans, dry (except soybean)	United States	Outdoor	Foliar	123	4	7	7
Beetroot	United States	Outdoor	Foliar	123	4	3	7
Maize	United States	Outdoor	Foliar	123	2	5	Grain and stover: 14 forage, fodder: 1
Maize	Brazil	Outdoor	Foliar	60	3	7	30
Tree nuts	United States	Outdoor	Foliar	123	3	7	5

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on blueberries, pepper, snap beans, dry beans, beetroot, maize and tree nuts. In all cases residues were measured and expressed as indoxacarb and its R enantiomer.

Bush berries

Blueberries

Thirteen residue trials were on blueberries from Canada and the United States in 2003 were provided to the Meeting. The trials were conducted with a WG formulation. A total of 4 applications with an individual application of approximately 123 g ai/ha were made. The re-treatment interval was 6–9 days.

Samples were collected 6–8 days after the last application and immediately frozen and stored at ≤ -18 ºC for up to 476 days prior to analysis.

Residues of indoxacarb and its R enantiomer in blueberries were determined using analytical method AMR 4271-96. Procedural recoveries were conducted at fortification levels of 0.02 and 1 mg/kg with recoveries in the range of 92–113 percent.

The three trials conducted in Fennville, MI, were conducted at the same trial site with similar dates of application and harvest. Therefore, these three trials are regarded as replicate trials.

A summary of the trials is outlined in Table 4.

Table 4 Residues of indoxacarb in blueberries from supervised trials conducted in Canada and the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	4 × 123	4 × 26 - 66	7	-	7	-	-	-
03-ME03 Jonesboro, ME, United States, 2003 Blueberry/ Low bush	130 129 128 128	53.8 53.7 53.8 54.0	- 7 9 6	Fruiting	6	Fruit	1.04. 1.03 (1.04)	IR-4 PR No 07038
03-NJ28 Bridgeton, NJ, United States, 2003 Blueberry/ Blueray	124 123 128 129	57.3 57.1 57.2 57.5	- 6 6 7	Fruiting	6	Fruit	0.32, 0.38 (0.35)	
03-MI31 Fennville, MI, United States, 2003 Blueberry/ Rubel Replicate trial 1a	120 123 120 121	26.3 26.4 26.4 26.4	- 7 7 7	Fruiting	7	Fruit	0.59, 0.56 (0.58)	
03-MI32 Fennville, MI, United States, 2003 Blueberry/Rubel Replicate trial 1b	123 124 118 118	26.4 26.4 26.2 26.4	- 7 7 7	Fruiting	7	Fruit	0.63, 0.51 (0.57)	
03-MI33 Fennville, MI, United States, 2003 Blueberry/ Rubel Replicate trials 1c	126 124 119 118	26.4 26.3 26.2 26.2	- 7 8 6	Fruiting	7	Fruit	0.53, 0.55 (0.54)	
03-GA*17 Alapaha, GA, United States, 2003 Blueberry/ Premier	124 123 122 123	60.2 60.0 59.8 59.8	- 6 6 8	Fruiting	6	Fruit	0.84, 0.84 (0.84)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
03-NC23 Castle Hayne, NC, United States 2003 Blueberry/ Summit	126 126 127 123	42.2 42.4 42.1 42.3	- 7 6 6	Fruiting	8	Fruit	0.20, 0.30 (0.25)	
03-WA15 Burlington, WA, United States, 2003 Blueberry/ Bluecrop	124 126 126 126	65.9 66.2 65.9 67.1	- 7 7 7	Fruiting	8	Fruit	0.58, 0.58 (0.58)	
03-OR16 Wilsonville, OR, United States, 2003 Blueberry/ Bluecrop	126 123 124 127	26.3 26.3 26.3 26.4	- 8 7 7	Fruiting	7	Fruit	0.38, 0.35 (0.37)	
03-QC09 St. Paul d'Abbotsford, QC, CAN, 2003 Blueberry/Northblue	123 126 123 124	52.7 52.9 52.6 52.6	- 6 6 6	Fruiting	6	Fruit	0.28, 0.27 (0.28)	
03-NS01 Mt. Thom, NS, CAN, 2003 Blueberry/ Lowbush	121 132 119 129	61.7 67.7 62.0 61.7	- 8 6 8	Fruiting	7	Fruit	0.81, 0.80 (0.81)	
03-NS02 East Village, NS, CAN, 2003 Blueberry/ Lowbush	122 121 130 131	61.7 62.0 61.6 61.8	- 7 6 8	Fruiting	7	Fruit	0.81, 0.73 (0.77)	
03-NS05 Parrsboro, NS, CAN, 2003 Blueberry/ Lowbush	118 129 133 128	61.9 61.7 62.0 61.7	- 8 6 8	Fruiting	6	Fruit	0.42, 0.52 (0.47)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

Fruiting vegetables, other than cucurbits

Peppers

Nine residue trials were conducted on bell (six trials) and non-bell peppers (three trials) in the United States in 1996–1997.

The trials were conducted with a WG formulation. A total of 4 applications with an individual application of 75 g ai/ha were made. The re-treatment interval was 4–5 days.

Samples were collected 0–21 days after the last application and immediately frozen and stored at ≤ -18 ºC for up to 7 months prior to analysis.

Residues of indoxacarb and its R enantiomer in peppers were determined using analytical method AMR 3493-95. Procedural recoveries were conducted at fortification levels of 0.02, 0.1 and 0.2 mg/kg with recoveries in the range of 80–114 percent.

The two trials conducted in 1996 in Bradenton, FL were conducted at the same trial site. However, the dates of application and harvest were > 30 days apart and hence the trials can be regarded as independent trials. The two trials conducted in 1997 in Bradenton, FL were also conducted at the same trial site. As the varieties of pepper tested are significantly different Bell pepper vs non-bell pepper), these trials are regarded as independent trials.

A summary of the trials is outlined in Tables 5 and 6 for bell and non-bell peppers respectively.

Table 5 Residues of indoxacarb in bell peppers from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
			-		-	-	AMR 3735-96
Trial 01 Seven Springs, NC, United States, 1996 Bell pepper/ Capistrano	75 75 75 75	- 5 5 5		3 7 14	Fruit	<0.02, < 0.02 (<0.02) <0.02, < 0.02 (<0.02) <0.02, < 0.02 (<0.02)	
Trial 02 Bradenton, FL, United States, 1996 Bell pepper/ Capistrano †	75 75 75 75	- 4 4 5		3 7 14	Fruit	<0.02, < 0.02 (<0.02) <0.02, < 0.02 (<0.02) <0.02, < 0.02 (<0.02)	
Trial 04 Genoa, OH, United States, 1996 Bell pepper/ Northstar	75 75 75 75	- 4 5 5		3 7 14	Fruit	<0.02, <0.02 (<0.02) 0.027, 0.029 (0.028) <0.02, <0.02 (<0.02)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 06 San Ardo, CA, United States, 1996 Bell pepper/ Cal Wonder	75 75 75 75	- 5 5 5		0 2 7 14 21	Fruit	0.062, 0.090 (<0.076) 0.026, 0.021 (<0.024) 0.056, 0.077 (0.067) 0.048, 0.065 (0.057) 0.039, 0.030 (0.035)	
Trial 07 Porterville, CA, United States 1996 Bell pepper/ Yolo Wonder	75 75 75 75	- 5 5 5		3 7 14	Fruit	0.072, 0.079 (0.076) 0.091, 0.059 (0.075) 0.027, 0.027 (0.027)	
Trial 08 Bradenton, FL, United States, 1997 Bell pepper/ Capostrano †	75 75 75 75	- 4 5 5		3 7 14	Fruit	<0.02, 0.02 (0.02) 0.034, <0.02 (0.027) <002, <0.02 (<0.02)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

Table 6 Residues of indoxacarb in non-bell pepper from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Indoxacarb (mg/kg)	Report Number
			-		-	-	AMR 3735-96
Trial 03 Bradenton, FL, United States, 1996 Non-Bell pepper/ Cayenne †	75 75 75 75	- 5 4 5		3 7 14	Fruit	<0.02, < 0.02 (<0.02) <0.02, < 0.02 (<0.02) <0.02, < 0.02 (<0.02)	
Trial 05 Donna, TX, United States, 1996 Non-Bell pepper/Anaheim	75 75 75 75	- 5 5 5		3 7 14	Fruit	0.039, 0.041 (0.04) 0.021, 0.040 (0.031) <0.02, <0.02 (<0.02)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Indoxacarb (mg/kg)	Report Number
Trial 09 Bradenton, FL, United States 1997 Non-Bell pepper/ Japapeno †	75 75 75 75	- 4 5 5		3 7 14	Fruit	0.093, 0.099 (0.096) 0.048, 0.037 (0.043) 0.024, 0.040 (0.032)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

† Trials regarded as independent based on treatments being > 30 days apart and/or different crop varieties.

Legume vegetables: Beans with pods

Snap beans/ common beans with pods

Nine residue trials were conducted on snap beans in the United States in 2005.

The trials were conducted with a WG formulation. A total of 4 applications in eight trials and 5 applications in one trial were made. The individual application rate was approximately 123 g ai/ha. The re-treatment interval was 5–8 days.

Samples were collected 0–13 days after the last application and immediately frozen and stored at ≤ -18 ºC for up to 203 days.

Residues of indoxacarb and its R enantiomer in snap beans were determined using analytical method AMR 271293. Procedural recoveries snap beans were conducted at fortification levels of 0.03 and 0.1 mg/kg with recoveries in the range of 91–109 percent.

The two trials conducted in Citra, FL were conducted at the same trial site. However, the application timings and harvest were > 30 days apart and hence the trials can be regarded as independent trials.

A summary of the trials is outlined in Table 7.

Table 7 Residues of indoxacarb in snap beans from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**4 × 123**	**4 × 13 - 66**	**7**	**-**	**3**	**-**	**-**	**-**
CA107Parlier, CA, United States, 2005 Snap beans/ charon	121 123 124 124	32.9 32.8 33.3 31.9	- 7 7 7	Fruiting Fruiting Fruiting Fruiting	0 2 7 13	Pods with seeds	0.79, 0.81 (0.80) 0.58. 0.59 (0.59) 0.42, 0.42 (0.42) 0.37, 0.37 (0.37)	IR-4 PR No. 08574

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
FL39 Citra, FL, United States, 2005 Snap beans/Leon †	129 127 130 126	37.8 37.6 37.6 37.5	- 7 7 7	Vegetative Blooming Flowering & fruiting Fruiting	3	Pods with seeds	0.12, 0.12 (0.12)	
FL53 Citra, FL, United States, 2005 Snap beans/Leon †	124 124 129 124	38 37.9 38 37.9	- 8 6 7	Blooming Blooming Flowering & fruiting Fruiting	2	Pods with seeds	0.19, 0.20 (0.20)	
MI21 Holt, MI, United States, 2005 Snap beans/Hercules	130 128 126 127	49.3 49.5 49.3 49.2	- 7 7 6	Vegetative Flowering Fruiting Fruiting	2	Pods with seeds	0.17, 0.17 (0.17)	
WI20 Arlington, WI, United States, 2005 Snap beans/Hystyle	127 133 128 124	35.1 36.4 35.4 36.9	- 7 7 7	Blooming Fruiting Fruiting Fruiting	3	Pods with seeds	0.11, 0.11 (0.11)	
ID20 Kimberly, ID, United States, 2005 Snap beans/Idelite garden beans	121 129 127 122	32.8 32.8 32.8 32.6	- 6 8 6	Pod set Maturing pod set Mature Mature	2	Pods with seeds	0.17, 0.17 (0.17)	
NC18 Clinton, NC, United States, 2005 Snap beans/Bronco	123 123 123 125	38.4 38.4 38.6 38.5	- 7 5 6	Vegetative Blooming Fruiting Flowering & fruiting	3	Pods with seeds	0.12, 0.13 (0.13)	
NJ22 Bridgeton, NJ, United States, 2005 Snap beans/ Bluelake 274	126 122 128 127	38.1 38 38.2 38.2	- 7 8 6	Blooming Blooming Flowering & fruiting Fruiting	2	Pods with seeds	0.15, 0.15 (0.15)	
OH12 Celeryville, OH, United States, 2005 Snap beans/Bluelake	122 121 121 122 121	26.3 26.4 26.3 26.4 26.5	- 7 7 7 7	Vegetative Vegetative Blooming Blooming Fruiting	2	Pods with seeds	0.05, 0.04 (0.05)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

† trials regarded as independent trails based on treatments being > 30 days apart.

Pulses

Dry beans

Sixteen residue trials were conducted on dry beans in the United States in 2006.

The trials were conducted with an EG formulation. A total of 4 applications in fifteen trials and 5 applications in one trial were made. The individual application rate was approximately 123 g ai/ha. The re-treatment interval was 6–8 days.

Samples were collected 0–22 days after the last application and immediately frozen and stored at ≤ -18 ºC for up to 225 days prior to analysis.

Residues of indoxacarb and its R enantiomer in dry beans were determined using analytical method AMR 2712 93. Procedural recoveries for dry beans were conducted at fortification levels of 0.01, 0.3 and 1 mg/kg with recoveries in the range of 69–120 percent.

For the trials conducted in Velva, ND, Moxee, WA and Arlington, WI, replicate trials were conducted at the same time but with a different formulation type (WG formulation).

A summary of the trials is outlined in Table 8.

Table 8 Residues of indoxacarb in dried beans from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**4 × 123**	**4 × 13 - 66**	**7**	**-**	**7**	**-**	**-**	IR-4 PR No. 09669
CA86 Davis, CA, U.S.A., 2006 Bean (dry)/ Calif. Early Light Red Kidney bean	127 127 123 122 119		- 6 8 6 7		0 8 13 22	Dry bean seed	0.048, 0.038 (0.053) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
CO10 Ft. Collins, CO, U.S.A., 2006 Bean (dry)/ Dry bean, Vision	129 122 123 122		- 6 7 7		6	Dry bean seed	<0.01, <0.01 (<0.01)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
CO11 Fruita, CO, United States, 2006 Bean (dry)/ Montrose Pinto bean	132 127 130 130		- 7 7 7		8	Dry bean seed	<0.01, <0.01 (<u><0.01</u>)	
ID12 Kimberly, ID, United States, 2006 Bean (dry)/ UI 911 Dry Bean	123 124 124 129		- 7 6 7		8	Dry bean seed	0.046, 0.093 (<u>0.070</u>)	
MI25 Holt, MI, United States, 2006 Bean (dry)/ Red Hawk Dark Red Kidney Bean	127 128 130 127		- 7 8 6		7	Dry bean seed	<0.01, <0.01 (<u><0.01</u>)	
MI41 Lansing, MI, United States, 2006 Bean (dry)/ Red Hawk Dark Red Kidney Bean	128 126 129 127		- 7 8 6		7	Dry bean seed	<0.01, <0.01 (<u><0.01</u>)	
ND05 Velva, ND, United States, 2006 Bean (dry)/ Maverick Pinto bean	121 124 126 123		- 7 7 7		7	Dry bean seed	<0.01, <0.01 (<0.01)	
ND05-03 Velva, ND, United States, 2006 Bean (dry)/Maverick Pinto bean	122 123 123 123		- 7 7 7		7	Dry bean seed	<0.01, 0.011 (<u>0.011</u>)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
ND06 Fargo, ND, United States, 2006 Bean (dry)/ Eclipse Black Bean	122 124 123 123		- 6 8 8		0 7 13 20	Dry bean seed	<0.01, <0.01 (<0.01) <0.01, <0.01 (<u>≤0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
ND17 Velva, ND, United States 2006 Bean (dry)/ Norstar Navy Bean	122 124 122 122		- 7 7 7		7	Dry bean seed	<0.01, <0.01 (<u>≤0.01</u>)	
NJ33 Bridgeton, NJ, United States, 2006 Bean (dry)/ Navy Bean, White Marrow	124 122 103 120		- 8 6 7		6	Dry bean seed	<0.01, <0.01 (<u>≤0.01</u>)	
OH16 Willard, OH, United States 2006 Bean (dry)/ Vermont Cranberry dry bean	126 126 122 127		- 7 7 8		7	Dry bean seed	<0.01, 0.01 (<u>0.01</u>)	
WA*27 Moxee, WA, United States 2006 Bean (dry)/ Othello Pinto bean	128 124 123 126		- 6 8 7		6	Dry bean seed	0.01, <0.01 (0.01)	
WA*27-03 Moxee, WA, United States, 2006 Bean (dry)/ Othello Pinto bean	126 127 124 124		- 6 8 7		6	Dry bean seed	0.02, 0.024 (<u>0.022</u>)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
WI19 Arlington WI, United States, 2006 Bean (dry)/ Dark Red Kidney bean	126 127 131 124		- 7 8 8		6	Dry bean seed	0.012, 0.016 (0.014)	
WI19- 03 Arlington, WI, United States, 2006 Bean (dry)/ Dark Red Kidney bean	127 126 131 126		- 7 8 8		6	Dry bean seed	0.028, 0.032 (0.030)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

Root and tuber vegetables

Beetroot

Five residue trials were conducted on beetroot in the United States in 2004.

The trials were conducted with a WG formulation. A total of 4 applications were made. The individual application rate was approximately 123 g ai/ha. The re-treatment interval was 3–4 days.

Samples were collected 3–14 days after the last application and immediately frozen and stored at ≤ -18 ºC for up to 515 days, for root samples, and up to 519 days, for beetroot tops, prior to analysis.

Residues of indoxacarb and its R enantiomer in beetroot were determined using analytical method AMR 271293. Procedural recoveries were conducted at fortification levels of 0.01, 0.1 and 1 mg/kg with recoveries in the range of 83–104 percent.

Information was also provided on the residue levels in the beetroot tops/ leaves, but as this is not consumed by humans or livestock, the information has not been presented.

A summary of the trials is outlined in Table 9.

Table 9 Residues of indoxacarb in beet from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**4 × 123**	**3**	**-**	**7**	**-**	**-**	IR-4 PR No 08870

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
NY12 Freeville, NY, United States, 2004 Beets (garden)/ Red Ace	129 128 119 124	- 4 4 3	8 leaf stage 8 leaf stage Tubers forming 2-4" tubers	7	Roots	0.18, 0.19 (0.19)	
OH09 Celeryville, OH, United States, 2004 Beetroots/ Beets (garden)/ Red Ace	122 120 121 123	- 4 4 3	Vegetative Vegetative Vegetative Vegetative	7	Roots	0.13, 0.12 (0.13)	
OR12 Aurora, OR United States, 2004 Beetroots/ Beets (garden)/ Detroit Dark Red St	126 127 127 123	- 4 2 3	1-2" beets 6-8 leaf stage 8-10 leaf stage 2-3" beets	7	Roots	0.11, 0.12 (0.12)	
TX*28 Weslaco, TX, United States, 2004 Beetroots/ Beets (garden)/ Detroit Dark Red	124 124 124 123	- 3 3 4	Forming bulbs Forming bulbs 1-3" bulbs 4" bulbs	3 7 10 14	Roots	0.29, 0.3 (0.30) 0.21, 0.22 (0.22) 0.17, 0.18 (0.18) 0.15, 0.15 (0.15)	
WI13 Arlington, WI, United States, 2004) Beetroots/ Beets (garden)/ Detroit Medium Top	127 123 129 127	- 4 3 4	Vegetative Vegetative Vegetative Vegetative	7	Roots	0.18, 0.18 (0.18)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

Cereal grains

Maize

Twenty one residue trials were conducted on maize in the United States in 2015.

The trials were conducted with an EC formulation. A total of 2 applications were made. The individual application rate was approximately 123 g ai/ha. The re-treatment interval was 4–6 days.

An adjuvant was used in the trials.

Grain samples were collected 12–14 days after the last application. samples were immediately frozen and stored at ≤ -18 ºC for up to 161 days prior to analysis.

Residues of indoxacarb and its R enantiomer were determined using analytical method DuPont 36189. Procedural recoveries were conducted at fortification levels of 0.01 and 0.1 mg/kg with recoveries in the range of 88–117 percent.

A summary of the trials is outlined in Table 10.

Table 10 Residues of indoxacarb in maize grain from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**2 × 123**	**5**	**-**	**14**	**-**	**-**	DuPont-44492
Trial 01 Germansville, PA, United States, 2015 Field Corn/ TA 105-00	129 129	- 4	- -	14	Grain	<0.01, <0.01 (<0.01)	
Trial 02 Chula, GA, United States, 2015 Field Corn/ DK 62-08	121 121	- 4	BBCH 87 -	12	Grain	<0.01, <0.01 (<0.01)	
Trial 03 Fitchburg, WI, United States, 2015 Field Corn/ GH 97 × 48-3111	126 125	- 5	- -	14	Grain	<0.01, <0.01 (<0.01)	
Trial 04 Northwood, ND, United States, 2015 Field Corn/ 01043620	126 127	- 5	BBCH 87 BBCH 87	14	Grain	0.01, 0.014 (0.012)	
Trial 05 Lime Springs, IA, United States, 2015 Field Corn/ DKC46-37R1B	123 124	- 5	BBCH 87 BBCH 87	13	Grain	<0.01, <0.01 (<0.01)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 06 Kirksville, MO, United States, 2015 Field Corn/ P1498AM	126 122	- 4	- -	12	Grain	<0.01, <0.01 (<u>≤0.01</u>)	
Trial 07 Richland, IA, United States, 2015 Field Corn/ P0506AM	124 124	- 5	-	14	Grain	0.012, <0.01, (<u>0.011</u>)	
Trial 08 St. Cloud, MN, United States, 2015 Field Corn/ DKC-41-32	124 123	- 5	BBCH 86 BBCH 86	14	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 09 Marysville, OH, United States, 2015 Field Corn/ 9603RR	124 121	- 5	- -	14	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 10 Lenexa, KS, United States, 2015 Field Corn/ 7224-UT3-PRIB	125 124	- 5	BBCH 85 BBCH 87	14	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 11 Highland, IL, United States, 2015 Field Corn/ A6517	125 128	- 5	BBCH 89 BBCH 89	14	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 12 Carlyle, IL, United States, 2015 Field Corn/ FS 63SV1 RIB	123 125	- 5	BBCH 89 BBCH 89	14	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 13 York, NE, United States, 2015 Field Corn/ P0876HR	123 124	- 4	BBCH 87 BBCH 87	13	Grain	<0.01, <0.01, (<u><0.01</u>)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 14 Fisk, MO, United States, 2015 Field Corn/ Channel 217-07VT2PRIB	125 123	- 4	BBCH 87 BBCH 87	13	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 15 Brunswick, NE, United States, 2015 Field Corn/ DKC 60-67 RIB	121 130	- 6	BBCH 87 BBCH 87	14	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 16 Clarence, MO, United States, 2015 Field Corn/ G01P529-GTA	123 123	- 5	- -	14	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 17 Stewardson, IL, United States, 2015 Field Corn/ DKC-62-98	124 126	- 5	BBCH 89 BBCH 89	13	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 18 Stilwell, KS, United States, 2015 Field Corn/ PK 110-10RR	126 122	- 5	BBCH 89 BBCH 89	13	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 19 Atlantic, IA, United States, 2015 Field Corn/ DKC 62-98 RIB	125 126	- 5	BBCH 89 BBCH 89	14	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 20 Geneva, MN, United States, 2015 Field Corn/ Pioneer 19526AMXT	123 122	- 5	BBCH 87 BBCH 87	14	Grain	<0.01, <0.01, (<u>≤0.01</u>)	
Trial 21 Uvalde, TX, United States, 2015 Field Corn/ DKC 64-69	123 123	- 5	BBCH 87 BBCH 87	14	Grain	<0.01, <0.01, (<u>≤0.01</u>)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

Tree nuts

Seventeen residue trials were conducted on almonds (six trials), pecan (six trials) and pistachios (five trials) in the United States in 2017.

The trials were conducted with three soil directed applications, at an individual rate of 0.757 g ai/ha, and with three foliar sprays, at an individual application rate of 124 g ai/ha. The soil directed applications were conducted with a RB formulation and the foliar applications were conducted with a WG formulation.

An adjuvant was used in the trials for the foliar applications. The label states that for best results an adjuvant should be used.

Samples of nutmeats were collected 0–15 days after the last application. Samples were immediately frozen and stored at ≤ -18 ºC for up to 319 days prior to analysis.

Residues of indoxacarb and its R enantiomer were determined using analytical method DuPont 36189. Procedural recoveries were conducted at fortification levels of 0.01 and 0.1 mg/kg with recoveries in the range of 93–108 percent.

The trials conducted in Fresno, CA, were conducted at different trial sites and are therefore regarded as independent trials.

A summary of the trials is outlined in tables 11, 12 and 13 for almonds, pecan and pistachios respectively.

Table 11 Residues of indoxacarb in almonds from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**3 × 123**	**9 - 26**	**7**	**-**	**5**	**-**	**-**	FMC-49392, Revision No. 1
Trial 01 Fresno, CA, United States, 2017 Almond/ non-Pareil	0.7578 0.7578 0.7578 123 123 123	- - - 9 9 9	- 30 30 - 7 7	BBCH 78 BBCH 82 BBCH 87 BBCH 84 BBCH 84 BBCH 87	5	Nutmeats	0.028, 0.018 (0.023)	
Trial 02 Madera, CA, United States, 2017 Almond/ Mission	0.7578 0.7578 0.7578 124 125 124	- - - 147 148 147	- 31 30 - 7 7	BBCH 77 BBCH 81 BBCH 88 BBCH 87 BBCH 87 BBCH 88	5	Nutmeats	0.02, 0.023 (0.022)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 03 Hickman, CA, United States, 2017 Almond/ Non-Pareil	0.7626 0.7626 0.7626 123 125 124	- - - 8 8 8	- 30 30 - 7 7	BBCH 79 BBCH 81 BBCH 89 BBCH 85 BBCH 85 BBCH 89	5	Nutmeats	0.013, 0.013 (0.013)	
Trial 04 Fresno, CA, United States, 2017 Almond/ Monterey	0.7622 0.7678 0.7523 123 121 123	- - - 23 22 22	- 28 32 - 7 7	Hull split Hull split Hull split Hull split Hull split Hull split	4	Nutmeats	<0.01, <0.01 (<0.01)	
Trial 05 Porterville, CA, United States, 2017 Almond/ Butte	0.7607 0.7596 0.7488 122 123 123	- - - 16 16 16	- 31 30 - 7 7	65 DBCH 35 DBCH BBCH 89 BBCH 81 BBCH 87 BBCH 89	-0 0 6 9	Nutmeat	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.01, 0.012 (0.011)	
Trial 06 Live Oak, CA, United States, 2017 Almond/ Non-Pareil	0.7877 0.7613 0.7585 122 124 124	- - - 24 25 25	- 29 31 - 7 7	BBCH 78 BBCH 85 BBCH 89 BBCH 87 BBCH 88 BBCH 88	-1 0 5 9 14	Nutmeat	<0.01, <0.01 (<0.01) 0.012, 0.015 (0.013) 0.014, 0.01 (0.012) 0.01, 0.012 (0.011) 0.014, 0.017 (0.016)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

DBCH = Days Before Commercial Harvest.

Table 12 Residues of indoxacarb in pecan from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**3 × 123**	**9 - 26**	**7**	**-**	**5**	**-**	**-**	FMC-49392, Revision No. 1
Trial 07 Bertrand, MO, United States, 2017 Pecan/ pawnee	0.7521 0.7646 0.7560 125 123 124	- - - 149 146 148	- 29 32 - 7 7	BBCH 81 BBCH 83-84 BBCH 87-88 BBCH 84-85 BBCH 85-86 BBCH 87-88	6	Nutmeat	0.01, 0.01 (0.01)	
Trial 08 Tifton, GA, United States, 2017 Pecan/ Sumner	0.7581 0.7581 0.7581 124 123 123	- - - 14 15 15	- 28 28 - 6 7	70 percent of final size Early shuck split 80 percent ripe Full shuck split 60 percent ripe 80 percent ripe	4	Nutmeats	0.032, 0.034 (0.033)	
Trial 09 Vienna, GA, United States, 2017 Pecan/ Oconee	0.7581 0.7581 0.7581 122 123 123	- - - 18 20 20	- 28 28 - 8 6	Early shuck split 20 percent ripe Ripe 70 percent ripe 90 percent ripe Ripe	5	Nutmeats	0.029, 0.027 (0.028)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 10 Colbert, GA, United States, 2017 Pecan/ Gloria Grand	0.7573 0.7573 0.7573 122 125 123	- - - 11 12 11	- 30 29 - 7 7	BBCH 75-7 BBCH 77-79 BBCH 87-89 BBCH 81-83 BBCH 83-85 BBCH 87-89	5	Nutmeats	0.018, 0.019 (0.019)	
Trial 11 Pearsall, TX, United States, 2017 Pecan/ Caddo	0.7568 0.7581 0.7581 126 125 123	- - - 20 22 21	- 29 31 - 6 7	Nuts full size Dough or later 100 percent shuck split 30 percent shuck split Complete shuck split 100 percent such split	4	Nutmeats	0.030, 0.038 (0.034)	
Trial 12 Lubbock, TX, United States, 2017 Pecan/ Western Schley	0.7165 0.7581 0.7581 124 121 126	- - - 24 24 24	- 31 30 - 6 7	Green shuck Green shuck Mature Shuck split Shuck split Mature	5	Nutmeats	<0.01, <0.01 (<0.01)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

DBCH = Days Before Commercial Harvest.

Table 13 Residues of indoxacarb in pistachios from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**3 × 123**	**9 - 26**	**7**	**-**	**5**	**-**	**-**	FMC-49392, Revision No. 1
Trial 13 West Sacramento, CA, United States, 2017 Pistachio/ Kerman	0.7679 0.7604 0.7604 123 124 124	- - - 25 25 25	- 28 28 - 7 7	BBCH 75 BBCH 79 BBCH 89 BBCH 85 BBCH 87 BBCH 89	6	Nutmeats	<0.01, 0.012 (0.011)	
Trial 14 Fresno, CA, United States, 2017 Pistachio/ Lost Hills	0.7578 0.7578 0.7578 123 123 123	- - - 146 146 146	- 30 30 - 7 7	BBCH 81 BBCH 83 BBCH 88 BBCH 86 BBCH 88 BBCH 88	5	Nutmeats	0.044, 0.046 (0.045)	
Trial 15 Richgrove, CA, United States, 2017 Pistachio/ Kerman/ Pioneer Root stock	0.7579 0.7689 0.7601 124 125 124	- - - 11 11 11	- 30 30 - 7 7	65 DBCH 35 DBCH BBCH 89 BBCH 81 BBCH 89 BBCH 89	-0 0 4 10 14	Nutmeat	0.014, 0.015 (0.014) 0.01, <0.01 (0.01) 0.022, 0.014 (0.018) <0.01, 0.012 (0.011) <0.01, <0.01 (<0.01)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 16 Arbuckle, CA, United States, 2017 Pistachio/ Kerman	0.7679 0.7583 0.7630 123 123 123	- - - 24 25 25	- 32 28 - 7 7	BBCH 75 BBCH 79 BBCH 89 BBCH 85 BBCH 87 BBCH 89	-0 0 6 10	Nutmeat	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<u><0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
Trial 17 Tipton, CA, United States, 2017 Pistachio/ Golden Hills	0.7580 0.7692 0.7606 124 124 125	- - - 13 13 13	- 28 31 - 7 7	BBCH 74 BBCH 76 BBCH 85 BBCH 78 BBCH 81 BBCH 85	-0 0 4 9 15	Nutmeat	0.018, 0.018 (0.018) 0.01, <0.01 (0.01) <0.01, <0.01 (<u><0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

DBCH = Days Before Commercial Harvest.

PRIMARY FEED COMMODITIES OF PLANT ORIGIN

Legume animal feeds

Bean forage

Nine residue trials were conducted on bean (snap beans) forage in the United States in 2005.

The trials were conducted with a total of 4 applications in eight trials and 5 applications in one trial. The individual application rate was approximately 123 g ai/ha. The re-treatment interval was 5– days.

Samples were collected 0–13 days after the last application, at the same time as the fresh seed with pods were harvested. The samples were immediately frozen and stored at ≤ -18 ºC for up 205 days, prior to analysis.

Residues of indoxacarb and its R enantiomer in snap bean forage was determined using analytical method AMR 271293. Procedural recoveries for the whole plant were conducted at fortification levels of 0.5 and 10 mg/kg with recoveries in the range of 82–95 percent.

The two trials conducted in Citra, FL were conducted at the same trial site. However, the application timings and harvest were > 30 days apart and hence the trials can be regarded as independent trials.

A summary of the trials is outlined in Table 14.

Table 14 Residues of indoxacarb in snap bean forage from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**4 × 123**	**4 × 13 - 66**	**7**	**-**	**3**	**-**	**-**	**-**	**-**
CA107Parlier, CA, United States, 2005 Snap beans/ charon	121 123 124 124	32.9 32.8 33.3 31.9	- 7 7 7	Fruiting Fruiting Fruiting Fruiting	0 2 7 13	Plant without pod	Fruiting	23.4, 23.9 (23.7) 14.6, 16.2 (15.4) 17.1, 17.6 (17.4) 13,6, 12.8 (13.2)	IR-4 PR No. 08574
FL39 Citra, FL, United States, 2005 Snap beans/Leon †	129 127 130 126	37.8 37.6 37.6 37.5	- 7 7 7	Vegetative Blooming Flowering & fruiting Fruiting	3	Whole plant	Fruiting	6.8, 6.8 (6.8)	
FL53 Citra, FL, United States, 2005 Snap beans/Leon †	124 124 129 124	38 37.9 38 37.9	- 8 6 7	Blooming Blooming Flowering & fruiting Fruiting	2	Whole plant	Fruiting	10.2, 10 (10.1)	
MI21 Holt, MI, United States, 2005 Snap beans/Hercules	130 128 126 127	49.3 49.5 49.3 49.2	- 7 7 6	Vegetative Flowering Fruiting Fruiting	2	Plant without pods	Fruiting	31.3, 32.1 (31.7)	
WI20 Arlington, WI, United States, 2005 Snap beans/Hystyle	127 133 128 124	35.1 36.4 35.4 36.9	- 7 7 7	Blooming Fruiting Fruiting Fruiting	3	Plant without pods	Fruiting	8.9. 9.1 (9.0)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
ID20 Kimberly, ID, United States, 2005 Snap beans/Idelite garden beans	121 129 127 122	32.8 32.8 32.8 32.6	- 6 8 6	Pod set Maturing pod set Mature Mature	2	Whole plant	Fruiting	12.4, 12.2 (12.3)	
NC18 Clinton, NC, United States, 2005 Snap beans/Bronco	123 123 123 125	38.4 38.4 38.6 38.5	- 7 5 6	Vegetative Blooming Fruiting Flowering & fruiting	3	Plant without pods	Fruiting	10.2, 10.2 (10.2)	
NJ22 Bridgeton, NJ, United States, 2005 Snap beans/ Bluelake 274	126 122 128 127	38.1 38 38.2 38.2	- 7 8 6	Blooming Blooming Flowering & fruiting Fruiting	2	Whole plant	Fruiting	16.1, 16.9 (16.5)	
OH12 Celeryville, OH, United States, 2005 Snap beans/Bluelake	122 121 121 122 121	26.3 26.4 26.3 26.4 26.5	- 7 7 7 7	Vegetative Vegetative Blooming Blooming Fruiting	2	Whole plant	Fruiting	1.3, 1.3 (1.3)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

† Trials regarded as independent as treatments > 30 days apart.

Maize forage and fodder (stover)

Twenty one residue trials were conducted on maize in the United States in 2015.

The trials were conducted with a total of 2 applications. The individual application rate was approximately 123 g ai/ha. The re-treatment interval was 4–7 days. At each trial site two replicate trials were conducted; one replicate to collect forage samples and one replicate to collect stover samples.

An adjuvant was used in the trials.

Forage samples were collected -0–29 days after the last application and stover samples were collected 12–14 days after the last application. Samples were immediately frozen and stored at ≤ -18 º for up to 161 days prior to analysis.

Residues of indoxacarb and its R enantiomer were determined using analytical method DuPont 36189. Procedural recoveries were conducted at fortification levels of 0.01, 0.1 and 5 mg/kg for forage

with recoveries in the range of 99–124 percent. For stover procedural recoveries were conducted at fortification levels of 0.01. 0.1 and 15 mg/kg with recoveries in the range of 67–108 percent.

A summary of the trials is outlined in Tables 15 and 16 for forage and stover respectively.

Table 15 Residues of indoxacarb in maize forage from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**2 × 123**	**5**	**-**	**1**	**-**		**-**	DuPont-44492
Trial 01 Germansville, PA, United States, 2015 Field Corn/ TA 105-00	121 126	- 5	BBCH 67 BBCH 71	1	Forage	BBCH 77	2.3, 3.2 (2.7)	
Trial 02 Chula, GA, United States, 2015 Field Corn/ DK 62-08	121 121	- 5	5 days before BBCH 71 BBCH 71	1	Forage	BBCH 77	1.4, 1.6 (1.5)	
Trial 03 Fitchburg, WI, United States, 2015 Field Corn/ GH 97 × 48-3111	126 125	- 5	BBCH 70 BBCH 71	1	Forage	BBCH 75	1.9, 1.3 (1.6)	
Trial 04 Northwood, ND, United States, 2015 Field Corn/ 01043620	126 127	- 5	BBCH 71 BBCH 71	1	Forage	BBCH 71	1.6, 1.8 (1.7)	
Trial 05 Lime Springs, IA, United States, 2015 Field Corn/ DKC46-37R1B	123 124	- 7	R2 BBCH 71-72	1	Forage	BBCH 75	1.7, 1.2 (1.5)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 06 Kirksville, MO, United States, 2015 Field Corn/ P1498AM	126 122	- 5	R2 blister R3/ BBCH 71	1	Forage	BBCH 71	1.4, 1.5 (1.5)	
Trial 07 Richland, IA, United States, 2015 Field Corn/ P0506AM	124 124	- 5	BBCH 71 BBCH 71	1	Forage	BBCH 71	2.3, 3.1 (2.7)	
Trial 08 St. Cloud, MN, United States, 2015 Field Corn/ DKC-41-32	124 123	- 5	BBCH 67 BBCH 71	1	Forage	BBCH 71	2.2, 2.3 (2.3)	
Trial 09 Marysville, OH, United States, 2015 Field Corn/ 9603RR	124 121	- 5	BBCH 65 BBCH 71	1	Forage	BBCH 71	2.0, 1.1 (1.5)	
Trial 10 Lenexa, KS, United States, 2015 Field Corn/ 7224-UT3-PRIB	125 124	- 5	BBCH 67 BBCH 71	-0 0 1 7 15 21 28	Forage	BBCH 71 - 79	0.33, 0.27 (0.3) 1.7, 2.6 (2.2) 2.4, 2.7 (2.6) 0.37, 0.35 (0.36) 0.18, 0.16 (0.17) 0.16, 0.15 (0.16) 0.16, 0.10 (0.13)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 11 Highland, IL, United States, 2015 Field Corn/ A6517	125 128	- 5	BBCH 71 BBCH 71	1 14	Forage	BBCH 71	1.4, 1.2 (1.3)	
Trial 12 Carlyle, IL, United States, 2015 Field Corn/ FS 63SV1 RIB	123 125	- 5	BBCH 71 BBCH 71	1	Forage	BBCH 71	0.66, 0.90 (0.78)	
Trial 13 York, NE, United States, 2015 Field Corn/ P0876HR	123 124	- 4	BBCH 69 BBCH 71	1	Forage	BBCH 71	1.2, 1.4 (1.3)	
Trial 14 Fisk, MO, United States, 2015 Field Corn/ Channel 217-07VT2PRIB	125 123	- 4	BBCH 67 BBCH 71	-0 0 1 8 14 21 29	Forage	BBCH 71-79	0.80, 0.78 (0.79) 2.5, 3.9 (3.2) 2.7, 3.0 (2.8) 0.40, 0.56 (0.48) 0.21, 0.21 (0.21) 0.17, 0.24 (0.20) 0.18, 0.15 (0.17)	
Trial 15 Brunswick, NE, United States, 2015 Field Corn/ DKC 60-67 RIB	121 130	- 6	BBCH 67 BBCH 71	1	Forage	BBCH 71	2.1, 1.4 (1.8)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 16 Clarence, MO, United States, 2015 Field Corn/ G01P529-GTA	123 123	- 5	R1 R2	1	Forage	Late Blister	3.1, 3.8 (3.4)	
Trial 17 Stewardson, IL, United States, 2015 Field Corn/ DKC-62-98	124 126	- 5	BBCH 67 BBCH 71	-0 0 1 7 14 21 28	Forage	BBCH 71 - 80	0.68, 0.73 (0.71) 1.6, 2.1 (1.9) 1.1, 1.3 (1.2) 0.50, 0.38 (0.44) 0.29, 0.25 (0.27) 0.16, 0.21 (0.18) 0.13, 0.19 (0.16)	
Trial 18 Stilwell, KS, United States, 2015 Field Corn/ PK 110-10RR	126 122	- 5	BBCH 69 BBCH 71	1	Forage	BBCH 71	2.5, 2.1 (2.3)	
Trial 19 Atlantic, IA, United States, 2015 Field Corn/ DKC 62-98 RIB	125 126	- 5	BBCH 69 BBCH 71	1	Forage	BBCH 71	1.9, 2.1 (2.0)	
Trial 20 Geneva, MN, United States, 2015 Field Corn/ Pioneer 19526AMXT	123 122	- 5	BBCH 67-69 BBCH 71	1	Forage	BBCH 71	1.1, 1.0 (1.1)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 21 Uvalde, TX, United States, 2015 Field Corn/ DKC 64-69	123 123	- 5	BBCH 69 BBCH 71	1	Forage	BBCH 71	1.4, 1.2 (1.3)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

Table 16 Residues of indoxacarb in maize stover from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**2 × 123**	**5**	**-**	**14**	**-**		**-**	DuPont-44492
Trial 01 Germansville, PA, United States, 2015 Field Corn/ TA 105-00	129 129	- 4	- -	14	Stover	BBCH 87	1.7, 2.0 (1.9)	
Trial 02 Chula, GA, United States, 2015 Field Corn/ DK 62-08	121 121	- 4	BBCH 87 -	12	Stover	BBCH 99	1.1, 1.2 (1.1)	
Trial 03 Fitchburg, WI, United States, 2015 Field Corn/ GH 97 × 48-3111	126 125	- 5	- -	14	Stover	BBCH 89	1.1, 1.2 (1.1)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 04 Northwood, ND, United States, 2015 Field Corn/ 01043620	126 127	- 5	BBCH 87 BBCH 87	14	Stover	BBCH 89	0.97, 0.95 (0.96)	
Trial 05 Lime Springs, IA, United States, 2015 Field Corn/ DKC46-37R1B	123 124	- 5	BBCH 87 BBCH 87	13	Stover	BBCH 97	1.3, 2.1 (1.7)	
Trial 06 Kirksville, MO, United States, 2015 Field Corn/ P1498AM	126 122	- 4	- -	12	Stover	BBCH 89	5.2, 5.0 (5.1)	
Trial 07 Richland, IA, United States, 2015 Field Corn/ P0506AM	124 124	- 5	 -	14	Stover	BBCH 89	2.4, 3.2 (2.8)	
Trial 08 St. Cloud, MN, United States, 2015 Field Corn/ DKC-41-32	124 123	- 5	BBCH 86 BBCH 86	14	Stover	BBCH 97	2.7, 2.2 (2.4)	
Trial 09 Marysville, OH, United States, 2015 Field Corn/ 9603RR	124 121	- 5	- -	14	Stover	BBCH 89	10, 8 (9.1)	
Trial 10 Lenexa, KS, United States, 2015 Field Corn/ 7224-UT3-PRIB	125 124	- 5	BBCH 85 BBCH 87	14	Stover	BBCH 83-85	6.0, 5.8 (5.9)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 11 Highland, IL, United States, 2015 Field Corn/ A6517	125 128	- 5	BBCH 89 BBCH 89	14	Stover	BBCH 89	1.5, 1.9 (1.7)	
Trial 12 Carlyle, IL, United States, 2015 Field Corn/ FS 63SV1 RIB	123 125	- 5	BBCH 89 BBCH 89	14	Stover	BBCH 91	3.3, 4.1 (3.7)	
Trial 13 York, NE, United States, 2015 Field Corn/ P0876HR	123 124	- 4	BBCH 87 BBCH 87	13	Stover	BBCH 89	1.3, 1.4 (1.3)	
Trial 14 Fisk, MO, United States, 2015 Field Corn/ Channel 217-07VT2PRIB	125 123	- 4	BBCH 87 BBCH 87	13	Stover	BBCH 83	3.9, 3.7 (3.8)	
Trial 15 Brunswick, NE, United States, 2015 Field Corn/ DKC 60-67 RIB	121 130	- 6	BBCH 87 BBCH 87	14	Stover	BBCH 89	1.2, 1.4 (1.3)	
Trial 16 Clarence, MO, United States, 2015 Field Corn/ G01P529-GTA	123 123	- 5	- -	14	Stover	BBCH 89	4.2, 2.8 (3.5)	
Trial 17 Stewardson, IL, United States, 2015 Field Corn/ DKC-62-98	124 126	- 5	BBCH 89 BBCH 89	13	Stover	BBCH 89	4.4, 3.3 (3.9)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Growth stage at harvest	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 18 Stilwell, KS, United States, 2015 Field Corn/ PK 110-10RR	126 122	- 5	BBCH 89 BBCH 89	13	Stover	BBCH 89	3.5, 4.0 (3.7)	
Trial 19 Atlantic, IA, United States, 2015 Field Corn/ DKC 62-98 RIB	125 126	- 5	BBCH 89 BBCH 89	14	Stover	BBCH 89	1.6, 1.6 (1.6)	
Trial 20 Geneva, MN, United States, 2015 Field Corn/ Pioneer 19526AMXT	123 122	- 5	BBCH 87 BBCH 87	14	Stover	BBCH 89	2.6, 3.4 (3.0)	
Trial 21 Uvalde, TX, United States, 2015 Field Corn/ DKC 64-69	123 123	- 5	BBCH 87 BBCH 87	14	Stover	BBCH 89	4.6, 3.8 (4.2)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

Almond hulls

Six residue trials were conducted on almonds the United States in 2017.

The trials were conducted with three soil directed applications, at an individual rate of 0.757 g ai/ha, and with three foliar sprays, at an individual application rate of 124 g ai/ha.

An adjuvant was used in the trials. The label states that for best results, use an adjuvant helps increase coverage, penetration and thus performance.

Samples of almond hulls were collected -0–15 days after the last application. Samples were immediately frozen and stored at ≤ -18 ºC for up to 319 days prior to analysis.

Residues of indoxacarb and its R enantiomer were determined using analytical method DuPont 36189. Procedural recoveries were conducted at fortification levels of 0.01, 0.1, 1 and 5 mg/kg with recoveries in the range of 86–104 percent.

The trials conducted in Fresno, CA were conducted at different trial sites and are therefore regarded as independent trials.

A summary of the trials is outlined in Table 17.

Table 17 Residues of indoxacarb in almond hulls from supervised trials conducted in the United States

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
GAP United States	**3 × 123**	**9 - 26**	**7**	**-**	**5**	**-**	**-**	FMC-49392, Revision No. 1
Trial 01 Fresno, CA, United States, 2017 Almond/ non-pareil	0.7578 0.7578 0.7578 123 123 123	- - - 9 9 9	- 30 30 - 7 7	BBCH 78 BBCH 82 BBCH 87 BBCH 84 BBCH 84 BBCH 87	5	Hulls	1.9, 2.1 (2)	
Trial 02 Madera, CA, United States, 2017 Almond/ Mission	0.7578 0.7578 0.7578 124 125 124	- - - 147 148 147	- 31 30 - 7 7	BBCH 77 BBCH 81 BBCH 88 BBCH 87 BBCH 87 BBCH 88	5	Hulls	3.8. 3.7 (3.8)	
Trial 03 Hickman, CA, United States, 2017 Almond/ Non-pareil	0.7626 0.7626 0.7626 123 125 124	- - - 8 8 8	- 30 30 - 7 7	BBCH 79 BBCH 81 BBCH 89 BBCH 85 BBCH 85 BBCH 89	5	Hulls	1.9, 1.6 (1.8)	
Trial 04 Fresno, CA, United States, 2017 Almond/ Monterey	0.7622 0.7678 0.7523 123 121 123	- - - 23 22 22	- 28 32 - 7 7	Hull split Hull split Hull split Hull split Hull split Hull split	4	Hulls	2.8, 2.9 (2.8)	

Trial identification Location, Country Year, Crop/Variety	Rate (g ai/ha)	Rate (g ai/hL)	Re-treatment Interval (days)	Growth stage at application	DALA (days)	Crop part	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Report Number
Trial 05	0.7607	-	-	65 DBCH	-0	Hulls	0.21, 0.80 (0.50)	
	0.7596	-	31	35 DBCH				
Porterville, CA, United States, 2017	0.7488	-	30	BBCH 89	0		0.58, 0.78 (0.68)	
	122	16	-	BBCH 81				
	123	16	7	BBCH 87				
Almond/ Butte	123	16	7	BBCH 89	6		0.34, 0.23 (0.29)	
					9		2.6, 2.4 (2.5)	
					15		1.2, 1.3 (1.2)	
Trial 06	0.7877	-	-	BBCH 78	-1	Hulls	2.4, 1.9 (2.2)	
	0.7613	-	29	BBCH 85				
Live Oak, CA, United States, 2017	0.7585	-	31	BBCH 89	0		2.7, 2.3 (2.5)	
	122	24	-	BBCH 87	5		3.5, 2.5 (3.0)	
	124	25	7	BBCH 88				
Almond/ Non-Pareil	124	25	7	BBCH 88	9		2.2, 2.4 (2.3)	
					14		2.3, 2.4 (2.4)	

Notes:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

FATE OF RESIDUES DURING PROCESSING

The nature of the residue on processing was considered by the JMPR in 2005. It was concluded that indoxacarb was generally stable on hydrolysis. Approximately 7–30 percent was lost during baking and boiling conditions with the products of hydrolysis being minor and polar in nature.

The current Meeting received information on procedures simulating commercial processing practices for maize. Samples of grain were taken from trials conducted at a high and low application rate.

High application rate

Samples were taken from three trials. The trials were conducted in the United States in 2015 at an application rate of 2 × 616 g ai/ha with a RTI of 5 days. Grain was collected 14 days after the final application and processed into flour, starch, grits, dry milling meal, dry milling oil, wet milling meal and wet milling oil.

Production of processed fractions

After the removal from the freezer, representative whole grain samples were collected from the bulk samples before processing and placed into frozen storage. The moisture content was determined, and if this was > 15 percent the samples were dried at 54–71 °C until the moisture content was < 15 percent. Samples were then cleaned by aspiration and screening, with light impurities removed. After aspiration,

samples were screened in a Enhance 2 screen cleaner to separate large and small foreign particles (screenings) from the cleaned grain.

Dry milling

Samples of whole grain were moisture conditioned to 20–22 percent and tempered for approximately 2 hours. After tempering, the samples were fed into a disc mill to crack the kernels. The corn stock from the mill was dried for around 30 minutes at 54–71 °C. The dried corn stock was screened/sieved to separate bran, germ, grits, meal and flour. Where necessary, germ was milled, screened and/or aspirated to remove endosperm and hull material.

Germ fractions were dried at 54–71 °C. Samples of grits, meal and flour were collected and stored frozen prior to analysis.

To produce crude oil, germ material was heated to 71–79 °C and then flaked using a flaking roll. The flaked material was placed in a stainless-steel extractor and submerged in hexane at 48–60 °C. After 30 minutes, hexane was drained from the crude oil and the extraction process repeated two more times with fresh hexane. Following the third extraction and draining, hexane was removed from the spent flakes using ambient air. The hexane was removed from the crude oil by vacuum evaporation followed by heating to 91–96 °C. The crude oil was collected for refining.

The crude oil was subject to alkali refining. Based on the free fatty acid content of the crude oil, a specified amount of sodium hydroxide was added. The mixture was then heated in a water bath for 15 minutes at 20–24 °C and then for 12 minutes at 63–67 °C. Neutralized oil and soapstock were separated by centrifugation.

The alkali refined oil was then subjected to bleaching. The refined oil was heated to 40–50 °C and an activated bleaching earth added. The solution was then placed under a vacuum and stirred. The temperature was increased to 85–100 °C and held for 10–15 minutes after which the solution was allowed to cool. The bleaching oil was filtered and then placed in a steam bath at 220–230 °C for approximately 30 minutes under vacuum. The oil was allowed to cool and 0.5 percent citric acid solution was added. The resulting fraction (bleached-deodorized oil–refined oil) was collected and stored frozen prior to analysis.

Wet milling

A representative sample of grain was steeped in 48–54 °C water containing 0.1–0.2 percent sulfur dioxide for 22- 48 hours. At the end of this process, the water was drained, and a representative fraction collected and stored frozen prior to analysis. The steeped grain was passed through a disc mill and a majority of the germ and hull was then removed by a hydroclone filter. Germ and hulls were dried at 74–91 °C to achieve a final moisture content of between 5–10 percent. After drying, the germ and hulls were separated using aspiration and screening.

Corn stock, collected from the disc mill, was screened, with the bran (hull material) collected and discarded. The process water (starch and gluten combined) passing through the screen was separated into starch and gluten by centrifugation. The starch was dried in an oven at 54–71 °C until the moisture content was less than 15 percent. The starch was then stored frozen until analysis.

Germ samples were moisture conditioned to 12 percent, heated to 88–104 °C, flaked using a flaking roll and then pressed to liberate crude oil along with press-cake (with residual crude oil).

The press-cake was placed in a stainless-steel extractor and submerged in hexane at 49–60 °C. After 30 minutes hexane was drained from the crude oil and the extraction process repeated two more

times with fresh hexane. Following the third extraction and draining, residual hexane was removed with ambient air. The resulting solvent extracted press-cake was collected and stored frozen prior to analysis.

Hexane was removed from the crude oil by vacuum evaporation and heating the samples to 91–96 °C. The crude oil was then subjected to alkali refining, bleaching and deodorizing using the same process as outlined for dry milling. The resulting refined oil was collected and stored frozen prior to analysis.

Low application rate

Samples were taken from three trial sites where the application rate was 2 × 123 g ai/ha (trials 7, 12 and 21 summarized in Table 10). Samples of grain were collected from the bulk samples and stored and then analysed separately, from the field trial samples, prior to processing. The grain samples were processed into aspirated grain fractions.

Generation of Aspirated Grain Fraction (AGF)

To generate aspirated grain fractions, the moisture content of the samples was determined and if it was > 13 percent, the samples were dried at 43–57 °C until the moisture content was < 13 percent. Each sample was placed in a dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. As the samples moved in the system (120 minutes), aspiration was used to remove light impurities (grain dust). Light impurities were categorized using sieves of 2360 microns, 200 microns, 1180 microns, 850 microns and 425 microns. The samples passing through the 2360 micron sieve were combined to produce the AGF sample.

Storage stability and analysis

Processed fractions were stored for up to 28 days prior to analysis. Samples were analysed using analytical method DuPont 36189. Procedural recoveries were conducted at fortification levels of 0.01 and 0.1 for all processed fractions. For aspirated grain, procedural recoveries were also conducted at 0.3 mg/kg. The procedural recoveries obtained were: aspirated grain (67–108 percent), flour (89–104 percent), starch (92–111 percent), grits (84–93 percent), dry milling meal (85–103 percent), dry milling oil (88–109 percent), wet milling meal (94–108 percent) and wet milling oil (98–112 percent).

The residues in the treated maize grain and processed fractions are outlined in tables 18 and 19.

Table 18 Residues of indoxacarb in maize grain and processed fractions

Country (Region) Crop (Variety)	Application Rate (g ai/ha)	**DALA** (days)	Commodity	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Processing Factor (Pf)
Richland, IA, United States Field Corn/ P0506AM	2 × 622	14	Grain	0.01, 0.012, 0.014 (0.012)	-
			Flour	0.032	2.7
			Starch	<0.01	<0.83
			Grits	<0.01	<0.83
			Dry milling meal	0.012	1
			Dry milling oil	0.022	1.8
			Wet milling meal	<0.01	<0.83
			Wet milling oil	0.023	1.9
York, NE, United States Field Corn/ P0876HR	614 613	13	Grain	0.1, 0.15, 0.15 (0.14)	-
			Flour	0.10	0.71
			Starch	<0.01	< 0.07
			Grits	<0.01	< 0.07
			Dry milling meal	<0.01	< 0.07
			Dry milling oil	0.016	0.11
			Wet milling meal	<0.01	< 0.07
			Wet milling oil	0.014	0.1
Uvalde, TX, United States Field Corn/ DKC 64-69	611 619	14	Grain	< 0.01	-
			Flour	<0.01	-
			Starch	<0.01	-
			Grits	<0.01	-
			Dry milling meal	<0.01	-
			Dry milling oil	<0.01	-
			Wet milling meal	<0.01	-
			Wet milling oil	<0.01	-

Notes:

Pf = residue level in processed commodity (mg/kg) ÷ residue level in RAC (mg/kg).

Table 19 Residues of indoxacarb in aspirated maize grain

Country (Region) Year Crop (Variety)	Application Rate (g ai/ha)	**DALA** (days)	Commodity	Residue level (sum of indoxacarb and its R enantiomer) (mg/kg)	Processing Factor (Pf)
Trial 07 Richland, IA, United States, 2015 Field Corn/ **P0506AM**	**2 × 124**	14	Grain AGF	<0.01, <0.01 (0.01) **0.21**	- **>21**
Trial 12 Carlyle, IL, United States, 2015 Field Corn/ **FS 63SV1 RIB**	**123** **125**	14	Grain AGF	<0.01, <0.01, (<0.01) **0.14**	- **>14**
Trial 21 Uvalde, TX, United States, 2015 Field Corn/ **DKC 64-69**	**2 × 123**	14	Grain AGF	<0.01, <0.01, (<0.01) **0.078**	- **>7.8**

Notes:

AGF: Aspirated grain fraction

Pf = residue level in processed commodity (mg/kg) ÷ residue level in RAC (mg/kg)

APPRAISAL

Indoxacarb is an indeno-oxadiazine insecticide that is used for the control of lepidopteran and other insect pests. Indoxacarb was first evaluated by the 2005 JMPR when an ADI of 0–0.01 mg/kg bw and an ARfD of 0.1 mg/kg bw were established. The residue definition for compliance with the MRL for plant and animal commodities and dietary risk assessment for plant commodities is the sum of indoxacarb and its R enantiomer.

The residue definition for dietary risk assessment for animal commodities is: *sum of indoxacarb, its R enantiomer and methyl 7-chloro-2,5-dihydro-2-[[[4-(trifluoromethoxy)phenyl]amino] carbonyl]indeno[1,2-e][1,3,4]oxadiazine-4a(3H)- carboxylate, expressed as indoxacarb (IN-JT333*).

The residue is fat soluble.

Indoxacarb has been evaluated for additional uses in 2007, 2009 and 2013. At the Fifty-first Session of the CCPR, indoxacarb was scheduled for the evaluation by the current Meeting for additional uses on bush berries, okra, beans with pods, pulses, beetroot, maize and tree nuts. The present Meeting

received information on residue analysis, storage stability, use pattern, supervised field trials and processing.

Methods of analysis

The Meeting received information on analytical method DuPont 36189, used in the supervised residue trials for maize and tree nuts. The method was also used to determine residues in the processed fractions of maize. The method involved extraction with hexane-acetonitrile with final determination achieved using LC-MS/MS. The Meeting concluded that the method was validated for dry commodities and crops of a high oil content with an LOQ of 0.01 mg/kg for the sum of indoxacarb and its R enantiomer.

The meeting also received additional validation data to support methods, previously considered by the JMPR, used in the residue trials and new storage stability studies. The Meeting concluded that the methods were validated and are suitable to measures indoxacarb and its R enantiomer in blueberries, snap beans (beans with pods), dry beans, beetroots and maize.

For peppers, no additional validation data were provided for analytical method AMR 3493-95. The Meeting noted that the JMPR previously concluded that this method was validated at an LOQ of 0.02 mg/kg for apple and that procedural recovery data generated in the trials for peppers (bell and non-bell) was acceptable. The Meeting concluded that the method was sufficiently validated for peppers with an LOQ of 0.02 mg/kg.

Stability of pesticide residues in stored analytical samples

The Meeting noted that the 2005 and 2009 JMPR considered storage stability data for a range of crops. Based on the overall data available, the Meeting concluded that indoxacarb (and its R enantiomer) are stable for at least 24 months (high water crops), 11 months (high protein), 18 (high starch) and 11 months (high oil crops).

This Meeting received additional information on storage stability of indoxacarb (and its R enantiomer) in blueberries, peppers, snap beans, dry beans, beetroots and maize. The Meeting agreed that the data were sufficient to support the storage stability of indoxacarb (and its R isomer) for at least 17 months (blueberries), 11 months (peppers), 7 months (fresh beans), 7 months (dry beans), 18 months (beetroots) and 13 months (maize grain).

Results of supervised residue trials on crops

Supervised trials were available for the use of indoxacarb on blueberries, peppers, snap beans, dry beans, beetroots, maize grain and tree nuts. The residue levels reported are the sum of indoxacarb and its R enantiomer.

Bushberries

Blueberries

The critical GAP for bushberries is from the United States and consists of 4 foliar applications each at 123 g ai/ha with a re-treatment interval (RTI) of 7 days and a PHI of 7 days.

Residues of indoxacarb in independent trials, conducted in the United States, approximating the critical GAP were (n= 11): 0.25, 0.28, 0.35, 0.37, 0.47, 0.58, 0.58, 0.77, 0.81, 0.84 and 1.04 mg/kg.

Noting that the registered use is on bushberries and blueberry is a representative of this subgroup the Meeting decided to make a recommendation for the Subgroup of bushberries. The Meeting

estimated a maximum residue level of 2 mg/kg, a STMR of 0.58 mg/kg and a HR of 1.04 mg/kg for indoxacarb in the Bushberries subgroup.

Legume vegetables: Beans with pods

Snap beans/ common beans with pods

The critical GAP for beans with pods, except soya beans is from the United States and consists of 4 foliar applications each at 123 g ai/ha with a RTI of 7 days and a PHI of 3 days.

Residues of indoxacarb in independent trials approximating the critical GAP were (n= 8): 0.11, 0.12, 0.13, 0.15, 0.17, 0.17, 0.20 and 0.59 mg/kg.

The Meeting noted that the registered use is on beans with pods, except soya beans. Therefore, the Meeting estimated a maximum residue level of 0.9 mg/kg, a STMR of 0.16 mg/kg and a HR of 0.59 mg/kg for indoxacarb in the subgroup of Beans with pods, except soya beans.

Pulses

Dry beans

The critical GAP for dry beans, except soya beans is from the United States and consists of 4 foliar applications each at 123 g ai/ha with a RTI of 7 days and a PHI of 7 days.

Residues of indoxacarb in independent trials, conducted in the United States, approximating the critical GAP were (n= 12): < 0.01 (7), 0.01, 0.011, 0.022, 0.03 and 0.07 mg/kg.

The Meeting estimated a maximum residue level of 0.09 mg/kg, a STMR of 0.01 mg/kg for indoxacarb in dry bean.

The Meeting noted that the 2005 JMPR estimated maximum residue levels of 0.5 mg/kg for soya bean (dry) and 0.2 mg/kg for mung bean (dry). The 2009 JMPR estimated a maximum residue level of 0.1 mg/kg for cowpea (dry). On the basis of the previous recommendations and the GAP considered by the current Meeting for dry beans being different to the GAPs previously considered for cowpea, mung beans and soya bean, the current Meeting estimated a maximum residue level of 0.09 mg/kg and a STMR of 0.01 mg/kg for indoxacarb in the subgroup of dry beans, except cowpea, mung bean and soya bean.

Root and tuber vegetables

Beetroot

The critical GAP for beetroot is from the United States and consists of 4 foliar applications each at 123 g ai/ha with a RTI of 3 days and a PHI of 7 days.

Residues of indoxacarb, in independent trials, conducted in the United States, approximating the critical GAP were (n= 5): 0.12, 0.13, 0.18, 0.19 and 0.22 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg, a STMR of 0.18 mg/kg and a HR of 0.22 mg/kg for indoxacarb in beetroots.

Cereal grains

Maize

The critical GAP for maize (field and popcorn) is from the United States and consists of 2 applications each at 123 g ai/ha with a RTI of 5 days and a PHI of 14 days.

Residues of indoxacarb in independent trials, conducted in the United States, approximating the critical GAP on field corn were (n= 21): < 0.01 (19), 0.011 and 0.012 mg/kg.

Noting that the registered use is on maize (field) and maize (popcorn) the Meeting decided to make a recommendation for the Subgroup of maize cereals. The Meeting estimated a maximum residue level of 0.015 mg/kg and a STMR of 0.01 mg/kg for indoxacarb in the subgroup of maize cereals.

Tree nuts

The critical GAP for tree nuts is from the United States and consists of 3 foliar applications each at 123 g ai/ha with a RTI of 7 days and a PHI of 5 days. The residue trials were conducted with 3 × 0.757 kg ai/ha, applied as a soil treatment, followed by 3 foliar applications at 124 g ai/ha with the RTI and PHI reflecting the GAP. The Meeting considered that the soil applications would not contribute significantly to residue levels at harvest and the trials could be used to support the GAP.

Almond

Six trials were conducted in the United States at a rate of 3 × 0.757 g ai/ha (applied as a soil treatment) along with 3 ×124 g ai/ha applied as a foliar treatment.

Residues of indoxacarb in independent trials approximating the critical GAP were (n=6): < 0.01, 0.011, 0.013, 0.016, 0.022 and 0.023 mg/kg

Pecan

Six trials were conducted in the United States at a rate of 3 × 0.757 g ai/ha (applied as a soil treatment) along with 3 × 124 g ai/ha applied as a foliar treatment

Residues of indoxacarb in independent trials approximating the critical GAP were (n=6): < 0.01, 0.01, 0.019, 0.028, 0.033 and 0.034 mg/kg

Pistachios

Residues of indoxacarb in independent trials approximating the critical GAP were (n=5): < 0.01 (2), 0.011, 0.018 and 0.045 mg/kg (highest individual value 0.046 mg/kg).

Summary–Tree nuts

Nothing that the median residues of indoxacarb for each tree nut type are within a 5- fold range and that there is no evidence of a difference in the residue populations for the different tree nuts by the Kruskal-Wallis test, the Meeting decided to make a recommendation for the Group of tree nuts based on the combined data.

Residues of indoxacarb in independent trials approximating the critical GAP were (n= 17): < 0.01 (5), 0.011 (3), 0.013, 0.016, 0.019, 0.022, 0.023, 0.028, 0.033, 0.034 and 0.045 mg/kg (highest individual value 0.046 mg/kg).

The Meeting estimated a maximum residue level of 0.07 mg/kg, a STMR of 0.013 mg/kg and a HR of 0.046 mg/kg for indoxacarb in tree nuts.

Residues in animal feed items

Bean forage

The critical GAP for bean forage is from the United States and consists of 4 applications each at 123 g ai/ha with a RTI of 7 days and a PHI of 3 days.

Residues of indoxacarb in independent trials, conducted in the United States, approximating the critical GAP were (n= 8): 6.8, 9, 10 (2), 12, 16.5, 17.4 and 32 mg/kg.

The Meeting estimated a median residue of 11 mg/kg and a highest residue of 32 mg/kg for indoxacarb in bean forage.

Maize forage

The critical GAP for maize forage is from the United States and consists of 2 applications each at 123 g ai/ha with a RTI of 5 days and a PHI of 1 day.

Residues of indoxacarb in independent trials, conducted in the United States, approximating the critical GAP were (n= 21): 0.78, 1.1, 1.2, 1.3 (3), 1.5 (4), 1.6, 1.7, 1.8, 2.0, 2.3 (2), 2.6, 2.7 (2), 2.8 and 3.4 mg/kg.

The Meeting estimated a median residue of 1.6 mg/kg (as received) and a highest residue of 3.4 mg/kg (as received) for indoxacarb in maize forage.

Maize stover

The critical GAP for maize fodder is from the United States and consists of 2 applications each at 123 g ai/ha with a RTI of 5 days and a PHI of 14 day.

Residues of indoxacarb in independent trials, conducted in the United States, approximating the critical GAP were (n= 21): 0.96, 1.1 (2), 1.3 (2), 1.6, 1.7 (2), 1.9, 2.4, 2.8, 3.0, 3.5, 3.7 (2), 3.8, 3.9, 4.2, 5.1, 5.9 and 9.1 mg/kg.

The Meeting noted that the residue levels in maize stover were covered by the GAP considered by the 2005 JMPR for sweet corn stover with a median residue of 3.7 mg/kg and a highest residue of 9.8 mg/kg. The Meeting noted that the previous recommendation was for maize fodder (dry). Therefore, the Meeting estimated a Maximum residue level of 25 mg/kg for maize stover based on the GAP for sweet corn stover and withdrew the previous recommendation of 25 mg/kg (dry) for maize fodder (dry).

Almond hulls

The critical GAP for tree nuts is from the United States and consists of 3 applications each at 123 g ai/ha with a RTI of 7 days and a PHI of 5 days.

Six trials were conducted in the United States at a rate of 3 × 0.757 g ai/ha (applied as a soil treatment) along with 3 × 124 g ai/ha applied as a foliar treatment. The residue trials were conducted with 3 × 0.757 kg ai/ha , applied as a soil treatment, followed by 3 foliar applications at 124 g ai/ha with the RTI and PHI reflecting the GAP. The Meeting considered that the soil applications would not contribute significantly to residue levels at harvest and the trials could be used to support the GAP.

Residues of indoxacarb in independent trials, conducted in the United States, approximating the critical GAP were (n= 6): 1.8, 2, 2.5, 2.8, 3.0 and 3.8 mg/kg.

The Meeting estimated a median residue of 2.65 mg/kg, a highest residue of 3.8 mg/kg and a maximum residue level of 9 mg/kg (dry weight) in almond hulls.

Fate of residues during processing

The Meeting received new information on the fate of indoxacarb (and its R enantiomer) residues during processing in maize. The Meeting noted that the individual processing factors for each processed fraction were significantly different and therefore decided not to estimate processing factors, MRLs, STMR-P and HR-P for processed commodities.

Residues in animal commodities

Farm animal feeding studies

The 2005 JMPR considered a lactating dairy cow feeding study and the 2009 JMPR considered a laying hen feeding study.

Farm animal dietary burden

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on the sum residue of indoxacarb and its R enantiomer and the feed items considered by the current Meeting and evaluated by the JMPR in 2005, 2007 and 2009. Some processed and forage commodities do not appear in the Recommendations Table (because no maximum residue level is needed), but they are used in estimating livestock dietary burdens.

The dietary burdens, estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO manual, are presented in Annex 6 of the 2021 JMPR Report and summarised below.

Table 19 Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden: indoxacarb (and its R enantiomer) ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	8.769	3.503	23.36	12.4	72.06❶	25.26❸	4.329	1.629
Dairy cattle	21.78	8.273	33.45	11.89	71.06❷	24.60❹	15.01	6.007
Poultry – broiler	0.094	0.094	0.04	0.037	0.06	0.06	0.009	0.099
Poultry – layer	0.094	0.094	0.705❺	0.182❻	0.06	0.06	0.022	0.012

Notes:

❶ Highest maximum beef cattle dietary burden suitable for MRL estimates for mammalian tissues

❷ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

❸Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

❺Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues and eggs.

❻Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Animal commodity maximum residue levels

Cattle

Table 20 Animal commodity maximum residue levels for cattle

	Feed Level (ppm) for milk and cream residues	Total residues (mg /kg) in milk	Total residues (mg /kg) in cream	Feed Level (ppm) for tissue residues	Total residues † (mg /kg)			
					Muscle	Liver	Kidney	Fat
	MRL Determination (beef or dairy cattle) - based on parent + R-enantiomer							
Feeding Study	75	0.163	2.2	75	0.093	0.019	0.049	1.9
Dietary burden and estimate of MRL	71.1	0.155	2.1	72.1	0.089	0.018	0.047	1.83

Notes:

† Total residue determined as indoxacarb and its R enantiomer in accordance with the residue definition for compliance with MRLs.

Table 21 Animal commodity highest and median residues for cattle

	Feed Level (ppm) for milk and cream residues	Total residues (mg /kg) in milk	Total residues (mg /kg) in cream	Feed Level (ppm) for tissue residues	Total residues† (mg /kg)			
					Muscle	Liver	Kidney	Fat
	HR Determination (beef or dairy cattle) based on parent + R + IN-JT333, expressed as parent							
Feeding Study	75	0.20	2.3	75	0.103	0.029	0.059	1.98
Dietary burden and estimate of highest residue	71.1	0.19	2.2	72.1	0.099	0.028	0.057	1.90
	STMR Determination (beef or dairy cattle) based on parent + R + IN-JT333, expressed as parent							
Feeding Study	75	0.173	2.1	75	0.076	0.028	0.049	1.95
	22.5	0.062	0.589	22.5	< 0.01	< 0.01 **	0.027	0.48
Dietary burden and estimate of median residue	24.6	0.068	0.68	25.3	0.026	< 0.01	0.03	0.66

Notes:

† Total residue determined as indoxacarb, its R enantiomer and IN-JT333, expressed as indoxacarb in accordance with the residue definition for risk assessment.

** Residues < 0.01 mg/kg except for one sample.

Based on indoxacarb and its R enantiomer in milk, the Meeting estimated a maximum residue of 0.2 mg/kg for indoxacarb for milk, replacing the previous recommendation of 0.1 mg/kg. For cream, the Meeting estimated a HR of 2.2 mg/kg based on the sum of indoxacarb and its R enantomer. On the assumption of 40 percent milk fat in cream, the Meeting estimated a maximum residue level of 6 mg/kg for milk fats, replacing the previous recommendation of 2 mg/kg.

Based on indoxacarb and its R enantiomer in tissues, the Meeting estimated maximum residue levels of 2 mg/kg for mammalian meat (fat) and of 0.05 mg/kg for edible offal (mammalian), which

confirms the previous recommendations made by the 2009 JMPR. For fat the Meeting estimated a maximum residue level of 2 mg/kg.

Based on the mean estimated residues of indoxacarb, its R isomer and the metabolite IN-JT333 in milk and tissues, the Meeting estimated STMRs of 0.07 for milk, of 0.15 mg/kg for mammalian meat, of 0.03 mg/kg for edible offal (mammalian) and of 0.66 mg/kg for mammalian fat. The Meeting estimated an STMR of 0.68 mg/kg for cream and on the assumption of a fat content of 40 percent estimated an STMR of 1.7 mg/kg for milk fats.

Based on the highest estimated residues of indoxacarb, its R isomer and the metabolite IN-JT333 in tissues, the Meeting estimated HR of 0.46 mg/kg for mammalian meat, of 0.06 mg/kg for edible offal (mammalian) and of 1.90 mg/kg for mammalian fat.

Poultry

The new feed items considered by the current Meeting did not contribute significantly to the dietary burden of poultry and the Meeting confirmed its previous recommendations.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *the sum of indoxacarb and its R enantiomer*

Definition of the residue for compliance with the MRL for animal commodities: *the sum of indoxacarb and its R enantiomer*

Definition of the residue for dietary risk assessment for animal commodities: *sum of indoxacarb, its R enantiomer and methyl 7-chloro-2,5-dihydro-2-[[[4- (trifluoromethoxy)phenyl]amino]carbonyl]indeno[1,2-e][1,3,4]oxadiazine-4a(3H)- carboxylate (IN-JT333), expressed as indoxacarb*.

The residue is fat-soluble.

Table 22 Residue levels suitable for establishing maximum residue limits and for IEDI and IESTI assessments

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
AM 0660	Almond hulls	9 (dw)	-	2.65	3.8
FB 2006	Bushberries	2	-	0.58	1.04
VD 2065	Beans, dry (except cowpea, mung bean and soya bean), subgroup	0.09	-	0.01	-
VP 2060	Beans with pods (except soya bean), subgroup	0.9	-	0.16	0.59
VR 0574	Beetroot	0.5	-	0.18	0.22
MO 0105	Edible offal (Mammalian)	0.05	0.05	0.03	0.06
MF 0100	Mammalian fats (except milk fats)	2	-	0.66	1.9
MM 0095	Meat (from mammals other than marine mammals)	2 (fat)	2 (fat)	0.15	0.46
GC 2091	Maize cereals (subgroup)	0.015	-	0.01	
AS 0645	Maize fodder (dry)	W	25	-	
AS 3558	Maize, stover	25 (dw)	-	Median: 3.7	Highest: 9.8
ML 0106	Milks	0.2	0.1	0.07	

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FM 0183	Milk fats	6	2	1.7	
TN 0085	Tree nuts	0.07	-	0.013	0.046
For dietary risk assessment and/or dietary burden calculations					
AL 1030	Bean forage	-	-	11(ar)	32(ar)
AS 0645	Maize forage	-	-	1.6(ar)	3.4(ar)

Notes:

(ar) – as received; (dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for indoxacarb is 0–0.01 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for indoxacarb were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 JMPR Report.

The IEDIs ranged from 2–20 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of indoxacarb from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for indoxacarb is 0.1 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for indoxacarb were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the current Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs varied from 0–20 percent of the ARfD for children and 0–10 percent of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of indoxacarb from uses considered by the current Meeting is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
AMR 3735-96.	Adams, G.M., Klemens, F.K	1997	Magnitude and decline of residues of DPX-KN128 and IN-KN127 in peppers following application of DPX-MP062 Experimental Insecticide at maximum label rates. McKenzie Laboratories Inc. Report No. AMR 3735-96. GLP: Yes. Unpublished
IR-4 PR No 08870	Corley, J	2007	Indoxacarb: Magnitude of the residue on beet (garden). IR-4 Project Rutgers, The State University of New Jersey. Report No. IR-4 PR No 08870. GLP: Yes. Unpublished
IR-4 PR No. 08574	Corley, J	2009	Magnitude of the residue on bean (snap). IR-4 Project Rutgers, The State University of New Jersey. Report No. IR-4 PR No. 08574. GLP: Yes. Unpublished.
Report No. IR-4 PR No. 09669	Corley, J	2011	Indoxacarb: Magnitude of the residue on bean (dry) Amended final report. IR-4 North Central Region Laboratory, Rutgers University. Report No. IR-4 PR No. 09669. GLP: Yes. Unpublished.

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
DuPont-44492	Dorsey, S	2016	Magnitude and decline of residues of DPX-KN128 (indoxacarb) in field corn and magnitude of residues of DPX-KN128 in field corn processed fractions following applications of DPX-KN128 150 g/L EC - United States, 2015. ABC Laboratories, Inc. (Missouri). Report No. DuPont-44492. GLP: Yes. Unpublished.
IR-4 PR No 07038	Dorschner, K	2007	Indoxacarb: Magnitude of the residue on blueberry. IR-4 Project Rutgers, The State University of New Jersey. Report No. IR-4 PR No 07038. GLP: Yes. Unpublished.
FMC-49392	Dorsey, S	2018	Magnitude and decline of indoxacarb (DPX-KN128) residues in tree nuts following foliar and soil applications of indoxacarb-containing formulations - U.S., 2017. ABC Laboratories, Inc. (Missouri). Report No. FMC-49392, Revision No. 1. GLP: Yes. Unpublished.
Dupont-6006	Guinivan, R., Kennedy, C. M. and Enriquez, M. A.	2003	Combined decline and magnitude of residues of DPX-KN128 (indoxacarb) togther with IN-KN127 in Maize (green plant and grain) following applications of DPX-MP062 30WG–Europe, season 2001. Report No. Dupont-6006. GLP: Yes. Unpublished.

INPYRFLUXAM (329)

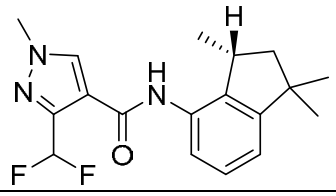
The first draft was prepared by Dr Chris Anagnostopoulos, Benaki Phytopathological Institute, Greece

EXPLANATION

Inpyrfluxam (S-2399) is a broad spectrum fungicide belonging to the succinate dehydrogenase inhibitor (SDHI) group of fungicides which have a mode of action that inhibits energy production processes in pathogenic fungi.

Inpyrfluxam was scheduled at the Fifty-second Session of the CCPR (2022), for toxicology and residue evaluation as a new compound by the 2020 JMPR. The Meeting received information on identity, physical-chemical properties, fate of residues in the environment, plant and animal metabolism, rotational crops, analytical methods, storage stability, use patterns, residues resulting from supervised trials, fate of residues during processing and livestock feeding studies.

IDENTITY

ISO common name:	Inpyrfluxam
IUPAC name:	3-(Difluoromethyl)-1-methyl-*N*-[(3*R*)-1,1,3-trimethyl-2,3-dihydro-1*H*-inden-4-yl]-1*H*-pyrazole-4-carboxamide
Chemical Abstract name:	3-(Difluoromethyl)-*N*-[(3*R*)-2,3-dihydro-1,1,3-trimethyl-1*H*-inden-4-yl]-1-methyl-1*H*-pyrazole-4-carboxamide
Other names and codes	S-2399
CAS No.:	1352994-67-2
CIPAC No.	1005
Synonyms:	No synonyms
Structural Formula:	
Molecular Formula:	$C_{18}H_{21}F_2N_3O$
Molecular Weight:	333.38 g/mol

Physical and chemical properties

Table 1 Physical and chemical properties of Inpyrfluxam

Property	Guideline and method	Test material specification and purity	Findings	Reference/ Remarks
Pure active ingredient				
Appearance	visual	*Inpyrfluxam* PAI (99.9%)	Beige granule	Butler, R. E., 2015; TPP-0006
	Munsell® Colour System		Colour: 10YR 9/2 R	
	odour		no discernible odour	
Vapour pressure	OECD 104, EC A4		3.8×10^{-8} Pa at 20 °C and 1.2×10^{-7} Pa at 25 °C	Butler, R. E., 2014; TPP-0003
Octanol-water partition coefficient	OECD 117 EC A8		4.45×10^3 at 25 °C (pH 7.1-7.3), log P_{ow} = 3.65	O'Connor, B. J., 2013; TPP-0002
Solubility (in water)	OECD 105, EC A6	*Inpyrfluxam* PAI (99.9%)	0.0164 g/L at 20 °C	O'Connor, B. J., 2013; TPP-0001
Specific gravity	OECD 109	*Inpyrfluxam* PAI	1.24×10^3 kg/m^3 at 20.0 ±	Walker, J. A., 2016; TPP-0010

Property	Guideline and method	Test material specification and purity	Findings	Reference/ Remarks
(density)		(99.9%)	1.0 °C	
Hydrolysis in sterile water in the dark	OCSPP 835.2120 OECD 111	pyrazolyl-4-^{14}C Inpyrfluxam	Hydrolytically stable at pH 4, 7 and 9 (50 °C). no isomerisation occurred.	Freedlander, S., 2016. TPM-0030
Photolysis in sterile water	(Protocol: aqueous phosphate buffer at pH 7 with <1% acetonitrile co-solvent in quartz tubes. The test solutions were irradiated at 25 ± 1 °C with a xenon lamp for up to 15 days.)	pyrazolyl-4-^{14}C Inpyrfluxam	The half-lives of inpyrfluxam could not be correctly calculated since inpyrfluxam was stable in the presence and in the absence of light irradiation but they were estimated to be over one year.	Ponte, M., 2015. TPM-0008
Dissociation constant	None	*Inpyrfluxam* PAI (99.9%)	The UV-visible spectra of inpyrfluxam recorded at pH values of 1, 7 and 12 contain no significant differences. This demonstrates that inpyrfluxam displays no dissociative activity in the pH range 1 to 12.	Butler, R. E., 2014; TPP-0005
Melting point	OECD 102 Melt microscope	*Inpyrfluxam* PAI (99.9%)	104 °C	Walker, J. A., 2016; TPP-0010
Boiling point	Siwoloboff method OECD 103	*Inpyrfluxam* PAI (99.9%)	Not determined. Decomposed at 237 °C at 100.4 to 101.1 kPa.	Walker, J. A., 2016; TPP-0010
Surface Tension	Ring method OECD 115	*Inpyrfluxam* PAI (99.9%)	not surface-active (60.4 mN/m at 21.3 ± 0.5 °C)	Walker, J. A., 2016; TPP-0010
Thermal Stability	DSC method OECD 113	*Inpyrfluxam* PAI (99.9%)	opposed to oxidative. Decomposed at 250°C	Walker, J. A., 2016; TPP-0010
Technical substance (TGAI):				
minimum purity		*Inpyrfluxam* TGAI (95.0%)	94% w/w	
Appearance	visual	*Inpyrfluxam* TGAI (95.0%)	White powder	Woolley, A. J., 2015; TPP-0007
Stability	OCSPP 830.6313 CITAC MT 46	*Inpyrfluxam* TGAI (95.0%)	Chemically stable when stored at ambient temperature and 54 °C for 14 days and chemically stable when stored at ambient temperature and 54 °C for 14 days in the presence of metals or metal ions.	Hasegawa, M., 2016; TPP-0008

Property	Guideline and method	Test material specification and purity	Findings	Reference/ Remarks
			Chemically stable when stored in its container (low-density polyethylene bag) at 25 °C and 50% relative humidity for one year. No corrosion to the container over the test period.	Hasegawa, M., 2016; TPP-0019
			Decomposition rate of inpyrfluxam in air = 45.8565 × 10^{-12} cm^3 $molecule^{-1}$ sec^{-1} (calculated) Half-life of inpyrfluxam in air = 2.799 hours (calculated)	Wojnarowicz, L. and Jarvis, T., 2017; TPP-0025
pH	OECD 122 CIPAC MT 75.3 OCSPP 830.7000	TGAI (95.0%, 1% dispersion)	5.61 at 25 °C	Walker, J. A., 2016; TPP-0012
Solubility in organic solvents	OECD 105	TGAI (95.0%) at 20 °C:	Acetone: 621 g/L	Walker, J. A., 2016; TPP-0012
			Dichloromethane:353 g/L	
			Ethyl acetate: 396 g/L	
			n-hexane : 0.982 g/L	
			Methanol: 368 g/L	
			n-octanol: 84.6 g/L	
			Toluene: 67.9 g/L	

Table 2 Physical and chemical properties of metabolites 3′-OH-S-2840, 1′-COOH-S-2840A and 1′-COOH-S-2840B.

Property	Guideline and method	Test material specification and purity	Findings	Reference/ Remarks
Pure active ingredient: 3′-OH-S-2840				
Octanol-water partition coefficient	OECD 117 EC A8	3′-OH-S-2840 (97.8 %)	2.53 at 25 °C (pH 6.5)	Foster, B., 2016; TPP-0020
Pure active ingredient: 1′-COOH-S-2840A and 1′-COOH-S-2840B				
Octanol-water partition coefficient	OECD 117 EC A8	1′-COOH-S-2840A (100%) 1′-COOH-S-2840B (99.6%)	<0.3 at 25 °C (pH 7 and 9) for both 0.84 at 25 °C (pH 5) for 1′-COOH-S-2840A 0.97 at 25 °C (pH 5) for 1′-COOH-S-2840B	Foster, B., 2016; TPP-0021

Formulations

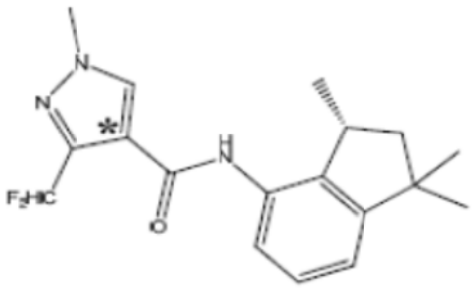
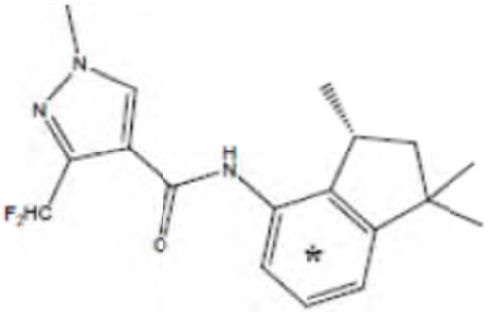
Inpyrfluxam (S-2399) is registered as a flowable concentrate (FC) formulation containing 34.05 percent w/v and as a suspension concentrate (SC) formulation containing 31.25 percent w/v or 37 percent w/v.

METABOLISM AND ENVIRONMENTAL FATE

The metabolism and environmental fate of inpyrfluxam was investigated in target crops (apples, soya bean, canola, corn, sorghum, rice and potatoes), rotational crops and livestock (laying hens and lactating

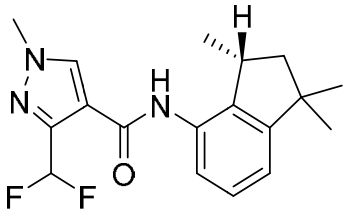
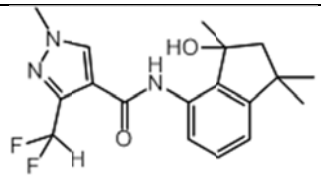
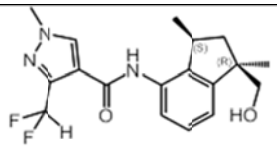
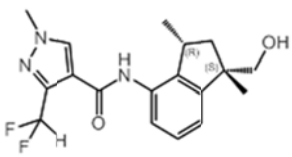
goats), soil, water and sediment systems (rice paddies). Chemical names, structures, and code names of metabolites and degradation products of inpyrfluxam are shown in Table 3. All the compounds were identified in at least one matrix in studies with radiolabelled inpyrfluxam.

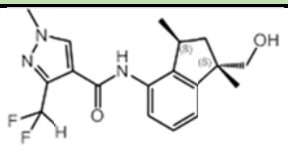
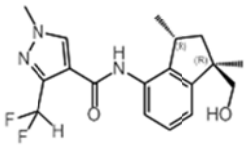
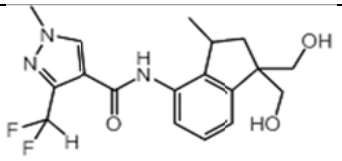
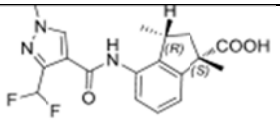
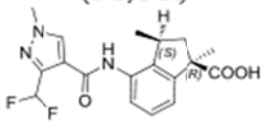
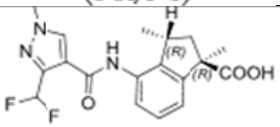
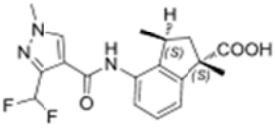
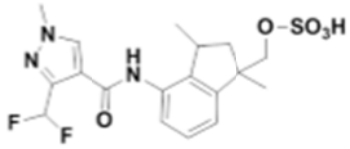
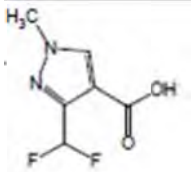
The test substance chemical structures with the position of [^{14}C] radiolabel are shown below.

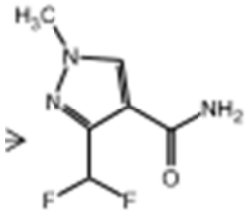
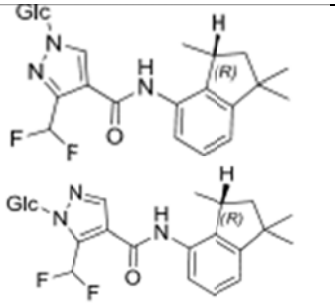
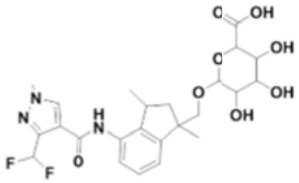
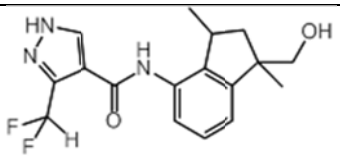
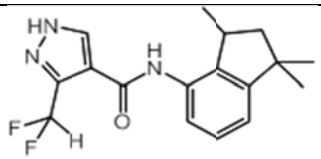
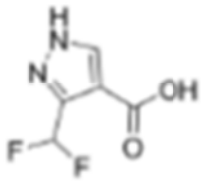
[Pyrazolyl-4-^{14}C] inpyrfluxam	[Phenyl-U-^{14}C] inpyrfluxam
	

Note:
* Indicates the position of the ^{14}C label.

Table 3 Known metabolites and degradation products of inpyrfluxam

Compound	Chemical structure	Found in
Inpyrfluxam		Plants: Apple, Soya , Rice, Potato, Lettuce (RC), Radish (RC), Sorghum (RC) Animals: Milk, Goat tissues, Eggs, Hen tissues Environment: Soil
3'-OH-S-2840		Plants: Apple, Soya , Rice, Potato, Lettuce (RC), Radish (RC), Sorghum (RC) Animals: Milk, Goat tissues, Eggs, Hen tissues Environment: Soil, Water
1'-CH_2OH-S-2840A	 	Plants: Rice, Potato, Lettuce (RC), Radish (RC), Sorghum (RC) Animals: Goat tissues, Eggs, Hen tissues Environment: Soil

Compound	Chemical structure	Found in
1'-CH_2OH-S-2840B	 	Plants: Apple, Soya, Rice, Potato, Lettuce (RC), Radish (RC), Sorghum (RC) Animals: Milk, Goat tissues, Eggs, Hen tissues Environment: Soil
1',1'-bis (CH_2OH)-S-2840		Animals: Goat tissues
1'-COOH-S-2840 A	 (1'S, 3'R)  (1'R, 3'S)	Plants: Potato, Lettuce (RC), Radish (RC), Sorghum (RC) Animals: Milk, Goat tissues, Eggs, Hen tissues Environment: Soil
1'-COOH-S-2840B	 (1'R, 3'R)  (1'S, 3'S)	Plants: Potato, Lettuce (RC), Radish (RC), Sorghum (RC) Animals: Milk, Goat tissues, Eggs, Hen tissues Environment: Soil
1'-CH_2OH-S-2840-sulfate (sum of isomers)		Animals: Eggs, Hen tissues
DFPA		Plants: Potato, Lettuce (RC), Radish (RC), Sorghum (RC) Animals: Goat tissues Environment: Soil, Water

Compound	Chemical structure	Found in
DFPA-$CONH_2$		Plants: Rice, Lettuce (RC), Radish (RC), Sorghum (RC) Animals: Milk, Goat tissues, Eggs, Hen tissues Environment: Water
Glc-NDM-inpyrfluxam (sum of isomers)		Plants: Soya
Glu-1'-CH_2OH-S-2840 (sum of isomers)		Animals: Goat tissues
N-des-Me-1'-CH_2OH-S-2840		Plants: Lettuce (RC), Radish (RC), Sorghum (RC)
N-des-Me-S-2840		Plants: Soya, Rice, Lettuce (RC), Radish (RC), Sorghum (RC) Animals: Eggs, Hen tissues hen tissues Environment: Soil
N-des-Me-DFPA		Plants: Soya, Rice, Potato, Lettuce (RC), Radish (RC), Sorghum (RC) Environment: Soil

Plant metabolism

The Meeting received studies depicting the metabolism of inpyrfluxam in apple (fruit crop), soya bean and canola (pulse and oilseed crops), maize, sorghum and rice (cereal crops) and potato (root crop).

Apple

The metabolism of inpyrfluxam in apples was investigated by Fleischmann (2018, Report TPM-0013). Inpyrfluxam was labelled at two positions; [pyrazolyl-4-^{14}C] inpyrfluxam (2.22 GBq/mmol, ≥ 96.7 percent purity) and [phenyl-U-^{14}C]inpyrfluxam (4.48 GBq/mmol, ≥ 98.3 percent purity) and applied in apple trees (Fuji variety) grown outdoors, at a test site in Madera, California, following foliar applications. A 40

percent SC formulation was isotopically diluted with distilled water to 21.4–22.1 mg ai/m^2 and applied three times (with a manually-operated, trigger-pulled, pump sprayer), with approximate 10-day intervals 35, 24 and 14 days before harvest (BBCH stage not specified in the report), at a nominal rate equivalent to 214–221 g ai/ha.

Samples of apple fruit and leaves were collected 14 days after the final application. Fruits were rinsed with acetonitrile and separated into peel and flesh before homogenisation. Processed samples were extracted twice with acetonitrile:water (1:1) and once with acetonitrile. Leaf samples although collected were not analysed.

All plant samples (extracts, rinses and post-extraction solids) were analysed by combustion/LSC to determine the total radioactive residues (TRR). The TRR of processed matrices were determined from combustion data and in the apple fruit is equal to the sum of fractions from the rinse, peel and pulp. The TRR in peel and flesh were determined from the summation of the residues in the extracts and the unextracted solids. Metabolites were identified by radio-HPLC, radio-TLC and LC-MS.

Apple peel extracts were initially stored in a freezer at approximately -17 °C until subjected to repeat analysis 665 days after the initial chromatography was performed. The profiles from the repeat analysis showed the overall ratio of inpyrfluxam, 3'-OH-S-2840 and 1'-CH_2OH-S-2840 to be similar indicating that resides were stable during the duration of the study.

The total radioactive residues (TRR) and extraction efficiency of the different solvent systems used in the study, as determined by LSC are presented in Table 4.

Table 4 Identification of residues in apple matrices treated with [pyrazolyl-4-^{14}C] inpyrfluxam and [phenyl-U-^{14}C] inpyrfluxam.

Fraction	[Pyrazolyl-^{14}C] radioactivity, %TRR (mg/kg)		[Phenyl-^{14}C] radioactivity, %TRR (mg/kg)	
	Apple rinse		Apple rinse	
Rinse with acetonitrile	64.0 (0.192)		58.2 (0.145)	
	Peel	Pulp	Peel	Pulp
Acetonitrile:water (Extract 1)	17.3 (0.052)	2.7 (0.008)	22.1 (0.055)	2.0 (0.005)
Acetonitrile:water (Extract 2)	8.0 (0.024)	1.3 (0.004)	9.6 (0.024)	1.6 (0.004)
Acetonitrile (Extract 3)	2.7 (0.008)	0.3 (0.001)	2.0 (0.005)	0.4 (0.001)
Total extractable, %TRR	*28.0 (0.084)*	*4.3 (0.013)*	*33.7 (0.084)*	*4.0*
PES	3.3 (0.010)	0.3 (0.001)	3.6 (0.009)	0.4 (0.001)
Total TRR[1], mg/kg	0.094	0.014	0.093	0.011
	Total fruit		Total fruit	
Total TRR[1], (mg/kg) in whole fruit including rinse	96.3 (0.3)		95.9 (0.25)	

Notes:
PES: Post-extraction solids
1 TRR based on sum of extracts 1-3 + PES

The total TRR of the fruit rinse, peel and flesh was 0.30 mg/kg eq for the [pyrazolyl-^{14}C] label and 0.25 mg/kg eq for the [phenyl-^{14}C] label. The majority of radioactivity was recovered in the fruit rinse (58–64 percent TRR, 0.14–0.19 mg/kg eq) and first extracts (17–22 percent TRR, 0.052–0.055 mg/kg eq) for both radiolabels. Radioactivity in the flesh was markedly lower than in the peel for both treatment groups. unextracted solids contained ≤0.014 mg/kg eq (4.7 percent TRR).

The chromatographic profiles for both labels were similar. Parent inpyrfluxam was the only major component found in all samples, ranging from 1.0 percent TRR (0.002 mg/kg) in flesh to 57 percent TRR

(0.13 mg/kg eq) in the fruit rinse. Minor metabolites identified were 3'-OH-S-2840, which was detected in all matrices at levels not exceeding 0.021 mg/kg eq (6.8 percent TRR) and 1-CH_2OH-S-2840, detected in the flesh and peel at levels ≤0.011 mg/kg eq (4.3 percent TRR). In pulp, several unidentified metabolites were observed for both radiolabels. However, no unidentified metabolites exceeded 1 percent TRR and 0.01 mg/kg eq. TLC and HPLC confirmed the presence of the identified metabolites (reference compounds used are presented in Table 3). The distribution of metabolites in each sample matrix is displayed in Table 5.

Chiral-HPLC analysis of inpyrfluxam from the test substance formulation and apple fruit extracts showed that no isomerisation of the chiral centre was observed during the course of the study.

Table 5 Nature of Residue in Apple Fruit Treated with [Pyrazolyl-4-^{14}C] inpyrfluxam and [Phenyl-U-^{14}C] inpyrfluxam by HPLC Analysis

Residue component	Apple rinse		Peel		Pulp		Total fruit	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
[pyrazolyl4-^{14}C] inpyrfluxam								
inpyrfluxam	0.171	57.2	0.061	20.4	0.004	1.5	0.236	79.1
3'-OH-S-2840	0.021	6.8	0.013	4.4	0.001	0.3	0.035	11.5
1'-CH_2OH-S-2840	<0.001	<0.001	0.010	3.2	0.004	1.4	0.014	4.6
Others [a]	<0.001	<0.001	ND	ND	ND	ND	ND	ND
unextracted solids	-	-	0.01	3.3	0.001	0.3	0.011	3.6
Total identified	0.192	64.0	0.084	28.0	0.009	3.2	0.28	95.2
[phenyl-U-^{14}C] inpyrfluxam								
inpyrfluxam	0.133	53.5	0.058	23.3	0.002	1.0	0.193	77.8
3'-OH-S-2840	0.012	4.7	0.015	6.1	0.001	0.2	0.028	11.0
1'-CH_2OH-S-2840	ND	ND	0.011	4.3	0.003	1.3	0.014	5.6
Others[b]	ND	ND	ND	ND	ND	ND	ND	ND
unextracted solids	-	-	0.009	3.6	0.001	0.4	0.01	4
Total identified	0.145	58.2	0.084	33.7	0.006	2.5	0.24	91.9

Notes:

[a] Sum of metabolites (N-des-Me-S-2840, DFPA. N-des-Me-DFPA, DFPA-CONH2) with largest component <1% TRR, <0.01 mg/kg.

[b] Sum of metabolites (N-des-Me-S-2840, ATMI) with largest component <1% TRR, <0.01 mg/kg.

The metabolism of inpyrfluxam in/on apples proceeds by oxidation to form the hydroxylated metabolites, 3'-OH-S-2840 and 1'-CH_2OH-S-2840 (Figure 1).

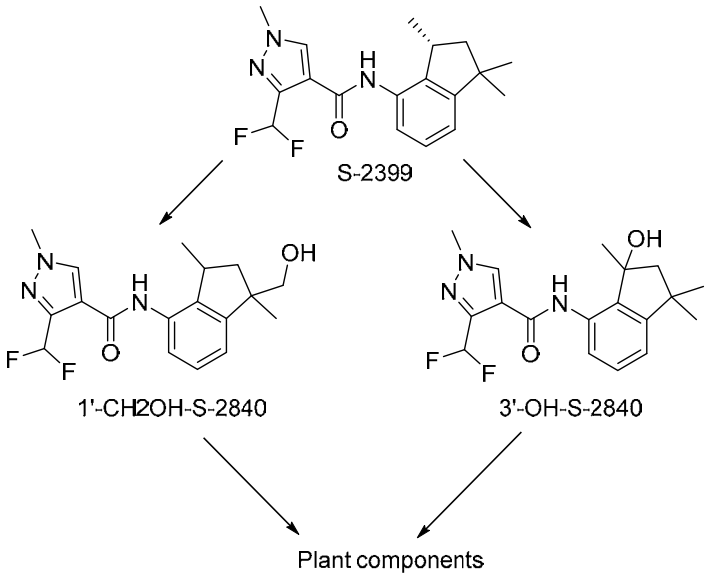


Figure 1 Proposed metabolic pathway of inpyrfluxam in apple after foliar application

Soya bean–foliar application (pulse and oilseed crops)

The metabolism of inpyrfluxam in soya beans was investigated by Fleischmann (2017, Report TPM-0015). The test system was soybean plants grown in a sandy loam soil in plots located outdoors at a test site in Madera, California.

Inpyrfluxam was labelled at two positions; [pyrazolyl-4-^{14}C] inpyrfluxam (2.22 GBq/mmol, ≥95.4 percent purity) and [phenyl-U-^{14}C] inpyrfluxam (4.48 GBq/mmol, ≥95.1 percent purity) in soya beans (var. Glycine max) following two foliar applications. A 40 percent SC formulation was isotopically diluted with distilled water to 10.7–11.3 mg ai/m^2 and applied twice with a 36-day interval (at BBCH 60 and 75) at a rate equivalent 107–113 g ai/ha.

Samples of soya bean forage and soya bean hay were collected 20 and 33 days after the final application (forage harvested at BBCH 75 and dried plants picked up as hay 3 days later), samples of immature pods were taken 11 days after the last application (BBCH 77) and mature pods were taken 53 days after the final application (BBCH 89). Samples were separated into pods and seeds and a portion of mature bean pods were rinsed before homogenisation.

In all samples, with the exception of soya bean hay samples from the [pyrazolyl-^{14}C] inpyrfluxam treatment group, residues were extracted twice with acetonitrile:water (1:1) and once with acetonitrile. Further extraction of the post-extraction solids (PES) was performed when >10 percent total radioactive residues (TRR) and >0.05 mg/kg was detected. Sequential extraction procedures solubilised plant natural components into a mixture of pectin (acetonitrile:0.1 M HCl (1:1)) followed by EGTA (Egtazic acid) in sodium acetate buffer, lignin (DMSO), hemicellulose (24 percent KOH) and cellulose (72 percent H_2SO_4) fractions. The hemicellulose extracts containing >10 percent TRR were partitioned with ethyl acetate. In soya bean hay samples from the [pyrazolyl-^{14}C] inpyrfluxam treatment group, initial extracts were purified by solid phase extraction (C_{18} cartridge), concentrated to dryness and reconstituted in 0.1 percent formic acid in water:acetonitrile for LC-MS/MS.

All plant samples (extracts, rinsates and post-extraction solids) were analysed by combustion/LSC to determine the total radioactive residues (TRR). Metabolites were identified by HPLC, TLC, LC-MS and LC-MS/MS (reference compounds used are presented in Table 3).

The total radioactive residues (TRR) and extraction efficiency of the different solvent systems used in the study, as determined by LSC are presented in Tables 6 and 7.

Table 6 Summary of radioactive residues in extracts of soya bean following treatment with Pyrazolyl-^{14}C] inpyrfluxam

Fraction	%TRR (mg/kg)					
	Forage	Hay	Edamame (immature)		Mature	
			Seed	Pods	Seed	Pods
Rinse	N/A	N/A	N/A	N/A	N/A	5.4 (0.065)
acetonitrile:water (Extract 1)	56.0 (0.779)	29.4 (0.700)	87.2 (0.095)	59.0 (0.419)	55.7 (0.122)	35.8 (0.430)
acetonitrile:water (Extract 2)	19.3 (0.268)	22.0 (0.523)	8.3 (0.009)	19.3 (0.137)	26.5 (0.058)	24.5 (0.294)
acetonitrile (Extract 3)	9.4 (0.131)	12.1 (0.288)	0.9 (0.001)	4.8 (0.034)	6.9 (0.015)	7.6 (0.091)
Total extracted, %TRR	*84.7 (1.18)*	*63.5 (1.5)*	*96.3 (0.1)*	*83.1 (0.59)*	*89.0 (0.2)*	*67.9 (0.88)*
PES (initial)	15.3 (0.213)	36.5 (0.867)	3.7 (0.004)	16.9 (0.120)	11.0 (0.024)	26.7 (0.321)
Total TRR†, mg/kg	1.391	2.378	0.109	0.710	0.219	1.201
PES analysis						
0.1 M HCl in ACN	3.6 (0.050)	6.6 (0.158)	N/A	4.4 (0.031)	3.7 (0.008)	5.4 (0.065)
Pectin (EGTA)	1.7 (0.024)	4.2 (0.099)	N/A	0.9 (0.006)	2.3 (0.005)	3.0 (0.036)
Lignin (DMSO)	1.8 (0.025)	4.6 (0.109)	N/A	1.7 (0.012)	0.5 (0.001)	1.4 (0.017)
Hemicellulose (24% KOH)	5.4 (0.075)	12.1 (0.288)	N/A	6.5 (0.046)	N/A	9.3 (0.112)
Aqueous fraction	N/A	N/A	N/A	N/A	N/A	*3.8 (0.045)*
Organic fraction	N/A	N/A	N/A	N/A	N/A	*5.6 (0.067)*
Cellulose (H_2SO_4)	2.7 (0.038)	9.0 (0.213)	N/A	3.7 (0.026)	N/A	7.5 (0.090)
Total extracted, in PES	*15.2 (0.21)*	*36.5 (0.87)*	*N/A*	*17.1 (0.12)*	*6.4 (0.014)*	*26.7 (0.16)*
Total extracted	*99.9 (1.39)*	*100 (2.37)*	*96.3 (0.1)*	*100 (0.71)*	*95.4 (0.2)*	*94.6 (1.04)*
Remaining unextracted solids	-	-	-	-	4.1 (0.009)	-

Table 7 Summary of radioactive residues in extracts of soya bean following treatment with Phenyl -^{14}C] inpyrfluxam

Fraction	%TRR (mg/kg)					
	Forage	Hay	Edamame (immature)		Mature	
			Seed	Pods	Seed	Pods
Rinse	N/A	N/A	N/A	N/A	N/A	7.4 (0.055)
ACN:water (Extract 1)	55.8 (0.869)	26.0 (0.583)	59.1 (0.013)	59.7 (0.379)	34.2 (0.013)	29.7 (0.220)
ACN:water (Extract 2)	19.4 (0.302)	21.8 (0.489)	9.1 (0.002)	19.8 (0.126)	18.4 (0.007)	21.7 (0.161)
ACN (Extract 3)	9.1 (0.142)	11.7 (0.263)	4.6 (0.001)	5.2 (0.033)	5.3 (0.002)	7.4 (0.055)
Total extracted, %TRR	*84.3 (1.3)*	*59.6 (1.33)*	*72.7 (0.016)*	*84.7 (0.54)*	*57.9 (0.022)*	*58.8 (0.49)*

Fraction	%TRR (mg/kg)					
	Forage	Hay	Edamame (immature)		Mature	
			Seed	Pods	Seed	Pods
PES (initial)	15.7 (0.244)	40.4 (0.906)	27.3 (0.006)	15.3 (0.097)	42.1 (0.016)	33.8 (0.251)
Total TRR[a], mg/kg	1.557	2.241	0.022	0.635	0.038	0.742
0.1 M HCl in ACN	3.9 (0.060)	8.7 (0.194)	N/A	3.5 (0.022)	5.3 (0.002)	4.0 (0.030)
Pectin (EGTA)	1.4 (0.022)	4.6 (0.103)	N/A	2.7 (0.017)	2.6 (0.001)	1.8 (0.013)
Lignin (DMSO)	2.1 (0.032)	5.4 (0.121)	N/A	1.6 (0.010)	N/A	2.3 (0.017)
Hemicellulose (24% KOH)	5.3 (0.082)	12.8 (0.287)	N/A	4.6 (0.029)	N/A	14.7 (0.109)
Aqueous fraction	*N/A*	*4.7 (0.106)*	*N/A*	*N/A*	*N/A*	*9.4 (0.070)*
Organic fraction	*N/A*	*8.1 (0.181)*	*N/A*	*N/A*	*N/A*	*5.3 (0.109)*
Cellulose (H_2SO_4)	3.0 (0.046)	8.9 (0.200)	N/A	3.2 (0.020)	N/A	10.9 (0.081)
Total extracted, in PES	*15.5 (0.18)*	*40.4 (0.38)*	*N/A*	*15.4 (0.07)*	*7.9 (0.001)*	*33.7 (0.4)*
Total extracted	*99.8 (1.5)*	*100 (1.71)*	*72.7 (0.016)*	*100 (0.61)*	*65.8 (0.023)*	*92.5 (0.89)*
Remaining unextracted solids	-	-	-	-	36.8 (0.014)	-

Note:
[a] TRR based on sum of rinse + extracts + PES (initial).

The sample extracts and rinsates were subjected to HPLC, TLC and LC-MS characterisation. Samples were stored in a freezer at approximately -17 °C until subjected to repeat extraction and analysis 364 days after the initial chromatographic analyses. The TRR values from the storage stability extractions were similar (within 5 percent) of the initial TRR of the immature rice plants. The chromatographic profiles from repeat analyses of the immature rice extracts showed a slight decrease in the level of 3'-OH-S-2840 in the [phenyl-^{14}C]inpyrfluxam labelled sample after freezer storage. Additional stability analyses performed on rice straw, rice hulls and rice grain extracts demonstrated similarity to the original results for both radiolabels.

Parent inpyrfluxam was extensively metabolised in soya beans. Characterisation of the residues showed the parent compound, inpyrfluxam accounted for the major part of the residue in forage samples (40.3–50.5 percent TRR, 0.561–0.786 mg/kg eq) but declined as the plant matured. Residues of inpyrfluxam in hay were between 0.424–0.495 mg/kg (17.8–22.1 percent TRR). In edamame (immature) pods, parent levels ranged from 0.241–0.414 mg/kg (34.0–65.2 percent TRR) and in mature pods ranged from 0.130–0.216 mg/kg (10.9–29.2 percent TRR), including the surface rinse fraction. Only trace levels of inpyrfluxam residues were detected in the soya bean seeds.

In forage, 3'-OH-S-2840 was present at the highest levels (15.3–22.1 percent TRR, 0.238–0.308 mg/kg). *N*-des-Me-S-2840 whilst 1'-CH_2OH-S-2840B was identified but in low levels below 3.7 percent TRR (0.058 mg/kg). As the plants matured, the residue levels of the metabolites decreased.

In hay, 3'-OH-S-2840 was present at the highest levels (14.3–14.7 percent TRR, 0.321–0.349 mg/kg), whilst *N*-des-Me-S-2840 was detected at low levels (≤2.4 percent TRR, 0.05 mg/kg). Also, the minor metabolites 1'-CH_2OH-S-2840B, which was exclusive to the [pyrazolyl-^{14}C]inpyrfluxam-treated hay and the sugar conjugate isomers, Glc-NDM-inpyrfluxam, exclusive to the [phenyl-^{14}C]inpyrfluxam labelled samples, were detected.

In immature pods metabolites, 3'-OH-S-2840 and *N*-des-Me-S-2840 were identified in both labels and 1'-CH_2OH-S-2840B was detected only in the [phenyl-^{14}C] label at levels below 10 percent TRR. Unretained polar components amounted to 26.8 percent TRR (0.191 mg/kg) for the [pyrazolyl-^{14}C] label.

In immature seeds, *N*-des-Me-DFPA and *N*-des-Me-S-2840 were detected but in all cases at levels were below 10 percent TRR. The majority of the residue (61.6 percent TRR, 0.067 mg eq/kg) was characterised as multiple polar components with a single component representing 4.6 percent TRR (0.005 mg eq/kg) and not further characterized.

In mature pods, for the [phenyl-^{14}C] label, 3'-OH-S-2840 was identified as the most dominant metabolite at 11.6 percent TRR (0.086 mg/kg), whilst *N*-des-Me-S-2840 and 1'-CH_2OH-S-2840B were also characterized at low levels <3.9 percent TRR. For the [pyrazolyl-^{14}C] label, polar components were present at high levels (48.9 percent TRR, 0.588 mg/kg), with 3'-OH-S-2840, *N*-des-Me-S-2840 and a *N*-des-Me-DFPA conjugates all detected at levels no greater than 2.1 percent TRR.

In seed, the major fraction contained unretained polar components (11.7-63.8 percent TRR, 0.004-0.140 mg/kg). In [pyrazolyl-^{14}C]inpyrfluxam treated seeds, the *N*-des-Me-DFPA conjugate (17.5 percent TRR, 0.038 mg/kg) was also detected, which was characterized as *N*-glycoside by acid hydrolysis. Following [phenyl-^{14}C]inpyrfluxam treatment, 3'-OH-S-2840, Glc-NDM-inpyrfluxam and 1'-CH_2OH-S-2840B were characterized at low levels <10 percent TRR.

The distribution of metabolites in each sample matrix is displayed in Table 8 and the proposed metabolic pathway of inpyrfluxam in soya bean after foliar application is shown in Figure 2.

Table 8 Nature of residue in soya beans treated with [Pyrazolyl-4-^{14}C] inpyrfluxam and [Phenyl-U-^{14}C] inpyrfluxam analysed by HPLC analysis

Residue component	Forage		Hay		Immature				Mature					
					Seed		Pods		Seed		Pods		Rinses	
	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR
[pyrazolyl-4-^{14}C] inpyrfluxam														
Inpyrfluxam	0.561	40.3	0.424	17.8	0.003	3.0	0.241	34.0	<0.001	<0.001	0.083	6.9	0.047	4.0
3'-OH-S-2840	0.308	22.1	0.349	14.7	<0.001	<0.001	0.065	9.2	<0.001	<0.001	0.025	2.1	0.017	1.4
1'-CH_2OH-S-2840B	0.05	3.6	0.087	3.7	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
N-des-Me-S-2840	0.032	2.3	0.054	2.3	<0.001	<0.001	0.032	4.6	<0.001	<0.001	0.024	2.0	0.001	0.1
N-des-Me-DFPA conjugate	<0.001	<0.001	<0.001	<0.001	0.01	9.0	<0.001	<0.001	0.038	17.5	0.022	1.8	<0.001	<0.001
Polars[a]	<0.001	<0.001	<0.001	<0.001	0.067	61.6	0.191	26.8	0.14	63.8	0.588	48.9	<0.001	<0.001
Max. single other	0.07	5.0	0.12	5.0	0.005	4.6	0.019	2.7	0.005	2.2	0.032	2.7	<0.001	<0.001
Total characterised	0.951	68.3	0.914	38.5	0.08	73.6	0.529	74.6	0.178	81.3	0.742	61.7	0.065	5.5
[phenyl-U-^{14}C] inpyrfluxam														
Inpyrfluxam	0.786	50.5	0.495	22.1	0.002	9.8	0.414	65.2	≤0.001	2.0	0.17	23.0	0.046	6.2
3'-OH-S-2840	0.238	15.3	0.321	14.3	ND	ND	0.057	9.0	≤0.001	0.8	0.086	11.6	0.008	1.1
1'-CH_2OH-S-2840B	0.058	3.7	ND	ND	ND	ND	0.026	4.0	0.002	5.2	0.021	2.8	<0.001	<0.001
N-des-Me-S-2840	0.044	2.8	0.053	2.4	≤0.001	4.6	0.042	6.6	ND	ND	0.029	3.9	ND	ND
Glc-NDM-inpyrfluxam[a]	<0.001	<0.001	0.113	5.1	<0.001	<0.001	<0.001	<0.001	≤0.001	1.6	<0.001	<0.001	<0.001	<0.001
Polars[b]	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.004	11.7	<0.001	<0.001	<0.001	<0.001
Max. single other	0.036	2.3	0.114	5.1	0.003	13.1	<0.001	<0.001	0.004	10.3	0.042	5.6	<0.001	<0.001

Residue component	Forage		Hay		Immature				Mature					
					Seed		Pods		Seed		Pods		Rinses	
	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR
Total characterised	1126	72.3	0.982	43.9	0.003	14.4	0.539	84.7	0.009	21.3	0.306	41.3	0.054	7.3

Notes:
ND: Not detected.
[a] Sum of isomers.
[b] Unretained by HPLC, TLC revealed multiple components present.

Chiral-HPLC analysis of inpyrfluxam from the test substance formulation and in the extracts was not performed in the current study.

Samples were stored frozen (-17 °C) between harvest and analysis. All samples were extracted and metabolite characterisation performed within 90 days of sampling, therefore storage stability data are not required. However, the chromatographic system for characterisation of the soluble residues was modified to provide better resolution of the component peaks. Therefore, selected sample extracts and matrices were reanalysed, providing storage stability data. Harvest 4 [pyrazolyl-^{14}C]inpyrfluxam mature seed extract was analysed for storage stability 624 days after the initial analysis and no substantial changes were observed, indicating that resides were stable during the duration of the study.

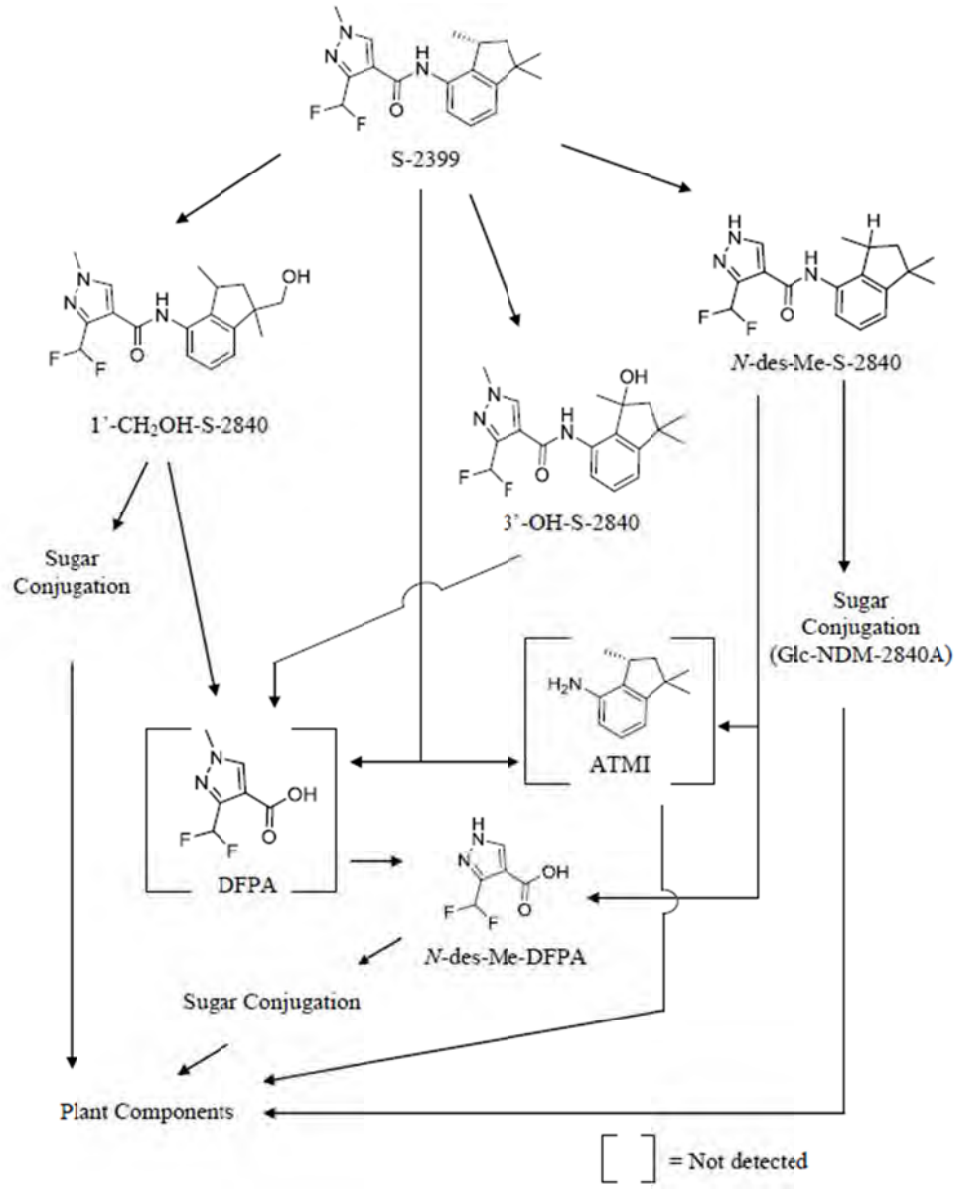


Figure 2 Proposed metabolic pathway for inpyrfluxam in soya bean after foliar application

Maize-seed application (cereal crops)

The metabolism of inpyrfluxam in corn (seed treatment) was investigated by Hguyen *et al*. (2016, Report TPM-0017). The test system was corn seeds grown in a sandy loam soil in plots located outdoors at a test site in Tulare County, California in the year 2014.

Inpyrfluxam was labelled at two positions; [pyrazolyl-4-^{14}C]inpyrfluxam (2.11 GBq/mmol, 97.8 percent purity) and [phenyl-U-^{14}C]inpyrfluxam (4.51 GBq/mmol, 99 percent purity) and applied in corn (var. TA 304-02) following seed application. Corn seeds were first coated uniformly with blank formulation before being individually spiked with the test substance and planted after 3–4 days. Treatments of either [pyrazolyl-^{14}C]inpyrfluxam or [phenyl-^{14}C]inpyrfluxam were made at a target rate of 0.014 mg ai/seed (49.6 g ai/tonne seeds).

Samples of forage, stover, grain, sweet corn were harvested from individual plots. Corn forage was sampled at late dough/early dent stage, sweet corn (kernels plus cob with husks removed at the

milk/succulent stage, approximately 95 days after planting) and stover and grains were harvested at maturity, when grain was separated from the cob (approximately 126 days after planting). Grain-free mature cobs and stalks were processed as corn stover.

Samples were homogenised and analysed to determine the total radioactive residue (TRR) by LSC however ^{14}C-residues were not found above the LOQ (0.005 mg/kg) in the sweetcorn, forage, stover and grain in both labels, thus no metabolite identification was required.

Sorghum-seed application (cereal crops)

The metabolism of inpyrfluxam in sorghum (seed treatment) was investigated by Hguyen *et al.* (2016, Report TPM-0017). The test system was sorghum seeds grown in a sandy loam soil in plots located outdoors at a test site in Tulare County, California in the year 2014.

Inpyrfluxam was labelled at two positions; [pyrazolyl-4-^{14}C]inpyrfluxam (2.11 GBq/mmol, 97.8 percent purity) and [phenyl-U-^{14}C]inpyrfluxam (4.51 GBq/mmol, 99 percent purity) and applied in sorghum (var. GA 3543) following seed application. Sorghum seeds were treated with a slurry prepared by mixing the test substance with the blank formulation and an appropriate volume of water. Treatments of either [pyrazolyl-^{14}C]inpyrfluxam or [phenyl-^{14}C]inpyrfluxam were made at a target rate of 50 g ai/tonne seeds.

Samples of sorghum were harvested from individual plots. Sorghum forage samples were collected at the soft dough to hard dough stage. The remaining plants were harvested at maturity and separated into grain and stover. Samples were homogenised and analysed to determine the total radioactive residue (TRR) by LSC. ^{14}C-residues were not found above the LOQ (0.005 mg/kg) in the sorghum forage, grain and stover samples in both labels, thus no metabolite identification was required.

Rice (cereal crops)–foliar treatment

The metabolism of inpyrfluxam in rice (foliar treatment) was investigated by Fleischmann *et al.* (2017b, Report TPM-0014). The test system was rice seeds (CM205 variety) grown in trays and transplanted (at the 4 leaf stage of growth) in plots (clay soil, pH 7.8) located outdoors at a test site in Madera, California.

Inpyrfluxam was labelled at two positions; [pyrazolyl-4-^{14}C]inpyrfluxam (2.22 GBq/mmol, ≥ 96.4 percent purity) and [phenyl-U-^{14}C]inpyrfluxam (4.48 GBq/mmol, ≥98.8 percent purity) in rice (var. *Oryza sativa L.*) following foliar application. A 40 percent SC formulation was isotopically diluted with distilled water to 95–108.1 mg ai/ha and applied once 28 days (at BBCH 77) before normal commercial harvest

Samples of the immature whole plant (BBCH not specified) were taken for analysis 14 days after application. Samples of rice heads and straw were collected at normal commercial harvest, 28 days after application and rice heads were separated into brown rice and hulls. Homogenised samples were extracted twice with acetonitrile:water (1:1) and once with acetonitrile. Further sequential extraction procedures solubilised plant natural components into a mixture of pectin (acetonitrile:0.1 M HCl (1:1) followed by EGTA (egtazic acid) in sodium acetate buffer, lignin (DMSO), hemicellulose (24 percent KOH) and cellulose (72 percent H_2SO_4) fractions. Sample extracts containing parent compound were partitioned with hexane:water to isolate the parent in the hexane fraction. The hexane extracts were combined, concentrated and subject to further analysis using chiral chromatography. The hemicellulose extracts containing >10 percent TRR were partitioned three times with ethyl acetate before analysis of the organic fractions by HPLC. All plant samples (extracts and post-extraction solids) were analysed by LSC to determine the total radioactive residues (TRR). Metabolites were identified by radio-HPLC and TLC-radioluminography.

The total radioactive residues (TRR) and extraction efficiency of the different solvent systems used in the study, as determined by LSC are presented in Table 9 and Table 10.

Table 9 Summary of radioactive residues in extracts of rice following treatment with pyrazolyl-^{14}C] inpyrfluxam (Report TPM-0014)

Fraction	%TRR (mg/kg)			
	Immature rice	Mature		
		Straw	Hulls	Grain
ACN:water (Extract 1)	50.2 (0.143)	51.6 (0.439)	45.6 (0.696)	64.1 (0.041)
ACN:water (Extract 2)	28.1 (0.080)	23.3 (0.198)	28.2 (0.431)	26.6 (0.017)
ACN (Extract 3)	11.6 (0.033)	11.2 (0.095)	11.7 (0.179)	4.7 (0.003)
Total extracted, %TRR	*89.8 (0.26)*	*86.0 (0.73)*	*85.5 (1.31)*	*95.3 (0.061)*
PES (initial)	10.2 (0.029)	14.0 (0.119)	14.5 (0.221)	4.7 (0.003)
Total TRR[a], mg/kg	0.285	0.851	1.527	0.064
0.1 M HCl in ACN	3.9 (0.011)	5.1 (0.043)	3.9 (0.060)	N/A
Pectin (EGTA)	1.4 (0.004)	1.9 (0.016)	1.1 (0.017)	N/A
Lignin (DMSO)	2.1 (0.006)	2.6 (0.022)	2.8 (0.043)	N/A
Hemicellulose (24% KOH)	1.8 (0.005)	2.8 (0.024)	4.2 (0.064)	N/A
Aqueous fraction	*N/A*	*1.8 (0.015)*	*2.6 (0.039)*	*N/A*
Organic fraction	*N/A*	*1.1 (0.009)*	*1.6 (0.025)*	*N/A*
Cellulose (H_2SO_4)	0.7 (0.002)	0.9 (0.008)	0.5 (0.007)	N/A
Total extracted, in PES	*9.9 (0.028)*	*13.3 (0.11)*	*12.5 (0.19)*	N/A
Total extracted	*99.7 (0.29)*	*99.3 (0.84)*	*98 (1.5)*	*95.3 (0.061)*
Remaining unextracted residues	0.7 (0.002)	0.6 (0.005)	2.0 (0.030)	N/A

Note:

[a] TRR based on sum of extracts + PES (initial).

Table 10 Summary of radioactive residues in extracts of rice following treatment with phenyl -^{14}C] inpyrfluxam (Report TPM-0014)

Fraction	%TRR (mg/kg)			
	Immature rice	Mature		
		Straw	Hulls	Grain
ACN:water (Extract 1)	48.3 (0.183)	42.7 (0.396)	40.1 (0.674)	N/A[b]
ACN:water (Extract 2)	27.4 (0.104)	26.9 (0.249)	28.9 (0.486)	N/A[b]
ACN (Extract 3)	11.4 (0.043)	11.9 (0.110)	14.0 (0.235)	N/A[b]
Total extracted, %TRR	*87.1 (0.33)*	*81.5 (0.76)*	*83.0 (1.4)*	*95.9 (0.047)*
Total TRR[a], mg/kg	0.379	0.927	1.680	0.049
PES (initial)	12.9 (0.049)	18.6 (0.172)	17.0 (0.285)	4.1 (0.002)
0.1 M HCl in ACN	5.0 (0.019)	4.8 (0.044)	4.1 (0.068)	N/A
Pectin (EGTA)	1.6 (0.006)	2.9 (0.027)	1.6 (0.026)	N/A
Lignin (DMSO)	2.7 (0.010)	3.4 (0.032)	3.2 (0.053)	N/A
Hemicellulose (24% KOH)	2.1 (0.008)	3.1 (0.029)	4.4 (0.073)	N/A
Aqueous fraction	*N/A*	*1.9 (0.018)*	*1.9 (0.032)*	*N/A*
Organic fraction	*N/A*	*1.2 (0.011)*	*2.4 (0.041)*	*N/A*
Cellulose (H_2SO_4)	0.8 (0.003)	1.5 (0.014)	0.6 (0.010)	N/A
Total extracted, in PES	*12.2 (0.043)*	*15.7 (0.68)*	*13.9 (0.22)*	*N/A (N/A)*
Total extracted	*99.3 (0.37)*	*97.2 (1.44)*	*96.9 (1.4)*	*95.9 (0.047)*
Remaining unextracted solids	0.8 (0.003)	2.8 (0.026)	3.2 (0.054)	N/A

Notes:

[a] TRR based on sum of extracts + PES (initial).

[b] Individual extracts were combined prior to radioanalysis.

All plant samples (extracts and post-extraction solids) were analysed by LSC to determine the total radioactive residues (TRR). Metabolites were identified by HPLC and TLC characterisation.

In immature rice, the only major component for both radiolabels detected in the neutral extract was parent inpyrfluxam, present at 81.2–86.7 percent TRR (0.247–0.308 mg/kg). The metabolite, 3′–OH-S2480 was detected at the next highest level, between 5.6–7.1 percent TRR (0.016–0.027 mg/kg), with trace levels of *N*-des-Me-S-2840 and 1'-CH_2OH-S-2840 (two isomers) also detected in both labelled extracts. The acidic acetonitrile extracts for both labels contained predominantly parent at 2.5 percent TRR (0.007 mg/kg) and 3.4 percent TRR (0.013 mg/kg) for the [pyrazolyl-^{14}C] and [phenyl-^{14}C] labels, respectively.

In rice grain and rice straw, chromatographic profiles were similar. In grain, inpyrfluxam accounted for 60.6 percent TRR (0.039 mg/kg) and 78.6 percent TRR (0.038 mg/kg) for the [pyrazolyl-^{14}C] and [phenyl-^{14}C] labels, respectively. 3'-OH-S-2840 was present at low levels between 5.9 percent TRR (0.004 mg/kg) and 7.0 percent TRR (0.003 mg/kg). The sugar conjugate, Gly-1'-CH_2OH-S-2840 was present at higher levels in the [pyrazolyl-^{14}C] treated samples at 16 percent TRR (0.010 mg/kg) than the [phenyl-^{14}C] treated samples (3.1 percent, 0.002 mg/kg).

Similarly, in the straw samples, inpyrfluxam was present as the major residue (67.7-77.8 percent TRR, 0.576-0.721 mg/kg) and 3'-OH-S-2840 was detected at the next highest level of 12 percent TRR (0.102 mg/kg) and 6 percent TRR (0.055 mg/kg) for the [pyrazolyl-^{14}C] and [phenyl-^{14}C] labels, respectively. In the [pyrazolyl-^{14}C] label, the conjugate, Gly-1'-CH_2OH-S-2840 and DFPA-$CONH_2$ were detected at 5.2 percent TRR (0.040 mg/kg) and 4.6 percent TRR (0.039 mg/kg), respectively. In both labels, trace levels of *N*-des-Me-S-2840 and 1'-CH_2OH-S-2840 were also found. Characterisation of the acidic acetonitrile extracts from both radiolabels showed that the majority of residues were from the parent, inpyrfluxam. Partitioning of the base soluble extracts with ethyl acetate resulted in 1.1 percent TRR (0.009 mg/kg) and 1.2 percent TRR (0.011 mg/kg) in the organic fractions from the [pyrazolyl-^{14}C]inpyrfluxam and [phenyl-^{14}C]inpyrfluxam treated samples, respectively.

In rice hulls, residues from both the [pyrazolyl-^{14}C]inpyrfluxam and [phenyl-^{14}C]inpyrfluxam treatments were characterised as a mixture of inpyrfluxam, at levels of 41.8 percent TRR (0.639 mg/kg) and 52.5 percent TRR (0.881 mg/kg), 1'-CH_2OH-S-2840, detected at 33.9 percent TRR (sum of isomers; 0.517 mg/kg) and 18 percent TRR (0.277 mg/kg) and 3'-OH-S-2840at 12 percent TRR (0.102 mg/kg) and 6 percent TRR (0.055 mg/kg), respectively. The major component in the acidified acetonitrile extracts for each label was inpyrfluxam, at 1.9-3.3 percent TRR (0.029-0.055 mg/kg).1'-CH_2OH-S-2840 (1.6 percent TRR, 0.024 mg/kg) was also detected in the extract from the [pyrazolyl-^{14}C] label. Partitioning of the base soluble fractions with ethyl acetate released 1.6 percent TRR (0.025 mg/kg) and 2.4 percent TRR (0.041 mg/kg) in the organic fractions from the [pyrazolyl-^{14}C]inpyrfluxam and [phenyl-^{14}C]inpyrfluxam treated samples, respectively. The main component in the organic fraction was inpyrfluxam. For both labels, there were also small amounts of undifferentiated peaks that were consistent with sugar conjugates of 1'-CH_2OH-S-2840.

The distribution of metabolites in each sample matrix is displayed in Table 11 and the proposed metabolic pathway of inpyrfluxam in rice after foliar application is shown in Figure 3.

Table 11 Nature of Residue in rice (foliar application) treated with [Pyrazolyl-4-^{14}C] inpyrfluxam and [Phenyl-U-^{14}C]inpyrfluxam by HPLC Analysis

Residue component	Immature rice		Mature					
			Straw		Hulls		Grain	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
[pyrazolyl-4-^{14}C]inpyrfluxam								
inpyrfluxam	0.247	86.7	0.576	67.7	0.639	41.8	0.039	60.6
3'-OH-S-2840	0.016	5.6	0.102	12.0	0.088	5.8	0.004	5.9
1'-CH_2OH-S-2840[a]	≤0.001	0.2	0.006	0.7	0.517	33.9	<0.001	<0.001
Gly- 1'-CH_2OH-S-2840	<0.001	<0.001	0.041	5.2	0.111	7.2	0.01	16.0
N-des-Me-S-2840	≤0.001	0.2	0.003	0.3	<0.001	<0.001	<0.001	<0.001
DFPA-$CONH_2$	<0.001	<0.001	0.039	4.6	<0.001	<0.001	<0.001	<0.001
Others	0.003	0.7	0.005	0.6	0.009	0.6	0.002	1.7
Max. single other	0.002	0.5	0.005	0.6	0.009	0.6	≤0.001	1.2
Total identified	0.265	92.7	0.767	90.5	1.355	88.7	0.053	82.5
[phenyl-U-^{14}C]inpyrfluxam								
inpyrfluxam	0.308	81.2	0.721	77.8	0.881	52.5	0.038	78.6
3'-OH-S-2840	0.027	7.1	0.055	6.0	0.087	5.6	0.003	7.0
1'-CH_2OH-S-2840[†]	0.011	3.0	0.014	1.5	0.277	18.0	ND	ND
Gly- 1'-CH_2OH-S-2840	ND	ND	ND	ND	0.119	7.1	0.002	3.1
N-des-Me-S-2840	0.002	0.5	0.005	0.5	ND	ND	ND	ND
DFPA-$CONH_2$	ND	ND	ND	ND	ND	ND	ND	ND
Others	≤0.001	0.3	0.005	0.5	0.074	4.4	≤0.001	0.6
Max. single other	≤0.001	0.3	0.003	0.3	0.049	2.9	≤0.001	0.6
Total identified	0.348	91.8	0.795	85.8	1.364	83.2	0.043	88.7

Note:
[a] Sum of isomers.

Chiral separation of the hexane soluble fractions showed only the R-enantiomer of inpyrfluxam was present in rice sample extracts. No isomerisation of the chiral carbon in inpyrfluxam occurred between the time of application and sample harvest in this study.

Samples were stored in a freezer at approximately -17 °C until subjected to repeat extraction and analysis 364 days after the initial chromatographic analyses. The TRR values from the storage stability extractions were similar (within 5 percent) of the initial TRR of the immature rice plants. The chromatographic profiles from repeat analyses of the immature rice extracts showed a slight decrease (5.2 percent) in the level of 3'-OH-S-2840 in the [phenyl-^{14}C]inpyrfluxam labelled sample after freezer storage. Additional stability analyses performed on rice straw, rice hulls and rice grain extracts demonstrated similarity to the original results for both radiolabels, indicating that resides were stable during the duration of the study.

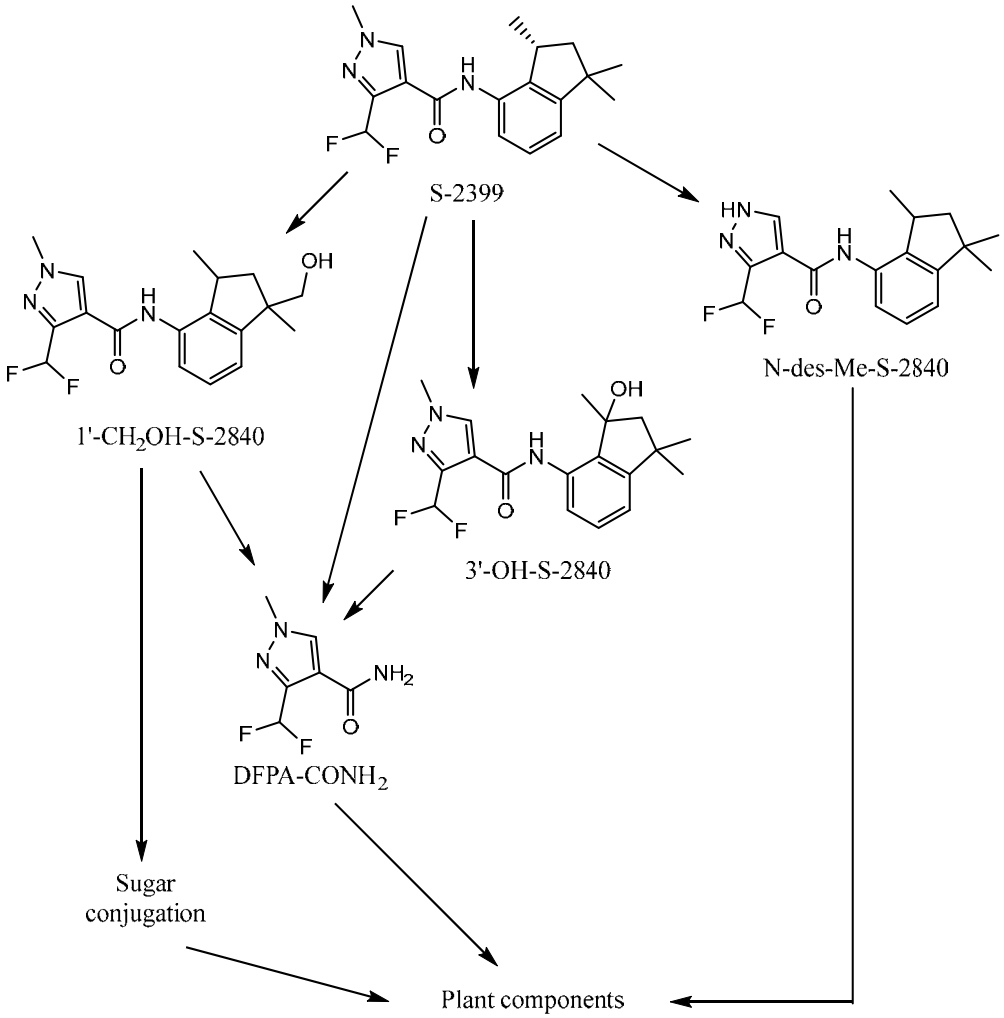


Figure 3 Proposed metabolic pathway of inpyrfluxam in rice after foliar application

Rice (cereal crops)–granular treatment

The metabolism of inpyrfluxam in rice (granular treatment) was investigated by Fleischmann (2017, Report TPM-0016). The test system was rice seeds (CM205 variety) grown in trays (above ground wooden boxes) and transplanted (at the 4 leaf stage of growth) in plots (clay soil, pH 7.8 and 1.1 percent organic matter) located outdoors without protection except a wind break at a test site in Madera, California. The crops were irrigated with 19–38mm water per irrigation event.

Inpyrfluxam was labelled at two positions; [pyrazolyl-4-^{14}C]inpyrfluxam (2.22 GBq/mmol, ≥ 97.1 percent purity) and [phenyl-U-^{14}C]inpyrfluxam (4.48 GBq/mmol, ≥ 95.9 percent purity) in rice (*Oryza sativa L.*) following granular application. A 4 percent granular formulation was applied in a single application (at BBCH 13–14) at a nominal rate equivalent to 400 g ai/ha (equivalent to 2 g ai/nursery box).

Immature rice plants were harvested 30 days after application (BBCH 30) and mature rice plants were harvested 132 days after treatment (BBCH 89) and were separated into straw, hulls and rice grain.

Homogenised samples were extracted twice with acetonitrile:water (1:1) and once with acetonitrile. Further sequential extractions solubilised plant natural components into a mixture of pectin (acetonitrile:0.1 M HCl (1:1)) followed by EGTA (egtazic acid) in sodium acetate buffer, lignin (DMSO), hemicellulose (24 percent KOH) and cellulose (72 percent H_2SO_4) fractions. Sample extracts containing

parent compound were partitioned with hexane:water to isolate the parent in the organic fraction. The hexane extracts were combined, concentrated and subjected to further analysis using a chiral chromatography. The hemicellulose extracts containing >10 percent TRR were partitioned three times with ethyl acetate before analysis of the organic fractions by HPLC.

The total radioactive residues (TRR) and extraction efficiency of the different solvent systems used in the study, as determined by LSC are presented in Table 12 and Table 13.

Table 12 Summary of radioactive residues in extracts of rice following treatment with pyrazolyl-^{14}C] inpyrfluxam (Report TPM-0016)

Fraction	[Pyrazolyl-^{14}C] radioactivity, %TRR (mg/kg)			
	Immature rice	Mature		
		Straw	Hulls	Grain
ACN:water (Extract 1)	36.3 (1.410)	38.6 (0.611)	37.7 (0.066)	44.4 (0.004)
ACN:water (Extract 2)	12.0 (0.465)	19.1 (0.302)	19.4 (0.034)	11.1 (0.001)
ACN (Extract 3)	3.0 (0.118)	6.3 (0.100)	5.7 (0.010)	NC
Total extracted	*51.3 (1,99)*	*64.0 (1,01)*	*62.8 (0,11)*	*55.6 (0,005)*
PES (initial)	48.7 (1.895)	36.0 (0.569)	37.1 (0.065)	44.4 (0.004)
Total TRR†, mg/kg	3.888	1.582	0.175	0.009
0.1 M HCl in ACN	8.9 (0.345)	3.9 (0.061)	4.0 (0.007)	N/A
Pectin (EGTA)	4.8 (0.185)	5.5 (0.087)	5.1 (0.009)	N/A
Lignin (DMSO)	1.8 (0.068)	2.0 (0.032)	2.8 (0.005)	N/A
Hemicellulose (24% KOH)	31.1 (1.208)	19.2 (0.303)	18.3 (0.032)	N/A
Aqueous fraction	*25.5 (0.992)*	*14.5 (0.230)*	*10.9 (0.019)*	*N/A*
Organic fraction	*5.6 (0.216)*	*4.6 (0.073)*	*7.4 (0.013)*	*N/A*
Cellulose (H_2SO_4)	2.3 (0.089)	5.4 (0.086)	N/A	N/A
Total extracted, in PES	*48.7 (1,89)*	*36.0 (0,57)*	*30.4 (0,053)*	*N/A*
Total extracted	*100 (3,88)*	*100 (1,58)*	*93,2 (0,16)*	*55.6 (0,005)*
Remaining unextracted solids	N/A	N/A	6.9 (0.012)	N/A

Note:
† TRR based on sum of extracts + PES (initial).

Table 13 Summary of radioactive residues in extracts of rice following treatment with phenyl-^{14}C] inpyrfluxam (Report TPM-0016)

Fraction	[Phenyl-^{14}C] radioactivity, %TRR (mg/kg)			
	Immature rice	Mature		
		Straw	Hulls	Grain
ACN:water (Extract 1)	38.4 (0.724)	36.6 (0.392)	60.3 (0.094)	26.7 (0.004)
ACN:water (Extract 2)	15.5 (0.292)	18.7 (0.200)		6.7 (0.001)
ACN (Extract 3)	4.7 (0.088)	7.2 (0.077)		NC
Total extracted	*58.6 (1,1)*	*62.5 (0,67)*	*60.3 (0,094)*	*33.4 (0,005)*
PES (initial)	41.5 (0.783)	37.6 (0.403)	39.7 (0.062)	66.7 (0.010)
Total TRR†, mg/kg	1.887	1.072	0.156	0.015
0.1 M HCl in ACN	4.1 (0.077)	4.3 (0.046)	5.8 (0.009)	6.7 (0.001)
Pectin (EGTA)	11.1 (0.210)	6.8 (0.073)	3.9 (0.006)	N/A
Lignin (DMSO)	3.7 (0.069)	2.6 (0.028)	2.6 (0.004)	N/A
Hemicellulose (24% KOH)	18.8 (0.355)	19.2 (0.206)	18.6 (0.029)	N/A

Fraction	[Phenyl-^{14}C] radioactivity, %TRR (mg/kg)			
	Immature rice	Mature		
		Straw	Hulls	Grain
Aqueous fraction	*14.1 (0.267)*	*15.4 (0.165)*	*10.9 (0.019)*	*N/A*
Organic fraction	*4.7 (0.088)*	*3.8 (0.041)*	*7.4 (0.013)*	*N/A*
Cellulose (H_2SO_4)	3.8 (0.071)	4.7 (0.050)	N/A	N/A
Total extracted, in PES	*41.4 (0,78)*	*37.6 (0,403)*	*30.8 (0,048)*	*6.7 (0,001)*
Total extracted	*100 (1,88)*	*100 (1,073)*	*91,1 (0,14)*	*40,4 (0,006)*
Remaining unextracted solids	N/A	N/A	9.6 (0.015)	60.0 (0.009)

Note:
† TRR based on sum of extracts + PES (initial).

All plant samples (extracts and post-extraction solids) were analysed by LSC to determine the total radioactive residues (TRR). Metabolites were identified by radio-HPLC and TLC-radioluminography. Which reference compounds were used.

In immature rice plants, parent inpyrfluxam accounted for a large proportion of the residues at 20 percent and 38.2 percent TRR (0.779 mg/kg and 0.721 mg/kg) for the [pyrazolyl-^{14}C] and [phenyl-^{14}C] labelled samples, respectively. In addition, 1'-CH_2OH-S-2480 (sum of isomers, 5.8–6.2 percent TRR, 0.109–0.241 mg/kg), 3'-OH-S-2840 (1.2-3.6 percent TRR, 0.023–0.142 mg/kg) and DFPA-$CONH_2$ (pyrazolyl label only, 2.2 percent TRR, 0.086 mg/kg) were also identified. Another dominant residue existed as the glycosidic derivative of 1'-CH_2OH-S-2840, which was present at 16.8–26.0 percent TRR (0.315–1.010 mg/kg).

In mature straw, 1'-CH_2OH-S-2840 and its glycosidic derivatives existed as the major components at 23.6–25.7 percent TRR (0.273–0.342 mg/kg) and 31.7–38 percent TRR (0.407–0.498 mg/kg), respectively. inpyrfluxam accounted for 0.030 mg/kg (1.9–2.8 percent TRR) for both radiolabels, whilst *N*-des-Me-DFPA, 3'-OH-S-2840 and DFPA-$CONH_2$ were identified in the [pyrazolyl-^{14}C] labelled sample extracts at levels ≤2.1 percent TRR (0.034 mg/kg).

In mature rice hulls residues from the [pyrazolyl-^{14}C]inpyrfluxam consisted of 1'-CH_2OH-S-2840 as the most dominant residue (40.1 percent TRR, 0.070 mg/kg), followed by DFPA-$CONH_2$ (17.5 percent TRR, 0.031 mg/kg) and *N*-des-Me-DFPA (5.3 percent TRR, 0.009 mg/kg). Parent was not detectable, indicating extensive metabolism in hulls. In the [phenyl-^{14}C] labelled hull samples, 1'-CH_2OH-S-2840 was detected at 50.8 percent TRR (0.080 mg/kg) and its glycosidic conjugates were present at 9.4 percent TRR (0.015 mg/kg).

In rice grain residues from the [pyrazolyl-^{14}C]inpyrfluxam label, were very low, with DFPA and/or *N*-des-Me-DFPA constituting the major residues (23.1 percent TRR, 0.002 mg/kg), whilst DFPA-$CONH_2$ (1.5 percent TRR, ≤ 0.001 mg/kg) and 1'-CH_2OH-S-2840 (4.7 percent TRR, ≤ 0.001 mg/kg) were also identified. Grain from the [phenyl-^{14}C]inpyrfluxam treated rice contained residues of 1'-CH_2OH-S-2840 (6.8 percent TRR, ≤ 0.001 mg/kg). No parent was detectable for either radiolabel.

The distribution of metabolites in each sample matrix is displayed in Table 14 and the proposed metabolic pathway of inpyrfluxam in rice after granule application is shown in Figure 4.

Table 14 Nature of Residue in rice (granular application) treated with [Pyrazolyl-4-^{14}C] inpyrfluxam and [Phenyl-U-^{14}C]inpyrfluxam by HPLC Analysis

Residue component	Immature rice		Mature					
			Straw		Hulls		Grain	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
[pyrazolyl-4-^{14}C]inpyrfluxam								
Inpyrfluxam	0.779	20.0	0.03	1.9	ND	ND	ND	ND
3'-OH-S-2840	0.142	3.6	0.009	0.6	ND	ND	ND	ND
1'-CH_2OH-S-2840B	0.241	6.2	0.276	25.7	0.07	40.1	≤0.001	4.7
Gly- 1'-CH_2OH-S-2840	1.010	26	0.498	31.7	ND	ND	ND	ND
N-des-Me-S-2840	ND	ND	0.025	1.6	0.009	5.3	0.002	23.1
DFPA-$CONH_2$	0.086	2.2	0.034	2.1	0.031	17.5	≤0.001	1.5
Total characterised	2.258	58.0	0.938	59.6	0.11	62.9	0.004	29.3
[phenyl-U-^{14}C]inpyrfluxam								
inpyrfluxam	0.721	38.2	0.03	2.8	ND	ND	ND	ND
3'-OH-S-2840	0.023	1.2	ND	ND	ND	ND	ND	ND
1'-CH_2OH-S-2840B	0.109	5.8	0.253	23.6	0.08	50.8	≤0.001	6.8
Gly- 1'-CH_2OH-S-2840	0.315	16.8	0.407	38.0	0.015	9.4	ND	ND
Total characterised	1.168	62.0	0.69	64.4	0.095	60.2	0.001	6.8

Chiral separation of the hexane soluble fractions showed only the R-enantiomer of inpyrfluxam was present in rice sample extracts. No isomerisation of the chiral carbon in inpyrfluxam occurred between the time of application and sample harvests in this study.

Samples were stored in a freezer at approximately -27 °C after harvest. Immature rice plants were extracted 48 days after harvest. Initial chromatographic profiling of the [pyrazolyl-4-^{14}C] residues from rice forage was performed 61 days after harvest. Enzyme hydrolysis of this extract, performed 307 days after the initial analysis showed a similar magnitude of inpyrfluxam to the 61 day extract, demonstrating stability of inpyrfluxam when stored frozen for up to 307 days. Comparison of residue components following re-extraction after 429 days of frozen storage showed that the magnitude of the major components inpyrfluxam, 3'-OH-S-2840 and 1'-CH_2OH-S-2840 were similar for the [pyrazolyl-^{14}C] label. Similarly, the [phenyl-^{14}C] label extracts showed the magnitude of the major components inpyrfluxam, 3-OH-S-2840, 1'-CH_2OH-S-2840 and the peak characterised as glycosides of 1'-CH_2OH-S-2840 were similar in rice forage stored frozen for up to 196 days. Additional stability analysis was performed on straw, hulls and grain extracts for both labels, demonstrating agreement between the initial and repeat analyses for the sugar conjugates of 1'-CH_2OH-S-2840, 1'-CH_2OH-S-2840 and inpyrfluxam. Analyses indicated stability of these compounds after 555 days of storage. Chromatographic profile comparison of the extracts indicated the stability of 1'-CH_2OH-S-2840 in rice hulls for 517 days and *N*-des-Me-DFPA, DFPA and 1'-CH_2OH-S-2840 in rice grain after 428 days of frozen storage. Based on the above it can be concluded that residues were stable in the whole duration of the study.

Oilseed - seed treatment (pulse and oilseed crops)

The metabolism of inpyrfluxam in canola (seed treatment) was investigated by Nguyen (2017, Report TPM-0031).

Inpyrfluxam was labelled at two positions; [pyrazolyl-4-^{14}C]inpyrfluxam (2.11 GBq/mmol, ≥95.4 percent purity) and [phenyl-U-^{14}C]inpyrfluxam (4.51 GBq/mmol, ≥97.7 percent purity) in canola (var. *Star*) following seed treatment. The test substance was mixed with an appropriate amount of inpyrfluxam FS formulation blank, isotopically diluted and applied at a target rate of 5 g ai/tonne of seed (4.68 or 5.13 mg ai/seed). Seeding was performed in containers with sandy loam soil in California, United States. The

period between treatment and seeding is 7 days and between seeding and harvesting is approximately 4 months.

Samples of mature seeds of canola, were harvested from individual plots at BBCH 97-99 (approximately 161 days after planting) and the total radioactive residues (TRR) analysed by LSC. ^{14}C-residues were not found above the LOQ (0.005 mg/kg) in canola seed samples in both labels, thus no metabolite identification was required.

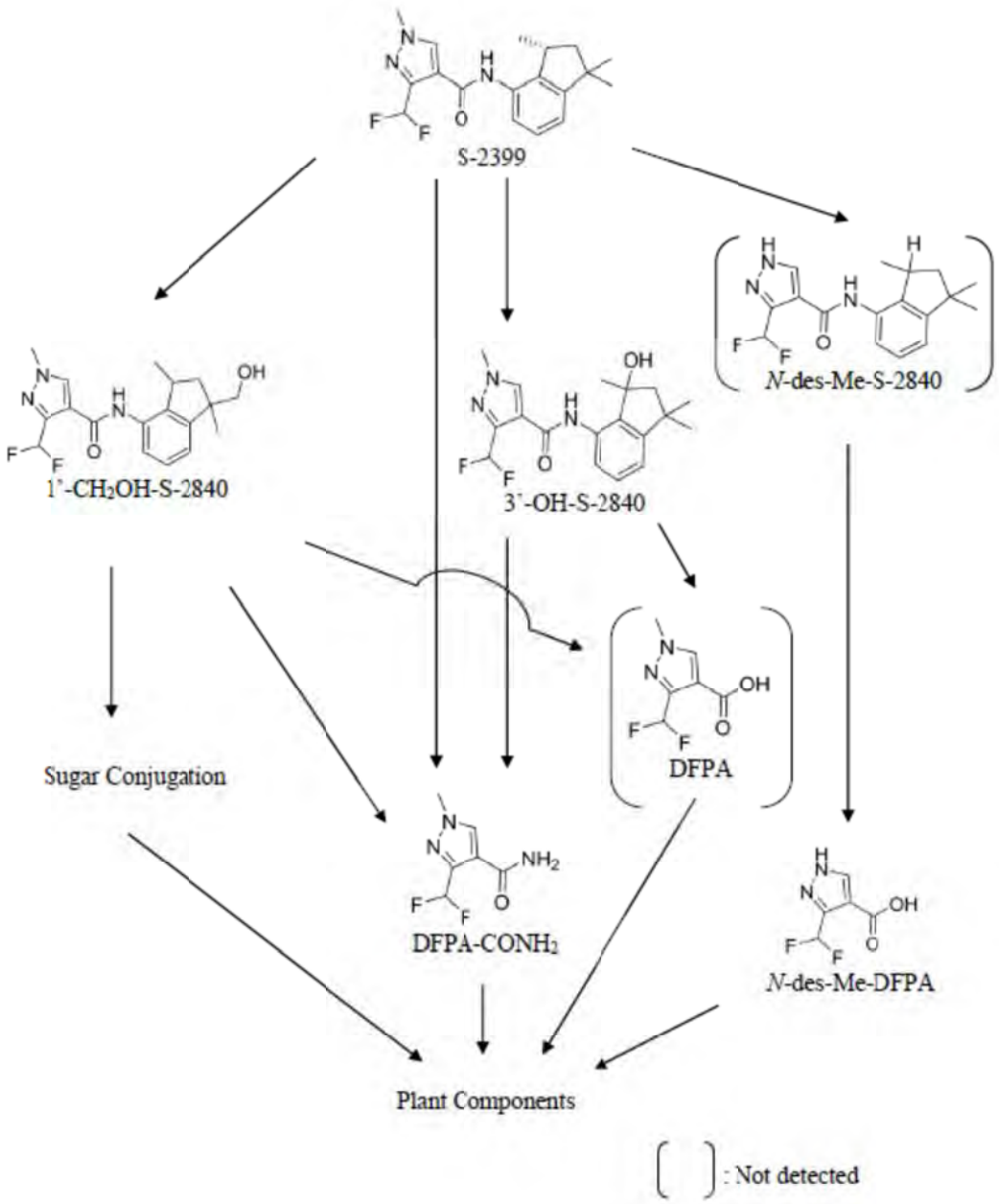


Figure 4 Proposed metabolic pathway of inpyrfluxam in rice after granular application.

Potatoes - seed treatment (root crops)

The metabolism of inpyrfluxam in potato (seed treatment) was investigated by Jalal (2017, Report TPM-0042). The test system was potatoes seeds (Red La Soda variety) planted in plots (loamy sand) located outdoors at a test site in Tulare, California.

Inpyrfluxam was labelled at two positions; [pyrazolyl-4-^{14}C]inpyrfluxam (2.11 GBq/mmol, ≥ 97.9 percent purity) and [phenyl-U-^{14}C]inpyrfluxam (4.51 GBq/mmol, ≥98.7 percent purity) in in potato (var. *Red La Soda*) following seed application. The test substance was mixed with an appropriate amount of inpyrfluxam 3.2 FS VTC-1412-39 formulation blank and isotopically diluted and applied at a target rate of 50 g ai/tonne of seed. Potato seeds were planted the same day after application.

Samples of the mature tubers were harvested at the appropriate growth stage (BBCH 49; 84–85 days from treatment and planting) and samples of foliage (collected at BBCH 48; 71–72 days from treatment and planting) were also taken from the plots. Surface radioactivity was extracted into acetone and homogenised tuber samples were extracted twice with acetone and twice with acetone:water (60:40). All plant samples (extracts and post-extraction solids) were analysed by LSC to determine the total radioactive residues (TRR). The TRR in treated foliage samples from the two radiolabels varied in the range of 0.151 mg/kg for the phenyl label and 0.385 mg/kg for the pyrazolyl label. No further analysis was made of the foliage samples.

To identify conjugates in tuber samples, the acetone extract was evaporated to dryness and dissolved in acetonitrile:water (1:1, v:v) before being subjected to acid hydrolysis with 2 M HCl (at 100 ºC for 2 hours). The hydrolysates were partitioned with ethyl acetate and the metabolites in the aqueous extract fractions were separated by fraction collection.

The total radioactive residues (TRR) and extraction efficiency of the different solvent systems used in the study, as determined by LSC are presented in Table 15 and Table 16.

Table 15 Summary of radioactive residues in extracts of potato tubers following treatment with pyrazolyl-^{14}C] inpyrfluxam (Report TPM-0042)

Fraction	[Pyrazolyl-^{14}C] radioactivity	
	%TRR	mg/kg
Acetone rinse	0.7	<0.001
Aqueous extract	7.9	0.003
Acetone extract	84.8	0.035
Acetonitrile fraction	32.7	0.014
Aqueous fraction (hydrolysis)	*0.8*	*<0.001*
Organic fraction (hydrolysis)	*31.9*	*0.013*
Aqueous fraction	52.8	0.022
Aqueous fraction (hydrolysis)	*4.6*	*0.002*
Organic fraction (hydrolysis)	*48.2*	*0.020*
Total extracted, %TRR	*93.4*	*0.039*
Remaining unextracted solids	6.6	0.003
Total TRR†, mg/kg	100	0.041

Note:
† TRR based on sum of extracts + PES.

Table 16 Summary of radioactive residues in extracts of potato tubers following treatment with phenyl-^{14}C] inpyrfluxam (Report TPM-0042)

Fraction	[Phenyl-^{14}C] radioactivity	
	%TRR	mg/kg
Acetone rinse	11.3	0.001
Aqueous extract	7.5	0.001
Acetone extract	69.0	0.008
Acetonitrile fraction	48.5	0.006
Aqueous fraction (hydrolysis)	*0.9*	*<0.001*
Organic fraction (hydrolysis)	*47.6*	*0.006*
Aqueous fraction	31.8	0.004
Aqueous fraction (hydrolysis)	*3.5*	*<0.001*
Organic fraction (hydrolysis)	*28.3*	*0.003*
Total extracted, %TRR	*87.9*	*0.011*
Remaining unextracted solids	12.1	0.001
Total TRR†, mg/kg	100	0.012

Note:
† TRR based on sum of extracts + PES.

The sample extracts were subjected to radio-HPLC and 2D-TLC characterisation. Characterisation of the acetonitrile fraction showed the presence of the parent and metabolites in free form, while the aqueous fraction contained polar conjugated metabolites that did not correspond to any of the available reference compounds. The acetonitrile fraction contained the parent, inpyrfluxam at a concentration of 0.002 mg/kg. This fraction also contained 3'-OH-S-2840, 1'-CH_2OH-S-2840 (isomers) and 1'-COOH-S-2840 (isomers) in free form, all in trace quantities (≤0.002 mg/kg). The acetonitrile fraction also contained some polar conjugated material (0.002 mg/kg) at or near the origin of the TLC plate. The entire radioactivity in the aqueous fraction (*ca.* 0.004 mg/kg) was polar residues that stayed immobile at the TLC origin.

For the [pyrazolyl-^{14}C] label, the acetonitrile fraction contained inpyrfluxam at a concentration of 0.002 mg/kg. This fraction also contained 3'-OH-S-2840, 1'-CH_2OH-S-2840, 1'-COOH-S-2840, DFPA and *N*-des-Me-DFPA in free form, all in trace quantities (≤0.002 mg/kg). The highest residue present was the metabolite 1'-COOH-S-2840, accounting for 22.3 percent TRR (0.009 mg/kg), of which 18.5 percent TRR was in conjugated form. The aqueous fraction contained a few polar conjugated components in addition to DFPA (0.001 mg/kg) and *N*-des-Me-DFPA (0.004 mg/kg). Unidentified components totalled 40.8 percent TRR (0.017 mg/kg).

In tubers from the [phenyl-^{14}C] label, parent accounted for 15 percent TRR (0.002 mg/kg) of residues. The metabolite, 1'-COOH-S-2840 (isomers) also accounted for a similar proportion, of which 9.2 percent TRR (0.001 mg/kg) was present in conjugated form. The other metabolites present included 3'-OH-S-2840 and 1'-CH_2OH-S-2840, each of which individually accounted for ≤0.001 mg/kg (≤6 percent TRR) and unknowns totalled 49.7 percent TRR (0.006 mg/kg).

By TLC, the ethyl acetate fractions showed the bulk of the polar conjugated residue at the TLC origin disappeared after acid hydrolysis, indicating that the metabolites were released from the conjugates during hydrolysis. Ethyl acetate fractions of the hydrolysates from the [phenyl-^{14}C] label samples could not be analysed further due to matrix interferences, low concentrations of residues (0.003 mg/kg) and large quantities of UV-active nonradioactive species. Similar problems occurred with the [pyrazolyl-^{14}C] labelled extracts, however the higher concentration of residues (0.020 mg/kg) meant that analysis could be performed by preparative HPLC.

Low TRR values in tubers indicated that the uptake of inpyrfluxam from the treated seeds was low. The absorbed test substance metabolised into a number of metabolites, none of which were present in any significant quantity (≥0.01 mg/kg) in the tuber tissue. inpyrfluxam was shown to metabolise via the routes of oxidation, amide bond cleavage and conjugation in potato tubers. The distribution of metabolites in tubers is displayed in Table 17 and the proposed metabolic pathway of inpyrfluxam in potatoes after seed treatment is shown in Figure 5.

Table 17 Nature of Residue in potato tubers (seed application) treated with [Pyrazolyl-4-^{14}C] inpyrfluxam and [Phenyl-U-^{14}C]inpyrfluxam by HPLC Analysis

Residue component[s]	Potato tuber	
	%TRR	mg/kg
[pyrazolyl-4-^{14}C]inpyrfluxam		
Inpyrfluxam	5.8	0.002
3'-OH-S-2840(free)	1.6	0.001
1'-CH_2OH-S-2840 (free)[b]	0.9	<0.001
1'-CH_2OH-S-2840 (conjugated)[b]	2.6	0.001
1'-CH_2OH-S-2840 (total)[b]	3.4	0.001
1'-COOH-S-2840 (free)[b]	3.7	0.002
1'-COOH-S-2840 (conjugated)[b]	18.5	0.008
1'-COOH-S-2840 (total)[b]	22.3	0.009
DFPA (free)	4.7	0.002
DFPA (conjugated)	4.5	0.002
N-des-Me-DFPA (free)	10.1	0.004
N-des-Me-DFPA (conjugated)	0.1	<0.001
Others[c]	40.8	0.017
Max. single other	7.8	0.003
Total identified	52.7	0.022
[phenyl-U-^{14}C]inpyrfluxam		
Inpyrfluxam	15.0	0.002
3'-OH-S-2840(free)	3.6	<0.001
3'-OH-S-2840(conjugated)[b]	2.4	<0.001
3'-OH-S-2840(total)	6.0	0.001
1'-CH_2OH-S-2840 (free)[c]	1.8	<0.001
1'-CH_2OH-S-2840 (conjugated)[c]	1.0	<0.001
1'-CH_2OH-S-2840 (total)[c]	2.7	<0.001
1'-COOH-S-2840 (free)[c]	5.3	0.001
1'-COOH-S-2840 (conjugated)[c]	9.2	0.001
1'-COOH-S-2840 (total)[c]	14.5	0.002
Others[d]	49.7	0.006
Max. single other	4.9	0.001
Total identified	38.2	0.005

Notes:

[a] 'Free' represents the levels present before acid hydrolysis and 'conjugated' represents levels after hydrolysis. The 'total' is the sum of the free and conjugated residue levels.

[b] Sum of isomers.

[c] Sum of >8 unidentified components.

Chiral-HPLC analysis of inpyrfluxam from the test substance formulation and in the extracts was not performed in the current study.

At 198 days after the initial extraction, representative freezer-stored potato tuber samples from both radiolabels were re-extracted and reanalysed to determine the storage stability of the samples. The results showed that the distribution of radioactive residues and the overall recoveries in the respective

extracts and in the Remaining unextracted solids were similar. The metabolite compositions were approximately similar in both extractions, indicating that the major inpyrfluxam metabolites in the potato tuber samples were stable during freezer storage for the duration of the study.

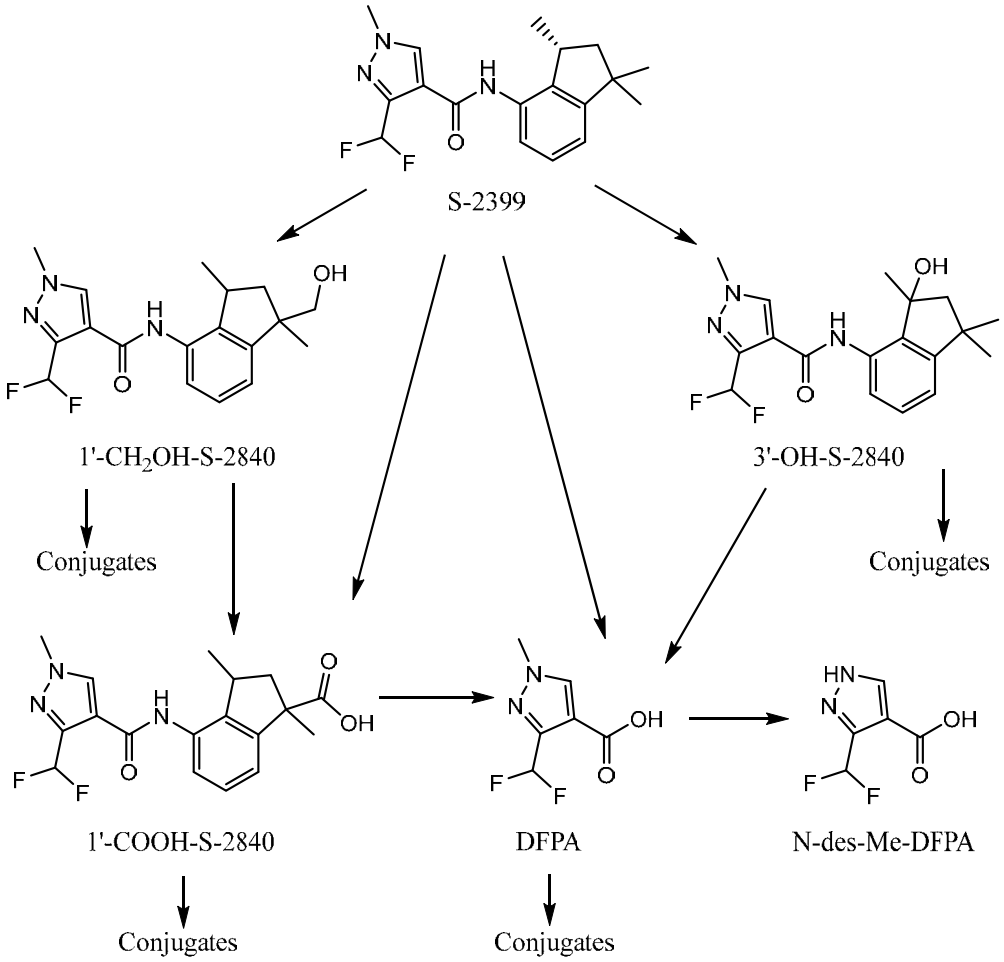


Figure 5 Proposed metabolic pathway of inpyrfluxam in potato tubers

Conclusions–Primary crops

Plant metabolism studies have been conducted with [pyrazolyl-4-^{14}C]inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam and applied to apple (fruit crop), soya bean and canola (pulse and oilseed crops), corn, sorghum and rice (cereal crops) and potato (root crop) at rates that accommodate the anticipated maximum total seasonal application rates. Uptake and transport of inpyrfluxam in the maize, sorghum, oilseed and potato studies were the seed was treated is low. In the rest of the studies in soya bean, rice and apple in which inpyrfluxam was applied via foliar application and residues were taken up by the plant, metabolism of the parent proceeds via oxidation to form the hydroxylated components, 3'-OH-S-2840 and 1'-CH_2OH-S-2840; the latter forming multiple glycoside conjugates. The glycoside conjugates are further transformed into plant constituents associated with pectin, lignin, hemicellulose and cellulose. DFPA-$CONH_2$ can be also formed from the degradation of 3'-OH-S-2840 and 1'-CH_2OH-S-2840, which is further metabolised into plant components. Additional minor pathways include the demethylation of inpyrfluxam or cleavage of the amide bond to DFPA and ATMI. DFPA is rapidly demethylated to *N*-des-Me-DFPA followed by sugar conjugation and metabolism into multiple high polarity components, whilst ATMI is

rapidly decomposed. Demethylation of inpyrfluxam generates *N*-des-Me-S-2840, which can be metabolised to *N*-des-Me-DFPA or conjugated to sugar to form Glc-NDM-2480, both of which are further metabolised into plant constituents. The proposed metabolic pathway for inpyrfluxam in plants is shown in Figure 6.

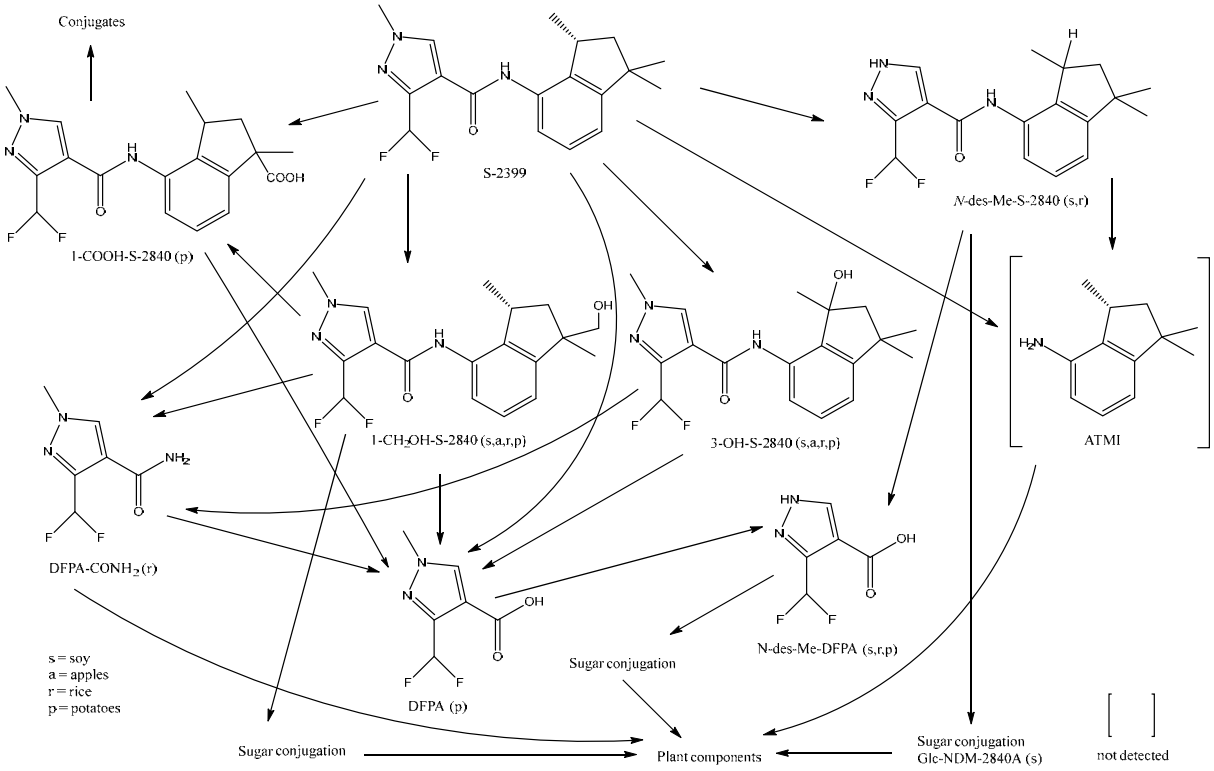


Figure 6 Proposed metabolic pathway of inpyrfluxam in primary crops

Animal metabolism

The Meeting received metabolisms studies in lactating goats (ruminants) and laying hens (poultry). Both the studies were conducted with [pyrazolyl-4-^{14}C]inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam.

Laboratory animals

Metabolism in laboratory animals was evaluated by the WHO Panel of the current Meeting.

Lactating goats

The metabolism of inpyrfluxam in lactating goats (Alpine breed) was investigated by Fleischmann *et. al* (2016, TPM-0024). In this study a target dose of 0.51 mg/kg body weight/day (13.74 ppm feed per day) for [pyrazolyl-^{14}C]inpyrfluxam and 0.64 mg/kg body weight/day (15.74 ppm feed per day) for the [phenyl-^{14}C] inpyrfluxam was given to a two goats (51.7 and 42.1 kg) on five consecutive days via gelatine capsules. Milk was collected twice daily during the dosing period, and the goat was sacrificed ca. 6–7 hours after the final dose on the 5th day. Tissue samples were analysed within 60 days of sacrifice and urine/faeces samples within 30 days of sacrifice.

Radioactivity in liquid samples (skimmed milk, urine and cage washes) was measured by LSC. Skimmed milk and fat were separated by centrifugation of the whole milk samples. TRR in milk fat, muscle, fat, liver and kidney samples were determined by solubilisation/LSC and in faeces by

combustion/LSC. Radiolabelled residues were characterised by HPLC using co-chromatography with reference standards. The identity of the radioactivity was assigned based on HPLC retention times with reference standards. Identities of components were confirmed by TLC using a comparison of the R_f values to standards which were analysed with the sample, or by LC-MS.

Total radioactive residues in milk, tissues and excreta are summarized in Table 18 and extraction efficacy of the solvent systems used are summarized in Table 19. The majority of the dosed radioactivity (≥ 76.5 percent) was recovered in excreta whilst the highest tissue radioactivity was in liver and kidney at 0.24–0.26 percent of the dose (0.33–0.35 mg/kg) and 0.02 percent of the dose (0.17 mg/kg), respectively. Radioactivity in the muscle and fat tissues were qualitatively similar for both radiolabels and residues in milk reached a plateau at day 1 and very low levels (0.09–0.12 percent of the dose) were excreted in whole milk. In all days the TRR in milk fat was <0.01 percent (0.011–0.042 mg/kg) and in skimmed milk 0.01–0.02 percent of the dose (0.013–0.041 mg/kg).

Different extraction procedures were employed depending on the sample. Muscle, liver and kidney samples were extracted twice with acetonitrile:water (1:1) and once with acetonitrile. To further investigate metabolites in the liver and kidney extracts, hydrolysis experiments were performed with the addition of β-glucuronidase and 0.5 M, 1 M and 2 M HCl to concentrated samples. Milk fat was extracted twice with hexane:acetone (4:1) and once with acetone, while skimmed milk was extracted once with acetone. Extracts were centrifuged, concentrated and analysed with HPLC. Fat samples were extracted once with hexane:acetone (4:1) and twice with acetone, combined, concentrated and partitioned between hexane and acetonitrile. The acetonitrile fraction was concentrated and analysed with HPLC.

The profile of the radioactive residues in tissues and milk are summarized in Table 20 and Table 21. In liver, the major residues characterised were 1'-COOH-S-2840 (35.3–42.1 percent TRR, 0.122–0.132 mg/kg) and the glucuronide conjugate of 1'-CH_2OH-S-2840 (15.9–19.2 percent TRR, 0.050–0.066 mg/kg). At lower levels, parent inpyrfluxam (4.9–5.9 percent TRR, 0.017–0.019 mg/kg) and metabolite 1'-CH_2OH-S-2840 (4.9–6.3 percent TRR, 0.015–0.022 mg/kg) were also found. Similar in kidney, the major residues characterized were 1'-COOH-S-2840 (45.4–49.7 percent TRR, 0.078–0.080 mg/kg) and Glu-1'-CH_2OH-S-2840 (24.5–33.5 percent TRR, 0.040–0.057 mg/kg). In muscle (flank and loin), the major residues characterized were 1'-COOH-S-2840 (27.1–46.4 percent TRR, 0.004–0.010 mg/kg) and Glu-1'-CH_2OH-S-2840 (16.0–28.6 percent TRR, 0.002–0.005 mg/kg). Metabolites DFPA-$CONH_2$ and 1'-CH_2OH-S-2840 were also found but at lower levels. Parent was not detected.

In fat, the major component of the residues was 1'-COOH-S-2840 28.8–39.7 percent TRR (0.003–0.018 mg/kg). inpyrfluxam was present at levels between 8.2–15.8 percent TRR (0.002–0.004 mg/kg).

Table 18 Total radioactive residues in milk, tissues and excreta following administration of radiolabelled inpyrfluxam to lactating goats at 14–16 mg/kg diet/day

Matrix		Total Radioactive Residues (TRR)	
		[Pyrazolyl-^{14}C] Label	[Phenyl-^{14}C] Label
		% of administered dose (mg/kg eq)	% of administered dose (mg/kg eq)
Skimmed milk / Milk fat Day 1	AM	ND / ND	ND / ND
	PM	0.01 (0.032) / ≤0.01 (0.022)	0.01 (0.036) / ≤0.01 (0.032)
Skimmed milk / Milk fat Day 2	AM	0.01 (0.014) / ≤0.01 (0.011)	0.01 (0.014) / ≤0.01 (0.014)
	PM	0.02 (0.034) / ≤0.01 (0.024)	0.01 (0.034) / ≤0.01 (0.032)
Skimmed milk / Milk fat Day 3	AM	0.01 (0.015) / ≤0.01 (0.013)	0.01 (0.014) / ≤0.01 (0.014)
	PM	0.01 (0.033) / ≤0.01 (0.025)	0.01 (0.034) / ≤0.01 (0.033)
Skimmed milk / Milk fat Day 4	AM	0.01 (0.017) / ≤0.01 (0.013)	0.01 (0.015) / ≤0.01 (0.015)
	PM	0.02 (0.038) / ≤0.01 (0.027)	0.01 (0.039) / ≤0.01 (0.037)
Skimmed milk / Milk fat Day 5	AM	0.01 (0.016) / ≤0.01 (0.013)	0.01 (0.013) / ≤0.01 (0.013)
	PM	0.01 (0.041) / ≤0.01 (0.030)	0.01 (0.039) / ≤0.01 (0.042)

Matrix	Total Radioactive Residues (TRR)	
	[Pyrazolyl-^{14}C] Label	[Phenyl-^{14}C] Label
	% of administered dose (mg/kg eq)	% of administered dose (mg/kg eq)
Total in milk (skimmed+fat)	0.12	0.09
Gastrointestinal tract	19.8 (1.678)	18.6 (1.893)
Liver	0.24 (0.334)	0.26 (0.350)
Kidney	0.02 (0.169)	0.02 (0.166)
Flank muscle	≤0.01 (0.015)	≤0.01 (0.024)
Loin muscle	≤0.01 (0.011)	0.01 (0.016)
Omental fat	≤0.01 (0.007)	≤0.01 (0.024)
Subcutaneous fat	≤0.01 (0.017)	≤0.01 (0.029)
Renal fat	≤0.01 (0.009)	≤0.01 (0.040)
Bile	0.23 (9.196)	0.05 (12.406)
Blood	≤0.01 (0.039)	≤0.01 (0.048)
Urine	35.4	33.4
Faeces	41.1	44.6
Cage wash	0.09	0.07
Total Recovery	97.0	97.1

Table 19 Extraction efficiency in animal tissues following administration of radiolabelled inpyrfluxam to goats at 14-16 mg/kg diet/day

Matrix	Extract	[Pyrazolyl-^{14}C] Label		[Phenyl-^{14}C] Label	
		%TRR	mg/kg eq	%TRR	mg/kg eq
Liver	TRR (mg/kg)[†]	0.313		0.344	
	Combined organic extracts	91.1	0.285	90.4	0.311
	Hexane phase	4.6	0.014	4.5	0.016
	Aqueous phase	86.6	0.271	85.9	0.295
	EtOAc phase	27.3	0.086	32.8	0.113
	Aqueous phase	59.2	0.185	53.0	0.182
	PES	9.0	0.028	9.6	0.033
Kidney	TRR (mg/kg)[†]	0.162		0.170	
	Combined organic extracts	98.2	0.159	97.7	0.166
	EtOAc phase	44.6	0.072	21.3	0.036
	Aqueous phase	53.5	0.087	76.3	0.13
	PES	1.9	0.003	2.4	0.004
Flank muscle	TRR (mg/kg)[†]	0.014		0.021	
	Combined organic extracts	100	0.014	95.2	0.020
	PES	-	≤0.001	4.8	0.001
Loin muscle	TRR (mg/kg)[†]	0.012		0.015	
	Combined organic extracts	91.7	0.011	93.3	0.014
	PES	8.3	0.001	6.7	0.001
Subcutaneous fat	TRR (mg/kg)[†]	0.012		0.029	
	Combined organic extracts	83.3	0.010	96.6	0.028
	PES	16.7	0.002	3.5	0.001
Omental fat	TRR (mg/kg)[†]	0.006		0.024	
	Combined organic extracts	83.3	0.005	87.5	0.021
	PES	16.7	0.001	12.5	0.003
Renal fat	TRR (mg/kg)[†]	0.007		0.041	
	Combined organic extracts	71.4	0.005	90.2	0.037
	PES	28.6	0.002	9.8	0.004
Skimmed milk	TRR (mg/kg)[†]	0.034		0.040	
	Combined organic extracts	100	0.034	100	0.040
	PES	-	≤0.001	-	≤0.001

Matrix	Extract	[Pyrazolyl-^{14}C] Label		[Phenyl-^{14}C] Label	
		%TRR	mg/kg eq	%TRR	mg/kg eq
Milk fat	TRR (mg/kg)†	0.029		0.018	
	Combined organic extracts	94.4	0.029	100	0.017
	PES	5.6	≤0.001	-	0.001

Notes:
-: Not calculated
† TRR values determined by the sum of fractions (extracts + PES).

Table 20 Summary of radioactive residues in goat tissues and milk after five daily doses of [pyrazolyl-4-^{14}C] inpyrfluxam

Compound	Skimmed milk		Milk fat		Liver		Kidney		Flank muscle		Loin muscle		Subcutaneous fat	
	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq
Inpyrfluxam	ND	ND	ND	ND	5.9	0.019	ND	ND	ND	ND	ND	ND	3.1	≤0.001
DFPA	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
DFPA-$CONH_2$	2.1	0.001	ND	ND	2.5	0.008	ND	ND	11.2	0.002	ND	ND	ND	ND
1'-COOH-S-2840	12.1	0.004	8.8	0.003	42.1	0.132	49.7	0.080	43.8	0.006	33.6	0.004	28.8	0.003
Glu-1'-CH_2OH-S-2840[a]	ND	ND	ND	ND	15.9	0.050	24.5	0.040	22.1	0.003	16.0	0.002	ND	ND
1',1'-bis-(CH_2OH)-S-2840	ND	ND	ND	ND	ND	ND	1.1[b]	0.002[b]	ND	ND	ND	ND	ND	ND
1'-CH_2OH-S-2840[a]	ND	ND	ND	ND	4.9	0.015	ND	ND	8.4	0.001	5.6	0.001	12.3	0.002
3'-OH-S-2840	ND	ND	ND	ND	ND	ND	3.1	0.005	ND	ND	ND	ND	<1.00	≤0.001
Other extracted	85.8	0.029	91.2	0.026	19.7	0.063	19.8	0.032	14.6	0.002	36.5	0.003	39.2	0.005
Max. single other	16.8	0.006	32.9	0.010	5.1	0.016	6.1	0.010	8.6	0.001	9.0	0.001	10.5	0.001
Total characterised	14.2	0.005	8.8	0.003	71.4	0.224	77.3	0.125	85.4	0.012	55.2	0.007	44.2	0.005

Table 21 Summary of radioactive residues in goat tissues and milk after five daily doses of [phenyl-U-^{14}C] inpyrfluxam

Compound	Skimmed milk		Milk fat		Liver		Kidney		Flank muscle		Loin muscle	
	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
Inpyrfluxam	ND	ND	9.1	0.002	4.9	0.017	ND	ND	ND	ND	ND	ND
1'-COOH-S-2840	15.9	0.006	5.5	0.001	35.3	0.122	45.4	0.078	46.4	0.010	27.1	0.004
Glu-1'-CH_2OH-S-2840	ND	ND	ND	ND	19.2	0.066	33.5	0.057	24.4	0.005	28.6	0.004
1'-CH_2OH-S-2840	ND	ND	3.0	0.001	6.3	0.022	3.4	0.006	7.8	0.002	7.0	0.001
3'-OH-S-2840	ND	ND	ND	ND	ND	ND	1.8	0.003	ND	ND	ND	ND
Other extracted	84.1	0.034	76.9	0.013	24.7	0.084	13.6	0.022	16.6	0.003	30.7	0.005
Max. single other	21.2	0.008	20.6	0.004	7.6	0.026	6.8	0.012	8.8	0.002	8.4	0.001

Compound	Skimmed milk		Milk fat		Liver		Kidney		Flank muscle		Loin muscle	
	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
Total characterised	15.9	0.006	17.6	0.004	65.7	0.227	84.0	0.144	78.7	0.017	62.6	0.009

Compound	Subcutaneous fat		Omental fat		Renal fat	
	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
Inpyrfluxam	6.4	0.002	15.8	0.004	8.2	0.004
1'-COOH-S-2840	32.3	0.009	33.8	0.008	39.7	0.018
Glu-1'-CH_2OH-S-2840	ND	ND	ND	ND	ND	ND
1'-CH_2OH-S-2840	10.4	0.003	8.6	0.002	ND	ND
3'-OH-S-2840	ND	ND	ND	ND	ND	ND
Other extracted	47.5	0.014	29.3	0.007	42.3	0.015
Max. single other	7.2	0.002	7.4	0.002	10.2	0.005
Total characterised	49.1	0.014	58.2	0.014	47.9	0.022

Laying hens

The metabolisms of inpyrfluxam in laying hens (Hyline Brown breed; mean body weights 1716-1777g on day 1) was investigated by Fleischmann (2016, TPM-0025). In the study a target dose of 12.44 ppm feed per day for the [pyrazolyl-^{14}C] label and 13.13 ppm feed per day for the [phenyl-^{14}C]inpyrfluxam label was given in 2 groups (10 hens each) on 7 consecutive days via gelatine capsules. Eggs and excreta were collected twice daily. The hens were sacrificed approximately 6 hours after the last dose administration and samples of liver, muscle (breast and thigh), fat (abdominal and subcutaneous) and gastrointestinal tracts with contents were taken. All samples were stored frozen (-20 °C) and analysed 40 days after sacrifice, In liver the identification of the metabolites took more than 6 months, thus extracted were stored and analysed after 286 days. The distribution of components in the chromatographic were similar with the postulated sulfate conjugates of 1'-CH_2OH-S-2840 unchanged (Percent ROI changed ≤ 6 percent).

Radioactivity in excreta and gastrointestinal tract was measured by combustion/LSC. TRR in liver, muscle and fat samples were determined by solubilisation/LSC. Radiolabelled residues were characterised by HPLC using co-chromatography based retention times of reference standards. Identities of components were confirmed by TLC using a comparison of the R_f values to standards which were analysed with the sample, or by LC-MS.

The total recovery of radioactivity was 82.7 percent in the [pyrazolyl-^{14}C] group and 84.6 percent in the [phenyl-^{14}C] group. The majority of the dosed radioactivity was eliminated in the excreta, accounting for 80.3 percent and 81.7 percent of the total dose for the [pyrazolyl-^{14}C] and [phenyl-^{14}C] groups, respectively.

Different extraction procedures were employed depending on the sample. Fat samples were extracted once with hexane/acetone (4:1) and then twice with acetone. The organic extracts were separated from solids by centrifugation. Extracts were combined, concentrated and partitioned three times with acetonitrile. The acetonitrile layers were concentrated prior to HPLC analysis. Excreta, egg, liver, thigh and breast muscle samples were extracted twice with acetonitrile:water (1:1) and then once with acetonitrile. Egg extracts were separated from solids by centrifugation, combined, concentrated and partitioned three times with hexane. The aqueous and hexane layers were radioassayed by LSC and the water extracts were concentrated for HPLC analysis. Excreta, liver, thigh and breast muscle organic extracts were separated from solids by centrifugation, concentrated and analysed by HPLC. Residues in liver extracts were subjected to enzyme or chemical hydrolysis. Neutral solvent extracts were treated with

β-glucuronidase and sulfatase to assess the hydrolysis of sulfate and hydrolysis of glucuronic acid ester. A portion of the unknowns, Rt37 and Rt39 was also treated with 1 M HCl.

The unknowns, Rt37 and Rt39 (both [pyrazolyl-^{14}C] and [phenyl-^{14}C] labelled metabolites) were isolated and purified using solid phase extraction (SPE) and analysed by high resolution LC-MS. A chemically hydrolysed product of one of the isolated metabolites was also analysed using LC-MS/MS.

The post-extraction solids (PES) for all matrices were combusted and radiocarbon quantified by LSC.

The profile of the radioactive residues in tissues and eggs are summarized in Table 22 and Table 23. The TRR were high in gastrointestinal tract at 0.8–1.1 percent of the dose (2.124–2.478 mg/kg) and liver at 0.11–0.22 percent of the dose (0.268–0.526 mg/kg). Combined subcutaneous and abdominal fat had lower radioactivity at 0.01–0.03 percent of the dose (0.069–0.109 mg/kg) whilst combined thigh and breast muscle had the lowest radioactivity concentrations of 0.012–0.022 mg/kg (representing 0.01–0.02 percent of the dose).

The profile of the radioactive residues in tissues and eggs are summarized in Table 24 and Table 25.

In eggs, the major residue components were parent inpyrfluxam at 11.5–11.9 percent TRR (0.002 mg/kg) and 1'–CH_2OH-S-2840 (sum of isomers, 29.8–31.6 percent TRR, 0.006–0.008 mg/kg). Sulfate conjugates of 1'-CH_2OH-S-2840, 1'-COOH-S-2840, 3'-OH-S-2840 (sum of isomers) and *N*-des-Me-S-2840 were present at ≤9.2 percent TRR (≤0.002 mg/kg).

In liver the major residues characterized were the sulfate conjugates of 1'-CH_2OH-S-2840 (44.0–51.7 percent TRR, 0.112–0.164 mg/kg) and metabolites *N*-des-Me-S-2840 (4.6–9.5 percent TRR, 0.015–0.024 mg/kg) and 1'-COOH-S-2840 (6.5–11.0 percent TRR, 0.020–0.028 mg/kg) were identified at lower levels. Inpyrfluxam was not detected.

In muscle (thigh and breast), the major residues characterized were sulfate conjugates of of 1'-CH_2OH-S-2840 (10.2–47.7 percent TRR, 0.002–0.011 mg/kg), 1'-COOH-S-2840 (9.8–16.4 percent TRR, 0.002–0.003 mg/kg), 1'-CH_2OH-S-2840 (3.4–11.1 percent TRR, 0.001–0.002 mg/kg) and DFPA-$CONH_2$ (11.8–14.5 percent TRR, 0.001–0.002 mg/kg). inpyrfluxam (≤4.9 percent TRR, ≤0.001 mg/kg) was also identified as minor component.

In the abdominal and subcutaneous fat, parent inpyrfluxam (55.0–80.7 percent TRR, 0.045–0.075 mg/kg) was the major part of the residue in contrast with other tissues. Other components identified at low levels were 1'-CH_2OH-S-2840 (2.3–2.7 percent TRR, 0.002–0.003 mg/kg), *N*-des-Me-S-2840 (2.5–3.3 percent TRR, 0.002–0.003 mg/kg), 3'-OH-S-2840 (1.5–2.7 percent TRR, 0.001–0.002 mg/kg) and 1'-COOH-S-2840 (1.2–3.2 percent TRR, 0.001–0.003 mg/kg).

Table 22 Total radioactive residues in eggs, tissues and excreta following administration of [pyrazolyl-4-^{14}C]inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam to laying hens at 12–13 mg/kg diet/day

Matrix		Total Radioactive Residues (TRR)	
		[Pyrazolyl-14C] Label	[Phenyl-14C] Label
		% of administered dose (mg/kg)	% of administered dose (mg/kg)
Egg Day 1	AM	<0.01	ND
	PM	<0.01	ND
Egg Day 2	AM	<0.01 (0.024)	<0.01 (0.012)
	PM	<0.01 (0.012)	<0.01 (0.014)
Egg Day 3	AM	-	<0.01 (0.007)
	PM	0.01 (0.019)	0.01 (0.017)

Matrix		Total Radioactive Residues (TRR)	
		[Pyrazolyl-14C] Label	[Phenyl-14C] Label
		% of administered dose (mg/kg)	% of administered dose (mg/kg)
Egg Day 4	AM	<0.01 (0.021)	<0.01 (0.012)
	PM	0.01 (0.020)	0.01 (0.022)
Egg Day 5	AM	<0.01 (0.032)	<0.01 (0.017)
	PM	0.01 (0.022)	0.01 (0.028)
Egg Day 6	AM	-	<0.01 (0.019)
	PM	0.01 (0.031)	0.01 (0.033)
Egg Day 7	AM	0.01 (0.032)	<0.01 (0.023)
	PM	0.01 (0.033)	0.02 (0.031)
Total in eggs		0.06 (0.025)	0.06(0.020)
Gastrointestinal tract		0.8 (2.124)	1.1 (2.478)
Liver		0.22 (0.526)	0.11 (0.268)
Thigh muscle		0.01 (0.013)	0.01 (0.012)
Breast muscle		0.01 (0.012)	0.02 (0.022)
Abdominal fat		0.01 (0.069)	0.03 (0.107)
Subcutaneous fat		0.01 (0.109)	0.01 (0.086)
Total in excreta		80.3	81.7
Cage wash		1.3	1.6
Total Recovery		82.7	84.6

Table 23 Extraction efficiency in poultry tissues following administration of [pyrazolyl-4-^{14}C]inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam to laying hens at 12-13 mg/kg diet/day

Matrix	Extract	[Pyrazolyl-^{14}C] Label		[Phenyl-^{14}C] Label	
		%TRR	mg/kg eq	%TRR	mg/kg eq
Liver	TRR (mg/kg)†	0.317		0.255	
	Combined organic extracts	94.3	0.299	91.4	0.233
	PES	5.7	0.018	8.6	0.022
Breast muscle	TRR (mg/kg)†	0.012		0.023	
	Combined organic extracts	91.7	0.011	91.3	0.021
	PES	8.3	0.001	8.7	0.002
Thigh muscle	TRR (mg/kg)†	0.013		0.015	
	Combined organic extracts	92.3	0.012	80.0	0.012
	PES	7.7	0.001	20.0	0.003
Abdominal fat	TRR (mg/kg)†	0.064		0.094	
	Combined organic extracts	98.4	0.063	96.8	0.091
	PES	1.6	0.001	3.2	0.003
Subcutaneous fat	TRR (mg/kg)†	0.102		0.081	
	Combined organic extracts	99.0	0.101	97.5	0.079
	PES	1.0	0.001	2.5	0.002
Egg†	TRR (mg/kg)‡	0.023		0.020	
	Combined organic extracts	91.3	0.021	90.0	0.018
	PES	8.7	0.002	10.0	0.002
Excreta	TRR (mg/kg)†	13.719		21.263	
	Combined organic extracts	98.2	13.475	98.2	20.872
	PES	1.8	0.244	1.8	0.391

Notes:

† Representative composite samples

‡ TRR values determined by the sum of fractions (extracts + PES).

Table 24 Summary of radioactive residues in tissues and eggs of hens after seven daily doses of [pyrazolyl-4-^{14}C]inpyrfluxam

Compound	Eggs		Liver		Breast muscle		Thigh muscle		Abdominal fat		Subcutaneous fat	
	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq
Applied to HPLC	91.3	0.021	94.3	0.299	91.7	0.011	0.012	92.3	98.44	0.063	99.0	0.101
Inpyrfluxam	10.5	0.002	ND	ND	2.9	≤0.001	4.9	0.001	69.9	0.045	73.7	0.075
DFPA-CONH$_2$	5.0	0.001	ND	ND	11.8	0.001	14.5	0.002	ND	ND	ND	ND
1'-COOH-S-2840†	ND	ND	6.5	0.020	11.0	0.002	9.8	0.002	1.2	0.001	ND	ND
1'-CH$_2$OH-S-2840-sulfate†	5.1	0.001	51.7	0.164	10.2	0.002	11.4	0.002	1.1	0.001	3.2	0.003
1'-CH$_2$OH-S-2840†	31.6	0.008	ND	ND	5.6	0.001	11.1	0.001	2.7	0.002	2.6	0.003
3'-OH-S-2840	1.9	≤0.001	ND	ND	ND	ND	ND	ND	2.7	0.002	2.2	0.002
N-des-Me-S-2840	5.0	0.001	4.6	0.015	ND	ND	ND	ND	3.3	0.002	3.2	0.003
Total characterised	59.1	0.014	62.8	0.199	41.5	0.007	51.7	0.008	80.9	0.053	84.9	0.086
Unknowns*	29.9	0.008 (5)	20.66	0.066 (4)	25.21	0.005 (5)	14.7	0.002 (2)	7.67	0.006 (5)	7.23	0.007 (6)

Notes:
ND: Not detected
† Sum of isomers
* number of unknowns shown in parentheses

Table 25 Summary of radioactive residues in in tissues and eggs of hens after seven daily doses of [phenyl-U-^{14}C] inpyrfluxam

Compound	Eggs		Liver		Breast muscle		Thigh muscle		Abdominal fat		Subcutaneous fat	
	%TRR	mg/kg eq	mg/kg eq	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	TRR	mg/kg eq	%TRR	mg/kg eq
Applied to HPLC	90.0	0.018	91.4	0.233	91.3	0.021	80.00	0.012	96.8	0.091	97.53	0.079
inpyrfluxam	10.9	0.002	ND	ND	ND	ND	2.2	≤0.001	55.0	0.052	80.7	0.065
1'-COOH-S-2840†	4.7	≤0.002	11.0	0.028	10.6	0.003	16.4	0.003	3.2	0.003	ND	ND
1'-CH$_2$OH-S-2840-sulfate†	9.2	≤0.002	44.0	0.112	47.7	0.011	25.2	0.004	8.4	0.008	16.9	0.014
1'-CH$_2$OH-S-2840†	29.8	0.006	ND	ND	3.4	0.001	10.8	0.002	2.3	0.002	ND	ND
3'-OH-S-2840	2.5	≤0.001	ND	ND	ND	ND	ND	ND	1.5	0.001	ND	ND
N-des-Me-S-2840	5.6	0.001	9.5	0.024	ND	ND	ND	ND	2.5	0.002	ND	ND
Total characterised	62.7	0.014	64.5	0.164	61.7	0.015	54.6	0.010	72.9	0.068	97.6	0.079
Unknowns*	18.7	0.004 (4)	17.86	0.046 (4)	20.81	0.005 (4)	25.4	0.005 (4)	12.5	0.011 (5)	-	-

Notes:
ND: Not detected
† Sum of isomers
* number of unknowns shown in parentheses

Conclusions–Animals

Animal metabolism have been conducted with [pyrazolyl-4-^{14}C] inpyrfluxam and [phenyl-U-^{14}C] inpyrfluxam in laying hens (poultry) and lactating goats (ruminants). The majority of the administered dose was rapidly excreted and parent inpyrfluxam was extensively metabolised in several poultry and ruminant matrices, proceeding via several pathways:

- Oxidation to form 1'-CH$_2$OH-S-2840 isomers, which is further transformed by conjugation to sulfate or glucuronic acid, or by oxidation to form 1'-COOH-S-2840 isomers and to 1',1'-bis-(CH$_2$OH)-S-2840;

- *N*-demethylation to form *N*-des-Me-S-2840, amide cleavage to form DFPA-$CONH_2$ or oxidation to 3'-OH-S-2840.

The proposed metabolic pathway for inpyrfluxam in livestock is shown in Figure 7.

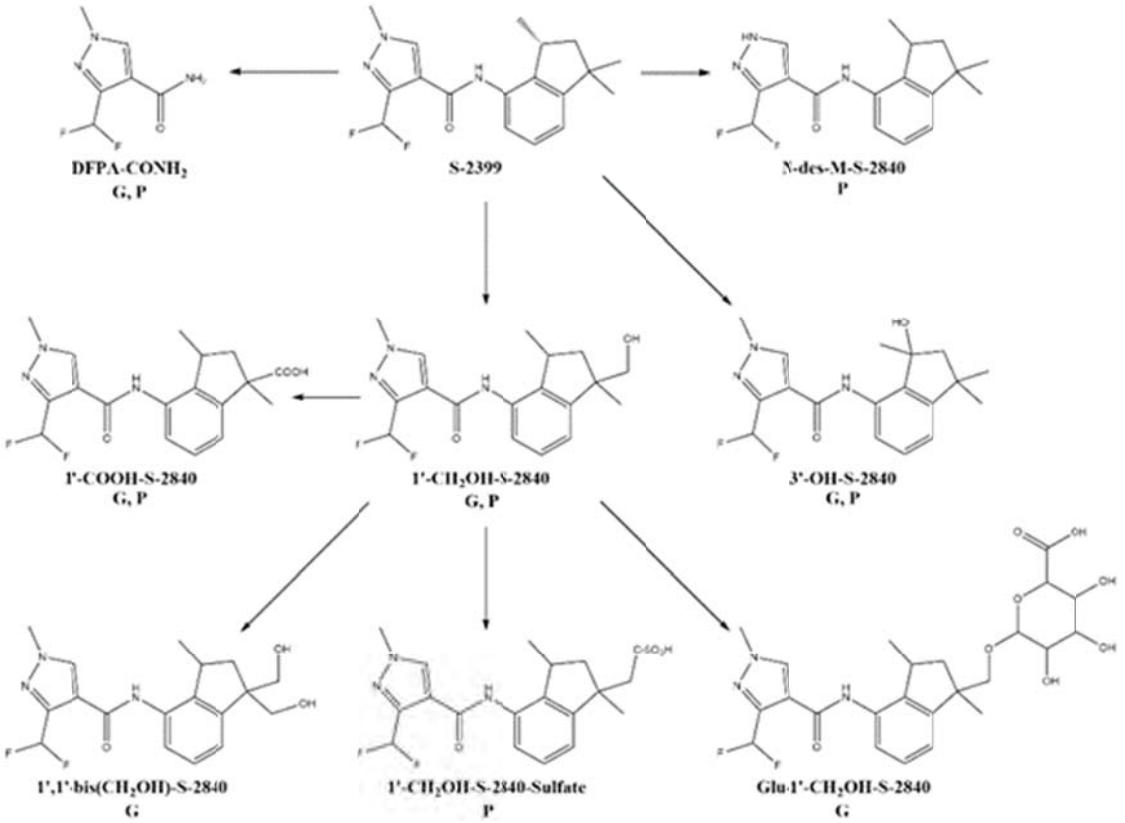


Figure 7 Proposed metabolic pathway of inpyrfluxam in livestock

Notes:
G= goat.
P=poultry.

Rotational crop studies

The Meeting received data from a radiolabelled, confined study in rotational crops (sorghum, lettuce and radish) as well as field rotational crop studies for lettuce (leafy crop), carrot (root crop) and wheat or barley (grain crops).

Confined rotational crop studies

A confined rotational crop study was conducted in California (United States) during the years 2015–2017 (Jalal, 2017; Report: TPM-0047). inpyrfluxam as [phenyl-^{14}C] inpyrfluxam and -[pyrazolyl-^{14}C]inpyrfluxam was applied outdoor to bare, soil (sandy loam) as a spray application formulated in 40 SC formulation at a rate of nominally 235 g ai/ha. Rotational crops were planted into the treated soil 30, 120 and 365 days after application. Samples of immature and mature lettuce leaves, radish roots and tops and sorghum forage, stover and grain were harvested. In addition, surface soil samples were collected immediately after application and at the time of sowing for each crop.

Total radioactivity in the sample matrices was measured by combustion/LSC. For samples with radioactivity levels greater than 0.01 mg/kg eq, samples were extracted twice with acetonitrile, twice with

water and a final rinse was performed with acetonitrile (using a solvent extractor). Post-extraction solids (PES) were measured by combustion/LSC. Representative PES samples containing significant radioactivity were characterised by sequential acidic hydrolytic treatments (2M HCl in 100 °C for 2–4 hours). In samples containing significant TRR (≥ 0.01 mg/kg), further analysis was conducted to identify the components of the radioactivity using HPLC and 2D-TLC by co-injection of reference standards.

Residues in all crops declined over time (Table 26). In lettuce, TRRs ranged from 0.012 mg/kg (mature, 365 DAT) to 0.103 mg/kg (immature, 120 DAT). For radish, the TRRs ranged between 0.022 mg/kg (mature roots, 365 DAT) to 0.367 mg/kg (mature tops, 120 DAT). In sorghum, total residues ranged from 0.012 mg/kg (sorghum grain, 30 and 120 DAT) to 1.074 mg/kg (sorghum stover, 120 DAT).

As to ensure that residues are stable during the study period, freezer-stored crop samples (30 DAT mature lettuce, mature radish tops and roots, sorghum stover and grain) from both radiolabels were re-extracted and analysed 17–21 months after the initial extraction. The distribution of residues in the extracts and PES was similar for the initial and final extractions.

Total radioactive residues in soil of the treated plots were 0.8–2.3 mg/kg. In the aerobic soil degradation study by Jalal (2017, Report: TPM-0023) an average half-life (DT_{50}) of inpyrfluxam in aerobic soil was over 365 days, thus varying portions of the applied inpyrfluxam or its soil metabolites are available to the crops planted 30, 120 and 365 DAT. As soil metabolites 3'-OH-S-2840 and 1'-COOH-S-2840 were the major degradates of inpyrfluxam in soil under aerobic conditions, thus uptake of these metabolites cannot be excluded.

Table 26 Summary of total radioactive residues in rotational crop matrices following soil application of [^{14}C]inpyrfluxam at a rate of 235 g ai/ha

Crop	Crop matrix	TRR (mg/kg eq)					
		[Pyrazolyl-^{14}C]			[Phenyl-^{14}C]		
		30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT
Lettuce	Immature (BBCH 44)	0.080	0.103	0.039	0.045	0.052	0.023
	Mature (BBCH 49)	0.074	0.093	0.025	0.094	0.069	0.012
Radish	Immature tops (BBCH 44)	0.139	0.230	0.101	0.112	0.106	0.088
	Immature roots (BBCH 44)	0.040	0.059	0.024	0.033	0.029	0.021
	Mature tops (BBCH 49)	0.228	0.367	0.073	0.136	0.117	0.092
	Mature roots (BBCH 49)	0.065	0.108	0.022	0.044	0.030	0.028
Sorghum	Forage (BBCH 85)	0.209	0.180	0.047	0.102	0.135	0.035
	Stover (BBCH 89)	0.703	0.945	0.236	0.692	1.074	0.133
	Grain (BBCH 89)	0.048	0.058	0.014	0.012	0.012	0.014

Notes:
DAP: Time between planting and harvest.
DAT: Time between treatment and harvest.

In lettuce (Table 27), inpyrfluxam was present in the sample extracts from both radiolabels at each sampling interval at levels between 5.5 percent and 28.9 percent TRR (0.001–0.029 mg/kg) and 17.4 percent and 46.2 percent TRR (0.011–0.019 mg/kg) for the [pyrazolyl-^{14}C] and [phenyl-^{14}C] labels, respectively. Major metabolites present at 30 and 120 DAT for both labels were the conjugated isomers of 1'-CH_2OH-S-2840 (7.6–21.2 percent TRR, 0.008–0.021 mg/kg). For the [pyrazolyl-^{14}C] label, free DFPA was present in all samples at levels between 9.9 percent and 22.4 percent TRR (0.004–0.022 mg/kg) and conjugated DFPA was detected at >10 percent TRR in mature lettuce at 30 and 120 DAT (12.5–16.6 percent TRR, 0.009–0.014 mg/kg). At 365 DAT, *N*-des-Me-DFPA was present in its free form at 28 percent TRR (0.006 mg/kg). In the [phenyl-^{14}C] label, metabolite 3'-OH-S-2840 was identified at 120 and 365 DAT (7.7–11.7 percent TRR; 0.001–0.008 mg/kg) and conjugated 1'-CH_2OH-S-2840 (13.3–21.2 percent TRR;

0.007–0.021 mg/kg) a 30 and 120 DAT. Free 1'-CH_2OH-S-2840 was also detected but at DFPA derivatives were detected.

Table 27 Nature of residue in lettuce as a rotational crop treated with [Pyrazolyl-4-^{14}C] inpyrfluxam and [Phenyl-U-^{14}C]inpyrfluxam by HPLC analysis

Residue component	%TRR (mg/kg eq)					
	Immature lettuce			Mature lettuce		
	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT
[pyrazolyl-^{14}C]inpyrfluxam						
Inpyrfluxam	27.4 (0.021)	28.9 (0.029)	8.3 (0.003)	12.2 (0.009)	11.4 (0.01)	5.5 (0.001)
3'-OH-S-2840 (free)	2.8 (0.002)	5.2 (0.005)	2.3 (0.001)	2.8 (0.002)	3.7 (0.003)	-
3'-OH-S-2840 (conj)	0.9 (0.001)	3.7 (0.004)	-	0.9 (0.001)	1.6 (0.001)	-
N-des-Me-S-2840, free	0.7 (0.001)	-	5.9 (0.002)	-	0.6 (0.001)	-
N-des-Me-S-2840, conj	2.0 (0.002)	-	-	2.2 (0.002)	-	-
1'-CH_2OH-S-2840,free [a]	1.4 (0.002)	0.8 (0.001)	-	3.3 (0.003)	-	-
1'-CH_2OH-S-2840, conj [a]	14.0 (0.01)	7.6 (0.008)	-	13.5 (0.01)	11.3 (0.01)	-
1'-COOH-S-2840,free [a]	0.7 (<0.001)	1.2 (0.001)	-	1.9 (0.002)	1.9 (0.002)	-
1'-COOH-S-2840,conj [a]	2.8 (0.003)	3.0 (0.003)	-	7.3 (0.005)	8.1 (0.007)	-
DFPA, free	19.1 (0.014)	22.4 (0.022)	9.9 (0.004)	13.0 (0.01)	11.7 (0.01)	15.6 (0.004)
DFPA, conj	5.9 (0.004)	6.4 (0.006)	-	12.5 (0.009)	16.6 (0.014)	-
N-des-Me-DFPA, free	7.6 (0.006)	6.8 (0.007)	18.5 (0.007)	8.4 (0.006)	8.1 (0.007)	28.0 (0.006)
N-des-Me-DFPA, conj	-	-	-	1.2 (0.001)	-	-
DFPA-$CONH_2$, free	3.3 (0.002)	2.7 (0.003)	4.5 (0.002)	3.2 (0.002)	2.5 (0.002)	3.1 (0.001)
Others	6.2 (0.005)	4.8 (0.005)	44.0 (0.016)	6.9 (0.005)	9.6 (0.008)	35.2 (0.008)
Total extracted	94.9 (0.072)	93.4 (0.093)	93.4 (0.034)	89.1 (0.066)	87.0 (0.076)	87.5 (0.02)
Total identified	88.7 (0.067)	88.7 (0.089)	49.4 (0.018)	82.2 (0.061)	77.4 (0.067)	52.2 (0.012)
Total unidentified	6.2 (0.005)	4.8 (0.005)	44 (0.016)	6.9 (0.005)	9.6 (0.008)	35.2 (0.008)
[phenyl-^{14}C]inpyrfluxam						
inpyrfluxam	46.2 (0.019)	41.8 (0.022)	26.4 (0.005)	26.9 (0.027)	17.4 (0.011)	27.2 (0.003)
3'-OH-S-2840, free	8.5 (0.004)	10.9 (0.006)	10.5 (0.002)	7.5 (0.008)	11.7 (0.008)	7.7 (0.001)
3'-OH-S-2840, conj	3.1 (0.001)	4.2 (0.002)	-	2.9 (0.003)	5.9 (0.004)	-
N-des-Me-S-2840, free	1.0 (<0.001)	0.6 (<0.001)	-	-	0.8 (<0.001)	1.3 (<0.001)
N-des-Me-S-2840, conj	2.6 (0.001)	-	-	2.8 (0.003)	-	-
1'-CH_2OH-S-2840,free [a]	2.6 (0.002)	2.2 (0.001)	5.6 (0.001)	3.6 (0.003)	9.0 (0.006)	2.4 (<0.001)
1'-CH_2OH-S-2840, conj [a]	15.7 (0.007)	13.3 (0.007)	-	21.2 (0.021)	14.8 (0.01)	-
1'-COOH-S-2840,free [a]	1.2 (<0.001)	2.9 (0.002)	4.3 (0.001)	1.5 (0.002)	6.0 (0.004)	3.1 (<0.001)
1'-COOH-S-2840,conj [a]	4.2 (0.002)	3.9 (0.002)	-	7.9 (0.008)	8.6 (0.005)	-
N-des-Me-1'-CH_2OH-S-2840, conj	-	-	-	0.9 (0.001)	-	-
Others	9.8 (0.004)	15.7 (0.008)	46.8 (0.01)	18.1 (0.018)	19.1 (0.012)	51 (0.005)
Total extracted	95.0 (0.04)	95.6 (0.049)	93.7 (0.019)	93.4 (0.093)	93.2 (0.06)	93.8 (0.01)
Total identified	85.3 (0.036)	79.9 (0.041)	46.8 (0.01)	75.3 (0.075)	74.1 (0.047)	42.8 (0.004)
Total unidentified [b]	9.8 (0.004)	15.7 (0.008)	46.8 (0.01)	18.1 (0.018)	19.1 (0.012)	51.0 (0.005)

Notes:
[a] Sum of isomers.
[b] Contains no more than 12 components, of which the largest was 25.7% TRR, 0.005 mg/kg.

In radish tops (Table 28), inpyrfluxam represents the major component of the residue at 30 DAT (6.1–15 percent TRR, 0.014–0.017 mg/kg) and one sample of mature radish at 365 DAT (10.5 percent TRR, 0.009 mg/kg). In the [pyrazolyl-^{14}C] label, metabolites *N*-des-Me-S-2840, DFPA-$CONH_2$ and conjugates of DFPA, *N*-des-Me-1'-CH_2OH-S-2840 and 1'-COOH-S-2840 were detected at significant levels (up to 17.1 percent TRR, up to 0.038 mg/kg). In the [phenyl-$^{1,\ 4}$C] label *N*-des-Me-S-2840 (free) and

conjugates of *N*-des-Me-1'-CH_2OH-S-2840, 3'-OH-S-2840 and 1'-COOH-S-2840 were detected up to levels of 22.1 percent TRR (0.019 mg/kg).

In radish roots (Table 28), inpyrfluxam represents the major component of the residue at 30, 120 and 365 DAT of both immature (28.2–58.9 percent TRR, 0.006–0.019 mg/kg) and mature samples (33.0–54.8 percent TRR, 0.007–0.045 mg/kg). Other major components (>10 percent TRR) were identified as non-conjugated DFPA, 3'-OH-S-2840, 1'-COOH-S-2840 at 23.6 (0.005 mg/kg), 11.9 (0.003 mg/kg) and 25.7 (0.006 mg/kg) percent TRR, respectively in immature roots. The PES fractions from the [pyrazolyl-^{14}C] and [phenyl-^{14}C] labelled immature and mature radish roots from all planting periods contained low levels of residues (≤0.004 mg/kg) and were therefore not analysed further.

Table 28 Nature of residue in radish as a rotational crop treated with [pyrazolyl-4-^{14}C] inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam by HPLC analysis

Residue component	%TRR (mg/kg eq)											
	Immature radish						Mature radish					
	Tops			Roots			Tops			Roots		
	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT
[pyrazolyl-^{14}C]inpyrfluxam												
Inpyrfluxam	14.0 (0.019)	7.9 (0.017)	6.5 (0.006)	52.1 (0.022)	46.7 (0.027)	28.2 (0.006)	6.1 (0.014)	6.9 (0.025)	7.6 (0.005)	57.3 (0.038)	41.2 (0.045)	33.0 (0.007)
3'-OH-S-2840(free)	1.8 (0.002)	1.2 (0.003)	-	5.1 (0.002)	6.6 (0.004)	4.4 (0.001)	0.8 (0.002)	1.4 (0.005)	-	4.8 (0.003)	4.0 (0.004)	4.5 (0.001)
3'-OH-S-2840 (conj)	0.3 (<0.001)	2.0 (0.004)	0.3 (<0.001)	-	-	-	1.0 (0.002)	2.7 (0.010)	2.0 (0.001)	-	0.2 (<0.001)	-
N-des-Me-S-2840 (free)	11.3 (0.015)	12.6 (0.027)	7.4 (0.007)	1.0 (<0.001)	1.6 (0.001)	2.1 (<0.001)	10.3 (0.023)	10.2 (0.038)	12.8 (0.009)	1.8 (0.001)	7.6 (0.008)	1.8 (<0.001)
N-des-Me-S-2840 (conj)	1.4 (0.002)	-	-	-	-	-	2.5 (0.006)	-	-	-	-	-
1'-CH_2OH-S-2840† (free)	0.9 (0.001)	-	-	2.0 (0.001)	3.0 (0.002)	-	-	1.7 (0.006)	-	2.2 (0.001)	3.5 (0.004)	-
1'-CH_2OH-S-2840† (conj)	2.9 (0.004)	6.5 (0.013)	4.6 (0.005)	-	-	-	7.9 (0.018)	8.3 (0.030)	6.4 (0.005)	-	4.5 (0.005)	-
1'-COOH-S-2840† (free)	1.8 (0.002)	0.9 (0.002)	-	8.7 (0.004)	6.2 (0.004)	14.3 (0.003)	0.7 (0.002)	1.9 (0.006)	2.7 (0.002)	3.1 (0.003)	6.3 (0.007)	4.6 (0.001)
1'-COOH-S-2840† (conj)	1.0 (0.002)	3.2 (0.007)	12.0 (0.011)	-	-	-	5.5 (0.012)	5.3 (0.020)	8.1 (0.006)	-	7.1 (0.008)	-
N-des-Me-1'-CH_2OH-S-2840† (free)	2.5 (0.003)	2.1 (0.004)	-	-	0.8 (<0.001)	-	-	2.6 (0.010)	-	-	0.5 (0.001)	-
N-des-Me-1'-CH_2OH-S-2840† (conj)	6.2 (0.008)	12.3 (0.026)	5.1 (0.005)	-	-	-	13.0 (0.029)	12.4 (0.046)	5.4 (0.004)	-	0.3 (<0.001)	-
DFPA (free)	3.5 (0.005)	3.0 (0.006)	6.7 (0.006)	11.0 (0.005)	11.2 (0.007)	23.6 (0.005)	2.0 (0.005)	3.3 (0.012)	4.7 (0.003)	4.2 (0.003)	6.1 (0.007)	13.7 (0.003)
DFPA (conj)	10.2 (0.014)	3.7 (0.008)	8.4 (0.008)	-	-	-	8.3 (0.019)	6.2 (0.023)	8.7 (0.006)	-	6.5 (0.007)	-
N-des-Me-DFPA (free)	1.8 (0.002)	1.5 (0.003)	6.3 (0.014)	3.3 (0.001)	3.2 (0.002)	-	1.7 (0.004)	-	6.6 (0.005)	1.1 (0.001)	1.2 (0.001)	-
N-des-Me-DFPA (conj)	3.0 (0.004)	2.9 (0.006)	1.3 (0.001)	-	-	-	4.7 (0.011)	3.4 (0.012)	2.7 (0.002)	-	0.6 (0.001)	-
DFPA-$CONH_2$ (free)	18.5 (0.025)	17.1 (0.037)	14.3 (0.013)	-	3.2 (0.002)	-	10.3 (0.023)	7.2 (0.027)	9.9 (0.007)	-	1.4 (0.001)	-
DFPA-$CONH_2$ (conj)	0.5 (0.001)	-	-	-	-	-	-	-	-	-	-	-
Others	13.1 (0.017)	17.8 (0.038)	20.9 (0.020)	12.4 (0.005)	16.4 (0.010)	24.2 (0.005)	18.3 (0.041)	16.0 (0.059)	16.4 (0.012)	21.7 (0.014)	5.1 (0.006)	36.7 (0.008)
Total extracted	*94.6 (0.126)*	*94.8 (0.202)*	*93.8 (0.089)*	*95.7 (0.040)*	*95.8 (0.056)*	*96.8 (0.022)*	*93.1 (0.210)*	*89.5 (0.331*	*94.0 (0.067)*	*96.0 (0.063)*	*96.1 (0.104)*	*94.3 (0.019)*

Residue component	%TRR (mg/kg eq)											
	Immature radish						Mature radish					
	Tops			Roots			Tops			Roots		
	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT
Total identified	*81.5 (0.108)*	*77.0 (0.164)*	*72.9 (0.069)*	*83.3 (0.035)*	*79.4 (0.047)*	*72.6 (0.017)*	*74.8 (0.169)*	*73.5 (0.272)*	*77.6 (0.055)*	*74.4 (0.049)*	*91.0 (0.099)*	*57.6 (0.012)*
Total unidentified ‡	*13.1 (0.017)*	*17.8 (0.038)*	*20.9 (0.020)*	*12.4 (0.005)*	*16.4 (0.010)*	*24.2 (0.005)*	*18.3 (0.041)*	*16.0 (0.059)*	*16.4 (0.012)*	*21.7 (0.014)*	*5.1 (0.006)*	*36.7 (0.008)*
[phenyl-^{14}C]inpyrfluxam												
inpyrfluxam	15.0 (0.017)	9.9 (0.010)	6.4 (0.005)	58.9 (0.019)	43.1 (0.012)	40.2 (0.009)	12.3 (0.016)	8.3 (0.009)	10.5 (0.009)	54.8 (0.026)	34.9 (0.010)	48.7 (0.012)
3'-OH-S-2840 (free)	3.4 (0.004)	3.6 (0.004)	3.5 (0.003)	9.8 (0.003)	11.9 (0.003)	11.0 (0.002)	3.1 (0.004)	3.6 (0.004)	3.6 (0.003)	8.7 (0.004)	9.9 (0.003)	11.1 (0.003)
3'-OH-S-2840 (conj)	0.7 (0.001)	6.5 (0.007)	-	-	-	-	10.2 (0.013)	2.5 (0.003)	6.4 (0.005)	-	-	-
N-des-Me-S-2840 (free)	14.3 (0.016)	12.4 (0.013)	10.2 (0.009)	1.9 (0.001)	1.1 (<0.001)	-	10.7 (0.014)	10.9 (0.011)	13.6 (0.012)	2.0 (0.001)	1.6 (<0.001)	-
N-des-Me-S-2840 (conj)	0.8 (0.001)	-	-	-	-	-	0.7 (0.001)	-	-	-	-	-
1'-CH2OH-S-2840† (free)	-	-	-	2.6 (0.001)	2.3 (0.001)	-	-	1.0 (0.001)	-	2.7 (0.001)	2.3 (0.001)	-
1'-CH2OH-S-2840† (conj)	6.2 (0.007)	3.8 (0.004)	7.7 (0.007)	-	-	-	7.9 (0.010)	5.4 (0.005)	8.4 (0.007)	-	-	-
1'-COOH-S-2840† (free)	1.3 (0.001)	1.0 (0.001)	4.0 (0.003)	5.8 (0.002)	11.7 (0.003)	25.7 (0.006)	1.8 (0.002)	2.5 (0.003)	-	9.8 (0.005)	15.7 (0.005)	19.1 (0.005)
1'-COOH-S-2840† (conj)	2.5 (0.002)	7.0 (0.008)	21.6 (0.018)	-	-	-	5.5 (0.007)	13.4 (0.014)	22.1 (0.019)	-	-	-
N-des-Me-1'-CH2OH-S-2840† (free)	1.7 (0.002)	2.2 (0.002)	-	1.6 (0.001)	0.6 (<0.001)	-	1.4 (0.002)	2.4 (0.002)	-	-	-	-
N-des-Me-1'-CH2OH-S-2840† (conj)	13.0 (0.015)	8.2 (0.008)	7.6 (0.007)	-	-	-	9.8 (0.012)	11.5 (0.012)	7.9 (0.007)	-	-	-
Others	31.4 (0.036)	38.9 (0.039)	34.0 (0.029)	12.9 (0.004)	19.1 (0.005)	18.7 (0.004)	27.0 (0.035)	30.9 (0.032)	22.4 (0.019)	16.8 (0.008)	29.1 (0.009)	17.8 (0.004)
Total extracted	*90.3 (0.104)*	*93.5 (0.095)*	*95.0 (0.081)*	*93.4 (0.030)*	*89.8 (0.024)*	*95.6 (0.021)*	*90.3 (0.116)*	*92.4 (0.095)*	*94.9 (0.081)*	*94.6 (0.045)*	*93.4 (0.028)*	*96.7 (0.024)*
Total identified	*58.9 (0.068)*	*54.6 (0.055)*	*61.0 (0.052)*	*80.5 (0.026)*	*70.7 (0.019)*	*77.0 (0.017)*	*63.3 (0.081)*	*61.5 (0.063)*	*72.5 (0.062)*	*77.8 (0.037)*	*64.3 (0.019)*	*78.9 (0.020)*
Total unidentified ‡	*31.4 (0.036)*	*38.9 (0.039)*	*34.0 (0.029)*	*12.9 (0.004)*	*19.1 (0.005)*	*18.7 (0.004)*	*27.0 (0.035)*	*30.9 (0.032)*	*22.4 (0.019)*	*16.8 (0.008)*	*29.1 (0.009)*	*17.8 (0.020)*

Notes:
DAT: Days after treatment.
Conj: Conjugate.
† Sum of isomers.
‡ Contains no more than 11 components, of which the largest was 15.8% TRR, 0.005 mg/kg.

In sorghum forage (Table 29), the parent compound inpyrfluxam accounted for only 4.1 percent TRR and 0.007 mg/kg, with only trace amounts observed at 365 DAT in the [pyrazolyl-^{14}C] label. At 30 DAT, the only major component (>10 percent TRR) was free 1'-CH_2OH-S-2840 which was present in the [phenyl-^{14}C] label only (13.8 percent TRR, 0.013 mg/kg).

At 120 DAT, conjugated 1'-CH_2OH-S-2840 (13 percent TRR, 0.015 mg/kg) and *N*-des-Me-1'-CH_2OH-S-2840 conjugate (12.3 percent TRR, 0.015 mg/kg) were identified. Last at 365 DAT, *N*-des-Me-1'-CH_2OH-S-2840 conjugate was the only major component detectable (10.1 percent TRR, 0.004 mg/kg) in the phenyl-^{14}C label). In the [pyrazolyl-^{14}C] labelled samples, the only major metabolite detected was the conjugated DFPA (11.1 percent TRR, 0.021 mg/kg) at 120 DAT.

In sorghum stover, inpyrfluxam was present at minor levels and accounted for no more than 1.8 percent TRR (0.020 mg/kg) in both radiolabels. At 30 DAT, the conjugated 1'-CH_2OH-S-2840 was identified in both the [phenyl-^{14}C] label (13.5 percent TRR, 0.092 mg/kg) and the [pyrazolyl-^{14}C] label (10.3 percent TRR, 0.078 mg/kg). Also present in the [pyrazolyl-^{14}C] labelled extracts at major levels was the conjugated DFPA conjugate at 11 percent TRR (0.083 mg/kg). At 120 DAT, the conjugated 1'-CH_2OH-S-2840 was detected (13.3 percent TRR, 0.167 mg/kg) in the [phenyl-^{14}C] label and the conjugated DFPA (11.6 percent TRR, 0.129 mg/kg) in the [pyrazolyl-^{14}C] label. At 365 DAT, no metabolites were detected at levels exceeding 10 percent TRR.

In sorghum grain, the total residue in the acetonitrile extracts from both radiolabels of all planting intervals was below 0.001 mg/kg. In the 30 DAT aqueous extract of the [phenyl-^{14}C] label, residue levels were low (0.006 mg/kg) and not analysed further, whilst for the [pyrazolyl-^{14}C] label, 59.9 percent TRR (0.029 mg/kg) was extracted. Chromatographic identification and acid hydrolysis showed that the extracted residue contained the conjugated forms of DFPA (22.6 percent TRR, 0.011 mg/kg) and *N*-des-Me-DFPA (8.6 percent TRR, 0.004 mg/kg). For the 120 DAT samples labelled with [pyrazolyl-^{14}C]inpyrfluxam, the aqueous extract contained 49 percent TRR (0.031 mg/kg) and contained conjugated material which was postulated to contain the same metabolites as were present in the 30 DAT extracts. Residues from the [phenyl-^{14}C] label were not analysed further due to low levels present (0.005 mg/kg). For the 365 DAT samples, residue levels in the aqueous extracts were below 0.01 mg/kg and were therefore not analysed further. The 365 DAT [phenyl-^{14}C] and 120 DAT [pyrazolyl-^{14}C] labelled sorghum grain contained 0.006 mg/kg and 0.031 mg/kg (48 percent and 50 percent TRR), respectively as bound residues in PES. Acid hydrolysis and solvent partitioning of the [phenyl-^{14}C] labelled grain PES with ethyl acetate released 0.002 mg/kg of the bound residues. The extract was not further analysed due to low radioactivity levels. Metabolite identification of the acid hydrolysed PES from the [pyrazolyl-^{14}C] label showed DFPA as the major component (14 percent TRR, 0.008 mg/kg) with other minor metabolites below 4 percent TRR. The base hydrolysed protein and lignin fractions from the [phenyl-^{14}C] labelled samples accounted for <0.002 mg/kg and the fractions from the [pyrazolyl-^{14}C] labelled samples accounted for 0.007 mg/kg and 0.001 mg/kg, respectively. The unhydrolysed cellulose-containing fractions remaining after acid and base hydrolysis of each PES retained approximately 0.001–0.002 mg/kg of residue.

The results in sorgun commodities are shown in Table 29.

Table 29 Nature of residue in sorghum as a rotational crop treated with [pyrazolyl-4-^{14}C] inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam by HPLC analysis

Residue component	% TRR (mg/kg eq)								
	sorghum forage			Sorghum stover			sorghum grain		
	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT
[pyrazolyl-^{14}C]inpyrfluxam									
Inpyrfluxam	3.4 (0.007)	1.7 (0.003)	0.5 (<0.001)	1.0 (0.008)	1.8 (0.020)	1.0 (0.002)	-	-	-
3'-OH-S-2840(free)	3.1 (0.006)	3.8 (0.007)	1.4 (0.001)	1.2 (0.009)	1.6 (0.017)	2.1 (0.004)	-	-	-
3'-OH-S-2840 (conj)	0.5 (0.001)	-	3.6 (0.002)	4.3 (0.032)	3.4 (0.038)	5.5 (0.012)	-	-	-
N-des-Me-S-2840 (free)	0.4 (0.001)	0.3 (0.001)	-	0.1 (<0.001)	-	-	-	-	-
N-des-Me-S-2840 (conj)	-	-	-	-	-	1.6 (0.003)	-	-	-
1'-CH_2OH-S-2840[†] (free)	-	-	-	0.7 (0.004)	0.3 (0.003)	-	-	-	-

Residue component	% TRR (mg/kg eq)								
	sorghum forage			Sorghum stover			sorghum grain		
	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT
1'-CH_2OH-S-2840[†] (conj)	7.8 (0.016)	3.5 (0.006)	7.8 (0.003)	10.3 (0.078)	7.2 (0.080)	3.1 (0.006)	-	-	-
1'-COOH-S-2840[†] (free)	-	-	-	-	-	-	-	-	-
1'-COOH-S-2840[†] (conj)	1.7 (0.003)	0.6 (0.002)	3.9 (0.002)	2.4 (0.018)	3.4 (0.038)	2.2 (0.005)	-	-	-
N-des-Me-1'-CH_2OH-S-2840[†] (free)	-	-	3.8 (0.002)	0.4 (0.003)	-	-	-	-	-
N-des-Me-1'-CH_2OH-S-2840[†] (conj)	7.5 (0.015)	3.9 (0.007)	2.5 (0.001)	5.5 (0.041)	4.2 (0.046)	2.3 (0.005)	-	-	-
DFPA (free)	1.8 (0.004)	2.2 (0.004)	0.7 (<0.001)	0.7 (0.005)	0.3 (0.004)	-	-	-	-
DFPA (conj)	9.3 (0.019)	11.1 (0.021)	9.8 (0.005)	11.0 (0.083)	11.6 (0.129)	6.1 (0.013)	22.6 (0.011)	-	-
N-des-Me-DFPA (free)	0.7 (0.001)	1.4 (0.003)	-	0.3 (0.003)	0.2 (0.002)	-	-	-	-
N-des-Me-DFPA (conj)	7.6 (0.015)	7.1 (0.013)	5.8 (0.003)	4.8 (0.036)	4.5 (0.059)	3.8 (0.008)	8.6 (0.004)	-	-
DFPA-$CONH_2$ (free)	1.8 (0.004)	2.9 (0.006)	1.8 (0.001)	0.3 (0.002)	1.4 (0.016)	1.4 (0.003)	-	-	-
Others	40.4 (0.081)	39.3 (0.073)	35.8 (0.017)	33.7 (0.253)	28.9 (0.430)	49.7 (0.104)	28.7 (0.014)	49.8 (0.031)	47.4 (0.008)
Total extracted	*85.9 (0.171)*	*77.9 (0.145)*	*77.4 (0.036)*	*76.7 (0.577)*	*78.8 (0.871)*	*78.7 (0.165)*	*59.9 (0.029)*	*49.8 (0.031)*	*47.4 (0.008)*
Total identified	*45.5 (0.091)*	*38.6 (0.072)*	*41.7 (0.020)*	*43.0 (0.324)*	*39.9 (0.441*	*28.9 (0.061)*	*31.2 (0.016)*	-	-
Total unidentified [‡]	*40.4 (0.081)*	*39.3 (0.073)*	*35.8 (0.017)*	*33.7 (0.253)*	*38.9 (0.430)*	*49.7 (0.104)*	*28.7 (0.014)*	*49.8 (0.031)*	*47.4 (0.008)*
[phenyl-^{14}C]inpyrfluxam									
inpyrfluxam	3.6 (0.003)	4.1 (0.005)	-	0.8 (0.006)	0.9 (0.011)	1.3 (0.002)	-	-	-
3'-OH-S-2840(free)	2.7 (0.003)	4.4 (0.005)	1.4 (0.001)	1.1 (0.008)	2.6 (0.033)	3.5 (0.005)	-	-	-
3'-OH-S-2840 (conj)	3.1 (0.003)	-	3.6 (0.001)	5.5 (0.038)	7.8 (0.098)	8.6 (0.012)	-	-	-
N-des-Me-S-2840 (free)	0.3 (<0.001)	-	-	<0.1 (<0.001)	-	-	-	-	-
N-des-Me-S-2840 (conj)	-	-	-	-	-	2.1 (0.003)	-	-	-
1'-CH_2OH-S-2840[†] (free)	13.8 (0.013)	-	-	0.4 (0.003)	2.2 (0.027)	-	-	-	-
1'-CH_2OH-S-2840[†] (conj)	-	13.0 (0.015)	9.7 (0.003)	13.5 (0.092)	13.3 (0.167)	3.7 (0.005)	-	-	-
1'-COOH-S-2840[†] (free)	4.4 (0.004)	-	-	-		-	-	-	-
1'-COOH-S-2840[†] (conj)	-	1.6 (0.002)	4.6 (0.002)	3.7 (0.025)	3.8 (0.048)	4.5 (0.006)	-	-	-
N-des-Me-1'-CH_2OH-S-2840[†] (free)	9.0 (0.009)	-	-	0.3 (0.002)	0.5 (0.006)	-	-	-	-
N-des-Me-1'-CH_2OH-S-2840[†]	-	12.3 (0.015)	10.1 (0.004)	7.6 (0.051)	8.9 (0.111)	3.9 (0.005)	-	-	-

Residue component	% TRR (mg/kg eq)								
	sorghum forage			Sorghum stover			sorghum grain		
	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT
(conj)									
Others	45.7 (0.043)	49.1 (0.058)	51.0 (0.018)	43.5 (0.297)	42.1 (0.528)	49.9 (0.068)	49.9 (0.006)	51.7 (0.005)	52.4 (0.007)
Total extracted	*82.6 (0.078)*	*84.5 (0.100)*	*80.3 (0.028)*	*76.6 (0.523)*	*81.9 (1.029)*	*77.6 (0.105)*	*49.9 (0.006)*	*51.7 (0.005)*	*52.4 (0.007)*
Total identified	*36.9 (0.035)*	*35.4 (0.042)*	*29.3 (0.010)*	*33.1 (0.226)*	*39.8 (0.501)*	*27.7 (0.038)*	-	-	-
Total unidentified ‡	*45.7 (0.043)*	*49.1 (0.058)*	*51.0 (0.018)*	*43.5 (0.297)*	*42.1 (0.528)*	*49.9 (0.068)*	*49.9 (0.006)*	*51.7 (0.005)*	*52.4 (0.007)*

Notes:
DAT: Days after treatment.
Conj: Conjugate.
† Sum of isomers.
‡ Contains no more than 11 components, of which the largest was 15.8% TRR, 0.005 mg/kg.

The proposed metabolic pathway is shown in Figure 6. Inpyrfluxam was extensively metabolised into a large number of metabolites, many of which formed complex conjugates with naturally occurring compounds and became incorporated as unextractable residues in various plant constituents. Parent inpyrfluxam underwent a number of transformation processes including oxidation, demethylation, amide bond cleavage, as well as combinations of these processes. The primary oxidation products of inpyrfluxam were 3'-OH-S-2840, 1'-CH_2OH-S-2840and 1'-COOH-S-2840 and were present in both free and conjugated forms. The amide bond cleavage of the parent inpyrfluxam and its metabolites produced DFPA and DFPA-$CONH_2$. The metabolites, *N*-des-Me-S-2840, *N*-des-Me-1'-CH_2OH-S-2840 and *N*-des-Me-DFPA were produced as a result of the loss of the *N*-methyl group in the pyrazolyl ring of the parent inpyrfluxam, its oxidation products and the cleavage product, respectively. The radioactivity from both radiolabels were distributed extensively in various fractions of the PES including starch, proteins, lignin and cellulose.

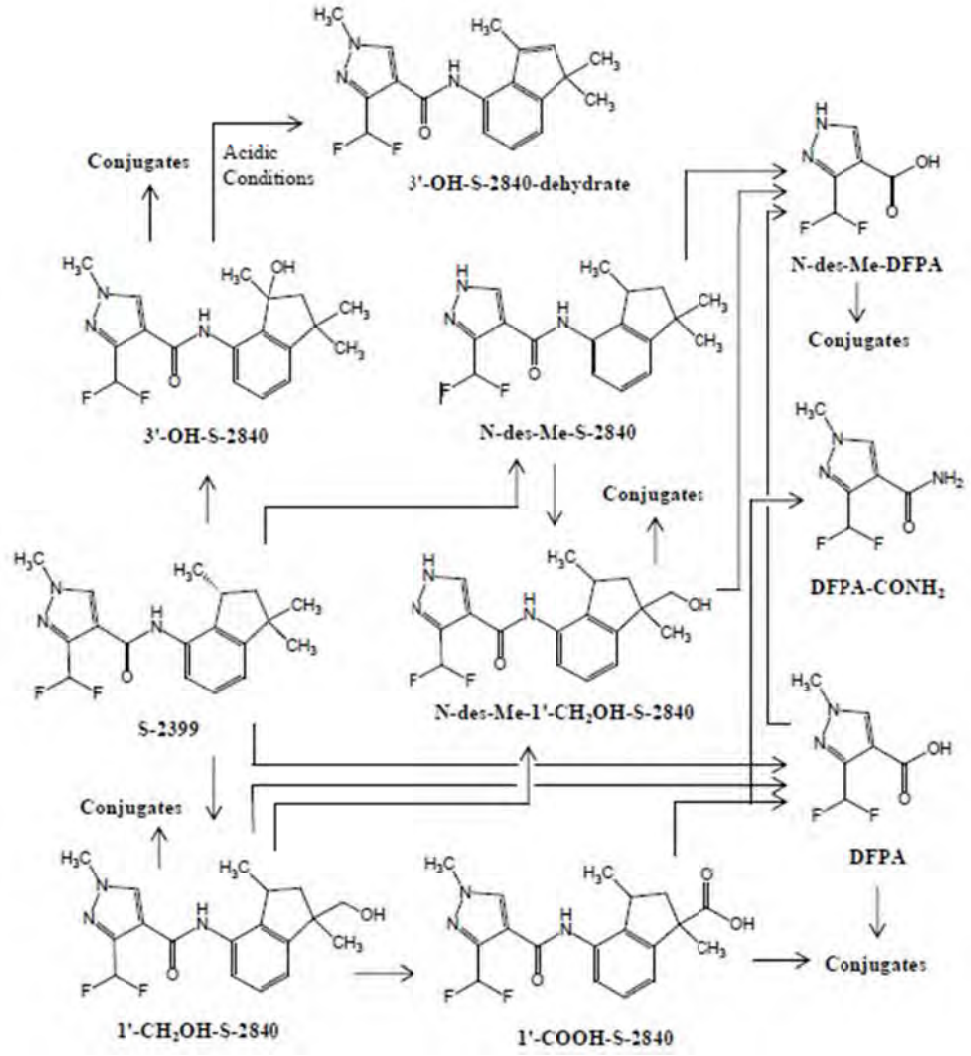


Figure 8 Proposed metabolic pathway for inpyrfluxam in rotational crops

Field rotational crop studies

Six field rotational trials conducted to investigate the magnitude of residues in rotational crops in Europe, Canada and the United States of American were submitted to the Meeting.

In the study conducted in Europe during 2016-2017 (Bousquet, 2018; TPR-0080), one foliar application of inpyrfluxam as a 40 SC was made at a rate of 240 g ai/ha and at BBCH 30 to 65, to winter barley (260-2016 GE01; treated GS 30 or 51-55) and spring wheat (260-2016 IT02; treated GS 31 or 65). In 260-2016 GE01, soil texture was loam with a pH of 6.9 and organic matter content of 2.25 percent. In 260-2016 IT02, soil texture was sandy loam with a pH of 6.8 and organic matter content of 4.98 percent. The primary crop was destroyed to simulate crop failure and incorporated in the soil whilst preparing the soil for rotational crops within 13–14 days of application.

Rotational crops of lettuce (leafy crop), carrot (root crop) and wheat or barley (grain crops) were sown or planted at three plant-back intervals (PBIs) of 28 ± 2, 120 ± 5 and 350 ± 15 days after application and sampled at normal commercial harvest.

Inpyrfluxam and its metabolites were analysed using the previously validated analytical methods, SUM-1601V and SUM-1701V (validation was performed within the reported study). The LOQ was 0.01 mg/kg (0.005 mg/kg for metabolite isomers) for inpyrfluxam, 3'-OH-S-2840, DFPA-$CONH_2$, *N*-des-Me-S-2840 and DFPA. Procedural recoveries for parent and metabolites in lettuce and carrot matrices fortified at the LOQ and 10× LOQ were in the acceptable range of 70–120 percent. Samples were stored frozen for a maximum of 371 days before analysis, which is covered by the storage stability (cucumber/apple, wheat/maize grain and potato tuber).

Residues of inpyrfluxam and its metabolites were < 0.01 mg/kg in all rotated crop matrices at PBIs of 120 ± 5 days and 350 ± 15 days. At the shortest PBI of 28 ± 2 days, all residues in lettuce, carrot roots and tops and cereal grain were <0.01 mg/kg. In cereal straw, residues at the LOQ (0.01 mg/kg) were detected for 3'-OH-S-2840, at 0.017 and 0.019 mg/kg for 1'-COOH-S-2840, at 0.023 mg/kg for 1'-CH_2OH-S-2840 and at 0.09 and 0.1 mg/kg for DFPA (Table 30).

Table 30 Residues of inpyrfluxam and its metabolites in rotational crops (lettuce, carrot, cereals) planted after foliar application with inpyrfluxam (40 SC) at 240 g ai/ha on cereals (crop failure after 13-14 days) in Europe.

Trial ID and Location (Year)	Crop (variety)	Commodity	Harvest DAP[a]	PBI[b] (days)	Residues (mg/kg eq)							
					Inpyrfluxam	3'-OH-S-2840	DFPA-$CONH_2$	*N*-des-Me-S-2840	DFPA	1'-COOH-S-2840[c]	1'-CH_2OH-S-2840[c]	*N*-des-Me-1'-CH_2OH-S-2840[c]
265-2016 GE01 Uedem, North Rhine-Westphalia, Germany, 2016/17	Winter barley (KWS Keeper)	Whole plant	-	-	3.4-8.0	-	-	-	-	-	-	-
	Lettuce (Casanova)	Whole plant	34	33	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Carrot (Almaro)	Roots Tops	113		<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	Spring wheat (Tybalt)	Straw Grain	107		<0.01 <0.01	0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.09 <0.01	0.019 <0.01	0.023 <0.01	<0.01 <0.01
	Lettuce (Zendria)	Whole plant	39	115	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Carrot (Almaro)	Roots Tops	109		<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	Winter barley (KWS Keeper)	Straw Grain	290	120	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	Lettuce (Carasco)	Whole plant	47	363	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Carrot (Almaro)	Roots Tops	93		<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
	Spring wheat (Tybalt)	Straw Grain	144	349	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Trial: 265-2016 IT02 Santa Venerina, Sicily, Italy, 2016/17	Spring wheat (Valbona)	Whole plant	-	0	5.6-7.6	-	-	-	-	-	-	-
	Lettuce (Canasta)	Whole plant	40	27	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Carrot (Berlicum 2)	Roots Tops	103		<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01

Trial ID and Location (Year)	Crop (variety)	Commodity	Harvest DAP[a]	PBI[b] (days)	Residues (mg/kg eq)							
					Inpyrfluxam	3'-OH-S-2840	DFPA-$CONH_2$	*N*-des-Me-S-2840	DFPA	1'-COOH-S-2840[c]	1'-CH_2OH-S-2840[c]	*N*-des-Me-1'-CH_2OH-S-2840[c]
	Spring wheat (Timilia)	Straw	104		<0.01	<0.01	<0.01	<0.01	0.10	0.017	0.023	0.019
		Grain			<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Lettuce (Nauplus)	Whole plant	43	120	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Carrot (Berlicum 2)	Roots	160		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		Tops			<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Winter wheat (Marco Aurelio)	Straw	272		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		Grain			<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Lettuce (Paspartu)	Whole plant	53	336	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Carrot (Berlicum 2)	Roots	132		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		Tops			<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	Spring wheat (Timilia)	Straw	103		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
		Grain			<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Notes:
[a] Days between sowing and sampling the succeeding crop.
[b] Plant-back interval = days between the last application to the treated plot and sowing of succeeding crop.
[c] Analytically determined as two separate groups of isomers but presented summed; 1'-COOH-S-2840A + 1'-COOH-S-2840B; 1'-CH2OH-S-2840A + 1'-CH2OH-S-2840B; N-des-Me-1'-CH2OH-S-2840A + N-des-Me-1'-CH2OH-S-2840B.

In the study conducted in Manitoba (CAN) during 2015 and 2016 (Bitter, 2017, 201700151), one foliar outdoor application of inpyrfluxam as a 2.84 SC was made on wheat (*var.* WFT 603) at BBCH 39-45 at a rate of 100 g ai/ha. Soil texture was sandy clay loam with a pH (water) of 7.7 and organic matter content of 3.1 percent. Sampling of the primary crop was performed after growth to maturity and the crop was collected and destroyed–the protocol states that minimum soil tillage should take place before planting the following crop. Wheat and field peas were planted into the plot 328 days after the last application of test substance formulation and canola was planted 339 days after the last application.

Wheat forage, wheat hay, wheat grain, wheat straw, field pea vines, field pea hay, field pea seed and canola seed were analysed for inpyrfluxam and conjugates using a modification of the Valent analytical method RM-50C. The limit of quantification (LOQ) for each analyte was 0.01 mg/kg in food commodities and 0.02 mg/kg in feed items. The limit of detection (LOD) was 0.005 mg/kg in food commodities and 0.01 mg/kg in feed items. Samples were stored for a maximum period of 157 days prior to analysis, which is supported by the available storage stability studies.

Residues were not detected in any of the matrices from the 11 month PBI. Procedural recoveries for parent and aglycones of DFPA and 1'-CH_2OH-S-2840 (fortified at levels between 0.01 and 0.2 mg/kg) were in the acceptable range of 70-120 percent (Table 31).

Table 31 Residues of inpyrfluxam and its metabolites in rotational crops (wheat, pea, canola) planted after foliar application with inpyrfluxam (2.84 SC) on wheat (crop failure) in Canada.

Trial ID and Location (Year)	Total rate g ai/ha	Crop (variety)	Commodity	Harvest DAP[a]	PBI[b] (days)	Residues (mg/kg)		
						Inpyrfluxam	DFPA[c]	1'-CH_2OH-S-2840B[c]
Trial: VP-38948-A Elgin, Manitoba, Canada 2015	100	Wheat (Cardale)	Forage	36	328	<0.02	<0.02	<0.02
			Hay	64		<0.02	<0.02	<0.02
			Grain	106		<0.01	<0.01	<0.01
			Straw	106		<0.02	<0.02	<0.02
		Field pea (Agassiz)	Vines	61	328	<0.02	<0.02	<0.02
			Hay	61		<0.02	<0.02	<0.02
			Seed	103		<0.01	<0.01	<0.01
		Canola (73-45RR)	Seed	87	339	<0.01	<0.01	<0.01

Notes:
[a] Days between sowing and sampling the succeeding crop.
[b] Plant-back interval = days between the last application to the treated plot and sowing of succeeding crop.
[c] Analysed as aglycones.

In the study conducted in North Dakota (the United States) during 2015 and 2016 (Bitter, 2017, 201700152), one foliar outdoor application of inpyrfluxam as a 2.84 SC was made on wheat (*var.* WFT 603) at a rate of 120 g ai/ha and BBCH 61. Sampling of the primary crop was performed after growth to maturity and the crop was harvested and tilled into the ground near the treated plot . Wheat and canola were planted into the plot 328 days and 312 days after the last application of test substance formulation, respectively.

Canola seed and wheat forage, hay, grain and straw were analysed for inpyrfluxam and conjugates using a modification of the Valent analytical method RM-50C. The limit of quantification for each analyte was 0.01 mg/kg in food commodities and 0.02 mg/kg in feed items. Samples were stored for a maximum period of 143 days prior to analysis, which is supported by the available storage stability studies.

Residues were not detected in any of the matrices from the 10 month PBI (Table 32). Procedural recoveries for parent and aglycones of DFPA and 1'-CH_2OH-S-2840 (fortified at levels between 0.01 and 0.2 mg/kg) were in the acceptable range of 70–120 percent.

Table 32 Residues of inpyrfluxam and its metabolites in rotational crops (wheat, canola) planted after foliar application with inpyrfluxam (2.84 SC) on wheat in United States in 2015

Trial and Location	Total rate g ai/ha	Crop (variety)	Commodity	Harvest DAP[a]	PBI[b] (days)	Residues (mg/kg)		
						Inpyrfluxam	DFPA[c]	1'-CH_2OH-S-2840B[c]
Trial: VP-38944-A Velva, North Dakota	117	Wheat (RB-07)	Forage	38	312	<0.02	<0.02	<0.02
			Hay	63		<0.02	<0.02	<0.02
			Grain	110		<0.01	<0.01	<0.01
			Stover	110		<0.02	<0.02	<0.02
		Canola (Invigor L-140P)	Seed	94	328	<0.01	<0.01	<0.01

Notes:
[a] Days between sowing and sampling the succeeding crop.
[b] Plant-back interval = days between the last application to the treated plot and sowing of succeeding crop.
[c] Analysed as aglycones.

In the study conducted in Oklahoma (United States) during 2015 and 2016 (Bitter, 2017, 01700154), two foliar outdoor application (14 day interval) of inpyrfluxam as a 2.84 SC was made on soya bean (*var.* P39T67R) at a total rate of 210 g ai/ha at BBCH 63–65 and 71. Sampling of the primary crop was performed after growth to maturity and the crop was destroyed; protocol states that after harvest the soil should be lightly tilled following usual agricultural practices. Sorghum and cotton were planted into the plot 273 days after the last application of test substance formulation.

Sorghum forage, grain and stover were analysed for inpyrfluxam and conjugates (using a modification of the Valent analytical method RM-50C. Unginned cotton was collected, processed into cottonseed and cotton gin trash and the seed was analysed for the same metabolites. The limit of quantification for each analyte was 0.005 mg/kg in food commodities and 0.01 mg/kg in feed items. Samples were stored for a maximum period of 110 days prior to analysis, which is supported by the available storage stability studies.

Residues were not detected in any of the matrices from the 9 month PBI (Table 33). Procedural recoveries for parent and aglycones of DFPA and 1'-CH_2OH-S-2840 (fortified at levels between 0.01 and 0.2 mg/kg) were in the acceptable range of 70-120 percent.

Table 33 Residues of inpyrfluxam and its metabolites in rotational crops (sorghum, cotton) planted after foliar application with inpyrfluxam (SC) at 209 g ai/ha the United States in 2015

Trial ID and Location	Crop (variety)	Commodity	Harvest DAP[a]	PBI[b] (days)	Residues (mg/kg)		
					Inpyrfluxam	DFPA[c]	1'-CH_2OH-S-2840B[c]
Trial: VP-38977-A Hinton, Oklahoma	Sorghum (DKS 29-28)	Forage	84	273	<0.02	<0.02	<0.02
		Grain	107		<0.01	<0.01	<0.01
		Stover	107		<0.02	<0.02	<0.02
	Cotton (PHY 367 WRF)	Seed	176		<0.02	<0.02	<0.02
		Gin trash	176		<0.02	<0.02	<0.02

Notes:
[a] Days between sowing and sampling the succeeding crop.
[b] Plant-back interval = days between the last application to the treated plot and sowing of succeeding crop.
[c] Analysed as aglycones.

In the study conducted in Louisiana (United States) during 2015 and 2016 (Bitter, 2017, 201700155) two foliar outdoor application (14 day interval) of inpyrfluxam as a 2.84 SC was made on soya bean (*var.* Asgrow AG 5335) at a total rate of 220 g ai/ha. Sampling of the primary crop was performed after growth to maturity (GS unstated) and the crop was destroyed ; protocol states that after harvest the soil should be lightly tilled following usual agricultural practices. Sorghum and cotton were planted into the plot 267 days after the last application of test substance formulation.

Sorghum forage, grain and stover were analysed for inpyrfluxam and conjugates using a modification of the Valent analytical method RM-50C. Unginned cotton was collected, processed into cottonseed and cotton gin trash and the seed was analysed for the same metabolites. The limit of quantification for each analyte was 0.005 mg/kg in food items and 0.01 mg/kg in feed items. Samples were stored for a maximum period of 111 days prior to analysis, which is supported by the available storage stability studies.

Residues were not detected in any of the matrices from the 9 month PBI (Table 34). Procedural recoveries for parent and aglycones of DFPA and 1'-CH_2OH-S-2840 (fortified at levels between 0.01 and 0.2 mg/kg) were in the acceptable range of 70-120 percent.

Table 34 Residues of inpyrfluxam and its metabolites in rotational crops planted after foliar application with inpyrfluxam (SC) at 219 g ai/ha on soya in the United States in 2015

Trial ID and Location	Crop (variety)	Commodity	Harvest DAP[a]	PBI[b] (days)	Residues (mg/kg)		
					Inpyrfluxam	DFPA[c]	1'-CH_2OH-S-2840B[c]
Trial: VP-39056-A Cheneyville, Louisiana	Sorghum (Pioneer 83G19)	Forage	77	267	<0.02	<0.02	<0.02
		Grain	103		<0.01	<0.01	<0.01
		Stover	103		<0.02	<0.02	<0.02
	Cotton[d] (ST4946GLB2)	Seed	147		<0.02	<0.02	<0.02
		Gin trash	147		<0.02	<0.02	<0.02

Notes:
[a] Days between sowing and sampling the succeeding crop.
[b] Plant-back interval = days between the last application to the treated plot and sowing of succeeding crop.
[c] Analysed as aglycones.
[d] Unginned cotton was processed into seed and gin trash after harvest.

Environmental fate in soil

The Meeting received studies depicting the environmental fate of inpyrfluxam in soils. Soil studies included laboratory under aerobic and anaerobic conditions, photodegradation studies, and field dissipation studies were submitted.

Aerobic degradation in soil

The route of degradation of inpyrfluxam in soil under aerobic condition was investigated by Jalal (2017, TPM-0023) in Penn series soil and by Gohre (2017, TPM-0044) in Atwater, Newhaven and Woodside farm soils. In addition, the route of degradation of metabolites 3'-OH-S-2840 and 1'-COOH-S-2840 were investigated by Cooper (2017, TPM-0033 and TPM-0049).

Inpyrfluxam was labelled at two positions; [pyrazolyl-4-^{14}C]inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam. The study parameters are summarized in Table 35.

Table 35 Summary of soil metabolism and dissipation studies conducted with inpyrfluxam under aerobic conditions

Study ID	Radiolabel position	Application, g ai/ha	Application, mg ai/kg soil dry weight basis, DM	Soil types	Incubation	Sampling (days)
TPM-0023	[pyrazolyl-4-^{14}C]inpyrfluxam	234	0.650	Penn Series	20 ± 2°C	0, 7, 14, 30, 63, 93, 120, 150 and 182
	[phenyl-U-^{14}C]inpyrfluxam	228	0.634		50 ± 10%	
TPM-0044	[pyrazolyl-4-^{14}C]inpyrfluxam	215	0.603	Atwater, Newhaven, Woodside farm	20 ± 2°C	0, 14, 30, 61, 90, 120

The soil extracts were analysed by LSC and 2D TLC and occasionally by HPLC to quantify and identify major radioactive components. In addition, 1 M NaOH traps were included to collect $^{14}CO_2$ and tetraethylene glycol dimethyl ether traps were included to trap radioactive organic volatiles.

In study TPM-0023, the rate and route of inpyrfluxam metabolism was investigated in Penn Series soil. The principal degradation routes were oxidation of the 3'-position in the indenyl ring to

produce 3'-OH-S-2840 (14.4 percent AR phenyl, 15.5 percent AR pyrazolyl) and the oxidation of one of the 1'-CH_3 groups of the indenyl ring to form 1'-COOH-S-2840 (6.6 percent AR phenyl, 5.9 percent AR pyrazolyl). Minor amount of *N*-des-Me-DFPA was observed (<5 percent AR). No isomerization from inpyrfluxam *R*-isomer to its *S*-isomer occurred during the study period. The distribution of the applied radioactivity in inpyrfluxam and its metabolites is presented in Table 36.

Table 36 Distribution (percent of Applied Radioactivity) in inpyrfluxam, in its metabolites, in volatiles and in bound residue fractions of soil at various sampling intervals

	Days After Treatment (DAT)																	
	0		7		14		30		63		93		120		150		182	
Label	Ph	Py	Ph	Py	Ph	Py	Ph	Py	Ph	Py	Ph	Py	Ph	Pyr	Ph	Py	Ph	Py
inpyrfluxam	93.2	93.9	89.4	88.4	87.2	85.8	83.1	82.9	72	72.3	64.5	65.3	62.6	62.1	55.4	56.4	53.5	51.7
3'-OH-Dehydrate	0	0	0.1	0	0.2	0.1	0.2	0.5	1.8	1.2	4.2	1.6	1.4	0.9	1.6	1.3	5.4	3.1
3'-OH-S-2840	1	1.9	1.9	1.8	2.2	2.3	5.1	3.7	9	7.8	7.2	11.3	11.2	11.6	12.6	12.2	14.4	15.5
3'-OH-S-2840 (incl. dehydrate)	1	1.9	2	1.9	2.4	2.4	5.4	4.2	10.8	9	11.4	12.9	12.6	12.5	14.2	13.6	19.7	18.6
N-des-Me-S-2840	0	0	0	0	0.2	0	0.1	0	0.3	0.1	0.3	0.2	0.5	0.4	0.7	0.6	0.8	0.6
1'-COOH-S-2840	0	0	2.5	2	3.8	3.9	5.2	4.6	6	5.2	6.2	5.3	6	5.4	6.6	5.4	6.6	5.9
DFPA	-	0	-	1	-	0.8	-	1	-	0.9	-	0.5	-	0.6	-	0.4	-	0.4
N-des-Me-DFPA	-	0	-	0	-	0	-	0.3	-	0.6	-	0.9	-	0.9	-	1.2	-	1.6
Total Identified	94.2	95.8	93.9	93.2	93.6	93	93.7	92.9	89	88	82.5	85	81.8	81.9	76.8	77.5	80.6	78.9
Total Unknowns [a]	1.4	1.9	2	2.2	1.3	1.6	2.4	1.8	4.3	4.3	6.9	5.6	6	6.5	7.3	6.8	7.4	8.9
Total Extracted	95.6	97.7	95.9	95.5	94.8	94.6	96.2	94.7	93.3	92.3	89.4	90.6	87.8	88.4	84.1	84.3	88	87.7
Bound in PES	0.1	0.1	1.6	1.7	2.8	2.6	3.5	3.3	6.6	6	7.1	7	7.9	8.3	8.8	9.2	8.9	9.5
Volatiles ($^{14}CO_2$)	-	-	0	0	0.1	0	0.2	0.1	0.3	0.1	0.4	0.2	0.5	0.2	0.6	0.3	0.7	0.3
Total Residue (Mass Balance)	95.7	97.8	97.6	97.2	97.7	97.2	99.8	98	100.2	98.4	96.8	97.8	96.2	96.9	93.4	93.8	97.5	97.5

Notes:
Ph: [phenyl-U-^{14}C]inpyrfluxam; Py: [pyrazolyl-4-^{14}C]inpyrfluxam.
[a] Individual peaks did not exceed 2% of applied radioactivity at any sampling interval.

The DT_{50} of inpyrfluxam was 213 days, 240 days, and 242 days, using the SFO, DFOP and IORE kinetic models, respectively. The corresponding DT_{90} were 707, 1149 and 3102 days, respectively. The PestDF results showed that the representative half-life (Slow $t_{1/2}$) DT_{50} was 398 days. The degradation of inpyrfluxam in soil under aerobic conditions was a gradual process, resulting in the formation of one major and many minor metabolites, which were mineralized slowly into the soil lattice of fulvic acid, humic acid and humin.

In the study TPM-0044, the rate and route of inpyrfluxam metabolism was investigated in three soils (Atwater, Newhaven and Woodside farm) under aerobic condition using [pyrazolyl-4-^{14}C]inpyrfluxam (PYR-label) at a rate of 0.603 mg/kg (DM). Treated soil samples were incubated at 20 ± 2 °C and approximately 50 percent of the maximum water holding capacity (MWHC) in the dark for a maximum of 120 days and were periodically collected and extracted. Duplicate soil samples were taken at 0, 14, 30, 61, 90 and 120 days after treatment. The test systems were equipped with NaOH traps for the collection of evolved $^{14}CO_2$ and tetraethylene glycol dimethyl ether traps to trap organic volatiles. The soil extracts were analysed by LSC and by HPLC and 2D TLC to quantify and identify major radioactive components. In addition, radioactive volatiles from the soil were quantified by LSC. The post-extraction soil (PES) was analysed by combustion analysis. The PES sample containing significant radioactivity was fractionated into fulvic acid, humic acid and humin and the residue in each fraction was analysed by LSC and/or combustion analysis and when appropriate, by HPLC.

The average material balance for the study was 98.7 ± 2.2, 98.1 ± 3.5 and 99.3 ± 1.5 percent AR, for Atwater, Newhaven and Woodside farm soils, respectively. The majority of the applied radioactivity was extracted at 120 DAT and only a small portion was bound (9.1 percent AR for the Atwater soil, 12.2 percent AR for the Newhaven soil and 9.3 percent for the Woodside farm soil). The $^{14}CO_2$ produced was insignificant (0.3, 0.8 and 0.3 percent AR for the Atwater, Newhaven and Woodside farm soil respectively).

The principal degradation routes were oxidation of the 1′ methyl group and at the 3-carbon on the indene ring to form 1'-COOH-S-2840 and 3'-OH-S-2840. The degradation products with the largest proportion observed in the study were 3'-OH-S-2840 (maximum 20.7 percent AR) and 1'-COOH-S-2840 (maximum 9.6 percent AR) for the Atwater soil; 1'-COOH-S-2840 (22.8 percent AR), 3'-OH-S-2840(10.5 percent AR) for the Newhaven soil; and 1'-COOH-S-2840 (30.1 percent AR) and 3'-OH-S-2840(8.4 percent AR) for the Woodside farm soil.

Hydrolysis of the central amide linkage or dealkylation of the N-methyl group on the pyrazolyl ring to yield DFPA, DFPA-$CONH_2$ and *N*-des-Me-DFPA or *N*-des-Me-S-2840 was observed in minor yields. No isomerization from inpyrfluxam *R*-isomer to its *S*-isomer occurred during the study period.

The distribution of the applied radioactivity in inpyrfluxam, its metabolites is presented in Table 37.

Table 37 Radioactivity distribution in total soil extracts for the Atwater, Newhaven and Woodside soil trials as percent of applied radioactivity.

	Percent (%) of applied radioactivity [1]						
	Time after application (days)						
	Compound	0	14	30	61	90	120
Atwater	N-des-Me-DFPA	0	0	0	1	1.4	2
	DFPA-CONH2	0	0	0	0.4	0.8	0.8
	DFPA	0	1.3	2	1.9	2	2.4
	1'-CH2OH-S-2840A[2]	0	0	0	0	0	0
	1'-COOH-S-2840A	0	1.8	2.2	3.4	5.7	5.3
	1'-CH2OH-S-2840B	0	0	0	0.1	0	0
	1'-COOH-S-2840B	0	2.5	2.7	3.4	3.9	2.2
	3'-OH-S-2840	2.1	4.2	6.3	11	16	21
	N-des-Me-S-2840	0	0	0	0.2	0	3.3
	inpyrfluxam	95	85.2	80	67	55	48
	Total other unknowns	1.7	0.7	0.6	1.3	4.6	6
	Total	99	95.7	94	91	90	91
	Bound in PES	0	2.3	3.9	5.6	7.8	9.1
	Volatiles ($^{14}CO_2$)	NA	0	0.1	0.1	0.2	0.3
	Total Residue (Mass balance)	99.0	98.5	99.0	96.2	98.8	101
Newhaven	N-des-Me-DFPA	0	0.7	1.1	1.4	1.3	1.2
	DFPA-CONH2	0	0	0	0	0	0
	DFPA	0	0	0	0	0	0
	1'-CH2OH-S-2840A[2]	0	0	0	0	0	0.7
	1'-COOH-S-2840A	0	4.2	4	5.1	4.7	4.7
	1'-CH2OH-S-2840B	0	0.1	0	0	0	0
	1'-COOH-S-2840B	0	11.2	14	18	15	15
	3'-OH-S-2840	2	7.3	7	9.4	9.3	11
	N-des-Me-S-2840	0	0.2	0.2	0.3	0.5	1.5
	inpyrfluxam	96	69.4	57	48	47	46
	Total other unknowns	1.5	4.2	1.6	4.9	4.4	5.6
	Total	100	97.4	88	87	83	85
	Bound in PES	0.2	4.6	6.5	9.9	10.8	12.2
	Volatiles ($^{14}CO_2$)	NA	0.1	0.3	0.5	0.7	0.8

	Percent (%) of applied radioactivity [1]					
	Time after application (days)					
	Compound	0	14	30	61	90	120
	Total Residue (Mass balance)	101.6	102.3	93.9	97.4	94.6	98.9
Woodside	N-des-Me-DFPA	0	0	0.3	1.5	2	1.5
	DFPA-CONH2	0	0	0	0	0	0
	DFPA	0	1.6	1.6	1.4	1.4	1.4
	1'-CH2OH-S-2840A[2]	0	0	0	0	0	0
	1'-COOH-S-2840A	0	2.2	3.1	5.2	5.7	5.2
	1'-CH2OH-S-2840B	0	0	0	0	0	0
	1'-COOH-S-2840B	0	7.8	12	19	22	25
	3'-OH-S-2840	2	5.6	6.3	7.4	7.9	8.4
	N-des-Me-S-2840	0	0	0	0	0	0
	inpyrfluxam	95	79.6	69	54	47	42
	Total other unknowns	2.4	0.8	0.7	3.1	4.4	5.8
	Total	99	97.5	93	91	90	89
	Bound in PES	0.2	3.0	3.9	6.3	7.4	9.3
	Volatiles ($^{14}CO_2$)	NA	0	0.1	0.1	0.2	0.3
	Total Residue (Mass balance)	99.7	101.9	98.7	98.0	98.5	99.0

Notes:
[1]: Duplicate samples were analysed at each timepoint.
[2] Chromatographically, 1'-CH2OH-S-2840A, 1'-COOH-S-2840A, 1'-CH2OH-S-2840B and 1'-COOH-S-2840B elute close together between 32-34 minutes by HPLC and tentative assignments have been made. A definitive assignment was made by 2D-TLC analysis.

DT_{50}s were estimated at 121, 101 or 331 days (SFO, Single First Order) for the Atwater, Newhaven or Woodside Farm sites, respectively. A DT_{50} of 1,720 days (DFOP, Double First-Order in Parallel, slow $t_{1/2}$) was also calculated for the Newhaven site. PestDF chose the DFOP value as its best fit, which is not realistic. A visual inspection of the data reveals that around 50 percent loss of inpyrfluxam was observed by 60 days. It is well understood, that one of the deficiencies of closed laboratory studies is that there is no carbon renewal, hence the rate of degradation at later intervals can be lower due to decreased microbial activity. The biomass values for the Newhaven soil suggest that the soil was less microbially active at 120 days. In this case, the DFOP model overemphasizes the slow degradation rate (k1). Therefore, the appropriate value for modelling would be the SFO value of 101 days.

The proposed metabolic pathway for inpyrfluxam in soil under aerobic conditions is shown in Figure 8.

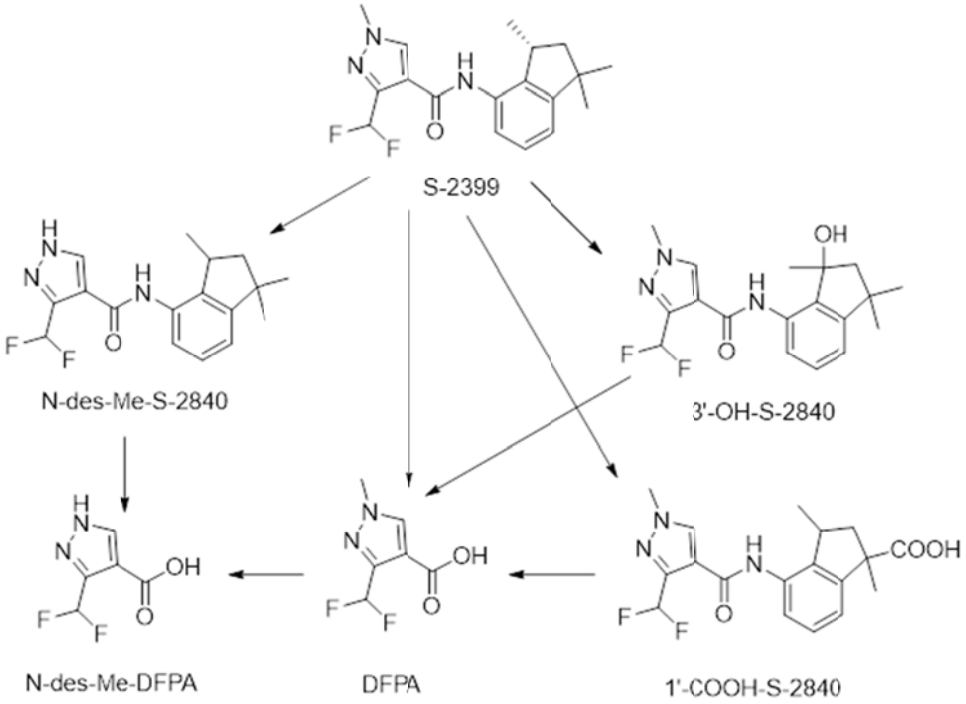


Figure 8 Proposed aerobic soil degradation pathway of inpyrfluxam.

In the study TPM-0057, the data of the rate of degradation of inpyrfluxam in aerobic soil from the studies TPM-0023 and TPM-0044 were normalised and the CAKE 3.2 software was used to determine the normalised degradation rate of inpyrfluxam following the FOCUS Kinetics Guidance (2014). The data were fitted to SFO and DFOP kinetics for inpyrfluxam only and the data normalised to 20 °C and pF are given in the Table 38 below.

Table 38 Normalization of laboratory DT_{50} values for inpyrfluxam

Soil name	Classification	pH	Study temp (°C)	Study matrix potential (% MWHC)	Actual study moisture content (% w/w)	Moisture content at pF2 (w/w)	Moisture correction factor	Experimental SFO DT_{50} (d)	DT_{50} (d) at 20 °C and pF 2
Penn	Loam	6.8	20	50	24.1	23.8	1	213	213
Atwater	Sandy loam	7.5	20	50	16.1	14.3	1	121	121
Newhaven	Silt loam	5.7	20	50	38.4	33.0	1	1720	1720
Woodside	Loam	7.5	20	50	29.0	28.6	1	331	331
Geomean DT_{50} value									348

In the same study, attempts were then made to additionally fit the metabolite (1'-COOH-S-2840 and 3'-OH-S-2840) data, using the best fit kinetics for the parent molecule. Given that the metabolites did not show significant decline during the incubation there is clearly significant uncertainty in the calculated DT_{50} values. This is particularly the case for 3'-OH-S-2840 at Atwater and 1'-COOH-S-2840 at Penn where the fitted DT_{50} values of >10,000 days are clearly nonsensical. The fitted formation fractions are subject to lesser uncertainty since the decline of the parent can be linked to the formation of the metabolite. The absence of robust DT_{50} values for the metabolite results in some uncertainties in the formation fractions but since degradation is generally expected to be slow, then these uncertainties are less than would otherwise occur. In Table 41 the results of the kinetic determinations for metabolites 1'-COOH-S-2840 and 3'-OH-S-2840 in four laboratory soils are summarized.

Table 39 Kinetic determinations for metabolites 1'-COOH-S-2840 and 3'-OH-S-2840

Soil	Penn	Atwater	Newhaven	Woodside
Parent kinetic	SFO	SFO	DFOP	DFOP
Overall Chi^2 error%	4.75	2.55	3.13	2.21
Parent Chi^2 error%	1.7	1.14	1.12	0.77
DT_{50} inpyrfluxam	212	121	653	226
3'OH-S-2840 Chi^2 error%	9.71	3.93	9.01	10.7
Visual fit	Good	Good	Good	Acceptable
Statistical fit	Acceptable	Unacceptable	Unacceptable	Acceptable
DT_{50} 3'OH-S-2840	241	>10,000	843	78
ff 3'-OH-S-2840	0.561	0.433	0.212	0.282
1'COOH-S-2840Chi^2 error%	32	11.6	5.1	2.5
Visual fit	Unacceptable	Good	Good	Good
Statistical fit	Unacceptable	Acceptable	Acceptable	Unacceptable
DT_{50} 1'-COOH-S-2840	>10,000	34.2	197	699
ff 1'-COOH-S-2840	0.197	0.517	0.540	0.613

Soil photolysis

Photodegradation of inpyrfluxam was investigated by Schick (2014, TPM-0005). Inpyrfluxam was labelled at two positions; [pyrazolyl-4-^{14}C]inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam and applied to thin layers of not sterilized soil (2.15 mg/kg) in individual photolysis vessels. The physicochemical characteristics of the soil are presented in Table 43. Light-exposed samples were irradiated with a Xenon lamp (wavelengths <290 nm filtered out, similar spectrum to natural sunlight) continuously for up to 13 days. All samples were maintained at 20 ± 3.3 °C and continuously aerated throughout the incubation period. Dark control samples were also analysed. Traps to collect organic volatiles and CO_2 were included.

Table 40 Soil physicochemical properties used in the study TPM-0005

Soil name	Classification	CEC (meq/100 g soil)	pH (1:1 soil:water ratio)	Organic matter (%)
Penn Soil	Loam	7.6	6.8	1.9

The DT_{50} and DT_{90} results for the pyrazolyl and phenyl labeled inpyrfluxam set are shown in the Table 44 below. The degradation rate constant (k) and estimated half-life (DT_{50}) of [^{14}C] inpyrfluxam were calculated assuming first-order kinetics using Microsoft Excel® version 2000. The degradation constants were calculated using the percent inpyrfluxam in each sample from the following equation:

$\ln C = -kt + \ln C0$ ($y = mx + b$) (m = slope)

where:

k = degradation rate constant

C = chemical concentration (inpyrfluxam expressed as percent of applied radiocarbon)

t = time

C_0 = initial concentration

The DT_{50} of [^{14}C] inpyrfluxam was calculated using the following equations:

$$DT_{50} = \frac{\ln 2}{-m} = \frac{0.693}{k}$$

Since the degradation in dark control samples was similar to that in the light exposed samples, the degradation in the light-exposed samples was corrected to account only for the effect of photolysis on degradation. The adjusted degradation rate constant was calculated as follows, and DT_{50} and DT_{90} shown in Table 41

$$k_{photolysis} = k_{light} - k_{dark}$$

Table 41 DT_{50} and DT_{90} results for the pyrazolyl and phenyl labelled inpyrfluxam

Sample set	DT_{50} hours (days)	DT_{90} hours (days)	R2
[phenyl-U-^{14}C]inpyrfluxam			
Light	2057 (86)	6833 (285)	0.2866
Dark	11951 (498)	39700 (1654)	0.0294
Photolysis	2484 (104)	8253 (344)	N/A
[pyrazolyl-4-^{14}C]inpyrfluxam)			
Light	2773 (116)	9210 (384)	0.8226
Dark	2795 (116)	9285 (387)	0.5191
Photolysis	346574 (14441)	1151293 (47971)	N/A

Using the light intensity of the Xenon lamp from 290-800 nm and 290-400 nm, the DT_{50} and DT_{90} for irradiated samples can be converted to equivalent solar days (30-50°N global summer day as per OECD, and summer days at 30, 40, and 50 °N). A summary of the results is presented in Table 42.

Table 42 DT_{50} and DT_{90} results for the pyrazolyl and phenyl labelled inpyrfluxam expressed as days in artificial light, summer days and global solar days.

	Days under Continuous Artificial Light	Summer days at 30°N (290-800 nm)	Summer days at 40°N (290-800 nm)	Summer days at 50°N (290-800 nm)	Global solar days (290-400 nm)
[phenyl-U-^{14}C]inpyrfluxam					
DT_{50}	104	279	252	235	235
DT_{90}	344	928	837	780	780
[pyrazolyl-4-^{14}C]inpyrfluxam)					
DT_{50}	14441	38956	35149	32762	32761
DT_{90}	47971	129409	116762	108833	108829

[^{14}C]inpyrfluxam was observed to photodegrade very slowly on a loam soil, with a nominal photolytic half-life (determined as the half-life in light-exposed samples corrected for the degradation in dark controls) of between 104 and 14441 days, indicating that photolysis is not a significant loss mechanism. The main degradation product was 3-'OH-S-2840 (mean maximum of 8.3 percent AR at day 13). Soil bound residues represented less than 3 percent AR. No isomerization from inpyrfluxam *R*-isomer to its *S*-isomer occurred throughout the study.

Field dissipation

The Meeting received nine field dissipation studies from which, five were conducted in North America (Foster, 2017; TPR-0031, TPR-0032, TPR-0053 Bitter, 2017; TPR-0034, TPR-0033) and four in Europe (LeBrun, 2018; TPR-0085). In these studies, the dissipation and mobility of inpyrfluxam and its

metabolites 3'-OH-S-2840, 1'-COOH-S-2840A and 1'-COOH-S-2840B were assessed following two applications of inpyrfluxam at rate of 115 g ai/ha and 100 g ai/ha or one application at a rate of 200 g ai/ha in bare uncropped soil. In all cases inpyrfluxam was applied as a SC formulation.

Soil samples (cores) from each plot and the control were taken on at several time period depending on the study. At each sampling event, five soil cores per subplot were taken. For each core, the 0–15 cm segment was collected and only in study TPR–0031, samples were also collected followed for the 15–90 cm core. Samples were frozen at the site and stored at the laboratory (maximum storage length 705 days) and covered by storage stability studies in soil (727 days). Samples were then cut into 15 cm segments (0–15 cm, 15–30 cm, 30–45 cm, 45–60 cm, 60–75 cm and 75–90 cm) and corresponding depth segments from each plot hand mixed and were analysed using the validated Method RM-50S (TPA-0070). The LOQ was 0.01 mg/kg for each analyte. Overall, concurrent recovery samples for inpyrfluxam and its metabolites gave results ranging from 90.2 to 96.6 percent recovery, with relative standard deviations up to 10.7 percent.

In the trials conducted in North America, inpyrfluxam residues in the 15–cm (6-inch) surface soil layer were 55–87.4 percent of the amount applied. The highest detected individual plot residue was 0.190 mg/kg (study TPR-0031) which was detected immediately after the second application. Average residues declined to < LOD (0.005 mg/kg) by 210–730 days after the second application. Residues of 3'-OH-S-2840 were above the LOQ at several DALA depending on the study (Table 43). Residues of 1'-COOH-S-2840A and 1'-COOH-S-2840B were not detected (Table 44). Similar in the trials conducted in Europe, movement of inpyrfluxam was not observed from the 0-30 cm soil segment to lower layers.

In study TPR-0031, residues of inpyrfluxam in the 15–30 cm soil segment was detected above the LOQ (0.01 mg/kg) in one sample (0.0137 mg/kg) at 29 days after the second application; all other samples were less than the LOQ for depths of 15–90 cm. In this trial flooding of the treated plot was observed that might lead to leaching of residues. However, the leaching potential is expected to be low in loam soil and under the environmental conditions of the region (Mississippi) since minimal residues were found in the lower levels even after the trial plot had received excessive rainfall.

Table 43 Residues of inpyrfluxam and metabolite 3'-OH-S-2840 in field dissipation studies.

		Residues (mg/kg eq)[b]					
Study	DALA[a]	Inpyrfluxam	3'-OH-S-2840	Inpyrfluxam	3'-OH-S-2840	Inpyrfluxam	3'-OH-S-2840
		0–15 cm		15–30 (10–20) cm		20–30 cm	
TPR-0031	-1[c]	<0.01	<0.01	<0.01	<0.01		
	0[e]	0.067	<0.01	<0.01	<0.01		
	13[c]	0.073	<0.01	<0.01	<0.01		
	0[e]	0.081	<0.01	0.0059	<0.01		
	1	0.028	<0.01	<0.01	<0.01		
	8	0.037	<0.01	<0.01	<0.01		
	13	0.026	<0.01	<0.01	<0.01		
	22	0.050	0.0115	<0.01	<0.01		
	29	0.025	<0.01 (0.0066)[f]	<0.01 (0.0062)[f]	<0.01		
	61	0.017	<0.01	<0.01	<0.01		
	89/96[g]	0.012	<0.01	<0.01	<0.01		
	120	0.010	<0.01	<0.01	<0.01		
	146	0.015	<0.01	<0.01	<0.01		
	272	0.012	<0.01	<0.01	<0.01		
	365	0.005	<0.01	<0.01	<0.01		
	429	N.D.	<0.01	<0.01	<0.01		
	488	N.D.	<0.01	<0.01	<0.01		

Study	DALA[a]	Residues (mg/kg eq)[b]					
		Inpyrfluxam	3'-OH-S-2840	Inpyrfluxam	3'-OH-S-2840	Inpyrfluxam	3'-OH-S-2840
		0–15 cm		15–30 (10–20) cm		20–30 cm	
TPR-0032	-1[c]	N.D.[d]	<0.01				
	0[e]	0.029	<0.01				
	13[c]	0.011	<0.01				
	0[e]	0.040	<0.01				
	1	0.026	<0.01				
	7	0.017	<0.01				
	14	0.024	<0.01 (0.0073)[f]				
	21	0.016	<0.01				
	30	0.013	<0.01				
	60	0.006	<0.01				
	90	0.005	<0.01				
	120	N.D.	<0.01				
	151	N.D.	<0.01				
	210	N.D.	<0.01				
	270	N.D.	<0.01				
TPR-0034	-1[c]	<0.01[d]	<0.01				
	0[e]	0.043	<0.01				
	13[c]	0.039	<0.01				
	0[e]	0.049	<0.01 (0.0071)[f]				
	1	0.087	0.0104				
	7	0.046	0.0061				
	14	0.047	0.0068				
	22	0.042	<0.01				
	30	0.019	<0.01				
	61	0.020	<0.01				
	92	0.006	<0.01				
	117	0.014	<0.01				
	–[g]	–	–				
	294	0.008	<0.01				
	365	0.008	<0.01				
	426	<0.01	<0.01				
	478	<0.01	<0.01				
TPR-0053	-1[c]	<0.01[d]	<0.01				
	0[e]	0.038	<0.01				
	13[c]	0.026	<0.01				
	0[e]	0.058	<0.01				
	1	0.054	<0.01				
	7	0.042	<0.01				
	14	0.043	<0.01				
	21	0.035	<0.01				
	30	0.034	<0.01				
	62	0.025	<0.01				
	91	0.017	<0.01				
	120	0.014	<0.01				
	149	0.011	<0.01				
	268	0.011	<0.01				
	365	<0.01 (0.0098)[f]	<0.01				
	426	0.005	<0.01				

Study	DALA[a]	Residues (mg/kg eq)[b]					
		Inpyrfluxam	3'-OH-S-2840	Inpyrfluxam	3'-OH-S-2840	Inpyrfluxam	3'-OH-S-2840
		0–15 cm		15–30 (10–20) cm		20–30 cm	
	485	0.010	0.0052				
	630	0.013	0.0070				
	730	<0.01	<0.01				
TPR-0033	-1[c]	N.D.[d]	<0.01				
	0[e]	0.052	<0.01				
	13[c]	0.024	0.0051[f]				
	0[e]	0.064	0.0052				
	1	0.052	<0.01				
	7	0.040	0.0054				
	14	0.025	0.0050				
	21	0.025	0.0051				
	31	0.025	<0.01				
	59	0.018	0.0065				
	89	0.023	0.0082				
	119	0.018	0.0098				
	—[g]	—	—				
	—[g]	—	—				
	367	0.0093	0.0056				
	419	0.0080	0.0060				
	489	0.0075	0.0058				
267-2016GE01	0	0.1500	<0.01	<0.01	<0.01	<0.01	<0.01
	3	0.1100	<0.01	<0.01	<0.01	<0.01	<0.01
	7	0.0900	<0.01	<0.01	<0.01	<0.01	<0.01
	20	0.1300	0.0064	0.0287	<0.01	<0.01	<0.01
	28	0.0987	0.00558	0.00206	<0.01	N.D.	N.D.
	61	0.0750	0.0079	<0.01	<0.01	0.097	0.00086
	91	0.0610	0.00818	<0.01	<0.01	<0.01	<0.01
	176	0.0350	0.00969	<0.01	<0.01	<0.01	<0.01
	270	0.0390	0.0106	<0.01	<0.01	<0.01	<0.01
	359	0.0240	0.00759	<0.01	<0.01	<0.01	<0.01
	448	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	543	0.0094	0.00461	<0.01	<0.01	<0.01	<0.01
	629	0.0130	0.00633	<0.01	<0.01	<0.01	<0.01
	728	0.0098	0.00561	<0.01	<0.01	<0.01	<0.01
267-2016CZ02	0	0.0967	<0.01	0.00209	<0.01	0.0033	N.D.
	3	0.1490	0.0025	<0.01	<0.01	<0.01	<0.01
	7	0.1130	0.0027	<0.01	<0.01	<0.01	<0.01
	15	0.1250	0.0023	<0.01	<0.01	<0.01	<0.01
	28	0.0834	0.0024	<0.01	<0.01	0.0037	<0.01
	61	0.0810	0.005	<0.01	<0.01	N.D.	<0.01
	92	0.0814	0.0075	<0.01	<0.01	<0.01	<0.01
	182	0.0716	0.009	<0.01	<0.01	<0.01	<0.01
	265	0.0842	0.012	<0.01	<0.01	<0.01	<0.01
	366	0.0594	0.01	<0.01	<0.01	<0.01	<0.01
	455	0.0441	0.0089	<0.01	<0.01	<0.01	<0.01
	540	0.0343	0.0087	<0.01	<0.01	<0.01	<0.01
	629	0.0323	0.0086	<0.01	<0.01	<0.01	<0.01
	733	0.0109	0.003	<0.01	<0.01	<0.01	<0.01
267-2018IT03	0	0.054	<0.01	<0.01	<0.01	<0.01	<0.01
	3	0.037	<0.01	<0.01	<0.01	<0.01	<0.01
	6	0.03	<0.01	<0.01	<0.01	<0.01	<0.01

Study	DALA[a]	Residues (mg/kg eq)[b]					
		Inpyrfluxam	3'-OH-S-2840	Inpyrfluxam	3'-OH-S-2840	Inpyrfluxam	3'-OH-S-2840
		0–15 cm		15–30 (10–20) cm		20–30 cm	
	14	0.051	<0.01	<0.01	<0.01	<0.01	<0.01
	28	0.044	0.0024	<0.01	<0.01	<0.01	<0.01
	60	0.044	0.0025	<0.01	<0.01	<0.01	<0.01
	89	0.033	0.0025	<0.01	<0.01	<0.01	<0.01
	180	0.028	0.003	<0.01	<0.01	<0.01	<0.01
	272	0.032	0.0044	<0.01	<0.01	<0.01	<0.01
	358	0.024	0.004	<0.01	<0.01	<0.01	<0.01
	455	0.016	0.0026	<0.01	<0.01	<0.01	<0.01
	550	0.015	0.004	<0.01	<0.01	<0.01	<0.01
	637	0.02	0.0049	<0.01	<0.01	<0.01	<0.01
	728	0.0087	0.0029	<0.01	<0.01	<0.01	<0.01
267-2016SP04	0	0.168	<0.01	<0.01	<0.01	<0.01	<0.01
	3	0.166	0.003	<0.01	<0.01	<0.01	<0.01
	7	0.133	0.0025	<0.01	<0.01	<0.01	<0.01
	14	0.124	0.004	<0.01	<0.01	<0.01	<0.01
	28	0.106	0.007	<0.01	<0.01	<0.01	<0.01
	62	0.079	0.01	<0.01	<0.01	<0.01	<0.01
	89	0.066	0.009	<0.01	<0.01	<0.01	<0.01
	176	0.05	0.011	<0.01	<0.01	<0.01	<0.01
	266	0.047	0.01	<0.01	<0.01	<0.01	<0.01
	361	0.047	0.006	<0.01	<0.01	<0.01	<0.01
	454	0.028	0.0088	<0.01	<0.01	<0.01	<0.01
	538	0.025	0.0087	<0.01	<0.01	<0.01	<0.01
	629	0.0206	0.007	<0.01	<0.01	<0.01	<0.01
	740	0.017	0.006	<0.01	<0.01	<0.01	<0.01

Notes:
[a] Days after last application.
[b] For sampling intervals containing residues and non-detects, "N.D." was replaced with 1/2LOD (0.0025 mg/kg) to calculate the mean.
[c] One day prior to the next application.
[d] Not detected, below LOD (0.005 mg/kg).
[e] Cores sampled after the application when the spray had dried—typically collection started between 1 and 3 hours after application.
[f] Values in parentheses are <LOQ (0.01 mg/kg).
[g] Samples collected at 89 days after the last application for Plots A and B and at 96 days after the last application for Plot C.

Table 44 Residues of metabolites 1'-COOH-S-2840A in field dissipation studies.

Study	DALA[a]	Residues (mg/kg)[b]					
		1'-COOH-S-2840A	1'-COOH-S-2840B	1'-COOH-S-2840A	1'-COOH-S-2840B	1'-COOH-S-2840A	1'-COOH-S-2840B
		0–15 cm		15–30 cm (10-20 cm)		20–30 cm	
TPR-0031	-1[c]	<0.01	<0.01	<0.01	<0.01		
	0[e]	<0.01	<0.01	<0.01	<0.01		
	13[c]	<0.01	<0.01	<0.01	<0.01		
	0[e]	<0.01	<0.01	<0.01	<0.01		
	1	<0.01	<0.01	<0.01	<0.01		
	8	<0.01	<0.01	<0.01	<0.01		
	13	<0.01	<0.01	<0.01	<0.01		
	22	<0.01	<0.01	<0.01	<0.01		

Study	DALA[a]	Residues (mg/kg)[b]					
		1'-COOH-S-2840A	1'-COOH-S-2840B	1'-COOH-S-2840A	1'-COOH-S-2840B	1'-COOH-S-2840A	1'-COOH-S-2840B
		0–15 cm		15–30 cm (10-20 cm)		20–30 cm	
	29	<0.01	<0.01	<0.01	<0.01		
	61	<0.01	<0.01	<0.01	<0.01		
	89/96[g]	<0.01	<0.01	<0.01	<0.01		
	120	<0.01	<0.01	<0.01	<0.01		
	146	<0.01	<0.01	<0.01	<0.01		
	272	<0.01	<0.01	<0.01	<0.01		
	365	<0.01	<0.01	<0.01	<0.01		
	429	<0.01	<0.01	<0.01	<0.01		
	488	<0.01	<0.01	<0.01	<0.01		
TPR-0032	-1[c]	<0.01	<0.01				
	0[e]	<0.01	<0.01				
	13[c]	<0.01	<0.01				
	0[e]	<0.01	<0.01				
	1	<0.01	<0.01				
	7	<0.01	<0.01				
	14	<0.01	<0.01				
	21	<0.01	<0.01				
	30	<0.01	<0.01				
	60	<0.01	<0.01				
	90	<0.01	<0.01				
	120	<0.01	<0.01				
	151	<0.01	<0.01				
	210	<0.01	<0.01				
	270	<0.01	<0.01				
TPR-0034	-1[c]	<0.01	<0.01				
	0[e]	<0.01	<0.01				
	13[c]	<0.01	<0.01				
	0[e]	<0.01	<0.01				
	1	<0.01	<0.01				
	7	<0.01	<0.01				
	14	<0.01	<0.01				
	22	<0.01	<0.01				
	30	<0.01	<0.01				
	61	<0.01	<0.01				
	92	<0.01	<0.01				
	117	<0.01	<0.01				
	–[g]	–	–				
	294	<0.01	<0.01				
	365	<0.01	<0.01				
	426	<0.01	<0.01				
	478	<0.01	<0.01				
TPR-0053	-1[c]	<0.01	<0.01				
	0[e]	<0.01	<0.01				
	13[c]	<0.01	<0.01				
	0[e]	<0.01	<0.01				
	1	<0.01	<0.01				
	7	<0.01	<0.01				
	14	<0.01	<0.01				
	21	<0.01	<0.01				
	30	<0.01	<0.01				

Study	DALA[a]	Residues (mg/kg)[b]					
		1'-COOH-S-2840A	1'-COOH-S-2840B	1'-COOH-S-2840A	1'-COOH-S-2840B	1'-COOH-S-2840A	1'-COOH-S-2840B
		0–15 cm		15–30 cm (10-20 cm)		20–30 cm	
	62	<0.01	<0.01				
	91	<0.01	<0.01				
	120	<0.01	<0.01				
	149	<0.01	<0.01				
	268	<0.01	<0.01				
	365	<0.01	<0.01				
	426	<0.01	<0.01				
	485	<0.01	<0.01				
	630	<0.01	<0.01				
	730	<0.01	<0.01				
TPR-0033	-1[c]	<0.01	<0.01				
	0[e]	<0.01	<0.01				
	13[c]	<0.01	<0.01				
	0[e]	<0.01	<0.01				
	1	<0.01	<0.01				
	7	<0.01	<0.01				
	14	<0.01	<0.01				
	21	<0.01	<0.01				
	31	<0.01	<0.01				
	59	<0.01	<0.01				
	89	<0.01	<0.01				
	119	<0.01	<0.01				
	–[g]	–	–				
	–[g]	–	–				
	367	<0.01	<0.01				
	419	<0.01	<0.01				
	489	<0.01	<0.01				
267-2016GE01	0	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	3	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	7	<0.01	0.0015[f]	<0.01	<0.01	<0.01	<0.01
	20	(<0.01) 0.0026[f]	(<0.01) 0.005[f]	<0.01	<0.01	<0.01	<0.01
	28	0.025	(<0.01) 0.004[f]	<0.01	<0.01	<0.01	<0.01
	61	0.0013[f]	(<0.01) 0.003[f]	<0.01	<0.01	<0.01	<0.01
	91	<0.01	(<0.01) 0.002[f]	<0.01	<0.01	<0.01	<0.01
	176	<0.01	(<0.01) 0.002[f]	<0.01	<0.01	<0.01	<0.01
	270	<0.01	(<0.01) 0.001[f]	<0.01	<0.01	<0.01	<0.01
	359	<0.01	(<0.01) 0.0011[f]	<0.01	<0.01	<0.01	<0.01
	448	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	543	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	629	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	728	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
20 16 03	0	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Study	DALA[a]	Residues (mg/kg)[b]					
		1'-COOH-S-2840A	1'-COOH-S-2840B	1'-COOH-S-2840A	1'-COOH-S-2840B	1'-COOH-S-2840A	1'-COOH-S-2840B
		0–15 cm		15–30 cm (10-20 cm)		20–30 cm	
	3	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	7	<0.01	(<0.01) 0.0009[f]	<0.01	<0.01	<0.01	<0.01
	15	(<0.01) 0.0013[f]	(<0.01) 0.0012[f]	<0.01	<0.01	<0.01	<0.01
	28	(<0.01) 0.0028[f]	(<0.01) 0.00278[f]	<0.01	<0.01	<0.01	<0.01
	61	(<0.01) 0.0037[f]	(<0.01) 0.004[f]	<0.01	<0.01	<0.01	<0.01
	92	(<0.01) 0.0037[f]	(<0.01) 0.004[f]	<0.01	<0.01	<0.01	<0.01
	182	(<0.01) 0.0019[f]	(<0.01) 0.002[f]	<0.01	<0.01	<0.01	<0.01
	265	(<0.01) 0.0024[f]	(<0.01) 0.0027[f]	(<0.01) 0.0015[f]	(<0.01) 0.0015[f]	<0.01	(<0.01) 0.0004[f]
	366	(<0.01) 0.002[f]	(<0.01) 0.002[f]	<0.01	(<0.01) 0.0004[f]	<0.01	<0.01
	455	(<0.01) 0.0022[f]	(<0.01) 0.0028[f]	<0.01	<0.01	<0.01	<0.01
	540	(<0.01) 0.001[f]	(<0.01) 0.0014[f]	<0.01	<0.01	<0.01	<0.01
	629	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	733	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
267-2018IT03	0	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	3	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	6	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	14	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	28	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	60	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	89	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	180	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	272	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	358	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	455	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	550	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	637	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	728	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
267-2016SP04	0	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	3	(<0.01) 0.0011[f]	(<0.01) 0.0014[f]	<0.01	<0.01	<0.01	<0.01
	7	(<0.01) 0.002[f]	(<0.01) 0.0029[f]	<0.01	<0.01	<0.01	<0.01
	14	(<0.01) 0.0036[f]	(<0.01) 0.0048[f]	<0.01	<0.01	<0.01	<0.01
	28	(<0.01) 0.004[f]	(<0.01) 0.006[f]	<0.01	<0.01	<0.01	<0.01
	62	(<0.01) 0.005[f]	(<0.01) 0.0087[f]	<0.01	<0.01	<0.01	<0.01
	89	(<0.01) 0.0048[f]	(<0.01) 0.0085[f]	<0.01	<0.01	<0.01	<0.01
	176	(<0.01) 0.0056[f]	0.01	<0.01	<0.01	<0.01	<0.01
	266	(<0.01) 0.0057[f]	0.0107	<0.01	<0.01	<0.01	<0.01
	361	(<0.01) 0.0059[f]	0.0104	<0.01	<0.01	<0.01	<0.01
	454	(<0.01) 0.0059[f]	0.011	<0.01	<0.01	<0.01	<0.01
	538	(<0.01) 0.0044[f]	(<0.01) 0.0009[f]	<0.01	<0.01	<0.01	<0.01
	629	(<0.01) 0.0043[f]	(<0.01) 0.0087[f]	<0.01	<0.01	<0.01	<0.01

Study	DALA[a]	Residues (mg/kg)[b]					
		1'-COOH-S-2840A	1'-COOH-S-2840B	1'-COOH-S-2840A	1'-COOH-S-2840B	1'-COOH-S-2840A	1'-COOH-S-2840B
		0–15 cm		15–30 cm (10-20 cm)		20–30 cm	
	740	(<0.01) 0.0035[f]	(<0.01) 0.0076[f]	(<0.01) 0.0007[f]	(<0.01) 0.0014[f]	<0.01	<0.01

Notes:
[a] Days after last application.
[b] For sampling intervals containing residues and non-detects, "N.D." was replaced with 1/2LOD (0.0025 mg/kg) to calculate the mean.
[c] One day prior to the next application.
[d] Not detected, below LOD (0.005 mg/kg).
[e] Cores sampled after the application when the spray had dried—typically collection started between 1 and 3 hours after application.
[f] Values in parentheses are <LOQ (0.01 mg/kg).
[g] Samples collected at 89 days after the last application for Plots A and B and at 96 days after the last application for Plot C.

First order half-life estimates based on the field trials in North America (Table 45) and Europe (Table 46 and Table 47) indicate that inpyrfluxam are expected to be persist. The overall geomean $DegT_{50}$ (following time step normalisation of the data) of all sites was 117 days.

Table 45 $DegT_{50}$ values (SFO model) for inpyrfluxam from field studies in North America

Parameter	California Site	Ontario Site	North Dakota Site	Washington Site
Visual fit	Acceptable	Acceptable	Acceptable	Acceptable
$DegT_{50}$ (d)	74	104	41.6	113
$DegT_{90}$ (d)	246	344	138	374
χ^2 error (%)	19.6	15.6	20.8	17.3
k ($days^{-1}$)	0.009365	0.00669	0.01667	0.00615
P value	2.00E-6	6.47E-4	4.74E-6	5.79E-13
Geomean SFO $DegT_{50}$	**78 day**			

Table 46 $DegT_{50}$ values for inpyrfluxam from field studies in Europe.

Parameter	German Site	Czech Site	Italian Site	Spanish Site
Model	SFO	SFO	SFO	SFO
χ^2 error (%)	17.6	14.4	14.9	18.0
k ($days^{-1}$)[1]	0.008048 ($4.3x10^{-10}$)	0.00384 ($7.8x10^{-13}$)	0.001653 ($3.3x10^{-7}$)	0.00888 ($1.5x10^{-8}$)
Statistical fit	Good	Good	Good	Good
Visual fit	Good	Good	Acceptable	Poor
$DegT_{50}$ (d)	86.1	181	419	78.1
$DegT_{90}$ (d)	286	599	1390	259
Model	DFOP	DFOP	DFOP	DFOP
χ^2 error (%)	15.4	15.3	15.9	5.69
k1*($days^{-1}$)	3.91 (0.43)	0.0595 (0.38)	0.2494 (0.38)	0.1463 (0.014)
k2*($days^{-1}$)	0.00740 ($1.4x10^{-10}$)	0.00358 ($2.9x10^{-6}$)	0.001613 ($5.9x10^{-10}$)	0.004808 ($4.9x10^{-5}$)
G	0.217	0.07813	0.0495	0.4692
Statistical fit	Unacceptable	Unacceptable	Unacceptable	Good
Visual fit	Good	Good	Acceptable	Good
$DegT_{50}$ (d) (overall)[2]	60.6 (93.7)	171 (194)	398 (430)	21.3 (144)
$DegT_{90}$ (d) (overall)	278	620	1400	347
Geomean $DegT_{50}$	**175 days (DFPO model in Spain)**			

Notes:
[1]: P value from the t-test is given in brackets.

[2:] Values in brackets represent slow phase.

Table 47 $DegT_{50}$ values for inpyrfluxam and its metabolites from field studies in Europe

Parameter	German Site	Czech Site	Italian Site	Spanish Site
Inpyrfluxam kinetic	SFO	SFO	SFO	DFOP
Overall Chi^2 error%	29.1	24.5	24.7	11.4
Parent Chi^2 error%	17.8	14.6	14.9	7.54
Visual fit	Good	Good	Good	Good
Statistical fit	Good	Good	Acceptable	Good
$DegT_{50}$ inpyrfluxam	79.5	168	421	38 (111)
3'OH-S-2840 Chi^2 error%	27.4	20.2	24.0	15.5
Visual fit	Acceptable	Acceptable	Acceptable	Acceptable
Statistical fit	Acceptable	Acceptable	Acceptable	Acceptable
$DegT_{50}$ 3'OH-S-2840	73.8	96.5	204	148
ff 3'-OH-S-2840	0.209	0.276	0.335	0.128
1'COOH-S-2840 Chi^2 error%	25.9	24.1	-	6.44
Visual fit	Acceptable	Acceptable	-	Acceptable
Statistical fit	Acceptable	Acceptable	-	Acceptable
$DegT_{50}$ 1'COOH-S-2840	6.46	25.2	-	224
ff 1'-COOH-S-2840	0.680	0.502	-	0.169

Environmental fate in water/sediment systems

The Meeting received studies investigating the rate and route of hydrolysis and photodegradation in water.

Hydrolysis in water

In study by Freedlander (TPM-0030) the rate and route of hydrolysis of inpyrfluxam was studied in three aqueous sterile buffer solutions (at pH 4, 7and 9) using [pyrazolyl-4-^{14}C]inpyrfluxam applied at 1.00 µg/mL. Solutions were incubated in the dark at 50 ± 0.5 °C and samples were analysed at 0 and 5 days of incubation by LSC and HPLC/RAM. In all three buffers inpyrfluxam was hydrolysed less than 10 percent (after 5 days), thus additional investigation was not performed. Isomerization in the chiral carbon was not observed. In conclusion, [^{14}C]inpyrfluxam is considered to be hydrolytically stable.

Aqueous photolysis

In study by Ponte (TPM-0008) the photodegradation of inpyrfluxam was studied in sterilized aqueous phosphate buffer (pH 7, 0.01 M sodium phosphate monobasic monohydrate) irradiated at 25 ± 1 °C with a xenon lamp (wavelengths <290 nm filtered out, similar spectrum to natural sunlight) for up to 15 days). Samples were analysed by LSC, HPLC and TLC in duplicate immediately after treatment (time 0) and 1, 3, 6, 9, 13 and 15 days after treatment.

Minimal degradation of inpyrfluxam was observed in light-exposed and dark control samples throughout the study period. inpyrfluxam ranged from 95.7 to 102.1 percent AR in light exposed samples and from 97.9 to 104.5 percent AR in dark controls. Although 3'-OH-S-2840 slightly increased up to 5.8 and 4.1 percent AR in light exposed and dark controls throughout the 15-day irradiation/incubation period, the degradate was detected in the purity check and T0 samples as an impurity. The half-lives of inpyrfluxam could not be correctly calculated since inpyrfluxam was stable to degradation in pH 7 buffer in the presence and in the absence of light irradiation but they were estimated to be over one year. In

conclusion, the results of the study indicate that photolysis is not a significant mode of dissipation of inpyrfluxam in aqueous solutions.

In another study by Ponte (TPM-0010) the photodegradation of inpyrfluxam was studied in sterilized natural water at 0.98 mg/L [pyrazolyl-4-^{14}C]inpyrfluxam and 1.00 mg/L [phenyl-^{14}C]inpyrfluxam. Solutions were subjected to continuous irradiation at 25 ± 1 °C with a xenon lamp (wavelengths <290 nm filtered out, similar spectrum to natural sunlight) for up to 16 days. Samples were analysed by LSC, HPLC and TLC in duplicate immediately after treatment (time 0) and 1, 3–4, 7, 10, 14 and 16 days after treatment.

Total recoveries for pyrazolyl labelled samples averaged 102.6 ± 2.3 and 104.3 ± 2.1 percent AR for light exposed and dark control sets, respectively.

In phenyl labelled samples, total recoveries averaged 98.7 ± 2.5 and 98.6 ± 1.3 percent AR for light exposed and dark control sets, respectively. inpyrfluxam degraded slowly in light-exposed samples and represented an average of 86.0 and 70.7 percent AR in PYR and PH labelled samples, respectively, following 16 days of continuous irradiation.

In PYR labelled samples, 3'-OH-S-2840, which was an impurity at time 0 (3.7 percent AR), reached an average of 6.8 percent AR by Day 14. DFPA-$CONH_2$ was present as a maximum average of 3.4 percent AR by Day 16 and DFPA represented a maximum average of 4.7 percent AR on Day 14. Radiocarbon recovered in the traps for volatiles represented <0.3 percent AR throughout the study.

In the PH labelled light exposed samples, the degradate 3'-OH-S-2840 was also observed at a maximum average of 8.6 percent AR by the end of the study. An area of radioactivity eluting in the polar region by HPLC represented up to 6.2 percent AR at Day 16 which was comprised of at least two polar degradates. The organic volatiles represented <0.3 percent AR, while $^{14}CO_2$ represented an average of 3.7 percent AR at the end of the study.

The half-life of inpyrfluxam in natural water are summarized in Table 48 and the proposed degradation pathway of inpyrfluxam in natural water when exposed to artificial sunlight in Figure 9.

Table 48 DT_{50} values for inpyrfluxam in natural water when exposed to artificial sunlight

Sample Set	DT_{50} (hours/days)[1]	DT_{50} in sunlight days		
		United States (40 °N summer)[2]	OECD (30-50 °N, summer)[3]	JMAFF (35 °N spring)[4]
[pyrazolyl-4-^{14}C]inpyrfluxam	2105/88	188	171	549
[phenyl-U-^{14}C]inpyrfluxam	857/36	77	69	223

Notes:
[1] Continuous Suntest irradiation.
[2] Average summer irradiation in the 300-800 nm range at 40 °N latitude.
[3] Average summer irradiation in the 300-400 nm range at 30-50 °N latitude.
[4] Average spring irradiation in the 300-400 nm range at 35 °N latitude in Tokyo.

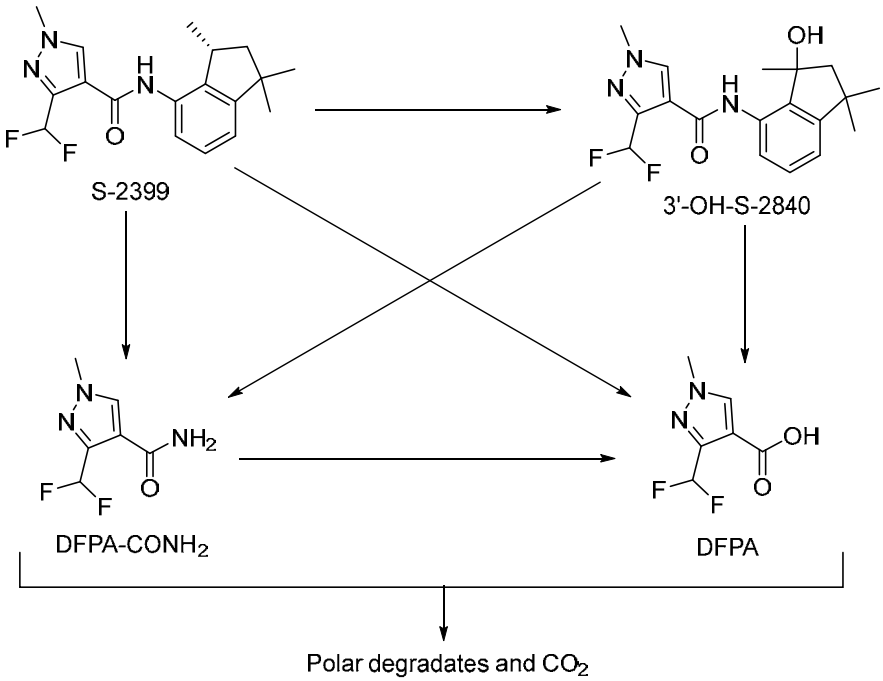


Figure 9 Proposed degradation pathway of inpyrfluxam in sterilized natural water exposed to artificial sunlight

Rice paddies

Since rice is included in the intended uses of inpyrfluxam, the Meeting received five studies investigating the degradation of inpyrfluxam in water/sediment systems (TPM-0041, TPM-0048, TPM-0050, TPM-0036, TPM-0038, TPM-0039).

In study by Gohre (TPM-0041) the biotransformation of inpyrfluxam in two water/sediment system (Golden Lake and Taunton River) was investigated under aerobic aquatic conditions using [phenyl-^{14}C] inpyrfluxam and [pyrazolyl-4-^{14}C] inpyrfluxam.

Two test systems (sediment and water) were collected from the top 0–5 cm layer (Golden Lake) and 0–2.5 cm layer (Taunton River). The sediment was thoroughly mixed and passed through a 2-mm mesh sieve with a minimum of air-drying. The sediment and water was stored in the dark before being used. The sediments characteristics are summarised in Table 49.

Table 49 Chemical and Physical characteristics of test sediments (TPM-0041)

Sediment characteristic	Golden Lake	Taunton River
USDA Particle size distribution		
% sand (50 µm - 2 mm)	80	58
% silt (2 µm - 50 µm)	15	36
% clay <2 µm	5	6
pH (H_2O)	7.8	5.9
% Moisture 1/3 bar	20.4	39.4
Cation exchange capacity (meq/100g)	13.0	6.5
% Organic carbon (Walkley Black)	1.6	3.7
% Organic Matter	2.8	6.3
USDA Textural class	Loamy sand	Sandy loam
Microbial Biomass Carbon (µg/g dry weight)	389 (0 DAT) 444 (112 DAT; untreated control)	401 (0 DAT) 503 (112 DAT; untreated control)

Sediment characteristic	Golden Lake	Taunton River
	460 (112 DAT; solvent control) 456 (112 DAT; inpyrfluxam control)	478 (112 DAT; solvent control) 480 (112 DAT; inpyrfluxam control)

A beaker (cylinder internal diameter of 5.2 cm) charged with 50 g (dry weight) of sediment and 165 mL of water has a water surface area of about 21.6 cm^2 (water column depth of about 6.3 cm (Golden Lake) and 5.1 cm (Taunton River). The aerobic aquatic test systems were dosed with a final water concentration of 0.018 µg/mL for both radiolabels in the Golden Lake systems, and a final water concentration of 0.015 µg/mL and 0.014 µg/mL for the PH-label and PY-label in the Taunton River systems, respectively. The test systems were incubated at 20 ± 2 °C in the dark with a constant humidified air flow for a maximum of 112 days and were periodically collected and extracted. The test systems were equipped with 1 M NaOH traps for the collection of evolved and tetraglyme/ethylene glycol traps for ^{14}C volatile capture. A pre-incubation period of 26–27 days was performed for the Golden Lake system and 14-15 days for the Taunton River system before dosing. Untreated control soils, organic solvent controls and non-radiolabelled inpyrfluxam samples were used to measure the effect on the microbial biomass at the end of sampling. Duplicate soil samples were taken at 0, 3, 7, 14, 30, 63 and 112 days after treatment (DAT). The trap solutions were analysed at the same points immediately after removal.

The physical parameters of the aerobic systems (oxygen concentration, redox and pH) were measured and the water separated from the sediment. The water phase and all extracts were analysed by LSC. The water phase was subjected to Solid Phase Extraction (SPE) and the organosoluble fraction was analysed by HPLC after concentration. Confirmation was performed by 2D-TLC.

The sediment samples were extracted with acetone, twice with acetone:water (3:2) and with acetone:water:HCl (c) (60:40:1). Activity in the neutral extracts were analysed by HPLC and confirmed by 2D-TLC after concentration. The acidic extracts contained < 4 percentAR and therefore no further analysis was performed. Representative Post-extracted sediments (PES) at 112 DAT were subjected to further additional sequential solvent extractions with ethyl acetate, dioxane and hexane. Total radioactivity in PES was determined by combustion. The $^{14}CO_2$ collected in the NaOH trapping solution and organic volatiles collected in tetraglyme/ethylene glycol traps were quantified by LSC. The potential isomerisation from [^{14}C] inpyrfluxam was evaluated using chiral HPLC analysis on the extracts obtained from the 112 DAT samples.

No significant change in the microbial biomass carbon was recognized between the initiation and termination of the incubation (Table 49). Thus, microbial viability was proved to be satisfactorily maintained during the incubation period.

It was confirmed that no isomerisation of [^{14}C] inpyrfluxam occurred during incubation period based on chiral HPLC analysis. The distribution and mass balance of applied radioactivity of [^{14}C] inpyrfluxam in water phase, extractable, sediment-bound and volatile fractions are summarised in Table 50 to Table 53. The quantification of inpyrfluxam and the degradates in the whole system is summarised in Table 54 to Table 57.

The average mass balance for the study was 98.6 ± 1.4 percent AR (PH-label) and 99.2 ± 1.0 percent AR (PY-label) for the Golden Lake system, and 98.8 ± 1.2 percent AR (PH-label) and 99.4 ± 1.9 percent AR (PY-label) for the Taunton River system. The radioactivity remaining in sediment following the neutral and acidic extractions, post extraction sediment (PES) or sediment-bound radioactivity, was insignificant, reaching 6 percent of the AR (Golden Lake) and 3 percent of the AR (Taunton River) by the end of the study (112 DAT). Additional extractions of the Golden Lake PES extracted low amounts of

activity from the sediment (~2 percent AR). No further analysis was performed on the PES. The cumulative production of $^{14}CO_2$ was insignificant reaching a maximum of 0.4 percent AR at 112 DAT in the Golden Lake system.

Two degradates were observed above 5 percent AR: 3´-OH-S-2840 (max. 6.8 percent AR), and 1'-COOH-S-2840, (max. 13.1 percent AR). N-demethylation of the pyrazolyl ring to produce N-des-Me-S-2840 was minor as well as hydrolysis of the amide bond to produce the pyrazolyl derivatives DFPA and DFPA-$CONH_2$.

Generally, all degradates were observed to be declining in both sediments by the end of the study (112 DAT) except 1'-COOH-S-2840 in the Golden Lake sediment. The aerobic aquatic metabolism degradation pathway of inpyrfluxam is summarized in Figure 9. The PestDF kinetics (consistent with the FOCUS approach) for Golden Lake and Taunton River were evaluated from 0–63 days (112 day data points were considered to have unacceptable data scatter). The total ^{14}C from the acidic extract was added to the identified inpyrfluxam from other extracts as a conservative approach. The calculated Single First Order DT_{50} values were 154 (χ^2 = 1.1) and 368 days (χ^2 = 1.5) for Golden Lake and Taunton River, respectively.

Inpyrfluxam degraded slowly in two sediment/water systems (Golden Lake and Taunton River) under aerobic aquatic conditions. The majority of the dose remained unchanged after 112 days of aerobic aquatic exposure and ultimate mineralization to bound residues and CO_2 was minor. Whole system aerobic aquatic half-lives were estimated at 154 and 368 days (SFO) for Golden Lake and Taunton River, respectively. Chi2 error values were 1.1 and 1.5, respectively. Degradate formation was primarily to 1'-COOH-S-2840 and 3'-OH-S-2840, formed at average maximums of 13.1 and 6.8 percent AR, respectively.

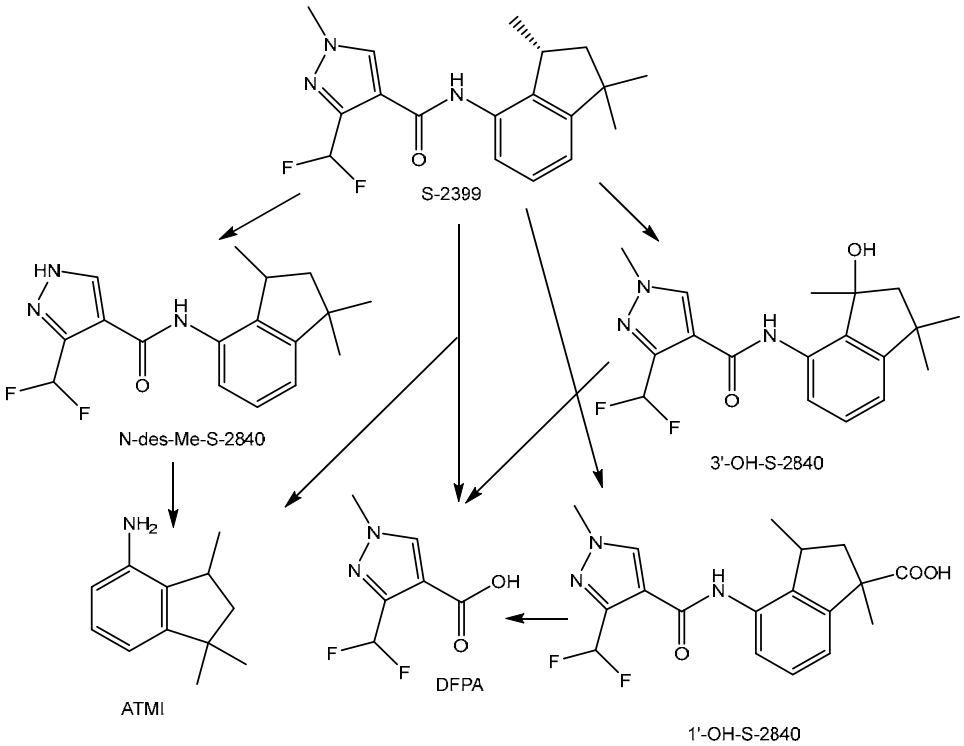


Figure 10 Proposed aerobic aquatic degradation pathways of inpyrfluxam

Table 50 Summary of the mass balance data for the Golden Lake sediment PH-label, as percent of Applied Radioactivity

Fraction	Days After Treatment (DAT)											
	0			3			7			14		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	89.3	86.8	88.0	51.5	49.8	50.6	37.3	39.1	38.2	33.7	34.9	34.3
Neutral Extract	12.3	13.1	12.7	43.9	46.5	45.2	58.4	57.5	57.9	60.0	60.2	60.1
Acidic Extract	0.0	0.0	0.0	0.6	0.7	0.7	1.0	1.0	1.0.	1.5	1.4	1.4
Total Ext.	12.3	13.2	12.7	44.6	47.2	45.9	59.3	58.5	58.9	61.5	61.6	61.6
Sediment-bound	0.0	0.0	0.0	0.6	0.7	0.6	1.3	1.2	1.3	2.0	2.1	2.1
Volatiles ($^{14}CO_2$)	NA	NA	NA	0.0	0.0	0.0	0.1	0.1	0.1	0.1	0.1	0.1
Total balance	101.6	100	100.8	96.7	97.7	97.2	98.0	98.9	98.5	97.4	98.7	98.0
Fraction	Days After Treatment (DAT)								
	30			63			112		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	30.5	30.7	30.6	24.3	23.6	24	21.5	22.8	22.2
Neutral Extract	61.5	63.7	62.6	66.1	66.6	66.4	68.6	67.7	68.2
Acidic Extract	2.2	2.3	2.2	2.9	3.1	3.0	3.8	3.1	3.4
Total Ext.	63.6	66	64.8	69	69.7	69.3	72.4	70.8	71.6
Sediment-bound	2.8	2.9	2.9	4.3	4.0	4.2	5.0	5.2	5.1
Volatiles ($^{14}CO_2$)	0.2	0.2	0.2	0.3	0.3	0.3	0.4	0.4	0.4
Total balance	97.2	99.9	98.6	98	97.5	97.7	99.3	99.2	99.2

Note:
NA: not analysed

Table 51 Summary of the mass balance data for the Golden Lake sediment PY-label, as percent of Applied Radioactivity

Fraction	Days After Treatment (DAT)											
	0			3			7			14		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	81.9	79.1	80.5	52.5	51.2	51.8	35.6	40.7	38.1	33.4	33.3	33.4
Neutral Extract	18.4	19.8	19.1	46.6	46.5	46.6	61.7	56.9	59.3	63.5	62.1	62.8
Acidic Extract	0.0	0.0.	0.0	0.8	0.7	0.7	1.0	1.0	1.0	1.7	1.6	1.6
Total Ext.	18.4	19.8	19.1	47.4	47.2	47.3	62.7	57.9	60.3	65.2	63.6	64.4
Sediment-bound	0.0	0.0	0.0	0.5	0.4	0.4	1.0	1.0	1.0	2.2	2.0	2.1
Volatiles, $^{14}CO_2$	NA	NA	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.1	0.1
Total balance	100.4	98.9	99.6	100.3	98.8	99.6	99.4	99.7	99.5	100.8	99.0	99.9
Fraction	Days After Treatment (DAT)								
	30			63			112		
Sample	Rep1	Rep 2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	30.8	33.6	32.2	22.5	23.1	22.8	22.5	21.3	21.9
Neutral Extract	64.0	59.8	61.9	69.6	67.8	68.7	66.7	66.1	66.4
Acidic Extract	2.1	1.9	2.0	3.5	3.1	3.3	3.4	3.7	3.5
Total Ext.	66.1	61.7	63.9	73.1	70.9	72.0	70.1	69.9	70
Sediment-bound	2.7	2.8	2.8	3.6	3.6	3.6	5.9	5.6	5.7
Volatiles, $^{14}CO_2$	0.1	0.1	0.1	0.2	0.2	0.2	0.4	0.4	0.4
Total balance	99.8	98.3	99.0	99.4	97.7	98.6	98.9	97.2	98.0

Note:
NA: not analysed

Table 52 Summary of the mass balance data for the Taunton River sediment PH-label, as percent of Applied Radioactivity

Fraction	Days After Treatment (DAT)											
	0			3			7			14		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	67.7	63.3	65.5	40.5	36.3	38.4	31.9	31.2	31.6	24.0	21.9	22.9
Neutral Extract	30.8	33.3	32.0	55.8	60.9	58.3	65.3	68.1	66.7	73.7	77.4	75.5
Acidic Extract	0.1	0.1	0.1	0.6	0.7	0.7	0.6	0.6	0.6	0.8	1.2	1.0
Total Ext.	30.9	33.3	32.1	56.4	61.6	59.0	65.9	68.7	67.3	74.5	78.5	76.5
Sediment-bound	0.1	0.1	0.1	0.3	0.3	0.3	0.5	0.4	0.5	0.6	0.9	0.8
Volatiles ($^{14}CO_2$)	NA	NA	NA	0.0	0.0	0.0	0.1	0.1	0.1	0.1	0.1	0.1
Total balance	98.7	96.7	97.7	97.2	98.2	97.7	98.3	100.5	99.4	99.2	101.4	100.3
Fraction	Days After Treatment (DAT)											
	30			63			112					
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Re 2	Avg			
Water Phase	17.1	15.8	16.5	8.0	10.0	9.0	6.1	6.3	6.2			
Neutral Extract	78.9	81.2	80.1	85.4	83.2	84.3	86.6	85.9	86.2			
Acidic Extract	1.2	1.2	1.2	2.7	2.5	2.6	4.1	3.6	3.9			
Total Ext.	80.1	82.4	81.2	88.1	85.7	86.9	90.6	89.5	90.1			
Sediment-bound	1.1	1.0	1.0	2.1	2.5	2.3	2.8	2.5	2.6			
Volatiles ($^{14}CO_2$)	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2			
Total balance	98.4	99.4	98.9	98.4	98.4	98.4	99.5	98.3	98.9			

Note:
NA: Not analysed.

Table 53 Summary of the mass balance data for the Taunton River sediment PY-label, as percent of Applied Radioactivity

Fraction	Days After Treatment (DAT)											
	0			3			7			14		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	65.3	74.3	69.8	36.1	36.5	36.3	32.3	35.2	33.8	28.1	26.6	27.4
Neutral Extract	34.1	22.8	28.4	61.2	59.3	60.3	68.4	63.4	65.9	70.8	73.1	72.0
Acidic Extract	0.0	0.0	0.0	0.5	0.4	0.4	0.4	0.4	0.4	0.7	0.6	0.7
Total Ext.	34.1	22.8	28.4	61.7	59.7	60.7	68.8	63.8	66.3	71.5	73.8	72.6
Sediment-bound	0.0	0.0	0.0	0.1	0.1	0.1	0.2	0.2	0.2	0.4	0.4	0.4
Volatiles, $^{14}CO_2$	NA	NA	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total balance	99.3	97.1	98.2	97.9	96.3	97.1	101.4	99.2	100.3	100.0	100.8	100.4

Fraction	Days After Treatment (DAT)								
	30			63			112		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	21.5	20.3	20.9	11.7	11.2	11.5	6.8	6.3	6.5
Neutral Extract	75.8	76.1	76.0	84.7	84.2	84.4	89.9	86.0	88.0
Acidic Extract	1.0	1.1	1.0	2.1	2.0	2.0	4.3	4.1	4.2
Total Ext.	76.8	77.2	77.0	86.7	86.1	86.4	94.2	90.1	92.1
Sediment-bound	0.7	0.8	0.7	1.5	1.4	1.4	2.8	2.5	2.7
Volatiles, $^{14}CO_2$	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total balance	99.1	98.3	98.7	100.0	98.7	99.4	103.8	98.9	101.4

Note:
NA: not analysed

Table 54 Radioactivity distribution from the water and sediment of Golden Lake PH-label total activity as percent of applied radioactivity

Fraction	Days After Treatment (DAT)											
	0			3			7			14		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Inpyrfluxam (water)	86.3	85.5	85.9	48.6	47.0	47.8	34.2	36.2	35.2	28.9	31.4	30.2
Inpyrfluxam (sediment)	12.3	13.1	12.7	43.2	45.9	44.5	57.3	56.5	56.9	60.0	60.2	60.1
1'-COOH-S-2840 total** (water)	0.0	0.0	0.0	0.0	0.0	0.0	0.8	1.6	1.2	2.8	2.1	2.5
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840 (water)	2.2	1.3	1.7	1.0	0.9	1.0	0.7	0.9	0.8	0.8	0.7	0.7
3'-OH-S-2840 (sediment)	0.0	0.0	0.0	0.8	0.6	0.7	1.1	1.0	1.0	0.0	0.0	0.0
N-des-Me-S-2840 (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Others (Total)*	0.8	0.0	0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total*	101.6	99.9	100.7	93.5	94.5	93.5	94.1	96.2	95.1	92.5	94.4	93.4
Fraction	Days After Treatment (DAT)								
	30			63			112		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep 2	Avg
Inpyrfluxam (water)	25.1	27.4	26.2	17.4	15.9	16.7	12.3	12.5	12.4
Inpyrfluxam (sediment)	58.7	60.9	59.8	57.7	44.6	51.2	63.7	60.3	62.0
1'-COOH-S-2840 total** (water)	3.0	1.5	2.2	6.4	5.3	5.9	7.9	9.6	8.8
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	2.1	7.5	4.8	2.6	3.9	3.2
3'-OH-S-2840 (water)	0.9	0.6	0.7	0.6	0.5	0.6	0.6	0.5	0.5
3'-OH-S-2840 (sediment)	2.8	2.8	2.8	3.7	6.3	5.0	2.4	2.8	2.6
N-des-Me-S-2840 (sediment)	0.0	0.0	0.0	0.0	4.1	2.1	0.0	0.7	0.3
Others (Total) *	0.0	0.0	0.0	2.5	4.0	3.3	0.0	0.0	0.0
Total*	90.4	93.2	91.8	90.6	88.5	89.5	89.4	90.4	89.9

Notes:

Chromatographically, 1'-CH_2OH-S-2840A, 1'-COOH-S-2840A, 1'-CH_2OH-S-2840B and 1'-COOH-S-2840B elute close together between ~32-34 minutes by HPLC and tentative assignments have been made. A definitive assignment was made by 2D-TLC analysis.
* Includes 1'-CH_2OH-S-2840 and unknowns, none of which individually exceeded 4% in the whole system
** As the sum of both isomers = 1'-COOH-S-2840 A + 1'-COOH-S-2840 B

Table 55 Radioactivity distribution from the water and sediment of Golden Lake PY-label total activity as percent of applied radioactivity

Fraction	Days After Treatment (DAT)											
	0			3			7			14		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Inpyrfluxam (water)	79.0	76.0	77.5	39.7	52.8	46.3	33.1	35.9	34.5	29.4	26.3	27.9
Inpyrfluxam (sediment)	18.4	19.8	19.1	45.3	45.2	45.2	58.8	55.1	56.9	60.1	62.1	61.1
1'-COOH-S-2840 total** (water)	0.0	0.0	0.0	0.9	1.1	1.0	1.1	1.6	1.3	1.2	4.3	2.7
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840 (water)	2.9	1.4	2.2	1.5	2.0	1.8	1.6	1.8	1.7	1.4	1.6	1.5
3'-OH-S-2840 (sediment)	0.0	0.0	0.0	1.3	1.3	1.3	2.9	1.9	2.4	3.3	0.0	1.7
N-des-Me-S-2840 (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Others (Total) *	0.0	1.7	0.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total*	100.4	98.8	99.6	88.7	102.5	95.6	97.5	96.2	96.8	95.4	94.3	94.9
Fraction	Days After Treatment (DAT)											
	30			63			112					
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg			
Inpyrfluxam (water)	20.9	26.6	23.7	14.5	15.3	14.9	11.4	10.5	10.9			
Inpyrfluxam (sediment)	57.0	53.0	55.0	57.0	52.8	54.9	58.5	59.2	58.9			
1'-COOH-S-2840 total** (water)	4.4	4.0	4.2	5.5	5.0	5.3	10.3	9.6	10.0			
1'-COOH-S-2840 total** (sediment)	2.8	2.6	2.7	2.5	3.5	3.0	3.3	2.7	3.1			
3'-OH-S-2840 (water)	0.7	1.2	1.0	0.8	0.6	0.7	0.5	0.6	0.6			
3'-OH-S-2840 (sediment)	4.1	4.2	4.2	5.1	4.8	4.9	3.6	3.6	3.6			
N-des-Me-S-2840 (sediment)	0.0	0.0	0.0	0.0	4.6	2.3	1.2	0.6	0.9			
Others (Total) *	0.0	0.0	0.0	6.5	3.2	4.8	0.0	0.4	0.2			
Total*	90.0	91.5	90.8	92.0	89.7	90.8	88.8	87.3	88.0			

Notes:
Chromatographically, 1'-CH_2OH-S-2840A, 1'-COOH-S-2840A, 1'-CH_2OH-S-2840B and 1'-COOH-S-2840B elute close together between ~32-34 minutes by HPLC and tentative assignments have been made. A definitive assignment was made by 2D-TLC analysis.
* Includes 1'-CH_2OH-S-2840 and unknowns, none of which individually exceeded 2.6% in the whole system
** As the sum of both isomers = 1'-COOH-S-2840 A + 1'-COOH-S-2840 B

Table 56 Radioactivity distribution from the water and sediment of Golden Lake PH-label total activity as percent of applied radioactivity

Fraction	Days After Treatment (DAT)											
	0			3			7			14		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Inpyrfluxam (water)	66.1	61.6	63.9	39.4	36.1	37.8	28.9	28.6	28.8	22.2	18.6	20.4
Inpyrfluxam (sediment)	30.8	33.3	32.0	55.8	60.0	57.9	64.3	67.0	65.7	73.7	75.5	74.6
1'-COOH-S-2840 total** (water)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.3	1.9	1.6
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840 (water)	1.5	1.7	1.6	1.0	0.9	1.0	1.2	1.1	1.1	0.5	0.5	0.5
3'-OH-S-2840 (sediment)	0.0	0.0	0.0	0.0	0.9	0.4	1.0	1.1	1.0	0.0	1.9	1.0
N-des-Me-S-2840 (Total)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Others (Total) *	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total*	98.5	96.6	97.5	96.2	97.9	97.0	95.4	97.8	96.6	97.7	98.4	98.0
Fraction	Days After Treatment (DAT)											
	30			63			112					
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep 2	Avg			
Inpyrfluxam (water)	15.9	14.0	14.9	7.3	7.7	7.5	5.1	5.7	5.4			
Inpyrfluxam (sediment)	69.1	77.3	73.2	72.6	72.0	72.3	81.6	82.8	82.2			
1'-COOH-S-2840 total** (water)	1.2	0.9	1.0	0.8	2.3	1.6	0.6	0.6	0.7			
1'-COOH-S-2840 total** (sediment)	3.3	0.0	1.6	3.8	4.1	4.0	2.2	1.0	1.6			
3'-OH-S-2840 (water)	0.0	0.6	0.3	0.0	0.1	0.1	0.4	0.2	0.3			
3'-OH-S-2840 (sediment)	4.5	3.9	4.2	6.0	4.8	5.4	2.7	2.1	2.4			
N-des-Me-S-2840 (Total)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
Others (Total) *	2.1	0.0	1.0	3.0	2.3	2.6	0.0	0.0	0.0			
Total*	95.9	96.7	96.3	93.5	93.3	93.4	92.7	92.3	92.5			

Notes:

Chromatographically, 1'-CH_2OH-S-2840A, 1'-COOH-S-2840A, 1'-CH_2OH-S-2840B and 1'-COOH-S-2840B elute close together between ~32-34 minutes by HPLC and tentative assignments have been made. A definitive assignment was made by 2D-TLC analysis.

* Includes ATMI, which never exceeded 3.0% in the whole system.

** As the sum of both isomers = 1'-COOH-S-2840 A + 1'-COOH-S-2840 B.

Table 57 Radioactivity distribution from the water and sediment of Taunton River PY-label total activity as percent of applied radioactivity

Fraction	Days After Treatment (DAT)											
	0			3			7			14		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Inpyrfluxam water)	62.7	71.2	66.9	32.9	33.1	33.0	23.9	34.3	29.1	23.3	23.1	23.2
Inpyrfluxam (sediment)	34.1	22.8	28.4	59.7	57.7	58.7	65.5	61.3	63.4	67.2	71.8	69.5
1'-COOH-S-2840 total** (water)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840 (water)	2.6	3.2	2.9	1.8	1.7	1.8	0.7	1.0	0.8	1.9	1.6	1.8
3'-OH-S-2840 (sediment)	0.0	0.0	0.0	1.5	1.6	1.6	2.9	2.0	2.5	3.6	1.3	2.4
N-des-Me-S-2840 (Total)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Others (Total) *	0.0	0.0	0.0	0.0	0.0	0.0	0.5	0.8	0.6	2.1	1.0	1.5
Total*	99.3	97.1	98.2	96.0	94.2	95.1	93.5	99.4	96.5	98.1	98.8	98.5
Fraction	Days After Treatment (DAT)											
	30			63			112					
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg			
Inpyrfluxam (water)	19.1	13.6	16.4	10.7	9.1	9.9	5.7	5.5	5.6			
Inpyrfluxam (sediment)	67.7	65.2	66.5	76.6	78.0	77.3	86.3	82.8	84.6			
1'-COOH-S-2840 total** (water)	0.3	1.6	0.9	0.0	0.6	0.3	0.0	0.0	0.0			
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	1.3	0.6	0.9			
3'-OH-S-2840 (water)	0.8	0.9	0.8	0.0	0.0	0.0	0.0	0.0	0.0			
3'-OH-S-2840 (sediment)	6.0	6.0	6.0	7.0	4.3	5.7	2.4	2.6	2.5			
N-des-Me-S-2840 (Total)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
Others (Total) *	3.0	6.3	4.7	2.0	2.8	2.3	0.9	0.9	0.9			
Total*	96.9	93.6	95.2	96.3	94.8	95.5	96.5	92.4	94.5			

Notes:
Chromatographically, 1'-CH_2OH-S-2840A, 1'-COOH-S-2840A, 1'-CH_2OH-S-2840B and 1'-COOH-S-2840B elute close together between ~32-34 minutes by HPLC and tentative assignments have been made. A definitive assignment was made by 2D-TLC analysis.
* Includes DFPA-$CONH_2$, DFPA and total unknowns which never individually exceeded 3.8% in the whole system.
** As the sum of both isomers = 1'-COOH-S-2840 A + 1'-COOH-S-2840 B.

In study TPM-0048 the mineralisation and degradation rate of inpyrfluxam in a natural surface water was performed using two radiolabels, [phenyl-^{14}C] inpyrfluxam and [pyrazolyl-4-^{14}C] inpyrfluxam, at 20 ± 2˚C and in the dark for a maximum of 61 days. Natural water was collected from The Lake at Studley Royal, Ripon UK and the test system was named 'Fountains Abbey'. Prior to use the water was stored in the dark in an environmental chamber routinely maintained at 4 ± 2°C, with free access to air. Water was 100 µm sieved prior to use and subsequent characterisation. The water characteristics are summarised in Table 58.

Table 58 Characteristics of the tested natural water

Water characteristic	Fountains Abbey water
Sampling water temperature	19.1°C
Sampling water oxygen content	10.14 mg/L
Sampling water pH	8.6
Water depth sampled	0-10 cm
Water depth above sediment	30 cm
Total Organic Carbon (TOC) 9mg/L)	0.13
Dissolved Organic Carbon (DOC) (mg/L)	0.00
pH	8.0
Suspended Solids (mg/L)	3
Electrical Conductivity (µS/cm)	285

The mineralisation and degradation rate of inpyrfluxam in a natural surface water was performed using two radiolabels, [phenyl-^{14}C] inpyrfluxam and [pyrazolyl-4-^{14}C] inpyrfluxam, at 20 ± 2 ˚C and in the dark for a maximum of 61 days. The test vessels were attached to a flow-through system for continuous aeration. Two concentrations were used for each label. The concentrations of the PY-label were 0.103 mg/L and 0.0102 mg/L, and the concentrations for the PH-label were 0.103 mg/L and 0.0101 mg/L. The test systems were equipped with 2 M NaOH traps for the collection of evolved $^{14}CO_2$ and ethanediol traps for ^{14}C volatile capture. Sterile samples were tested at the higher concentration (0.103 mg/L.) Untreated blank controls were used to measure oxygen content and pH. Solvent controls and reference controls using sodium [^{14}C] benzoate were used to demonstrate that the microbial population was viable in the test system.

Duplicate samples were taken at 0, 3, 7, 14, 21, 30 and 61 days after treatment (DAT). Duplicate of sterilised water samples were taken at 0, 14, 30 and 61 days after treatment (DAT). The trap solutions were analysed at the same points. Dissolved oxygen and pH were measured in the blank controls at each sampling point. The water from each vessel and the water used for rinsing the vessel (extract 1) were analysed for radioactive content by LSC and were analysed for test substance and metabolites by HPLC. Then, the vessel was rinsed with acetonitrile (extract 2), sonicated and the solution analysed by LSC. The $^{14}CO_2$ collected in the NaOH trapping solution and organic volatiles collected in ethanediol traps were counted by LSC. The potential isomerisation from [^{14}C] inpyrfluxam was evaluated using chiral HPLC.

During incubation, oxygen levels were > 8 mg/L at all sampling times, showing that the water samples were maintained under aerobic conditions. Recorded pH values were in the range 8.1 to 8.5 (no change with respect to original value). The profiles of mineralisation rates of sodium [^{14}C]benzoate in the reference and solvent controls were similar showing that the acetonitrile did not inhibit the microbial degradation of sodium [^{14}C]benzoate in the natural water. Mineralisation exceeded 50 percent AR within 7 days and therefore the natural water used was microbially active and the study was valid. It was confirmed that no isomerisation of [^{14}C] inpyrfluxam occurred during incubation period based on chiral HPLC analysis.

The distribution and mass balance of applied radioactivity of both radiolabelled [^{14}C] inpyrfluxam and at both concentrations in surface water, vessel wash and volatile fractions were very similar and are summarised in Table 59. Inpyrfluxam and metabolites were separately determined in all natural water samples collected. The quantification of inpyrfluxam and the degradates is summarised in Table 60.

Table 59 Ranged of average percent recovery of applied radioactivity recovered from natural water treated with [^{14}C] inpyrfluxam (low and high concentration)

Incubation group	% AR present in			
	Surface water	Vessel wash	Volatiles	Total
PH-label, low rate	90.0-95.9	0.4-2.0	≤ 0.2	92.1-96.2
PH-label, high rate	91.2-95.0	0.4-2.0	≤ 0.4	93.2-96.6
PY-label, low rate	*93.2-99.6	ND-1.7	ND	*94.3-100.4
PY-label, high rate	88.6-93.4	0.4-1.5	ND	90.1-94.0
Sterile (PH-label, high rate)	91.9-96.5	0.3-0.5	ND	92.8-97.0

Notes:
Values are the mean of two replicates and are ranges over the period 0 to 61 DAT
ND = Not detected, * One replicate from 61 DAT has been excluded due to low mass balance

Table 60 Average percent recovery of [^{14}C] inpyrfluxam and metabolites expressed as applied radioactivity from natural water samples

Incubation group	% AR present as		
	inpyrfluxam at 0 DAT	inpyrfluxam at 61 DAT	Unknowns (max)
PH-label, low rate	91.2	92.1	1.3
PH-label, high rate	92.4	90.6	3.3
PY-label, low rate	94.1	96.5*	3.9
PY-label, high rate	89.7	90.4	3.9
Sterile (PH-label, high rate)	91.3	94.2	0.6

Notes:
Values are the mean of two replicates, *One replicate excluded due to low mass balance

Ethanediol traps did not contain any detectable radioactivity. $^{14}CO_2$ was insignificant reaching a maximum of 0.4 percent AR for the phenyl-label at high concentration (30 DAT). The largest metabolite was present at ≤ 4 percent AR (maximum 3.9 percent AR, 14 DAT pyrazolyl-label at high and low concentration) and it was not identified. Degradation rates of [^{14}C] inpyrfluxam were obtained by CAKE software (Version 2.0) in line with FOCUS guidelines). The calculated Single First Order DT_{50} values are summarsied in Table 61, showing that no degradation of inpyrfluxam could be demonstrated under sterile and non-sterile conditions.

Table 61 Degradation rate of [^{14}C] inpyrfluxam

Group	DT_{50} (days)	DT_{90} (days)	X^2
PH-label, low rate	3190	10600	1.41
PH-label, high rate	1540	5120	1.10
PY-label, low rate*	5850	19400	2.10
PY-label, high rate	23600	78500	1.42
Sterile (PH-label, high rate)	3.45×10^{12}	1.15×10^{13}	1.6

Note:
* One replicate excluded due to low mass balance

No individual metabolites were detected at > 5 percent AR at any sampling interval (maximum 3.9 percent AR, 14 DAT pyrazolil-label at high and low concentration). Degradation rate DT_{50} values calculated as ≥ 1540 days under non-sterile and sterile conditions confirm that no significant degradation occurred during the study.

In study TPM-0036 the biotransformation of inpyrfluxam in two water/sediment system (Sharkey and Golden Lake) was investigated under anaerobic aquatic conditions using [phenyl-^{14}C] inpyrfluxam and [pyrazolyl-4-^{14}C] inpyrfluxam.

Two test systems (sediment and water) were collected from the top 5–10 cm layer (Sharkey) and 0–5 cm layer (Golden Lake). The sediment was thoroughly mixed and passed through a 2-mm mesh sieve with a minimum of air-drying. The sediment and water was stored in the dark before being used. The sediment characteristics are summarised in Table 62.

Table 62 Chemical and Physical characteristics of test sediments (TPM-0036)

Sediment characteristic	Sharkey	Golden Lake
USDA Particle size distribution		
% sand (50 µm - 2 mm)	18	89
% silt (2 µm - 50 µm)	19	6
% clay <2 µm	63	5
pH (H_2O)	6.4	8.0
% Moisture 1/3 bar	45.8	11.8
Cation exchange capacity (meq/100g)	32.6	10.1
% Organic carbon (Walkley Black)	1.45	0.93
% Organic Matter	2.5	1.6
USDA Textural class	Clay	Sand
Microbial Biomass Carbon as percent organic carbon	2.2 (1 DAT) 1.2 (103 DAT*; untreated control) 0.64 (103 DAT; solvent control) 0.61 (103 DAT; inpyrfluxam control)	0.35 (1 DAT) 0.17 (114 DAT; untreated control) 1.63 (114 DAT; solvent control) 2.13 (114 DAT; inpyrfluxam control)

Notes:
*The intended endpoint of the study was 102 DAT, but the study was extended to 180 days. Treatment inpyrfluxam controls were sampled at 209 DAT instead. The microbial biomass as percent organic carbon for this sample was 1.47%.

The anaerobic aquatic test systems (50 g of dry weight sediment with 181 mL water for Sharkey and 175 mL water for Golden Lake) were dosed after the anaerobic conditions were established (28–30 days for the Sharkey system and 41–43 days for the Golden Lake system). The concentrations applied to the Sharkey phenyl and pyrazolyl systems were 0.019 and 0.020 µg/mL, respectively, and 0.014 µg/mL for both radiolabels in the Golden Lake systems. The test systems were incubated at 20 ± 2 °C in the dark for a maximum of 180 days in the Sharkey system and 112 days in the Golden Lake system and were periodically collected and extracted. The physical parameters of the anaerobic system (oxygen concentration, redox and pH) were measured at each sampling date. The test systems were equipped with NaOH traps for the collection of evolved $^{14}CO_2$ and evolved $^{14}CH_4$ was analysed by oxidation of a sample of the biometer flask headspace gas. Untreated control soils, organic solvent controls and non-radiolabelled inpyrfluxam samples were used to measure the effect on the microbial biomass at the end of sampling.

Duplicate soil samples were taken at 0, 7, 14, 32, 67, 102 and 180 days after treatment (DAT) for the Sharkey system and at 0, 14, 30, 63 and 112 for the Golden lake system. All samples were measured for $^{14}CH_4$ and $^{14}CO_2$ production, dissolved oxygen concentration, redox potential, and pH at the time of analysis. The sediment and the water phases were separated by decantation and analysed separately. For the Sharkey samples, the water phase was centrifuged, decanted and aliquots analysed by LSC and HPLC, and representative samples by TLC. For the Golden Lake system, the water phase was centrifuged, decanted and aliquots analysed by LSC. After pH adjustment (pH 5), the water was passed through a reverse phase cartridge, washed with water and eluted with acetonitrile. The aliquots were analysed by LSC and HPLC. Representative samples were analysed by TLC. The sediment samples were extracted with acetone, twice with acetone:water (3:2) and with acetone:water:HCl (c) (60:40:1). Activity in the neutral

extracts were analysed by LSC and HPLC after concentration and representative samples were analysed by TLC. The acidic extracts were partitioned with ethyl acetate, concentrated and aliquots of both phases were counted by LSC. Final extracts were analysed by HPLC and representative samples by TLC. The post-extracted sediments (PES) at 180 DAT (Sharkey system) was subjected to additional sequential solvent extractions with ethyl acetate, dioxane and hexane, and a sonic dismembrator. Finally, it was fractionated into humin, humic acid and fulvic acid. Total radioactivity in PES was determined by combustion. The activity in the water phase and all extracts was counted by LSC. Water and sediment extracts were analysed by HPLC and 2D-TLC to identify and quantify [^{14}C] inpyrfluxam and its [^{14}C]labelled metabolites. The $^{14}CO_2$ collected in the NaOH trapping solution and headspace samples were counted by LSC. The potential isomerisation from [^{14}C] inpyrfluxam was evaluated using chiral HPLC analysis on the extracts obtained from the end of the study samples and on the test substances prior to application.

The microbial biomass and organic carbon values were viable and typical for sediment/water systems under anaerobic conditions. It was confirmed that no isomerisation of [^{14}C] inpyrfluxam occurred during incubation period based on chiral HPLC analysis. The distribution and mass balance of applied radioactivity of [^{14}C] inpyrfluxam in water phase, extractable, sediment-bound and volatile fractions are summarised in Table 63 to Table 66.

Table 63 Summary of the mass balance data for the Sharkey sediment system phenyl-label, as percent of Applied Radioactivity

Fraction	Days After Treatment (DAT)											
	0			7			14			32		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	65.5	68.5	67.0	53.1	64.3	58.7	60.0	48.9	54.5	41.6	35.7	38.7
Neutral Extract	29.1	25.5	27.3	44.5	32.3	38.4	34.5	44.6	39.5	47.0	53.1	50.1
Acidic Extract	1.9	2.7	2.3	1.9	1.3	1.6	2.9	3.6	3.2	5.5	7.7	6.6
Total Ext.	31.0	28.3	29.6	46.5	33.6	40.1	37.3	48.2	42.8	52.5	60.9	56.7
Sediment-bound	1.4	1.3	1.4	1.1	1.3	1.2	2.0	2.8	2.4	3.2	4.3	3.8
Volatiles ($^{14}CO_2$)	NA	NA	NA	0.0	0.0	0.0	0.0	0.1	0.0	0.1	0.0	0.0
Volatiles ($^{14}CH_4$)	NA	NA	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Biometer rinse	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.3	0.0	0.1
Total balance	98.0	98.2	98.1	100.7	99.2	100	99.3	100.0	99.7	97.5	100.9	99.2
Fraction	Days After Treatment (DAT)											
	67			102			180					
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg			
Water Phase	19.0	18.5	18.7	13.3	19.2	16.2	10.1	10.9	10.5			
Neutral Extract	64.7	66.4	65.5	63.4	57.9	60.7	66.7	61.8	64.2			
Acidic Extract	9.9	9.1	9.5	12.4	11.6	12.0	12.8	14.1	13.5			
Total Ext.	74.6	75.5	75.0	75.8	69.6	72.7	79.6	75.9	77.7			
Sediment-bound	5.8	6.7	6.2	7.5	7.0	7.2	8.0	11.3	9.6			
Volatiles ($^{14}CO_2$)	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.1			
Volatiles ($^{14}CH_4$)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
Biometer rinse	0.3	0.0	0.1	0.2	0.5	0.4	0.8	0.1	0.4			
Total balance	99.6	100.6	100.1	96.7	96.3	96.5	98.5	98.1	98.3			

Table 64 Summary of the mass balance data for the Sharkey sediment system pyrazolyl-label, as percent of Applied Radioactivity

Fraction	Days After Treatment (DAT)											
	0			7			14			32		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	63.6	76.0	69.8	50.5	55.0	52.8	43.8	39.7	41.8	38.5	36.0	37.3
Neutral Extract	30.5	18.5	24.5	41.2	39.6	40.4	46.0	40.0	43.0	49.6	49.2	49.4
Acidic Extract	1.7	1.0	1.3	1.5	1.5	1.5	4.7	3.3	4.0	4.5	7.6	6.0
Total Ext.	32.2	19.5	25.8	42.7	41.0	41.9	50.7	43.3	47.0	54.1	56.8	55.4
Sediment-bound	1.1	0.7	0.9	1.6	1.5	1.5	3.7	2.7	3.2	3.3	5.3	4.3
Volatiles ($^{14}CO_2$)	NA	NA	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Volatiles ($^{14}CH_4$)	NA	NA	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Biometer rinse	0.0	0.0	0.0	1.6	0.0	0.8	0.0	0.1	0.1	0.1	0.0	0.1
Total balance	96.9	96.2	96.6	96.4	97.5	97.0	98.3	85.8	92.0	96.0	98.1	97.1
Fraction	Days After Treatment (DAT)											
	67			102			180					
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg			
Water Phase	22.2	23.7	22.9	17.2	18.1	17.7	9.8	10.0	9.9			
Neutral Extract	60.3	57.9	59.1	60.0	59.9	60.0	67.3	68.9	68.1			
Acidic Extract	8.6	7.5	8.1	12.5	12.4	12.4	11.6	12.0	11.8			
Total Ext.	68.9	65.4	67.1	72.5	72.3	72.4	78.9	80.9	79.9			
Sediment-bound	5.4	7.2	6.3	7.7	8.8	8.3	9.8	8.3	9.0			
Volatiles ($^{14}CO_2$)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
Volatiles ($^{14}CH_4$)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
Biometer rinse	0.3	0.0	0.2	0.3	0.0	0.1	2.0	0.1	1.1			
Total balance	96.8	96.3	96.5	97.8	99.3	98.5	100.5	99.3	99.9			

Note: NA: Not analysed.

Table 65 Summary of the mass balance data for the Golden Lake sediment phenyl-label, as percent of Applied Radioactivity

Fraction	Days After Treatment (DAT)								
	0			14			30		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	81.5	83.8	82.6	26.5	29.8	28.1	20.8	23.8	22.3
Neutral Extract	16.0	13.7	14.9	68.1	67.0	67.5	74.3	68.2	71.3
Acidic Extract	0.3	0.3	0.3	1.5	1.2	1.3	2.2	1.8	2.0
Total Ext.	16.3	14.0	15.1	69.6	68.1	68.9	76.5	70.1	73.3
Sediment-bound	0.2	0.2	0.2	1.7	1.9	1.8	2.7	3.7	3.2
Volatiles ($^{14}CO_2$)	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.2	0.1
Volatiles ($^{14}CH_4$)	NA	NA	NA	0.0	0.0	0.0	0.0	0.0	0.0
Biometer rinse	0.1	0.1	0.1	1.8	0.1	1.0	0.2	0.7	0.5
Total balance	98.1	98.0	98.1	99.6	99.9	99.7	100.2	98.5	99.3

Fraction	Days After Treatment (DAT)						
	63			112			
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	
Water Phase	11.4	11.3	11.4	9.2	8.8	9.0	
Neutral Extract	79.2	80.5	79.8	85.4	86.3	85.8	
Acidic Extract	1.5	1.8	1.7	1.4	1.4	1.4	
Total Ext.	80.7	82.3	81.5	86.7	87.6	87.2	
Sediment-bound	3.5	3.9	3.7	3.8	3.5	3.7	
Volatiles ($^{14}CO_2$)	0.1	0.1	0.1	0.1	0.1	0.1	
Volatiles ($^{14}CH_4$)	0.0	0.0	0.0	0.0	0.0	0.0	
Biometer rinse	0.5	0.6	0.6	0.1	0.3	0.2	
Total balance	96.2	98.2	97.2	99.9	100.4	100.1	

Note: NA: not analysed

Table 66 Summary of the mass balance data for the Golden Lake sediment pyrazolyl-label, as percent of Applied Radioactivity

Fraction	Days After Treatment (DAT)								
	0			14			30		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1*	Rep2	Avg
Water Phase	86.1	83.8	84.9	34.3	30.2	32.2	NA	20.7	20.7
Neutral Extract	12.7	17.3	15.0	67.0	65.7	66.3	NA	74.9	74.9
Acidic Extract	0.1	0.3	0.2	1.1	1.2	1.2	NA	2.1	2.1
Total Ext.	12.8	17.6	15.2	68.2	66.9	67.5	NA	77.0	77.0
Sediment-bound	0.1	0.2	0.1	2.0	1.7	1.8	NA	2.9	2.9
Volatiles ($^{14}CO_2$)	0.0	0.0	NA	0.0	0.0	0.0	NA	0.0	0.0
Volatiles ($^{14}CH_4$)	NA	NA	NA	0.0	0.0	0.0	NA	0.0	0.0
Biometer rinse	0.1	0.0	0.1	0.1	0.8	0.5	NA	0.3	0.3
Total balance	99.1	101.7	100.4	104.6	99.6	102.1	NA	100.8	100.8

Fraction	Days After Treatment (DAT)						
	63			112			
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	
Water Phase	13.2	15.2	14.2	10.0	10.0	10.0	
Neutral Extract	77.6	83.4	80.5	85.7	87.0	86.3	
Acidic Extract	1.9	1.8	1.8	1.2	1.4	1.3	
Total Ext.	79.5	85.2	82.4	86.9	88.3	87.6	
Sediment-bound	3.9	3.8	3.8	3.2	3.2	3.2	
Volatiles ($^{14}CO_2$)	0.0	0.0	0.0	0.0	0.0	0.0	
Volatiles ($^{14}CH_4$)	0.0	0.0	0.0	0.0	0.0	0.0	
Biometer rinse	0.9	0.1	0.5	0.1	0.2	0.2	
Total balance	97.5	104.3	100.9	100.2	101.8	101.0	

Notes:
NA: Not analysed.
*This sample was not analysed as it was not dosed properly.

The quantification of inpyrfluxam and the degradates in the whole system is summarised in Table 67 to Table 70. The summary of the mass balance data for exhaustive extraction and fractionation of the Post-extracted sediments at 180 DAT (Sharkey system) is shown in Table 71.

Table 67 Radioactivity distribution from the Sharkey sediment system phenyl-label total activity (combined total sediment extracts and water extracts) as percent of applied radioactivity

Fraction	Days After Treatment (DAT)											
	0			7			14			32		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
ATMI	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.0
1'-COOH-S-2840/1'-CH_2OH-S-2840 *	NA	NA	0.0	0.2	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840	1.8	1.7	1.7	1.1	1.6	1.4	1.9	1.4	1.6	1.1	0.9	1.0
N-des-Me-S-2840	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.1
inpyrfluxam	92.2	91.6	91.9	94.7	94.1	94.4	92.0	91.3	91.6	91.7	94.1	92.9
Total other unknowns	0.0	0.0	0.0	0.7	0.0	0.3	0.0	0.0	0.0	0.2	0.0	0.1
Total	94.0	93.3	93.6	96.7	95.7	96.7	93.9	92.7	93.3	93.1	95.2	94.1
Fraction	Days After Treatment (DAT)											
	67			102			180					
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg			
ATMI	0.0	0.0	0.0	0.0	0.1	0.1	0.0	0.0	0.0			
1'-COOH-S-2840/1'-CH_2OH-S-2840*	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
3'-OH-S-2840	0.5	0.4	0.5	0.3	0.3	0.3	1.5	0.8	1.1			
N-des-Me-S-2840	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.2	0.1			
inpyrfluxam	91.0	91.4	91.2	86.8	86.6	86.7	87.6	86.1	86.9			
Total other unknowns	0.3	0.0	0.1	1.3	0.8	1.1	0.2	0.0	0.1			
Total	91.8	91.8	91.8	88.4	87.9	88.1	89.3	87.1	88.2			

Notes:
*Sum of 1'-CH_2OH-S-2840A, 1'-COOH-S-2840A, 1'-CH_2OH-S-2840B and 1'-COOH-S-2840B. Chromatographically, all compounds elute close together between ~32-34 minutes by HPLC.
NA = Not Analysed.

Table 68 Radioactivity distribution from the Sharkey sediment system pyrazolyl-label total activity (combined total sediment extracts and water extracts), as percent of applied radioactivity

Fraction	Days After Treatment (DAT)											
	0			7			14			32		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
N-des-Me-DFPA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
DFPA-$CONH_2$	0.0	0.0	0.0	0.0	1.5	0.7	0.6	0.0	0.3	0.0	0.0	0.0
DFPA	0.0	1.1	0.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1'-COOH-S-2840/1'-CH_2OH-S-2840 *	NA	NA	0.0	0.0	0.0	0.0	0.5	0.0	0.3	0.0	0.0	0.0
3'-OH-S-2840	4.1	4.1	4.1	2.7	5.0	3.8	3.0	3.2	3.1	2.9	3.1	3.0
N-des-Me-S-2840	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
inpyrfluxam	89.7	89.3	89.5	89.0	86.1	87.6	83.3	76.5	79.9	85.3	88.9	87.1
Total other unknowns	0.3	0.0	0.2	0.0	2.0	1.0	2.4	0.0	1.2	0.0	0.0	0.0
Total	94.1	94.5	94.3	91.7	94.5	91.7	89.8	79.7	84.7	88.1	92.0	90.1

Fraction	Days After Treatment (DAT)								
	67			102			180		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
N-des-Me-DFPA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
DFPA-$CONH_2$	0.3	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.0
DFPA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1'-COOH-S-2840/1'-CH_2OH-S-2840 *	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840	2.8	3.7	3.2	2.3	2.6	2.5	2.2	1.7	1.9
N-des-Me-S-2840	0.0	0.0	0.0	0.0	0.1	0.1	0.1	0.2	0.1
inpyrfluxam	86.7	84.1	85.4	87.0	86.1	86.5	85.5	88.9	87.2
Total other unknowns	1.7	1.1	1.4	0.5	1.0	0.7	0.0	0.0	0.0
Total	91.3	88.9	90.1	89.8	89.8	89.8	87.7	90.7	89.2

Notes:

*Sum of 1'-CH_2OH-S-2840A, 1'-COOH-S-2840A, 1'-CH_2OH-S-2840B and 1'-COOH-S-2840B. Chromatographically, all compounds elute close together between ~32-34 minutes by HPLC.

NA = Not Analysed.

Table 69 Radioactivity distribution from the Golden Lake sediment PH-label total activity (combined neutral sediment extracts and water extracts), as percent of applied radioactivity

Fraction	Days After Treatment (DAT)								
	0			14			30		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
ATMI	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1'-COOH-S-2840/1'-CH_2OH-S-2840 *	0.0	0.0	0.0	0.0	0.0	0.0	0.0	3.2	1.6
3'-OH-S-2840	2.6	3.0	2.8	3.1	2.8	2.9	1.2	3.7	2.5
N-des-Me-S-2840	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
inpyrfluxam	92.7	93.1	92.9	90.4	93.5	91.9	91.8	82.1	87.0
Total other unknowns	0.0	0.0	0.0	0.0	0.0	0.0	1.6	1.5	1.5
Total	95.4	96.1	95.7	93.5	96.2	93.5	94.6	90.4	92.5

Fraction	Days After Treatment (DAT)					
	63			112		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg
ATMI	0.0	0.0	0.0	0.0	0.0	0.0
1'-COOH-S-2840/1'-CH_2OH-S-2840 *	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840	3.0	3.0	3.0	3.0	3.3	3.2
N-des-Me-S-2840	0.0	0.0	0.0	0.0	0.0	0.0
inpyrfluxam	87.4	88.7	88.1	90.8	91.4	91.1
Total other unknowns	0.0	0.0	0.0	0.0	0.0	0.0
Total	90.4	91.7	91.1	93.8	94.7	94.2

Notes:

*Sum of 1'-CH_2OH-S-2840A, 1'-COOH-S-2840A, 1'-CH_2OH-S-2840B and 1'-COOH-S-2840B. Chromatographically, all compounds elute close together between ~32-34 minutes by HPLC.

Table 70 Radioactivity distribution from the Golden Lake sediment PY-label total activity (combined neutral extract and water extracts), as percent of applied radioactivity

Fraction	Days After Treatment (DAT)								
	0			14			30		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1**	Rep2	Avg
N-des-Me-DFPA	0.0	0.0	0.0	0.0	0.0	0.0	NA	0.0	0.0
DFPA-$CONH_2$	0.0	0.0	0.0	0.0	0.0	0.0	NA	0.0	0.0
DFPA	0.0	0.0	0.0	0.0	0.0	0.0	NA	0.5	0.5
1'-COOH-S-2840/1'-CH_2OH-S-2840 *	0.0	0.0	0.0	0.0	0.0	0.0	NA	0.0	0.0
3'-OH-S-2840	5.2	4.5	4.9	5.3	4.9	5.1	NA	5.3	5.3
N-des-Me-S-2840	0.0	0.0	0.0	0.0	0.0	0.0	NA	0.0	0.0
inpyrfluxam	90.5	93.3	91.9	97.5	89.9	93.7	NA	89.1	89.1
Total other unknowns	0.0	0.0	0.0	0.0	0.0	0.0	NA	0.0	0.0
Total	95.7	97.8	96.8	102.8	94.8	98.8	NA	94.9	94.9
Fraction	Days After Treatment (DAT)								
	63			112					
Sample	Rep 1	Rep 2	Avg	Rep 1	Rep 2	Avg			
N-des-Me-DFPA	0.0	0.0	0.0	0.0	0.0	0.0			
DFPA-$CONH_2$	0.0	0.0	0.0	0.0	0.0	0.0			
DFPA	0.7	0.0	0.4	0.0	0.6	0.3			
1'-COOH-S-2840/1'-CH_2OH-S-2840 *	0.0	0.0	0.0	0.0	0.0	0.0			
3'-OH-S-2840	5.0	5.1	5.0	4.9	4.8	4.9			
N-des-Me-S-2840	0.0	0.0	0.0	0.0	0.0	0.0			
inpyrfluxam	84.1	92.5	88.3	89.7	91.7	90.7			
Total other unknowns	0.2	0.0	0.1	0.0	0.0	0.0			
Total	90.1	97.6	93.8	94.6	97.1	95.9			

Notes:

*Sum of 1'-CH_2OH-S-2840A, 1'-COOH-S-2840A, 1'-CH_2OH-S-2840B and 1'-COOH-S-2840B. Chromatographically, all compounds elute close together between ~32-34 minutes by HPLC.

NA = Not Analysed

**This sample was not analysed as it was not dosed properly

Table 71 Summary of the mass balance data for exhaustive extraction and fractionation of sediment-bound activity in Sharkey sediment as percent of applied radioactivity

Sample description	Average of the two replicates (%AR)	
	180 DAT PH-label	180 DAT PY-label
Starting sediment bound (PES)	9.65	9.04
Exhaustive extraction		
Ethyl Acetate	3.32	2.88
Dioxane	1.53	1.27
Hexane	0.37	0.34
Dismembrator	2.21	2.12
Fractionation		
Humin	1.34	0.93
Humic acid	0.22	0.11
Fulvic acid	0.56	0.18
Total	9.55	7.82
% Recovery	99.11	86.27

The average material balance for the Sharkey studies was 98.9 ± 1.5 percent (phenyl) and 96.8 ± 3.4 percent (pyrazolyl) of the applied radioactivity (AR); and the average material balance for the Golden Lake studies was 98.9 ± 1.3 percent (phenyl) and 101.1 ± 2.3 percent (pyrazolyl) of the applied radioactivity (AR). Non-extractable radioactive residues (PES, post extraction sediment) increased to 10 percent of the AR (phenyl) and 9 percent of the AR (pyrazolyl) by the end of the study for the Sharkey system and to 4 percent of the AR (phenyl) and 3 percent of the AR (pyrazolyl) by the end of the study for the Golden Lake system. The production of ^{14}C-volatile activity ($^{14}CO_2$ and $^{14}CH_4$) was insignificant, ranging from 0.0 to 0.1 percent of the AR throughout the study.

In the Sharkey system, 3′-OH-S-2840 was the largest degradate found in both labels with a maximum average of 1.7 percent AR and 4.1 percent AR for the phenyl and pyrazolyl labels, respectively. Minor amounts (<1 percent AR) of ATMI, 1′-COOH-S-2840 and N-des-Me-S-2840 were observed in the phenyl label samples. DFPA-$CONH_2$, DFPA, 1′-COOH-S-2840 and N-des-Me-S-2840 were observed in minor amounts (≤2 percent AR) in the pyrazolyl samples. In the Golden Lake system 3′-OH-S-2840 was the largest degradate found in both labels with a maximum average of 3.2 percent AR and 5.3 percent AR for the phenyl and pyrazolyl labels, respectively. Only one other degradate, 1′-COOH-S-2840 (3 percent AR) was observed in one phenyl label sample. Minor amounts of DFPA (<1 percent AR) were observed in the pyrazolyl samples. These degradates were considered transitory because they were not seen in duplicate samples and disappeared rapidly once they were formed. No significant unknown degradates (>2 percent AR) were observed.

The DT_{50} and DT_{90} of inpyrfluxam in each soil was calculated using the USEPA PestDF kinetic software (this is consistent with the FOCUS approach). The whole system anaerobic aquatic half-life based on the SFO model was estimated at 3537 days (χ^2 = 1.5) for the phenyl and pyrazolyl labels combined for the Sharkey system and 3498 days (χ^2 = 1.8) for the Golden Lake system.

Inpyrfluxam degraded slowly in both sediment/water systems (Sharkey and Golden Lake) under anaerobic aquatic conditions. The majority of the dose remained unchanged after 180 days (Sharkey) or 112 days (Golden Lake) of anaerobic aquatic exposure (87–91 percent of the dose for both labels). No significant degradates (>5 percent of the dose) were formed and ultimate mineralization to bound residues and CO_2 was minor. Whole system anaerobic aquatic half-lives were estimated at 3537 and 3498 days (SFO) for Sharkey and Golden Lake, respectively. 3′-OH-S-2840 was the only degradate found consistently in both sediments and both labels. A variety of other transitory degradates were seen (ATMI, DFPA-$CONH_2$, DFPA, N-des-Me-S-2840 and 1′-COOH-S-2840) in very low amounts.

In study TPM-0038 and TPM-0039 the dissipation, mobility and degradation of inpyrfluxam and its transformation products was determined in an aquatic field dissipation study following planting of rice seed.

In study TPM-0038, treated and control paddies were sown with rice seeds at a drill rate of 83 kg/ha. The treated paddy was sown with inpyrfluxam 3.2 FS (flowable concentrate for seed treatment) treated seed (10 g ai/100 kg seed) which introduced 0.187 gram of inpyrfluxam to the paddy. The control plot was sown with untreated seed. The sediment and soil characteristics of the test site are summarised in Table 72. The irrigation water-source had a pH of 7.4, a conductivity of 0.66 mmhos/cm and a total suspended solids of 4 mg/L. No known compounds that would analytically interfere with inpyrfluxam were used over the three-year period before the study

Table 72 Chemical and Physical characteristics of test sediments and soil (TPM-0038 and TPM-0039)

Characteristic	Untreated Control		Treated	
	sediment	soil	sediment	soil
Soil depth	0-5 cm	5-15 cm	0-5 cm	5-15 cm
USDA Particle size distribution				
% sand (50 µm - 2 mm)	14	16	16	20
% silt (2 µm - 50 µm)	38	32	42	46
% clay <2 µm	48	52	42	34
pH	7.1	7.5	7.3	7.8
% Moisture 1/3 bar	30.9	37.3	30.3	30.2
Cation exchange capacity (meq/100g)	22.1	24.8	20.2	19.3
% Organic carbon	0.93	0.64	0.77	0.56
% Organic Matter	1.6	1.10	2.10	0.96
USDA Textural class	Clay	Clay	Silty clay	Clay loam

After sowing, both paddies were flooded (5 cm of water) in accordance with local agricultural practices. A permanent flood condition (12.7 cm of water) was established in the treated and untreated control paddies 32 days after sowing. At the late boot stage of rice crop development, a broadcast foliar application of inpyrfluxam 2.84 SC (suspension concentrate) was made to the rice canopy in the treated paddy at a nominal rate of 99.8 g as/ha (71 days after sowing). Paddy water, sediment (0–5 cm) and soil (5–15 cm) samples were collected for residue analysis at pre-determined intervals and analysed for inpyrfluxam and its major transformation products (3′-OH-S-2840, 1′-COOH-S-2840-A, and 1′-COOH-S-2840-B). The pre-determined intervals began on the day of broadcast foliar application (0 days after application [DAA]) and continued through 88 DAA. The permanent flood was maintained at a relatively constant depth until it was released 27 days after broadcast foliar application (DAA) (98 days after planting). Sediment and soil sampling continued after the flood release at pre-determined intervals through 88 DAA (159 days after sowing). Application verification pads (from treated and control paddies) were analysed at 0 DAA to confirm that the target application rate for inpyrfluxam was achieved.

Samples were taken (duplicates) at the day of broadcast application. Triplicate soil and sediment samples from the control and treated paddy were taken -78, 0, 1, 5, 14, 26, 42, 61 and 88 DAA (days after broadcast application). Paddy water samples were collected at -39, 0, 1, 3 (sample taken before adding water to reset the water depth), 3(sample taken after addition of water to reset the water depth), 5, 7, 14 and 26 DAA from the control paddy (2 replicates) first, followed by collection from the treated paddy (3 replicates). If an irrigation event was to take place the same day as a scheduled sampling event, water samples were collected prior to the addition of irrigation water. Samples were analysed according to the analytical method RM-50V.

Results from the analysis of saturation pad samples after the test substance application confirmed that the target application rate for inpyrfluxam was achieved. The transit stability test demonstrated the stability of inpyrfluxam during shipment, handling, and storage. In the paddy water, fortified samples showed average recoveries for inpyrfluxam, 3′-OH-S-2840, 1′-COOH-S-2840-A, and 1′-COOH-S-2840-B of between 81.3 percent and 120 percent. In the sediment and soil, fortified samples showed average recoveries for inpyrfluxam, 3′-OH-S-2840, 1′-COOH-S-2840-A, and 1′-COOH-S-2840-B of between 70.1 percent and 116 percent. Hence the methods were considered to be performing appropriately.

Results from the analysis of paddy water samples for inpyrfluxam, 3′-OH-S-2840 and 1′-COOH-S-2840 collected from the treated plot are summarized in Table 73. Treated paddy water was sampled 32 days after sowing to determine whether inpyrfluxam residues were present in the paddy water from the

seed treatment. Results from the analysis of sediment (0-5 cm) samples for inpyrfluxam, 3′-OH-S-2840 and 1′-COOH-S-2840 collected from the treated plot are summarized in Table 74. Results from the analysis of soil (5-15 cm) samples for inpyrfluxam, 3′-OH-S-2840 and 1′-COOH-S-2840 collected from the treated plot are summarized in Table 75.

Table 73 Mean inpyrfluxam and transformation product paddy water results

Actual DAA	Mean paddy water residues found (µg/L)		
	inpyrfluxam	3′-OH-S-2840	1′-COOH-S-2840(sum of A+B isomers)
-39*	ND	ND	ND
0	95.2	ND	ND
1	23.0	ND	ND[1]
3**	17.2	ND	3.9
3***	15.2	ND	3.2
5	7.9	ND	2.8
7	7.2	ND	4.2
14	4.6	ND[1]	5.1
26	1.3	ND	2.5[1]

Notes:
* Paddy flood.
**Sample taken prior to adding water to reset the water depth.
***Sample taken after addition of water to reset the water depth.
[1] Samples contained residues and non-detects; non-detects were replaced with 1/2 × LOD (0.25 ppb) to calculate the mean.
ND = not detected.
DAA = days after foliar application.

Table 74 Mean inpyrfluxam and transformation product sediment results (sampling depth 0–5 cm)

Actual DAA	Mean paddy sediment residues found (mg/kg)[1]		
	inpyrfluxam	3′-OH-S-2840	1′-COOH-S-2840(sum of A+B isomers)
-78	ND	ND	ND
0	0.021	ND	ND
1	0.027	ND	ND
5	0.021	ND	ND
14	0.020	ND	ND
26	ND	ND	ND
42	0.027	ND	ND
61	ND	ND	ND
88	ND	ND	ND

Notes:
1Mean dry-weight residue results reported
ND = not detected
DAA = days after application

Table 75 Mean inpyrfluxam and transformation product soil results (sampling depth 5–15 cm)

Actual DAA	Mean paddy soil residues found (mg/kg)[1]		
	inpyrfluxam	3′-OH-S-2840	1′-COOH-S-2840(sum of A+B isomers)
-78	ND	ND	ND
0	ND	ND	ND
1	ND*	ND	ND
5	ND	ND	ND
14	ND	ND	ND
26	ND	ND	ND
42	ND	ND	ND

Actual DAA	Mean paddy soil residues found (mg/kg)[1]		
	inpyrfluxam	3'-OH-S-2840	1'-COOH-S-2840(sum of A+B isomers)
61	ND	ND	ND
88	ND	ND	ND

Notes:
[1] Mean dry-weight residue results reported.
*Samples contained residues and non-detects; non-detects were replaced with 1/2 × LOD (0.25 ppb) to calculate the mean
ND = not detected.
DAA = days after application.

The only 1'-COOH-S-2840 observed was and only in the water phase at low-level concentrations (maximum 5.1 µg/L). S-2399 dissipated quickly from the paddy water with a calculated half-life (DT_{50}) of 0.485 days by the best fit model (IORE, χ^2 = 6.8). The dissipation of INPYRFLUXAM from the paddy sediment was considerably slower than from the paddy water with an estimated half-life (DT_{50}) of 105 days by the best fit model (SFO, χ^2 = 53).

The average mass of inpyrfluxam from duplicate paddy water samples and the mass of inpyrfluxam in the sediment were summed to obtain the mass of inpyrfluxam in the total system. inpyrfluxam dissipated rapidly from the total system with an estimated half-life (DT_{50}) of 0.86 days by the best fit model (DFOP, χ^2 = 5.7). Table 7.8.1/03-9 summarises the results from the total system.

The dissipation half-life of inpyrfluxam following foliar application to a flooded rice field was determined using the PestDF tool. inpyrfluxam dissipated quickly from the paddy water with a calculated half-life (DT_{50}) of 0.485 days by the best fit model (IORE, χ^2 = 6.8). The dissipation of S-2399 from the paddy sediment was considerably slower than from the paddy water with an estimated half-life (DT_{50}) of 105 days by the best fit model (SFO, χ^2 = 53). The average mass of S-2399 from duplicate paddy water samples and the mass of S-2399 in the sediment were summed to obtain the mass of S-2399 in the total system. S-2399 dissipated rapidly from the total system with an estimated half-life (DT_{50}) of 0.86 days by the best fit model (DFOP, χ^2 = 5.7). The results from paddy water, paddy sediment and from the total system are sumamrised in Table 76

Table 76 Dissipation of S-2399 in paddy water, paddy sediment and total system

Transformation model	DT_{50} (days)	DT_{90} (days)	χ^2	Parameters
Paddy Water				
SFO	0.962	3.19	25	k = 0.721
DFOP	0.398	5.45	8	f = 0.603, k_0 = 3.65, k_1 = 0.253
IORE	0.485	5.8	6.8	N = 2.24, k = 0.00983
Paddy Sediment				
SFO	105	349	53	k = 0.00659
DFOP	102	237	60	f = -0.685, k_0 = 0.243, k_1 = 0.0119
IORE	81.5	94.8	55	N = -1.82, k = 3.09
Total System				
SFO	1.18	3.92	24	k = 0.588
DFOP	0.86	20.4	5.7	f = 0.714, k_0 = 1.34, k_1 = 0.0515
IORE	0.822	21.3	6.9	N = 2.83, k = 0.000488

Notes:
SFO = Single First Order; DFOP = Double First Order in Parallel; IORE = Intermediate Order Rate Equation.

Inpyrfluxam declines rapidly in the water phase. Formation of the 1'-COOH-S-2840 transformation product in the water phase was observed at low-level concentrations, and its decline was evident within the study period. Inpyrfluxam was observed in the sediment and soil phases at very low concentrations,

with no transformation product residues observed in sediment and soil samples collected at any sampling event. The dissipation half-life of inpyrfluxam following foliar application to a flooded rice field was calculated using the PestDF tool. Inpyrfluxam dissipated quickly from the paddy water with a calculated half-life of 0.485 days by the best fit model (IORE). Dissipation of S-2399 from the paddy sediment was significantly slower with a calculated half-life of 105 days (SFO). S-2399 dissipated rapidly from the system as a whole (paddy water + paddy sediment) with a calculated half-life of 0.86 days by the best fit model (DFOP).

In study TPM-0039, treated and control paddies were sown with rice seeds by hand-broadcast (method equivalent to drilling) at a rate of 185 kg seed/ha. The treated paddy was sown with inpyrfluxam 3.2 FS (flowable concentrate for seed treatment) treated seed (10 grams of active ingredient per 100 kg of seed) which introduced 0.756 gram of S-2399 to the paddy. The control plot was sown with untreated seed. Two days after sowing, both paddies were flooded (average 11 cm of water). At the late boot stage of rice crop development (77 days after sowing), a broadcast foliar application of S-2399 2.84 SC (suspension concentrate) was made to the rice canopy in the treated paddy at a nominal rate of 100 g as/ha.

Paddy water, sediment (0-5 cm) and soil (5-15 cm) samples were collected for residue analysis at pre-determined intervals and analysed for inpyrfluxam and its major transformation products (3'-OH-S-2840 and 1'-COOH-S-2840). The pre-determined intervals began on the day of broadcast foliar application (0 days after application [DAA]) and continued through 90 DAA. The permanent flood was maintained at a relatively constant depth until it was released 28 days after broadcast foliar application (DAA). Sediment and soil sampling continued after the flood release at pre-determined intervals through 90 DAA. Application verification pads (from treated and control paddies) were analysed at 0 DAA to confirm that the target application rate for S-2399 was achieved.

Application verification pads were taken at the day of broadcast application of S-239 2.84SC. Triplicate soil and sediment samples from the control and treated paddy were taken -84, 0, 1, 5, 14, 28, 42, 60 and 90 DAA (days after broadcast application). Paddy water samples were collected at -32, 0, 1, 3 (sample taken before adding water to reset the water depth), 3 (sample taken after addition of water to reset the water depth), 5, 7, 14 and 28 DAA from the control paddy (2 replicates) first, followed by collection from the treated paddy (3 replicates). If an irrigation event was to take place the same day as a scheduled sampling event, water samples were collected prior to the addition of irrigation water. Samples were analysed according to the analytical method RM-50W.

For the application verification (saturation pad) samples, two fortified recovery samples were analysed with the entire batch of application verification samples. Recovery from fortified samples was 106 percent and 107 percent. For the paddy water matrix the overall average recoveries for S-2399, 3'-OH-S-2840 and 1'-COOH-S-2840 were between 78.6 and 120 percent for the fortified samples. For the sediment and soil matrices, the overall average recoveries for S-2399, 3'-OH-S- 2840 and 1'-COOH-S-2840 ranged from 82.1 to 101 percent, 79.9 to 95.4 percent, and 66.8 to 94.9 percent respectively for fortified samples. Hence the methods were considered to be performing appropriately Results from the analysis of paddy water samples for S-2399, 3'-OH-S-2840 and 1'-COOH-S-2840 collected from the treated plot are summarized in Table 77.

Table 77 Mean S-2399 and transformation product paddy water results

Actual DAA	Mean paddy water residues found (µg/L)		
	S-2399	3'-OH-S-2840	1'-COOH-S-2840 (sum of A+B isomers
-32	ND	ND	ND
0	51.8	ND	ND
1	7.8	ND	ND

Actual DAA	Mean paddy water residues found (µg/L)		
	S-2399	3′-OH-S-2840	1′-COOH-S-2840 (sum of A+B isomers
3*	4.9	ND	ND
3**	6.0	ND	ND[1]
5	6.3	ND	ND[1]
7	3.0	ND	ND[1]
14	ND	ND	ND
28	ND[1]	ND	ND

Notes:
*Sample taken prior to adding water to reset the water depth.
**Sample taken after addition of water to reset the water depth.
[1]Samples contained residues and non-detects; non-detects were replaced with 1/2 × LOD (0.25 ppb) to calculate the mean.
ND = not detected.
DAA = days after application.

Treated paddy water was sampled 45 days after sowing (32 days before foliar application) to determine whether S-2399 residues were present in the paddy water from the seed treatment. Results from the analysis of sediment (0–5 cm) samples for S-2399, 3′-OH-S-2840 and 1′-COOH-S-2840 collected from the treated plot are summarized in Table 78.

Table 78 Mean S-2399 and transformation product sediment results (sampling depth 0–5 cm)

Actual DAA	Mean paddy sediment residues found (mg/kg)[1]		
	S-2399	3′-OH-S-2840	1′-COOH-S-2840 (sum of A+B isomers)
-84	NA	NA	NA
0	0.016	ND	ND
1	0.012*	ND	ND
5	0.027	ND	ND
14	0.009*	ND	ND
28	ND	ND	ND
42	0.018	ND	ND
60	0.026	ND	ND
90	0.019	ND	ND

Notes:
[1]Mean dry-weight residue results reported.
ND = not detected.
DAA = days after application.
NA = Not applicable (sample not received).
*Samples contained residues and non-detects; non-detects were replaced with 1/2 × LOD (0.25 ppb) to calculate the mean.

Results from the analysis of soil (5–15 cm) samples for S-2399, 3′-OH-S-2840 and 1′-COOH-S-2840 collected from the treated plot are summarized in Table 79.

Table 79 Mean S-2399 and transformation product soil results (sampling depth 5–15 cm)

Actual DAA	Mean paddy soil residues found (mg/kg)[1]		
	S-2399	3′-OH-S-2840	1′-COOH-S-2840 (sum of A+B isomers)
-84	NA	NA	NA
0	ND	ND	ND
1	ND	ND	ND
5	0.007*	ND	ND
14	ND	ND	ND
28	ND	ND	ND
42	ND	ND	ND
60	ND	ND	ND

Actual DAA	Mean paddy soil residues found (mg/kg)[1]		
	S-2399	3'-OH-S-2840	1'-COOH-S-2840 (sum of A+B isomers)
90	ND	ND	ND

Notes:
1Mean dry-weight residue results reported.
ND = not detected.
*Samples contained residues and non-detects; non-detects were replaced with 1/2 × LOD (0.25 ppb) to calculate the mean.
ND = not detected.
DAA = days after application.
NA = Not applicable (sample not received).

Mass balance calculations were not possible as the test substance was not radiolabelled. No degradates were observed in the water phase, soil or sediment above the LOQ. The dissipation half-life of S-2399 following foliar application to a flooded rice field was determined using the PestDF tool. S-2399 dissipated quickly from the paddy water with a calculated half-life (DT_{50}) of 0.0312 days by the best fit model (DFOP, χ^2 = 7.4). There was no dissipation from the paddy sediment over the course of the study.

The average mass of Inpyrfluxam from duplicate paddy water samples and the mass of S-2399 in the sediment, were summed to obtain the mass of S-2399 in the total system. S-2399 dissipated rapidly from the total system with an estimated half-life (DT_{50}) of 0.0641 days by the best fit model (DFOP, χ^2 = 13). Table 80 summarises the results from the paddy water and total system.

Table 80 Dissipation of S-2399 in paddy water, paddy sediment and total system

Transformation model	DT_{50} (days)	DT_{90} (days)	χ^2	Parameters
Paddy Water				
SFO	0.387	1.28	25	k = 1.79
DFOP	0.0312	3.72	7.4	f = 0.831, k_0 = 29.4, k_1 = 0.141
IORE	0.0839	2.64	10	N = 2.97, k = 0.00381
Total System				
SFO	0.44	1.46	29	k = 1.58
DFOP	0.0641	13.1	13	f = 0.747, k_0 = 17.2, k_1 = 0.071
IORE	0.0844	13.4	16	N = 4.07, k = 0.0000429

Notes:
SFO = Single First Order; DFOP = Double First Order in Parallel; IORE = Intermediate Order Rate Equation.

Inpyrfluxam declines rapidly in the water phase. Inpyrfluxam was observed in the sediment and soil phases at very low concentrations. No transformation product residues were observed in the water phase, sediment and soil samples collected at any sampling event. Inpyrfluxam dissipated quickly from the paddy water with a calculated half-life of 0.0312 days by the best fit model (DFOP). There was no dissipation from the paddy sediment over the course of the study. S-2399 dissipated rapidly from the system as a whole (paddy water + paddy sediment) with a calculated half-life of 0.0641 days by the best fit model (DFOP).

In study TPM-0050 the biotransformation of inpyrfluxam in three water/sediment system (Goose River, Sharkey and Weweantic River) was investigated under aerobic aquatic conditions using [pyrazolyl-4-^{14}C]S-2399.

Three test systems (sediment and water) were collected from the top 0–5 cm layer (Goose River), 0–7.6 cm layer (Sharkey) and 0–7.6 cm layer (Weweantic River). The sediment was thoroughly mixed and passed through a 2-mm mesh sieve with a minimum of air-drying. The sediment and water were stored in the dark before being used. The sediments characteristics are summarised in Table 81.

Table 81 Chemical and physical characteristics of test sediments

Sediment characteristic	Goose River	Sharkey	Weweantic River
USDA Particle size distribution			
% sand (50 µm - 2 mm)	25	21	97
% silt (2 µm - 50 µm)	42	21	3
% clay <2 µm	33	58	0
pH	7.9	6.5	5.7
% Moisture 1/3 bar	49.9	52.5	10.6
Cation exchange capacity (meq/100g)	22.7	30.9	3.8
% Organic carbon			
% Organic Matter	3.5	2.4	0.9
	6.0	4.2	1.6
USDA Textural class	Clay loam	Clay	Sand
Microbial Biomass Carbon (µg/g dry weight)	72.9 (0 DAT) 73.6 (111 DAT; untreated control) 31.4 (111 DAT; solvent control) 45.3 (111 DAT; S-2399 control)	67.9 (0 DAT) 60.1 (111 DAT; untreated control) 48.9 (111 DAT; solvent control) 69.8 (111 DAT; S-2399 control)	10.4 (0 DAT) <0.1 (111 DAT; untreated control) < 0.1 (111 DAT; solvent control) 3.5 (111 DAT; S-2399 control)

The aerobic aquatic test systems consisted in an incubation apparatus containing 50 g of dry weight sediment and water collected from the same location in a sediment:water ratio of 0.30 (Goose River), 0.29 Sharkey and 0.33 (Weweantic River). The systems were dosed at dry sediment concentrations at *ca*. 0.05 mg/kg (Goose River), 0.06 mg/kg (Sharkey) and 0.06 mg/kg (Weweantic River). The test systems were incubated at 20 ± 2 °C in the dark for a maximum of 111 days and were periodically collected and extracted. The test systems were equipped with 1 M NaOH traps for the collection of evolved $^{14}CO_2$ and tetraglyme/ethylene glycol traps for ^{14}C volatile capture. A pre-incubation period of 8-12 days was performed for the Goose River system, 14 days for the Sharkey sediment system and 4 days for the Weweantic River system before dosing.

Duplicate samples were removed at 0, 1, 3, 7, 15, 30, 45, 62, 76, 91 and 111 DAT and analysed immediately. The physical parameters of the aerobic systems (oxygen concentration, redox and pH) were measured and the water separated from the sediment. The water phase and all eluents were analysed by LSC. The water phase was adjusted to pH 5 and subjected to Solid Phase Extraction (SPE) with water and acetonitrile. Representative samples were analysed by TLC. The sediment samples were extracted with acetone, twice with acetone:water (3:2) and with acetone:water:HCl (c) (60:40:1). The acidic extract rotary-evaporated to remove the acetone was adjusted to pH 5 and subjected to SPE with water and acetonitrile. The activity of the extracts was determined by LSC. The extracts were analysed by HPLC and representative samples by TLC. Selected 62 and 111 DAT samples from the acidic extraction were analysed by HPLC. Representative PES at 111 DAT were subjected to further additional sequential solvent extractions with ethyl acetate, dioxane and hexane, and with a dismembrator (5:1 acetone:0.5 M HCl) for comparison. The radioactivity was determined by LSC and the extract was analysed by HPLC. Total radioactivity in PES was determined by combustion. The Goose River PES was finally subjected to a Humin, Humic Acid and Fulvic Acid Fractionation. The $^{14}CO_2$ collected in the NaOH trapping solution and organic volatiles collected in tetraglyme/ethylene glycol traps were quantified by LSC. The potential isomerisation from [^{14}C]S-2399 was evaluated using chiral HPLC analysis on the extracts obtained from the 111 DAT samples.

No significant change in the microbial biomass carbon was recognized between the initiation and termination of the incubation (Table 81). Thus, microbial viability was proved to be satisfactorily

maintained during the incubation period. It must be noted that values for the Weweantic River are considered not atypical for the type of sediment.

It was confirmed that no isomerisation of [^{14}C]S-2399 occurred during incubation period based on chiral HPLC analysis.

The distribution and mass balance of applied radioactivity of [^{14}C]S-2399 in water phase, extractable, sediment-bound and volatile fractions are summarised in Table 82 to Table 84. The quantification of S-2399 and the degradates in the neutral extracts and water phase is summarised in Table 85 to Table 87. The acidic sediment extracts at 62 and 111 DAT showed only the presence of S-2399, and 3'-OH-S-2840 and other unknowns in only very low percentage of applied radioactivity (Table 82).

Table 82 Summary of the mass balance data for the Goose River system as percentage of Applied Radioactivity

Fraction	Days After Treatment (DAT)											
	0			1			3			7		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	91.4	90.8	91.1	57.9	64.5	61.2	42.7	45.1	43.9	30.7	33.4	32.0
Neutral Extract	6.6	7.2	6.9	37.7	30.1	33.9	53.3	48.8	51.1	61.0	59.1	60.0
Acidic Extract	0.1	0.1	0.1	0.8	0.7	0.8	2.1	2.1	2.1	4.0	3.4	3.7
Total Ext.	6.6	7.3	7.0	38.4	30.8	34.6	55.4	50.9	53.2	65.0	62.5	63.8
Sediment-bound	0.1	0.1	0.1	0.8	0.8	0.8	2.6	0.2	1.4	5.5	4.6	5.0
Volatiles ($^{14}CO_2$)	NA	NA	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total balance	98.2	98.2	98.2	97.1	96.2	96.6	100.6	96.3	98.5	101.2	100.4	100.8
Fraction	Days After Treatment (DAT)											
	15			30			45			62		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	19.7	18.4	19.1	15.0	10.3	12.6	7.9	8.6	8.3	7.4	7.8	7.6
Neutral Extract	68.6	70.7	69.7	75.7	77.8	76.7	78.0	78.7	78.3	73.9	75.7	74.8
Acidic Extract	5.9	5.5	5.7	5.1	5.0	5.0	5.1	5.2	5.1	6.6	8.0	7.3
Total Ext.	74.5	76.2	75.3	80.8	82.8	81.8	83.0	83.8	83.4	80.6	83.7	82.1
Sediment-bound	6.8	6.1	6.4	6.1	6.4	6.3	7.7	6.7	7.2	7.9	7.7	7.8
Volatiles ($^{14}CO_2$)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.2	0.2	0.2
Total balance	101.1	100.7	100.9	101.9	99.6	100.7	98.6	99.2	98.9	96.1	99.4	97.7
Fraction	Days After Treatment (DAT)											
	76			91			111					
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg			
Water Phase	8.1	5.7	6.9	6.3	6.8	6.5	6.9	6.6	6.8			
Neutral Extract	75.8	78.0	76.9	75.0	73.9	74.5	71.6	69.0	70.3			
Acidic Extract	6.8	7.5	7.2	10.3	9.8	10.0	9.9	9.6	9.8			
Total Ext.	82.6	85.5	84.1	85.3	83.7	84.5	81.5	78.6	80.0			
Sediment-bound	9.7	9.3	9.5	9.4	10.5	10.0	12.5	12.7	12.6			
Volatiles ($^{14}CO_2$)	0.3	0.3	0.3	0.4	0.4	0.4	0.5	0.5	0.5			
Total balance	100.7	100.8	100.7	101.4	101.4	101.4	101.4	98.4	99.9			

Note:
NA: not analysed

Table 83 Summary of the mass balance data for the Sharkey system as percentage of Applied Radioactivity

Fraction	Days After Treatment (DAT)											
	0			1			3			7		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	89.6	92.8	91.2	64.4	66.3	65.3	46.7	53.7	50.2	43.5	46.1	44.8
Neutral Extract	8.1	7.6	7.9	31.9	29.9	30.9	41.4	44.1	42.7	52.8	48.8	50.8
Acidic Extract	0.0	0.0	0.0	0.3	0.3	0.3	1.1	1.0	1.0	3.1	3.3	3.2
Total Ext.	8.1	7.6	7.9	32.3	30.2	31.2	42.4	45.1	43.8	55.9	52.1	54.0
Sediment-bound	0.0	0.0	0.0	0.4	0.2	0.3	0.6	0.5	0.6	1.6	1.6	1.6
Volatiles ($^{14}CO_2$)	NA	NA	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total balance	97.7	100.5	99.1	97.0	96.7	96.8	89.8	99.3	94.5	100.9	99.8	100.4

Fraction	Days After Treatment (DAT)											
	15			30			45			62		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	37.7	39.2	38.5	28.6	28.9	28.7	15.9	15.3	15.6	13.8	10.0	11.9
Neutral Extract	56.8	55.4	56.1	62.4	71.6	67.0	78.3	78.2	78.3	76.8	80.9	78.9
Acidic Extract	3.4	3.8	3.6	4.0	3.3	3.7	3.5	3.0	3.3	5.4	4.4	4.9
Total Ext.	60.3	59.2	59.7	66.4	74.9	70.6	81.8	81.2	81.5	82.2	85.4	83.8
Sediment-bound	1.9	2.2	2.0	2.4	3.4	2.9	3.1	3.5	3.3	3.2	4.1	3.7
Volatiles ($^{14}CO_2$)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total balance	99.9	100.6	100.2	97.4	107.2	102.3	100.9	100.1	100.5	99.3	99.6	99.4

Fraction	Days After Treatment (DAT)								
	76			91			111		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	12.0	10.6	11.3	10.5	9.0	9.8	10.1	10.5	10.3
Neutral Extract	80.1	81.7	80.9	81.3	81.6	81.4	79.2	79.7	79.5
Acidic Extract	5.2	5.3	5.3	6.3	6.0	6.2	5.9	5.1	5.5
Total Ext.	85.3	87.1	86.2	87.6	87.6	87.6	85.1	84.8	85.0
Sediment-bound	3.6	3.5	3.5	4.0	4.5	4.2	4.2	5.3	4.8
Volatiles ($^{14}CO_2$)	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Total balance	101	101.2	101.1	102.2	101.1	101.6	99.6	100.8	100.2

Note:
NA: Not analysed.

Table 84 Summary of the mass balance data for the Weweantic River system as percentage of Applied Radioactivity

Fraction	Days After Treatment (DAT)											
	0			1			3			7		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	79.7	76.9	78.3	65.4	63.9	64.7	54.4	56.5	55.5	41.7	44.4	43.1
Neutral Extract	18.7	21.2	20.0	31.4	34.3	32.9	43.7	43.2	43.4	56.1	54.4	55.2
Acidic Extract	0.0	0.0	0.0	0.3	0.2	0.3	0.5	0.5	0.5	1.1	1.1	1.1
Total Ext.	18.7	21.2	20.0	31.7	34.5	33.1	44.1	43.7	43.9	57.3	55.5	56.4
Sediment-bound	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.4	0.2	0.4	0.3	0.4
Volatiles ($^{14}CO_2$)	NA	NA	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total balance	98.5	98.2	98.3	97.1	98.5	97.8	98.6	100.6	99.6	99.4	100.2	99.8
Fraction	Days After Treatment (DAT)											
	15			30			45			62		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
Water Phase	37.9	41.0	39.5	34.1	33.5	33.8	24.6	23.6	24.1	23.5	22.3	22.9
Neutral Extract	60.6	57.7	59.2	64.3	61.8	63.0	69.9	71.3	70.6	68.9	71.2	70.1
Acidic Extract	1.4	1.3	1.3	2.0	1.9	2.0	2.5	2.2	2.4	4.2	3.7	4.0
Total Ext.	62.0	59.0	60.5	66.3	63.7	65.0	72.4	73.5	73.0	73.1	75.0	74.0
Sediment-bound	0.6	0.4	0.5	0.8	0.7	0,8	1.1	1.0	1.1	1.3	1.3	1.3
Volatiles ($^{14}CO_2$)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total balance	100.5	100.4	100.4	101.2	97.9	99.5	98.1	98.1	98.1	98.0	98.5	98.2
Fraction	Days After Treatment (DAT)											
	76			91			111					
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg			
Water Phase	19.4	19.7	19.6	16.6	19.5	18.1	15.6	15.1	15.4			
Neutral Extract	72.8	73.9	73.4	75.5	71.1	73.3	73.3	73.5	73.4			
Acidic Extract	5.1	4.8	4.9	6.3	7.1	6.7	7.4	6.7	7.0			
Total Ext.	77.9	78.7	78.3	81.8	78.2	80.0	80.6	80.2	80.4			
Sediment-bound	2.0	1.9	1.9	2.1	2.4	2.3	3.1	3.3	3.2			
Volatiles ($^{14}CO_2$)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
Total balance	99.3	100.3	99.8	100.6	100.1	100.4	99.4	98.7	99.0			

Note:
NA: not analysed

Table 85 Radioactivity distribution from the water and sediment of Goose River system (excluding acidic sediment extract) as percentage of applied radioactivity

Fraction	Days After Treatment (DAT)											
	0			1			3			7		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
S-2399 (water)	92.3	93.5	92.9	56.0	63.2	59.6	43.4	43.8	43.6	29.8	32.9	31.4
S-2399 (sediment)	6.6	7.2	6.9	37.7	30.1	33.9	52.8	48.2	50.5	60.3	58.4	59.3
1'-COOH-S-2840 total** (water)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840 (water)	0.0	0.0	0.0	0.6	0.5	0.6	0.4	0.3	0.3	0.0	0.4	0.2
3'-OH-S-2840	0.0	0.0	0.0	0.0	0.0	0.0	0.5	0.6	0.5	0.7	0.7	0.7

(sediment)												
Others (Total) *	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total*	98.8	100.7	99.8	94.3	93.8	94.0	97.1	92.9	95.0	90.8	92.4	91.6
Fraction	Days After Treatment (DAT)											
	15			30			45			62		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
S-2399 (water)	18.0	16.0	17.0	11.9	7.0	9.5	5.9	5.7	5.8	4.4	4.4	4.4
S-2399 (sediment)	67.4	69.6	68.5	74.5	76.1	75.3	74.8	74.2	74.5	69.8	71.9	70.9
1'-COOH-S-2840 total** (water)	1.0	1.5	1.3	1.7	1.8	1.8	2.1	2.5	2.3	2.5	2.9	2.7
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	1.3	2.4	1.9	2.0	3.1	2.5
3'-OH-S-2840 (water)	0.6	0.3	0.4	0.3	0.2	0.2	0.0	0.0	0.0	0.3	0.0	0.1
3'-OH-S-2840 (sediment)	1.3	1.2	1.2	1.2	1.7	1.4	1.8	2.0	1.9	2.2	2.3	2.2
Others (Total) *	0.0	0.0	0.0	0.2	0.4	0.3	0.0	0.0	0.0	0.0	0.0	0.0
Total*	88.2	88.4	88.3	89.8	87.2	88.5	86.0	86.8	86.4	81.1	84.6	82.8
Fraction	Days After Treatment (DAT)											
	76			91			111					
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg			
S-2399 (water)	4.3	2.8	3.6	2.1	2.3	2.2	2.2	1.7	1.9			
S-2399 (sediment)	71.0	73.7	72.3	68.9	68.0	68.5	64.3	62.0	63.2			
1'-COOH-S-2840 total** (water)	3.5	2.4	3.0	3.2	3.9	3.5	3.8	3.3	3.6			
1'-COOH-S-2840 total** (sediment)	2.3	2.2	2.3	3.2	3.1	3.1	4.3	4.2	4.2			
3'-OH-S-2840 (water)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
3'-OH-S-2840 (sediment)	2.1	2.2	2.1	2.8	2.9	2.9	2.9	2.8	2.9			
Others (Total) *	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
Total*	83.1	83.3	83.2	80.3	80.1	80.2	77.7	74.0	75.8			

Notes:

* Includes DFPA and total other unknowns which never individually exceed 0.2% in the whole system.

** As the sum of both isomers = 1'-COOH-S-2840 A + 1'-COOH-S-2840 B.

Table 86 Radioactivity distribution in the water and sediment of Sharkey system (excluding acidic sediment) as percentage of applied radioactivity

Fraction	Days After Treatment (DAT)											
	0			1			3			7		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
S-2399 (water)	86.0	92.0	89.0	63.7	64.5	64.1	44.0	53.9	49.0	42.1	44.0	43.1
S-2399 (sediment)	8.1	7.6	7.9	31.9	29.5	30.7	40.9	43.5	42.2	52.1	48.0	50.0
1'-COOH-S-2840 total** (water)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840 (water)	1.0	1.1	1.0	0.5	1.1	0.8	0.4	0.8	0.6	0.7	0.6	0.6
3'-OH-S-2840 (sediment)	0.0	0.0	0.0	0.0	0.4	0.2	0.5	0.6	0.5	0.7	0.8	0.7
Others (Total) *	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total*	95.2	100.7	97.9	96.2	95.5	95.8	85.8	98.8	92.3	95.7	93.4	94.5
Fraction	Days After Treatment (DAT)											
	15			30			45			62		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
S-2399 (water)	35.7	37.8	36.8	25.0	24.7	24.8	11.5	10.4	11.0	8.9	5.1	7.0
S-2399 (sediment)	55.9	54.2	55.0	61.1	70.2	65.6	74.9	75.4	75.1	72.0	77.4	74.7
1'-COOH-S-2840 total** (water)	0.0	0.0	0.0	2.6	2.4	2.6	3.3	2.1	2.7	2.6	3.6	3.1
1'-COOH-S-2840 total** (sediment)	0.3	0.0	0.2	0.0	0.0	0.0	1.6	1.2	1.4	3.0	1.7	2.4
3'-OH-S-2840 (water)	0.9	0.6	0.7	0.4	0.4	0.4	0.0	0.0	0.0	0.2	0.0	0.1
3'-OH-S-2840 (sediment)	0.6	0.8	0.7	1.3	1.4	1.3	1.9	1.6	1.7	1.4	1.8	1.6
Others (Total)*	0.0	0.3	0.2	0.9	0.6	0.7	0.5	0.8	0.7	1.3	0.7	1.0
Total*	93.4	93.8	93.6	91.3	99.8	95.5	93.8	91.6	92.7	89.4	90.3	89.9

Fraction	Days After Treatment (DAT)								
	76			91			111		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
S-2399 (water)	6.6	5.4	6.0	5.1	4.0	4.6	4.2	4.1	4.1
S-2399 (sediment)	74.8	77.9	76.4	74.7	76.3	75.5	73.5	74.4	74.0
1'-COOH-S-2840 total** (water)	3.9	3.5	3.7	3.2	4.0	3.7	4.3	4.1	4.2
1'-COOH-S-2840 total** (sediment)	2.7	1.4	2.0	2.7	2.0	2.3	3.0	1.7	2.4
3'-OH-S-2840 (water)	0.0	0.2	0.1	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840 (sediment)	1.8	1.8	1.8	2.4	2.8	2.6	2.7	2.6	2.7
Others (Total)*	1.9	1.9	1.8	3.1	1.4	2.2	1.1	2.5	1.8
Total*	91.7	91.9	91.8	91.2	90.6	90.9	88.7	89.5	89.1

Notes:

* Includes DFPA and total other unknowns which never individually exceed 2.4% in the whole system.

** As the sum of both isomers = 1'-COOH-S-2840 A + 1'-COOH-S-2840 B.

Table 87 Radioactivity distribution in water and sediment from Weweantic River system (excluding acidic sediment extract) as percentage of applied radioactivity

Fraction	Days After Treatment (DAT)											
	0			1			3			7		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
S-2399 (water)	79.3	76.0	77.6	64.8	63.2	64.0	55.3	55.7	55.5	39.6	40.6	40.1
S-2399 (sediment)	18.7	21.2	20.0	31.4	34.3	32.9	43.1	42.7	42.9	55.4	54.2	54.8
1'-COOH-S-2840 total** (water)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3'-OH-S-2840 (water)	1.7	1.7	1.7	0.8	1.6	1.2	0.7	0.5	0.6	0.6	0.6	0.6
3'-OH-S-2840 (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.6	0.5	0.5	0.7	0.2	0.5
Others (Total)*	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.2	0.1	0.0	0.5	0.3
Total*	99.7	98.9	99.3	97.0	99.0	98.0	99.7	99.6	99.6	96.3	96.1	96.2

Fraction	Days After Treatment (DAT)											
	15			30			45			62		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
S-2399 (water)	35.0	36.9	36.0	29.4	27.9	28.6	22.7	19.9	21.3	16.9	18.0	17.5
S-2399 (sediment)	59.4	57.1	58.2	63.0	60.8	61.9	68.9	69.8	69.4	66.4	67.7	67.0
1'-COOH-S-2840 total** (water)	0.9	0.9	0.9	2.4	2.7	2.6	1.9	1.5	1.7	4.6	2.2	3.4
1'-COOH-S-2840 total** (sediment)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.6	0.8	0.7
3'-OH-S-2840 (water)	0.7	0.8	0.8	0.8	0.6	0.7	0.4	0.4	0.4	0.5	0.7	0.6
3'-OH-S-2840 (sediment)	1.3	0.6	0.9	1.3	1.0	1.1	0.9	1.6	1.2	1.1	1.5	1.3
Others (Total)*	1.9	0.7	1.3	0.9	1.7	1.3	0.7	0.9	0.8	1.3	1.8	1.6
Total*	99.1	97.0	98.1	97.8	94.6	96.2	95.5	94.0	94.8	91.5	92.7	92.1

Fraction	Days After Treatment (DAT)								
	76			91			111		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg
S-2399 (water)	15.2	12.0	13.6	12.0	11.4	11.7	10.6	11.6	11.1
S-2399 (sediment)	70.6	72.7	71.7	73.7	69.1	71.4	70.0	70.2	70.1
1'-COOH-S-2840 total** (water)	3.2	3.3	3.2	1.7	3.5	2.6	2.4	1.7	2.0
1'-COOH-S-2840 total** (sediment)	0.4	0.0	0.2	0.0	0.4	0.2	1.2	0.5	0.9
3'-OH-S-2840 (water)	0.3	0.3	0.3	0.4	0.2	0.3	0.0	0.0	0.0
3'-OH-S-2840 (sediment)	0.9	0.8	0.9	1.5	1.6	1.6	1.2	2.0	1.6
Others (Total)*	1.6	2.3	2.1	1.6	1.7	1.7	2.3	1.5	1.9
Total*	92.3	91.5	91.9	91.0	87.9	89.4	87.7	87.5	87.6

Notes:

* Includes DFPA, DFPA-$CONH_2$, N-des-Me-S-2840 and total other unknowns which never individually exceed 1.3% in the whole system.

** As the sum of both isomers = 1'-COOH-S-2840 A + 1'-COOH-S-2840 B.

Table 88 Radioactivity distribution in acidic sediment extracts as percent of applied radioactivity

System	Goose River						Sharkey					
Fraction	Days After Treatment (DAT)											
	62			111			62			111		
Sample	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep2	Avg	Rep1	Rep 2	Avg
3'-OH-S-2840	0.0	0.0	0.0	0.0	0.5	0.2	0.0	0.0	0.0	0.0	0.0	0.0
S-2399	6.6	6.9	6.8	9.9	9.1	9.5	5.4	4.2	4.8	5.9	4.2	5.0
Total other unknowns	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.2	0.1	0.0	0.9	0.4
Total	6.6	6.9	6.8	9.9	9.6	9.8	5.4	4.4	4.9	5.9	5.1	5.5

System	Weweantic River					
Fraction	Days After Treatment (DAT)					
	62			111		
Sample	Rep1	Rep2	Avg	Rep1	Rep 2	Avg
3'-OH-S-2840	0.0	0.0	0.0	0.0	0.0	0.0
S-2399	4.2	3.1	3.7	7.2	6.7	6.9
Total other unknowns	0.0	0.6	0.3	0.2	0.0	0.1
Total	4.2	3.7	4.0	7.4	6.7	7.0

The average mass balance was 99.5 ± 1.9 percent AR for the Goose River system, 99.6 ± 3.1 percent for the Sharkey system and 99.2 ± 1.1 percent for the Weweantic River system. The radioactivity remaining in sediment following the neutral and acidic extractions was 13 percent of the AR (Goose River), 5 percent of the AR (Sharkey) and 3 percent of the AR (Weweantic River) by the end of the study (111 DAT). Additional extractions (ethyl acetate extract, 4.5 percent of the AR; dioxane and hexane extract, <1 percent of the AR; dismembrator extract, 6.4 percent of the AR) of the Goose River and fractioning into humin (5.5 percent of AR), humic acid (0.8 percent of AR) and fulvic acid (1.0 percent of AR) fractions was performed. The total amount of carbon dioxide generated during the study phase was negligible, reaching 0.5 percent of the AR in the Goose River system, 0.1 percent in the Sharkey system and 0.0 percent in the Weweantic River system.

Two degradates were observed above 2.5 percent AR: 3´-OH-S-2840, and 1'COOH-S-2840. The maximum observed 3´-OH-S-2840 and 1'-COOH-S-2840 (as 1'-COOH-S-2840 A + 1'-COOH-S-2840 B) were 2.9 and 8.1 percent AR respectively at 111 DAT in the Goose River system and 2.8 percent AR at 91 DAT and 7.3 percent AR at 111 DAT respectively in the Sharkey system. In the Weweantic River system, the maximum observed 3´-OH-S-2840 and 1'-COOH-S-2840 were 2.2 and 5.2 percent AR at 62 DAT. The aerobic aquatic metabolism degradation pathway of S-2399 is summarized in Figure 10. Proposed aerobic aquatic degradation pathways of S-2399.

A whole system aerobic aquatic half-life was estimated using the PestDF kinetics (this is consistent with the FOCUS approach) for the three systems. As a conservative approach the radioactivity in the acidic extract was added to the identified S-2399 in the water and neutral sediment extract, for the DT_{50} calculation. The estimated aerobic aquatic half-lives for the Goose River, Sharkey and Weweantic River sediment systems were 319, 563 and 704 days (SFO) respectively. Chi^2 error values were 1.6, 1.3 and 1.1 percent, respectively.

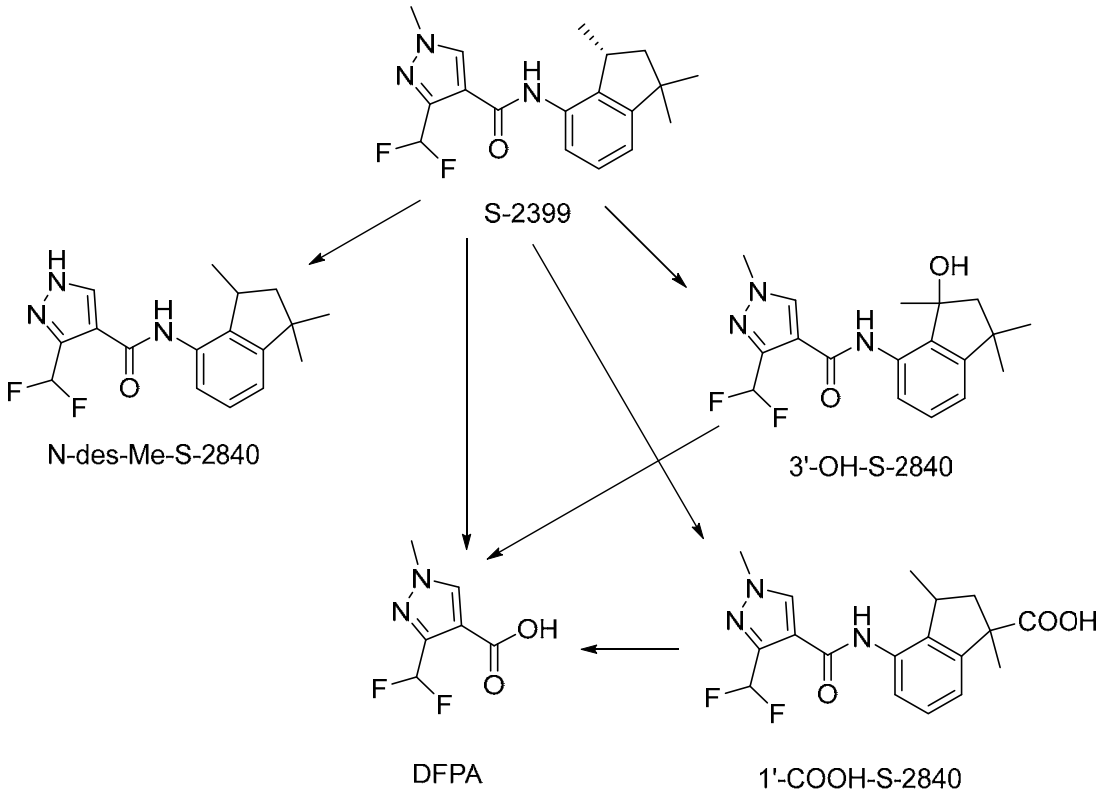


Figure 11 Proposed aerobic aquatic degradation pathways of Inpyrfluxam

The degradation pattern of Inpyrfluxam was similar in all three sediment systems. Inpyrfluxam declined to 75, 84 and 88 percent of the AR (total S-2399 in neutral and acidic extract and water) by the end of the 111 DAT study in the Goose River, Sharkey and Weweantic River sediment systems, respectively with aerobic aquatic half-lives estimated at 319, 563 and 704 days (SFO, PestDF). Degradate formation was primarily to 1'-COOH-S-2840 and 3'-OH-S-2840, formed at maximum levels of 8.1 and 2.9 percent AR respectively for the Goose River system, 7.3 and 2.8 percent AR respectively for the Sharkey system and 5.3 and 2.2 percent AR respectively for the Weweantic River system during the study.

RESIDUE ANALYSIS

Analytical methods

The meeting received analytical methods for the determination of inpyrfluxam and its metabolites in plant and livestock matrices. An overview of the analytical method is presented in Table 89.

Table 89 Overview of the analytical methods submitted for inpyrfluxam and its metabolites

Report ID Method ID	Matrix	Analytes[a]	Extraction	Hydrolysis step	Clean-up	Separation/ Analysis	LOQ	Purpose
RM-50C-1; 201700135	Apple fruit Apple wet pomace, Apple juice Soya bean seed (dry) Soya bean meal, Soya bean	inpyrfluxam 3'-OH-S-2840 1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B DFPA-$CONH_2$ 1'-COOH-S-2840-A 1'-COOH-S-2840-B	acetonitrile/ water (1:1) hexane/ethyl acetate (4:1).	Acid hydrolysis (HCl, 2M)	Strata-X column	HPLC-MS/MS	0.01 mg/kg (crops) 0.02 mg/kg (feed)	Trials in apple (TPR-0019), soya bean dry seeds (TPR-0056), sugar beet roots (TPR-0021), rice grains (TPR-0020), sweet

Report ID Method ID	Matrix	Analytes[a]	Extraction	Hydrolysis step	Clean-up	Separation/ Analysis	LOQ	Purpose
	hulls Soya bean oil Sweet corn Corn and rice grain, flour, grits, meal and oil (dry and wet) corn starch sugar beet roots sugar sugar beet dried pulp molassa Peanut							corn kernels, maize grains, maize forage, maize stover (TPR-0059), peanut nutmeat and peanut hay (TPR-0066), storage stability (TPR-0067), processing on apples (V-38516), dry soya bean seeds (VP-38537), sugar beet roots (V-38533), rice grains (V-38528/ V-38529), peanut nutmeat (TPR-0065), corn seeds (V-15-38939),
RM-50C-1; 201700100	Corn stover	inpyrfluxam 3'-OH-S-2840 1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B DFPA-$CONH_2$	acetonitrile/ water (1:1) hexane/ethyl acetate (4:1).	Acid hydrolysis (HCl, 2M)	Strata-X column	HPLC-MS/MS	0.02 mg/kg	ILV
RM-50C-1; 201600556	Corn forage	inpyrfluxam 3'-OH-S-2840 1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B DFPA-$CONH_2$	acetonitrile/ water (1:1) hexane/ethyl acetate (4:1).	Acid hydrolysis (HCl, 2M)	Strata-X column	HPLC-MS/MS	0.02 mg/kg	ILV
RM-50C-2; 201700099	Apple fruit Soya bean seed Corn grain Corn stover	*N*-des-Me-DFPA	acetonitrile/ water (1:1) hexane/ethyl acetate (8:1).	Acid hydrolysis (HCl, 2M) for 4 hours at 95 °C	Chem Elut SPE	HPLC-MS/MS	0.01 mg/kg	trials for sugar beet (TPR-0023, V-38533)
RM-50C-2a; 201700156	Soya bean seed dry Soya bean meal, Soya bean hulls, oil Peanut Sweet corn, Corn and rice grain, flour, grits, meal and oil (dry and wet) corn starch	*N*-des-Me-DFPA	acetonitrile/ water (1:1) hexane/ethyl acetate (8:1).	Acid hydrolysis for 4 hours at 95 °C	Oasis HLB SPE	HPLC-MS/MS	0.02 mg/kg	trial for soya bean dry seeds (TPR-0056), sweet corn kernels, maize grains, maize forage, maize stover (TPR-0059), peanut nutmeat, peanut hay (TPR-0066), processing on soya beans (VP-38537), .peanuts (V-15-38939) .
SUM-1601V; TPA-0057	Wheat (whole plant) Wheat (grain) Potato Grapes Soya bean	inpyrfluxam 3'-OH-S-2840 DFPA-$CONH_2$ *N*-des-Me-DFPA DFPA 1'-CH_2OH-S-2840-	acetonitrile/ water (1:1)	Acid hydrolysis for 4-6 hours at 100 °C	Oasis HLB SPE	HPLC-MS/MS	0.01 mg/kg for all analytes except 1'-CH_2OH-S-2840-A, 1'-CH_2OH-S-	rotational field study (TPR-0080) and storage stability (TPR-0093) in cucumber,

Report ID Method ID	Matrix	Analytes[a]	Extraction	Hydrolysis step	Clean-up	Separation/ Analysis	LOQ	Purpose
	seeds	A 1'-CH_2OH-S-2840-B 1'-COOH-S-2840-A 1'-COOH-S-2840-B					2840-B, 1'-COOH-S-2840-A and 1'-COOH-S-2840-B (0.005 mg/kg)	grapes, soybean seeds (dry), wheat grain and field bean
SUM-1701V; TPA-0053	Wheat (whole plant) Wheat (grain) Potato Grapes Soya bean seeds	*N*-des-Me-S-2840 *N*-des-Me-1'-CH_2OH-S-2840-A *N*-des-Me-1'-CH_2OH-S-2840-B	acetonitrile/ water (1:1)	No	No	HPLC-MS/MS	0.01 mg/kg for *N*-des-Me-S-2840, 0.005 mg/kg for *N*-des-Me-1'-CH_2OH-S-2840-A and B	rotational crop field study (TPR-0080), storage stability (TPR-0075) in cucumber, grapes, soybean seeds (dry), wheat grain and bean.
JP2015C239 TPR-0024	Apple	inpyrfluxam 3'-OH-S-2840 DFPA-$CONH_2$ 1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B *N*-des-Me-DFPA	Acetonitrile /water (1:1),	Acid hydrolysis for 4-6 hours at 100-105 °C	HLB or graphite carbon or macroporous diatomaceous earth or cation exchange mini-column	HPLC-MS/MS	0.5 mg/kg for all analytes except 1'-CH_2OH-S-2840-A and B (0.25 mg/kg)	trial for apple (JP2015C239) and storage stability studies (TPR-0029)
JP2014C288 TPR-0003	Apple	inpyrfluxam 3'-OH-S-2840 DFPA-$CONH_2$ 1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B *N*-des-Me-DFPA	Acetonitrile /water (1:1),	Acid hydrolysis for 4-6 hours at 100-105 °C	HLB or graphite carbon or macroporous diatomaceous earth or cation exchange mini-column	HPLC-MS/MS	0.01 mg/kg for all analytes except 1'-CH_2OH-S-2840-A and B (0.005 mg/kg)	trial for apple and storage stability studies (TPR-0003)
JP2016C334 TPR-0029	Apple	inpyrfluxam 3'-OH-S-2840 DFPA-$CONH_2$ 1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B *N*-des-Me-DFPA	acetonitrile/water (1:1),	Acid hydrolysis for 4-6 hours at 100-105 °C	HLB or graphite carbon or macroporous diatomaceous earth or cation exchange mini-column	HPLC-MS/MS	0.01 mg/kg for all analytes except 1'-CH_2OH-S-2840-A and B (0.005 mg/kg)	trial for apple and storage stability studies (TPR-0029)
SUM-1601V, SUM-1701V; TPA-0057	Lettuce (w/o roots) Carrot (roots) Carrot (leaves + tops)	inpyrfluxam 3'-OH-S840 DFPA-$CONH_2$ DFPA 1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B 1'-COOH-S-2840-A 1'-COOH-S-2840-B *N*-des-Me-S-2840 *N*-des-Me-1'-CH_2OH-S-2840-A *N*-des-Me-1'-CH_2OH-S-2840-B	acetonitrile/water (1:1),	Acid hydrolysis for 4-6 hours at 100-105 °C	HLB or graphite carbon or macroporous diatomaceous earth or cation exchange mini-column	HPLC-MS/MS	0.01 mg/kg for all except 1'-CH_2OH-S-2840-A, 1'-CH_2OH-S-2840-B, 1'-COOH-S-2840-A, 1'-COOH-S-2840-B, *N*-des-Me-1'-CH_2OH-S-2840-A and *N*-des-Me-1'-CH_2OH-S-2840-B (0.005 mg/kg)	rotational crop field study (TPR-0080) for lettuce (without root), carrot roots and carrot leaves and storage stability study (TPR-0093).
TPR-0015	Egg Hen muscle Hen liver Hen fat	inpyrfluxam 1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B 1'-COOH-S-2840-A 1'-COOH-S-2840-B	hexane/acetone (1:1) and acetone for eggs, acetonitrile/water (1:1) for tissues. hexane/acetone (4:1) for fat	Acid hydrolysis for 4 hours at 100 °C	Oasis HLB SPE	HPLC-MS/MS	0.005 mg/kg (all analytes except inpyrfluxam) 0.01 mg/kg (inpyrfluxam)	livestock feeding studies for poultry.
TPR-0013	Bovine liver,	inpyrfluxam	Acetone and	Acid hydrolysis	Oasis HLB SPE	HPLC-	0.005 mg/kg	livestock feeding

Report ID Method ID	Matrix	Analytes[a]	Extraction	Hydrolysis step	Clean-up	Separation/ Analysis	LOQ	Purpose
	kidney, muscle fat milk	1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B 1'-COOH-S-2840-A 1'-COOH-S-2840-B	acetone/water (1:1) and acetone for milk. acetonitrile/ water (1:1) and acetonitrile for tissues. hexane/ethyl acetate (4:1) and acetone for fat.	for 4 hours at 100 °C		MS/MS	(all analytes except inpyrfluxam) 0.01 mg/kg (inpyrfluxam)	studies for ruminants.
RM-50S (TPA-0028 and TPA-0070)	Soil	Inpyrfluxam, 3'-OH-S-2840, 1'-COOH-S-2840-A and 1'-COOH-S-2840-B	acetone/ water (4:1)	Acid hydrolysis with acetone/0.5M HCl (4:1 v/v)	partitioned into dichloromethane.	HPLC-MS/MS	0.01 mg/kg for all analytes)	Used for the environmental fate studies.
TPA-0043	Soil	Inpyrfluxam, 3'-OH-S-2840, 1'-COOH-S-2840-A and 1'-COOH-S-2840-B	acetone/ water (8:2)	Acid hydrolysis with acetone/0.5M HCl (8:2 v/v)	-	HPLC-MS/MS	0.01 mg/kg for all analytes)	environmental fate studies.
TPA-0027	Wheat grain Cucumber Soybean Grapes	inpyrfluxam	QuEChERS method	No	PSA	HPLC-MS/MS	0.01 mg/kg	Used for enforcement.
TPA-0048	Wheat grain Cucumber Soybean Grapes	inpyrfluxam	QuEChERS method	No	PSA	HPLC-MS/MS	0.01 mg/kg	ILV of TPA-0027
TPA-0049	whole milk Poultry eggs Bovine fat, muscle, liver, blood, urine	inpyrfluxam	QuEChERS method	No	PSA, C18	HPLC-MS/MS	0.01 mg/kg	Used for enforcement
TPA-0061	Bovine fat and liver	inpyrfluxam	QuEChERS method	No	PSA, C18	HPLC-MS/MS	0.01 mg/kg	ILV of TPA-0049

Notes:
[a] Conjugated forms of the metabolites were hydrolysed and detected as inpyrfluxam, 1'-CH_2OH-S-2840-A, 1'- CH_2OH-S-2840-B, 1'-COOH-S-2840-A, or 1'-COOH-S-2840-B.

Plant materials

In apple, corn grain, corn stover, corn forage and soya bean the method RM-50C-1 was used for data generation. The analytical method RM-50C-1 determines residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840-A, 1'-CH_2OH-S-2840-B, DFPA-$CONH_2$, 1'-COOH-S-2840-A and 1'-COOH-S-2840-B.

Residues are extracted from crops using three extractions with acetonitrile/water (1:1). The residues of inpyrfluxam, 3'-OH-S-2840 and 1'-CH_2OH-S-2840-A/-B are partitioned into hexane/ethyl acetate (4:1). The water fraction undergoes purification on a Strata-X column. The sample is split with one portion analysed for DFPA-$CONH_2$ and the other portion for the analysis of conjugates of 1'-COOH-S-2840-A, 1'-COOH-S-2840-B, 1'-CH_2OH-S-2840-A and 1'-CH_2OH-S-2840-B. The DFPA-$CONH_2$ portion is added to half of the hexane/ethyl acetate portion containing the other free compounds, evaporated to dryness, dissolved in methanol/water (1:1) and the residues are quantitated by HPLC-MS/MS. The remaining second fraction from the Strata-X column is hydrolysed with HCl (2 M) to free analytes from the

conjugates, purified on a Strata-X column, evaporated to dryness and dissolved in methanol/water (1:1) before being analysed by HPLC-MS/MS. The method also includes conditions for the optional use of internal standards.

Method RM-50C-1 was validated by fortification with inpyrfluxam, 3'-OH-S-2840, 1'-CH_2OH-S-2840-A, 1'-CH_2OH-S-2840-B, DFPA-$CONH_2$, 1'-COOHS-2840-A and 1'-COOH-S-2840-B at nominal concentrations of 0.01 mg/kg (LOQ) and 0.10 mg/kg to apples, corn grain and soya beans and 0.05 mg/kg for apple aglycone metabolites at nominal concentrations of 0.02 mg/kg (LOQ for livestock feed matrices) and 0.20 mg/kg to corn stover and forage.

For the analysis of metabolite *N*-des-Me-DFPA Method RM-50C-2 was used for data generation. Conjugated forms of the metabolite are cleaved by acid hydrolysis to form the free acid prior to analysis. This method is a supplemental procedure for extracts obtained utilising method RM-50C-1.

In method RM-50C-1, residues are removed from crops using three extractions of acetonitrile/water (1:1). In method RM-50C-2, the organic solvent is removed from the 10 mL extract aliquot and the total volume is returned to 10 mL using HPLC-grade water. The solution is partitioned with hexane/ethyl acetate (8:2). An aliquot of the aqueous layer is pH adjusted with concentrated HCl to create a 4 N acidic solution. The solution undergoes acid hydrolysis for 4 hours at 95 °C. The pH of the cooled solution is adjusted to pH 3 ± 1 using concentrated ammonium hydroxide and loaded onto an unbuffered, Agilent Chem Elut solid phase extraction (1 g, 20 cc) cartridge. *N*-des-Me-DFPA is eluted with ethyl acetate, rotary evaporated to dryness and reconstituted in methanol/water (1:1). An internal standard, *N*-des-Me-DFPA-13C3 was also used. Samples were analysed using HPLC/MS-MS.

Method RM-50C-2 was validated by fortification with *N*-des-Me-DFPA at nominal concentrations of 0.01 mg/kg (LOQ) and 0.10 mg/kg (10x LOQ) to apple, soya bean seed, corn grain and corn stover. The mass transitions employed in this method RM-50C-1 are summarised in Table 90.

Table 90 Mass transitions for analytes determined with the method RM-50C-1

Analyte	Ion Mode	Mass transition (m/z)	Quantification/Confirmation
Inpyrfluxam	ESI^+	334 → 238	Quantification
	ESI^+	334 → 294	Confirmation
Inpyrfluxam-*d3*	ESI^+	337 → 261	
DFPA-$CONH_2$	ESI^+	176 →136	Quantification
	ESI^+	176 → 156	Confirmation
DFPA-$CONH_2$-*d3*	ESI^+	179 → 139	
3'-OH-S-2840	ESI^-	348 →131	Quantification
	ESI^-	348 → 175	Confirmation
3'-OH-S-2840-*d3*	ESI^-	351 → 134	
1'-COOH-S-2840-A and -B	ESI^-	362 → 318	Quantification
	ESI^-	362 → 131	Confirmation
1'-COOH-S-2840-A-*d3* and -B-*d3*	ESI^-	365 → 321	
1'-CH_2OH-S-2840-A and -B	ESI^+	350 → 292	Quantification
	ESI^+	350 → 159	Confirmation
1'-CH_2OH-S-2840-A-*d3* and -B-*d3*	ESI^+	353 → 295	

Notes:
ESI+: Electron spray ionization mode in positive mode. ESI-: electron spray ionization mode in negative mode.

Due to problems identified with the consistency of the Chem Elut SPF cartridges using in method RM-50C-2 the method RM-50C-2a was created in which a Waters Oasis HLB is used instead.

In method RM-50-2a, the organic solvent is removed from a 10 mL (or 20 mL for feed items such as corn stover) extract aliquot and the total volume is adjusted to 10 mL using HPLC-grade water.

Residues are partitioned against hexane/ethyl acetate (8:2) and residues in the aqueous layer are hydrolysed using a 4 N HCl solution for 4 hours at 95 °C. The solution is then cooled, the pH adjusted to 3 ± 1 using concentrated ammonium hydroxide and residues are purified using a solid phase extraction (SPE, Waters Oasis HLB) cartridge conditioned with methanol and water (1:1) and eluted with acetone. Residues are concentrated, reconstituted in methanol/water (1:1) and quantified by HPLC-MS/MS using an internal standard. The method was validated by fortification with inpyrfluxam and *N*-des-Me-DFPA at nominal concentrations of 0.02 mg/kg (LOQ) and 0.10 mg/kg. The mass transitions employed in methods RM-50C-2 and RM-50C-2a are summarised in Table 91.

Table 91 Mass transitions for analytes determined with the method RM-50C-2 and RM-50C-2a

Analyte	Ion Mode	Mass transition (m/z)
N-des-Me-DFPA	ESI^+	161.0 → 141.0
N-des-Me-DFPA-13C3	ESI^+	164.0 → 144.0

Note:
ESI+: Electron spray ionization mode in positive mode

An additional method SUM-1601V (TPA-0057 and TPR-0080) was used for data generation that determines residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840-A, 1'-CH_2OH-S-2840-B, DFPA-$CONH_2$, 1'-COOH-S-2840-A and 1'-COOH-S-2840-B in wheat (whole plant), wheat (grain), potato (tubers), grapes, soya bean (seeds), lettuces (w/o roots), carrot (roots) and carrots (leaves + top).

Samples were homogenised with acetonitrile/water (1:1) and mechanically shaken for 30 minutes. The extracts are filtered through filter paper and Celite (40 g suspended is methanol (80 mL) in a Buchner funnel. The extraction/filtration process is repeated twice more, with the extracts combined and made up with acetonitrile/water (1:1).

For inpyrfluxam, 3'-OH-S-2840 and DFPA-$CONH_2$, an aliquot is transferred to a test tube, evaporated to dryness with nitrogen at 40 °C then reconstituted in water/methanol (3:1 v/v) prior to analysis.

For DFPA, 1'-COOH-S-2840-A and B and 1'-CH_2OH-S-2840-A and B, an aliquot of the combined extract is hydrolysed with 4 M hydrochloric acid for 4 hours at 100 °C, cooled to room temperature and cleaned up using an Oasis HLB cartridge. The analytes are eluted with methanol (10 mL), evaporated to 5 mL under nitrogen at 40 °C and diluted with water to 20 mL prior to analysis.

For *N*-des-Me-DFPA, an aliquot of the combined extract is hydrolysed with 6 M hydrochloric acid for 6 hours at 100 °C and diluted to 15 mL with water. The extract is cleaned up with a Chem Elut cartridge, eluted with ethyl acetate (4 × 25 mL) and evaporated to dryness under vacuum at 40 °C and reconstituted in water/methanol (3:1) with sonication before being made up to 10 mL with water/methanol (3:1). All samples are analysed by HPLC-MS/MS. The mass transitions employed in methods SUM-1601V are summarised in Table 92.

Table 92 Mass transitions for analytes determined with the method SUM-1601V

Analyte	Ion Mode	Mass transition (m/z)	Quantification/Confirmation
inpyrfluxam	ESI^+	334 → 294	Quantification
	ESI^+	334 → 238	Confirmation
DFPA-$CONH_2$	ESI^+	176 → 136	Quantification
	ESI^+	176 → 156	Confirmation
3'-OH-S-2840	ESI^-	348 → 175	Quantification
	ESI^-	348 → 131	Confirmation
N-des-Me-DFPA	ESI^-	161 → 141	Quantification

Analyte	Ion Mode	Mass transition (m/z)	Quantification/Confirmation
	ESI^-	161 → 66	Confirmation
DFPA	ESI^+	177 → 137	Quantification
	ESI^-	175 → 91	Confirmation
	ESI^+	177 → 137	Confirmation
1'-COOH-S-2840-A & B	ESI^+	364 → 278	Quantification
	ESI^+	364 → 318	Confirmation
	ESI^-	362 → 318	Confirmation
1'-CH_2OH-S-2840-A & B	ESI^+	350 → 292	Quantification
	ESI^+	350 → 312	Confirmation
	ESI^+	350 → 262	Confirmation

Notes:
ESI+: Electron spray ionization mode in positive mode. ESI-: electron spray ionization mode in negative mode

The method was validated by fortification with inpyrfluxam, 3'-OH-S-2840, DFPA-$CONH_2$, *N*-des-Me-DFPA and DFPA at nominal concentrations of 0.02 mg/kg (LOQ) and 0.10 mg/kg and with 1'-COOH-S-2840-A&B and 1'-CH_2OH-S-2840-A&B at nominal concentrations of 0.005 mg/kg (LOQ) and 0.10 mg/kg.

For the determination of *N*-des-Me-S-2840 residues in wheat (whole plant), wheat (grain), potato (tubers), grapes, soya bean (seeds), lettuces (w/o roots), carrot (roots) and carrots (leaves + top) the method SUM-1701V was used for data generation.

Samples were homogenised with acetonitrile/water (1:1) and mechanically shaken for 30 minutes. The extracts are filtered through filter paper and Celite (40 g suspended is methanol (80 mL) in a Buchner funnel. The extraction/filtration process is repeated twice more and the extracts combined and made up to 200 mL with acetonitrile/water (1:1). For *N*-des-Me-S-2840, an aliquot is transferred to a test tube, evaporated to dryness with nitrogen at 40 °C then reconstituted in water/methanol (3:1) prior to analysis. Samples are analysed by HPLC-MS/MS. The method was validated by fortification with *N*-des-Me-S-2840 at nominal concentrations of 0.01 mg/kg (LOQ) and 0.10 mg/kg. The mass transitions employed in methods SUM-1701V are summarised in Table 93.

Table 93 Mass transitions for analytes determined with the method SUM-1701V

Analyte	Ion Mode	Mass transition (m/z)	Quantification/Confirmation
N-des-Me-S-2840	ESI^-	318 > 278	Quantification
	ESI^+	320 > 280	Confirmation

Notes:
ESI+: Electron spray ionization mode in positive mode. ESI-: electron spray ionization mode in negative mode

An additional method JP2015C239 was used for the determination of inpyrfluxam, 3'-OH-S-2840, 1'-CH_2OH-S-2840-A, 1'-CH_2OH-S-2840-B, DFPA-$CONH_2$ and *N*-des-Me-DFPA in apples in studies JP2015C239, JP2016C334 and JP2014C288.

In brief, the homogenised samples were extracted with acetonitrile/water (1:1). For the analysis of inpyrfluxam and 3'-OH-S-2840, an aliquot of the extract was purified with a pre-conditioned HLB mini-column and then quantified by HPLC-MS/MS. For DFPA-$CONH_2$ an aliquot was purified with a graphite carbon mini-column and then quantified using HPLC-MS/MS. The metabolites 1'-CH_2OH-S-2840-A and 1'-CH_2OH-S-2840-B were hydrolysed with alkali and enzymes or 4 M hydrochloric acid for 4 hours at 100 °C followed by purification with a pre-conditioned HLB mini-column and then quantified using HPLC-MS/MS. For the extraction of the metabolite *N*-des-Me-DFPA, samples were refluxed with hydrochloric acid (6

hours, 105 °C) followed by purification with a macroporous diatomaceous earth column and cation exchange mini-column and then quantified by using HPLC-MS/MS.

The method was validated by fortification with inpyrfluxam, 3'-OH-S-2840, DFPA-$CONH_2$and 1'-CH_2OH-S-2840 (sum of 1'-CH_2OH-S-2840-A and 1'-CH_2OH-S-2840-B), at nominal concentrations of 0.01 mg/kg (LOQ) and 0.1 mg/kg and 1'-CH_2OH-S-2840-A and 1'-CH_2OH-S-2840-B at nominal concentrations of 0.005 mg/kg and 0.1 mg/kg. The mass transitions employed in method JP2015C239 are summarised in Table 94.

Table 94 Mass transitions for analytes determined with the method JP2015C239

Analyte	Ion Mode	Mass transition (m/z)
Inpyrfluxam	ESI^-	332 → 91
3'-OH-S-2840	ESI^-	348 → 175
DFPA-$CONH_2$	ESI^+	176 → 156
1'-CH_2OH-S-2840-A and -B	ESI^+	350 → 292
N-des-Me-DFPA	ESI^-	161 → 141

Notes:
ESI+: Electron spray ionization mode in positive mode. ESI-: electron spray ionization mode in negative mode.

In all methods, the mean recoveries were within the range of 70–120 percent and relative standard deviations (RSD) were less than 20 percent for all analytes tested. In all cases, the analytical procedures have been successfully validated in terms of specificity, linearity, precision, accuracy, and LOQ. A summary of method validation recovery data is presented in Table 95.

For enforcement, the multi-residue QuEChERS method using liquid chromatography coupled with tandem mass spectrum detection (LC-MS/MS) has been validated, in the study TPA-0027, at the LOQ of 0.01 mg/kg for determining inpyrfluxam in wheat (grain), cucumber, soya bean (seeds) and grapes. An independent laboratory validation was carried out and reported in TPA-0048.

Homogenised samples (5 g wheat grain or soya bean seeds, 10 g cucumber or grapes) were combined with water (*ca.* 10 mL; wheat grain and soya bean seeds only) and the samples were allowed to soak for 20 minutes at room temperature. All the samples were extracted with acetonitrile (*ca.* 10 mL) on a platform shaker for 15 minutes before the addition of magnesium sulfate (4.0 g), sodium chloride (1.0 g), trisodium citrate dihydrate (1.0 g) and disodium hydrogen citrate sesquihydrate (0.5 g). The samples were shaken by hand for approximately 1 minute followed by centrifugation.

For wheat grain and soya bean seed samples, after complete phase separation, aliquots of the upper acetonitrile phase were centrifuged and the supernatant separated. For all matrices, aliquots of acetonitrile phase (1.5 mL) were combined with 40 mg of primary secondary amine and 225 mg of magnesium sulfate. The samples were vortex mixed, shaken by hand for 30 seconds and centrifuged. Aliquots of the extracts were diluted to 10 mL with acetonitrile/0.1 percent formic acid (7:3).

The samples were analysed by HPLC-MS/MS in positive ion mode. The limit of quantification (LOQ) for inpyrfluxam, defined as the lowest fortification level at which acceptable recovery and repeatability data were obtained, was demonstrated to be 0.01 mg/kg. Quantification was performed using external standards. The ion transition m/z 334 → 258 $gmol^{-1}$ was used for quantification and the ion transition m/z 334 → 238 $gmol^{-1}$ was used for confirmation.

Mean recoveries were within the range of 70-120 percent and relative standard deviations (RSD) were less than 20 percent for all analytes tested. In all cases, the analytical procedures have been successfully validated in terms of specificity, linearity, precision, accuracy, and LOQ. A summary of method validation recovery data is presented in Table 95.

Table 95 Summary of method validation recovery data in plant materials

Analyte	N	Spiking level [mg/kg]	Quantification transition			Confirmation Transition			Reference
			Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
Corn Grain									
Inpyrfluxam	5	0.01	76.9-84.6	81.7	3.5	-	-	-	201700135; Bitter, 017
	5	0.1	81.2-90.7	86.0	4.9	-	-	-	
3'-OH-S-2840	5	0.01	70.5-78.4	75.6	4.1	-	-	-	
	5	0.1	81.9-90.5	86.7	4.1	-	-	-	
1'-CH_2OH-S-2840-A	5	0.01	73.8-85.1	82.2	5.8	-	-	-	
	5	0.1	81.8-91.7	85.2	4.4	-	-	-	
1'-CH_2OH-S-2840-B	5	0.01	74.2-84.3	80.6	4.7	-	-	-	
	5	0.1	82.2-87.2	84.6	2.4	-	-	-	
DFPA-$CONH_2$	5	0.01	73.3-86.2	80.1	5.8	-	-	-	
	5	0.1	80.1-86.3	83.6	3.4	-	-	-	
1'-COOH-S-2840-A	5	0.01	77.7-102.5	94.1	10.2	-	-	-	
	5	0.1	87.6-103.6	93.9	6.6	-	-	-	
1'-COOH-S-2840-B	5	0.01	80.3-106.5	96.8	10.3	-	-	-	
	5	0.1	87.5-100.3	92.8	5.9	-	-	-	
1'-CH_2OH-S-2840-A, (aglycone)	5	0.01	85.0-104.4	96.6	9.0				
	5	0.1	92.0-101.6	64.7	4.2				
1'-CH_2OH-S-2840-B (aglycon)	5	0.01	82.0-105.5	97.5	9.8				
	5	0.1	82.7-98.2	89.6	6.3				
N-des-Me-DFPA	5	0.01	82.9-90.6	87.2	3.5	-	-	-	201700099; Foster, 2017
N-des-Me-DFPA	5	0.1	77.2-83.1	76.9	5.3	-	-	-	
Soya bean seed									
Inpyrfluxam	5	0.01	73.9-80.7	78.1	3.7	-	-	-	201700135 (Bitter, 2017)
	5	0.1	74.9-81.8	78.0	3.9	-	-	-	
3'-OH-S-2840	5	0.01	73.4-88.8	82.2	6.9	-	-	-	
	5	0.1	86.2-93.6	90.8	3.8	-	-	-	
1'-CH_2OH-S-2840-A	5	0.01	765-86.7	82.5	4.7	-	-	-	
	5	0.1	85.2-102.6	95.2	7.4	-	-	-	
1'-CH_2OH-S-2840-B	5	0.01	73.7-82.3	79.0	4.4	-	-	-	
	5	0.1	81.4-84.6	82.8	1.6	-	-	-	
DFPA-$CONH_2$	5	0.01	70.1-90.0	76.2	10.4	-	-	-	
	5	0.1	74.8-85.6	79.9	5.3	-	-	-	
1'-COOH-S-2840-A	5	0.01	77.1-103.9	93.8	11.1	-	-	-	
	5	0.1	72.7-99.2	89.3	11.2	-	-	-	
1'-COOH-S-2840-B	5	0.01	91.7-106.4	100	7.5	-	-	-	
	5	0.1	76.3-99.7	90.5	9.5	-	-	-	
1'-CH_2OH-S-2840-A (aglycone)	5	0.01	88.4-106.5	102	7.6				
	5	0.1	72.4-97.3	89.5	11.3				
1'-CH_2OH-S-2840-B (aglycone	5	0.01	78.1-102.7	93.7	10.1				
	5	0.1	73.9-92.5	87.1	8.6				
N-des-Me-DFPA	5	0.01	83.2-94.3	87.8	4.8	-	-	-	201700099; Foster, 2017
	5	0.1	73.3-85.1	79.7	6.7	-	-	-	
Inpyrfluxam	5	0.01	103-114	110	4.1	-	-	-	TPA-0027; Lindner, Grewe, 2016
	5	0.1	101-108	104	3.1	-	-	-	
N-des-Me-DFPA	5	0.02	69.2-78.2	73.6	5.3	-	-	-	VP-38537 RM-50C-2a; Foster, 2017
	5	0.10	82.3-84.7	83.6	1.1	-	-	-	
Inpyrfluxam	5	0.01	93-104	101	4.5	91-105	98	6.3	SUM-1601V Lindner, 2017
	5	0.1	92-100	96	3.8	91-101	96	4.4	

Analyte	N	Spiking level [mg/kg]	Quantification transition			Confirmation Transition			Reference
			Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
DFPA-$CONH_2$	5	0.01	95-108	100	5.3	95-104	99	3.3	
	5	0.1	93-101	98	3.3	95-103	100	4.0	
3'-OH-S-2840	5	0.01	96-105	101	4.9	91-105	97	5.7	
	5	0.1	92-97	94	2.1	92-99	96	3.2	
N-des-Me-DFPA	5	0.01	86-97	92	4.9	93-106	101	5.2	
	5	0.1	84-91	88	3.0	83-89	87	2.9	
DFPA	5	0.01	84-112	102	11.0	82-107	97	10.0	
	5	0.1	81-84	82	1.4	78-84	81	3.7	
1'-CH_2OH-S-2840-A	5	0.005	87-93	90	2.7	79-91	85	6.4	
	5	0.05	80-83	82	1.9	79-83	82	2.0	
1'-CH_2OH-S-2840-B	5	0.005	96-106	101	3.6	96-111	106	6.6	
	5	0.05	81-85	83	1.3	82-85	84	1.6	
1'-COOH-S-2840-A	5	0.005	73-90	85	11.0	66-100	83	15	
	5	0.05	80-83	82	2.0	79-86	81	5.2	
1'-COOH-S-2840-B	5	0.005	74-84	81	5.2	68-87	79	10.0	
	5	0.05	81-85	83	2.7	81-85	83	2.2	
N-des-Me-S-2840	5	0.01	97-114	106	6.0	86-106	93	9.4	SUM-1701V Lindner, Grewe, 2017
	5	0.1	82-98	91	7.0	85-101	92	7.0	
Apples (whole fruit)									
Inpyrfluxam	5	0.01	79.9-96.5	87.3	7.3	-	-	-	201700135; Bitter 2017
	5	0.1	85.0-90.6	88.0	2.3	-	-	-	
3'-OH-S-2840	5	0.01	79.5-91.9	88.0	5.9	-	-	-	
	5	0.1	82.8-89.9	87.6	3.3	-	-	-	
1'-CH_2OH-S-2840-A	5	0.01	74.3-82.6	79.1	4.6	-	-	-	
	5	0.1	78.7-88.6	84.3	5.8	-	-	-	
1'-CH_2OH-S-2840-B	5	0.01	71.2-88.4	79.6	10.2	-	-	-	
	5	0.1	81.0-91.4	85.2	4.5	-	-	-	
DFPA-$CONH_2$	5	0.01	81.5-100.0	87.6	9.2	-	-	-	
	5	0.1	76.7-92.5	85.1	7.6	-	-	-	
1'-COOH-S-2840-A	5	0.01	84.3-89.1	87.0	2.6	-	-	-	
	5	0.05/0.1	85.3-91.2	88.4	3.1	-	-	-	
1'-COOH-S-2840-B	5	0.01	82.5-90.8	86.4	4.0	-	-	-	
	5	0.05/0.1	82.0-90.2	86.1	3.7	-	-	-	
1'-CH_2OH-S-2840-A (aglycone)	5	0.01	83.1-101.1	93.3	8.1				
	5	0.05/0.1	82.9-92.2	86.3	4.3				
1'-CH_2OH-S-2840-B (aglycone)	5	0.01	84.5-95.1	91.3	5.5				
	5	0.05/0.1	86.4-97.4	90.5	4.6				
N-des-Me-DFPA	5	0.01	81.4-87.5	85.1	2.8	-	-	-	201700099; Foster, 2017
	5	0.1	77.0-80.0	78.5	1.4	-	-	-	
Inpyrfluxam	6	0.01	91-98	95.8	2.7	-	-	-	JP2014C288; Takahashi, 2016
	6	0.5	83-94	91.0	4.4	-	-	-	
	6	2.0	87-96	90.3	4.1	-	-	-	
3'-OH-S-2840	6	0.01	92-97	94.3	1.9	-	-	-	
	6	0.5	81-96	92.8	6.3	-	-	-	
DFPA-$CONH_2$	6	0.01	99-99	99.0	0.0	-	-	-	
	6	0.5	96-99	96.8	1.4	-	-	-	
1'-CH_2OH-S-2840-A	6	0.005	90-100	95.5	4.0	-	-	-	
	6	0.25	96-102	98.8	2.3	-	-	-	
1'-CH_2OH-S-2840-B	6	0.005	95-107	102.0	4.4	-	-	-	
	6	0.25	95-99	97.2	2.1	-	-	-	
N-des-Me-DFPA	6	0.01	81-91	85.5	4.4	-	-	-	

Analyte	N	Spiking level [mg/kg]	Quantification transition			Confirmation Transition			Reference
			Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
	6	0.5	86-90	88.5	1.7	-	-	-	
Inpyrfluxam	5	0.01	82-88	85	3	-	-	-	JP2015C239; Takahashi, 2016
	5	0.5	95-103	98	3	-	-	-	
	5	1.0	85-92	89	3	-	-	-	
3'-OH-S-2840	5	0.01	83-92	88	4	-	-	-	
	5	0.5	89-96	93	3	-	-	-	
DFPA-$CONH_2$	5	0.01	93-103	99	4	-	-	-	
	5	0.5	91-104	98	5	-	-	-	
1'-CH_2OH-S-2840-A	5	0.005	66-102	78	18	-	-	-	
	5	0.25	80-86	84	3	-	-	-	
1'-CH_2OH-S-2840-B	5	0.005	74-100	88	12	-	-	-	
	5	0.25	81-88	85	3	-	-	-	
N-des-Me-DFPA	5	0.01	87-96	91	4	-	-	-	
	5	0.5	81-90	85	4	-	-	-	
Apple (edible part									JP2016C334 Takahashi, 2016
Inpyrfluxam	5	0.01	86-96	91	4	-	-	-	
	5	0.5	91-99	95	3	-	-	-	
	5	2.0	91-100	94	4	-	-	-	
3'-OH-S-2840	5	0.01	85-89	87	2	-	-	-	
	5	0.5	90-97	93	3	-	-	-	
DFPA-$CONH_2$	5	0.01	93-108	98	6	-	-	-	
	5	0.5	97-101	99	2	-	-	-	
1'-CH_2OH-S-2840-A	5	0.005	71-84	76	7	-	-	-	
	5	0.25	83-88	86	2	-	-	-	
1'-CH_2OH-S-2840-B	5	0.005	87-101	94	7	-	-	-	
	5	0.25	80-89	86	4	-	-	-	
N-des-Me-DFPA	5	0.01	85-91	88	3	-	-	-	
	5	0.5	82-100	87	8	-	-	-	
Corn stover									
Inpyrfluxam	5	0.02	75.6-80.9	78.6	2.9	-	-	-	201700135 Bitter, 2017
	5	0.2	70.1-73.7	72.1	2.2	-	-	-	
3'-OH-S-2840	5	0.02	71.7-91.2	82.1	10.4	-	-	-	
	5	0.2	73.1-77.1	75.3	2.0	-	-	-	
1'-CH_2OH-S-2840-A	5	0.02	71.3-92.3	79.7	9.6	-	-	-	
	5	0.2	77.4-82.0	79.5	2.1	-	-	-	
1'-CH_2OH-S-2840-B	5	0.02	73.0-78.8	79.6	7.9	-	-	-	
	5	0.2	76.0-78.8	77.9	1.4	-	-	-	
DFPA-$CONH_2$	5	0.02	81.2-95.7	86.7	6.4	-	-	-	
	5	0.2	82.6-92.1	86.9	4.1	-	-	-	
1'-COOH-S-2840-A	5	0.02	86.8-110.0	98.8	8.4	-	-	-	
	5	0.2	88.1-96.4	93.2	3.5	-	-	-	
1'-COOH-S-2840-B	5	0.02	93.9-107.4	102.2	6.1	-	-	-	
	5	0.2	90.7-100.7	93.6	4.5	-	-	-	
1'-CH_2OH-S-2840-A (aglycone)	5	0.02	99.4-117.6	107.8	8.0				
	5	0.2	92.9-101.6	95.8	3.7				
1'-CH_2OH-S-2840-B (aglycone)	5	0.02	88.9-110.6	102.2	8.9				
	5	0.2	87.4-98.5	91.5	4.7				
Inpyrfluxam	5	0.02	88.3-93.8	90.9	2.5	-	-	-	201700100; Powley, 2017
	5	0.2	82.1-97.5	90.6	8.3	-	-	-	
3'-OH-S-2840	5	0.02	90.5-96.5	93.7	2.7	-	-	-	
	5	0.2	79.2-96.0	88.4	8.5	-	-	-	

Analyte	N	Spiking level [mg/kg]	Quantification transition			Confirmation Transition			Reference
			Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
1'-CH_2OH-S-2840-A	5	0.02	89.0-94.0	92.0	2.7	-	-	-	
	5	0.2	86.1-98.5	93.9	5.5	-	-	-	
1'-CH_2OH-S-2840-B	5	0.02	89.5-93.5	91.2	1.8	-	-	-	
	5	0.2	86.6-99.0	93.6	5.9	-	-	-	
DFPA-$CONH_2$	5	0.02	89.3-95.3	92.5	2.6	-	-	-	
	5	0.2	90.1-99.5	95.9	4.1	-	-	-	
N-des-Me-DFPA	5	0.02	77.2-83.1	80.9	2.9	-	-	-	201700099 (Foster, J.; 2017)
N-des-Me-DFPA	5	0.2	75.3-79.7	77.6	2.6	-	-	-	
Corn forage									
Inpyrfluxam	5	0.02	94.5-123.0	106.0	11.6	-	-	-	201600556 (Malayappan, B.; 2017)
	5	0.2	91.0-101.0	95.6	4.1	-	-	-	
3'-OH-S-2840	5	0.02	93.0-122.0	104.0	12.2	-	-	-	
	5	0.2	89.9-99.4	94.8	4.0	-	-	-	
DFPA-$CONH_2$	5	0.02	92.5-126.0	103.0	14.4	-	-	-	
	5	0.2	93.0-102.0	98.6	3.3	-	-	-	
1'-CH_2OH-S-2840-A	5	0.02	93.5-123.0	104	13.1	-	-	-	
	5	0.2	87.0-99.5	93.5	4.9	-	-	-	
1'-CH_2OH-S-2840-B	5	0.02	94.0-123.0	105	12.3	-	-	-	
	5	0.2	87.99.0	93.9	4.6	-	-	-	
Wheat (whole plant)									
Inpyrfluxam	5	0.01	103-112	108	4.0	99-110	104	3.9	SUM-1601V (Lindner, M.; 2017)
	5	0.1	96-98	97	0.9	91-99	96	3.2	
DFPA-$CONH_2$	5	0.01	95-107	101	4.2	91-104	100	5.4	
	5	0.1	89-96	93	2.9	90-95	92	2.4	
3'-OH-S-2840	5	0.01	94-119	104	9.5	93-112	103	6.7	
	5	0.1	90-96	92	2.7	90-99	94	3.6	
N-des-Me-DFPA	5	0.01	72-78	75	2.9	75-88	83	6.0	
	5	0.1	64-76	71	6.6	62-79	71	9.3	
DFPA	5	0.01	103-124	115	7.5	98-109	106	4.2	
	5	0.1	97-108	104	4.3	97-113	104	5.6	
1'-CH_2OH-S-2840-A	5	0.005	99-111	105	5.0	97-117	106	7.0	
	5	0.05	94-101	97	3.1	95-99	97	1.7	
1'-CH_2OH-S-2840-B	5	0.005	89-115	105	9.5	97-111	105	5.6	
	5	0.05	91-99	96	3.6	96-100	97	2.0	
1'-COOH-S-2840-A	5	0.005	97-106	102	3.7	92-121	107	12.0	
	5	0.05	93-99	95	2.4	94-99	97	2.4	
1'-COOH-S-2840-B	5	0.005	100-110	104	3.7	91-110	102	7.4	
	5	0.05	94-96	95	0.7	94-96	95	1.2	
Wheat (grain)									
Inpyrfluxam	5	0.01	90-108	97	7.2	93-104	97	5.0	
	5	0.1	88-102	91	6.6	85-101	91	6.7	
DFPA-$CONH_2$	5	0.01	107-114	110	2.6	104-116	110	4.6	
	5	0.1	99-103	101	1.6	100-104	102	1.5	
3'-OH-S-2840	5	0.01	104-118	109	4.8	105-119	110	4.8	
	5	0.1	98-107	102	3.9	94-104	99	4.4	
N-des-Me-DFPA	5	0.01	87-93	90	2.6	86-94	92	3.8	
	5	0.1	79-83	82	2.1	81-85	83	2.2	
DFPA	5	0.01	87-115	96	11	93-118	106	11	
	5	0.1	78-91	84	6.7	95-107	100	5.7	
1'-CH_2OH-S-2840-A	5	0.005	92-112	102	7.3	102-112	106	4.5	

Analyte	N	Spiking level [mg/kg]	Quantification transition			Confirmation Transition			Reference
			Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
	5	0.05	85-96	89	5.1	86-92	88	2.9	
1'-CH_2OH-S-2840-B	5	0.005	102-111	106	4.0	94-115	102	7.7	
	5	0.05	86-91	90	5.5	82-99	89	7.7	
1'-COOH-S-2840-A	5	0.005	86-101	93	6.1	83-107	92	10	
	5	0.05	81-96	88	6.3	81-97	87	7.5	
1'-COOH-S-2840-B	5	0.005	85-106	93	8.6	90-105	95	6.1	
	5	0.05	81-93	86	5.7	82-94	86	5.6	
N-des-Me-S-2840	5	0.01	74-100	87	12.0	73-94	84	11.0	SUM-1701V Lindner, Grewe, 2017
	5	0.1	67-96	83	14.0	70-91	83	11.0	
Inpyrfluxam	5	0.01	106-111	109	1.9	106-113	108	3.1	TPA-0027; Lindner, Grewe, 2016
	5	0.1	95-109	102	5.1	98-106	101	3.2	
Cucumber									
Inpyrfluxam	5	0.01	99-105	103	2.1	99-106	103	2.8	
	5	0.1	93-104	99	4.1	96-103	100	3.0	
Grapes									
Inpyrfluxam	5	0.01	89-107	99	6.5	90-105	101	6.2	
	5	0.1	88-109	102	8.1	99-106	103	2.8	
N-des-Me-S-2840	5	0.01	93-109	103	6.9	102-118	107	6.1	SUM-1701V Lindner, Grewe, 2017
	5	0.1	83-93	89	4.3	84-97	91	5.7	
Inpyrfluxam	5	0.01	98-108	103	4.5	93-110	101	6.8	SUM-1601V Lindner, 2017
	5	0.1	97-105	101	3.1	96-109	102	5.0	
DFPA-$CONH_2$	5	0.01	104-116	109	4.3	101-113	108	4.1	
	5	0.1	96-115	106	6.5	96-118	105	8.1	
3'-OH-S-2840	5	0.01	103-118	109	5.3	101-121	109	7.4	
	5	0.1	97-105	102	3.2	95-108	100	5.2	
N-des-Me-DFPA	5	0.01	90-98	94	3.5	85-104	83	14	
	5	0.1	76-86	80	5.5	76-87	81	5.9	
DFPA	5	0.01	97-113	104	5.6	98-122	105	12.0	
	5	0.1	99-104	102	2.2	93-110	102	6.6	
1'-CH_2OH-S-2840-A	5	0.005	69-94	85	11.0	82-104	92	9.9	
	5	0.05	78-91	86	6.3	83-92	87	5.1	
1'-CH_2OH-S-2840	5	0.005	79-109	94	12	83-109	93	11	
	5	0.05	83-101	92	7.1	87-103	94	6.3	
1'-COOH-S-2840-A	5	0.005	69-101	82	14	70-11	81	8.2	
	5	0.05	74-88	82	7.5	75-89	82	13.0	
1'-COOH-S-2840-B	5	0.005	63-94	77	14.0	67-95	80	5.6	
	5	0.05	79-90	83	5.7	78-89	84	15	
Potato (tuber)									
Inpyrfluxam	5	0.01	90-113	102	9	92-111	104	7.2	
	5	0.1	90-97	92	3.4	88-99	93	4.4	
DFPA-$CONH_2$	5	0.01	100-115	107	5.4	92-105	98	5.4	
	5	0.1	100-113	106	5.4	92-106	97	6.4	
3'-OH-S-2840	5	0.01	87-114	102	10	90-95	92	2.0	
	5	0.1	98-111	103	3.4	89-96	93	3.4	
N-des-Me-DFPA	5	0.01	58-86	70	15.0	56-89	70	17.0	
	5	0.1	60-84	77	13.0	60-83	77	13.0	
DFPA	5	0.01	108-114	110	2.2	99-105	103	2.3	
	5	0.1	103-118	109	6.5	103-117	110	4.9	
1'-CH_2OH-S-2840-A	5	0.005	88-115	101	9.8	91-107	99	6.1	

Analyte	N	Spiking level [mg/kg]	Quantification transition			Confirmation Transition			Reference
			Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
	5	0.05	92-102	96	4.4	91-100	96	3.4	
1'-CH_2OH-S-2840-B	5	0.005	88-111	103	9.1	97-108	102	4.6	
	5	0.05	90-102	95	4.6	93-104	97	4.7	
1'-COOH-S-2840-A	5	0.005	91-114	98	9.4	94-117	103	8.4	
	5	0.05	85-99	90	6.1	87-97	91	5.1	
1'-COOH-S-2840-B	5	0.005	88-98	94	4.2	87-102	94	6.2	
	5	0.05	86-96	90	4.5	86-96	90	4.5	
N-des-Me-S-2840	5	0.005	82-114	102	12.0	79-112	101	13.0	SUM-1701V Lindner, Grewe, 2017
	5	0.05	88-101	94	6.1	88-103	96	7.0	
Barley (whole plant)									
N-des-Me-S-2840	5	0.01	102-108	105	2.5	99-118	107	6.7	
	5	0.1	91-107	100	6.3	94-105	101	4.3	
Lettuce (w/o roots)									
Inpyrfluxam	3	0.01	94-111	104	8.4	85-110	102	14	TPR-0080 (Bousquet, C.; 2016)
	3	0.1	97-105	102	4.1	95-105	102	5.7	
3'-OH-S-2840	3	0.01	90-110	102	10	88-113	102	12	
	3	0.1	96-103	99	3.8	93-103	99	5.5	
DFPA-$CONH_2$	3	0.01	92-110	101	8.9	96-109	103	6.3	
	3	0.1	95-110	96	15	94-108	102	7.1	
N-des-Me-S-2840	3	0.01	91-107	101	8.8	93-111	102	8.9	
	3	0.1	97-108	104	5.7	88-104	99	9.4	
DFPA	3	0.01	99-101	100	1.0	93-104	97	6.0	
	3	0.1	96-110	104	6.8	94-105	98	6.2	
1'-COOH-S-2840-A	3	0.005	91-105	96	8.4	99-104	102	2.6	
	3	0.05	83-99	92	8.8	81-101	93	11	
1'-COOH-S-2840-B	3	0.005	90-97	94	3.8	98-102	100	2.1	
	3	0.05	82-97	91	8.9	82-108	95	14	
1'-CH_2OH-S-2840-A	3	0.005	87-109	99	11	88-98	93	5.4	
	3	0.05	85-99	94	8.1	81-95	90	8.9	
1'-CH_2OH-S-2840-B	3	0.005	98-104	101	3.0	90-104	98	7.5	
	3	0.05	83-98	93	9.3	82-99	92	9.7	
Carrot (roots)									
inpyrfluxam	3	0.01	87-95	91	4.4	87-105	94	10	
	3	0.1	96-101	98	2.7	97-99	98	1.0	
3'-OH-S-2840	3	0.01	93-100	97	3.9	97-104	99	4.1	
	3	0.1	96-104	100	4.0	98-104	102	3.2	
DFPA-$CONH_2$	3	0.01	76-99	88	13	75-104	92	16	
	3	0.1	91-96	95	3.7	92-100	95	4.4	
N-des-Me-S-2840	3	0.01	97-107	101	5.5	85-95	91	5.7	
	3	0.1	89-100	96	6.3	93-102	98	4.8	
DFPA	3	0.01	94-110	105	8.8	99-114	107	7.1	
	3	0.1	87-98	93	5.9	95-103	100	4.2	
1'-COOH-S-2840-A	3	0.005	100-107	104	3.5	98-102	100	2.1	
	3	0.05	94-110	100	9.0	94-97	95	1.6	
1'-COOH-S-2840-	3	0.005	73-104	90	18	84-96	92	7.5	
	3	0.05	82-106	98	7.5	89-94	92	2.9	
1'-CH_2OH-S-2840-A	3	0.005	102-109	106	3.6	92-108	101	8.0	
	3	0.05	97-99	98	1.2	97-102	100	2.5	
1'-CH_2OH-S-2840-B	3	0.005	98-104	101	3.0	101-108	104	3.7	
	3	0.05	83-98	93	9.3	99-106	102	3.4	

Analyte	N	Spiking level [mg/kg]	Quantification transition			Confirmation Transition			Reference
			Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
Carrot (leaves + top)									
Inpyrfluxam	3	0.01	85-98	92	7.2	91-100	94	3	
	3	0.1	74-105	93	18	72-106	93	20	
3'-OH-S-2840	3	0.01	78-100	92	13	88-110	96	12	
	3	0.1	72-104	93	20	73-108	95	20	
DFPA-$CONH_2$	3	0.01	95-100	98	2.9	92-100	96	4.2	
	3	0.1	77-105	95	17	77-106	96	17	
N-des-Me-S-284	3	0.01	87-99	93	6.5	83-104	93	11	
	3	0.1	70-104	91	20	72-99	90	17	
DFPA	3	0.01	96-109	103	6.4	94-110	104	8.6	
	3	0.1	97-109	102	6.1	93-107	98	7.7	
1'-COOH-S-2840-A	3	0.005	92-110	99	9.5	91-95	92	2.5	
	3	0.05	93-102	97	4.9	89-96	92	3.8	
1'-COOH-S-2840-B	3	0.005	101-108	105	3.4	95-110	101	7.9	
	3	0.05	90-98	94	4.3	93-96	94	1.6	
1'-CH_2OH-S-2840-A	3	0.005	92-105	101	7.5	92-103	97	5.7	
	3	0.05	87-101	96	8.1	94-96	95	1.2	
1'-CH_2OH-S-2840-B	3	0.005	93-109	100	8.2	104-109	107	2.5	
	3	0.05	95-97	96	1.2	97-103	100	3.0	

Animal matrices

The analytical method TPR-0013 analyses inpyrfluxam and its metabolites 1'-CH_2OH-S-2840-A, 1'-CH_2OH-S-2840-B, 1'-COOH-S-2840-A and 1'-COOH-S-2840-B in samples of poultry egg, muscle, liver and fat. Milk/skim/cream samples were extracted with acetone, liver/kidney/muscle samples with acetonitrile/water (1:1) and fat samples were extracted with hexane/acetone (4:1).

The extraction was repeated twice more, firstly for milk/skim/cream/fat samples with acetone and for liver/kidney/muscle samples with acetonitrile/water (1:1). The final extraction was carried out for milk/skim/cream samples with acetone/water (1:1), for liver/kidney/muscle samples with acetonitrile or fat with acetone. The samples were centrifuged after each partition to remove and combine the supernatants. The "initial extract" is then used to prepare two separate final extracts, one for analysis of inpyrfluxam, 1'-COOH-S-2840-A and -B residues and another for analysis of 1'-CH_2OH-S-2840-A and -B residues.

For the determination of residues of inpyrfluxam, 1'-COOH-S-2840-A and -B in all matrices except fat, an aliquot of the "initial extract" was diluted with methanol/water (1:1) then filtered. For fat, an aliquot of the "initial extract" was diluted with hexane and partitioned twice with acetonitrile. The acetonitrile layers were combined and concentrated to near dryness before being reconstituted in methanol/water (1:1) prior to analysis.

For the determination of 1'-CH_2OH-S-2840-A and -B residues in all matrices except fat, an aliquot of the "initial extract" was acidified (1 M HCl) and hydrolysed for 4 hours at 100 °C to release free forms of 1'-CH_2OH-S-2840-A and -B metabolites from potential conjugates. For fat extracts, an aliquot of the "initial extract" is first concentrated to near dryness, then reconstituted with acetonitrile/water (1:1) prior to acidification and hydrolysis as above. For all matrices, the resulting hydrolysed extract is cleaned up using a Waters Oasis HLB solid phase extraction (SPE) cartridge. Samples are analysed by HPLC-MS/MS

in both positive and negative modes. Quantification is performed using external standards. The LOQ was determined to be 0.01 mg/kg for inpyrfluxam and 0.005 mg/kg for metabolites 1'-COOH-S-2840-A/-B and 1'-CH_2OH-S-2840-A/-B. The following mass transitions were employed:

Analyte	Ion Mode	Mass transition (m/z)	Quantification/Confirmation
inpyrfluxam	ESI^+	334 → 238	Quantification
	ESI^+	334 → 258	Confirmation
1'-COOH-S-2840-A and -B	ESI^-	362 → 318	Quantification
	ESI^-	362 → 131	Confirmation
1'-CH_2OH-S-2840-A and -B	ESI^+	350 → 292	Quantification
	ESI^+	350 → 312	Confirmation

Notes:
ESI+: Electron spray ionization mode in positive mode. ESI-: electron spray ionization mode in negative mode.

An additional analytical method (TPR-0015) was used for analysis of inpyrfluxam and its metabolites 1'-CH_2OH-S-2840-A, 1'-CH_2OH-S-2840-B, 1'-COOH-S-2840-A and 1'-COOH-S-2840-B in samples of bovine milk, liver, fat, muscle and kidney for data generation.

Homogenised tissue samples (muscle, liver, fat or egg) were extracted using vigorous mechanical shaking with different extraction solvents. Egg/white/yolk samples were extracted with hexane/acetone (1:1), while liver/muscle samples were extracted with acetonitrile/water (1:1) and fat was extracted with hexane/acetone (4:1). The extractions were repeated a further two times on the pellet with egg/white/yolk/fat samples using acetone and acetonitrile/water (1:1) for muscle/liver samples. The samples were then centrifuged each time to remove and combined supernatants. The "initial extract" was used to prepare two separate final extracts, one for analysis of inpyrfluxam, 1'-COOH-S-2840-A and B residues and another for analysis of 1'-CH_2OH-S-2840-A and B residues.

For the determination of residues of inpyrfluxam, 1'-COOH-S-2840-A and -B in eggs and fat samples, an aliquot of the "initial extract" was diluted with hexane then partitioned twice with acetonitrile. The acetonitrile layers were combined and concentrated to near dryness. The resulting concentrate was reconstituted in methanol/water (1:1) before being further diluted/filtered as necessary and presented for analysis.

For the determination of 1'-CH_2OH-S-2840-A and B residues in eggs and fat samples, an aliquot of the "initial extract" was first concentrated to near dryness, before being reconstituted with acetonitrile/water (1:1), acidified (1 M HCl) and then hydrolysed for 4 hours at 100 °C to release 1'-CH_2OH-S-2840-A and -B metabolites from potential conjugates.

For muscle and liver extracts, an aliquot of the "initial extract" is directly acidified (without concentration), then hydrolysed as above. For all matrices, the resulting hydrolysed extract is purified using a Waters Oasis HLB solid phase extraction (SPE) cartridge. The resulting final extract is presented for analysis.

Samples are analysed by HPLC-MS/MS in both positive and negative ion modes. Quantification is performed using external standards. Analyte identity was confirmed by comparison of the retention time of the analyte with that of a reference standard. The following mass transitions were the same as shown previously.

In both methods, the mean recoveries were within the range of 70–120 percent and relative standard deviations (RSD) were less than 20 percent for all analytes tested in most cases. An exception was observed in the method TPR-0015 for 1'-COOH-S-2840-A at the 0.005 mg/kg level where the mean recovery was marginally outside these levels in egg and muscle matrices. In principle, the analytical

procedures have been successfully validated in terms of specificity, linearity, precision, accuracy, and LOQ. A summary of method validation recovery data for inpyrfluxam is presented in Table 96.

For enforcement, the multi-residue QuEChERS method using LC-MS/MS has been validated, in the study TPA-0049, at the LOQ of 0.01 mg/kg for determining inpyrfluxam in bovine whole milk, poultry eggs, bovine fat, bovine muscle meat and bovine liver. In addition, this method was validated at an LOQ of 0.01 mg/L in bovine blood and urine. An ILV (TPA-0061) of the method TPA-0049 was also provided in which an LOQ for inpyrfluxam was demonstrated to be 0.01 mg/kg in bovine fat and bovine liver.

For bovine whole milk, poultry eggs, bovine muscle, bovine liver, bovine fat (5 g) and bovine blood (5 mL), water (*ca.* 6-10 mL) and acetonitrile (*ca.* 10 mL) were added. For urine, only acetonitrile (*ca.* 10 mL) was added. The samples were shaken for 15 minutes on a platform shaker (with heating in a water bath at 60 °C for bovine fat). The contents of QuEChERS Bekolut Citrate Kit 01 (magnesium sulfate (4.0 g), sodium chloride (1.0 g), trisodium citrate dehydrate (1.0 g) and disodium hydrogen citrate sesquihydrate (0.5 g)) were added and the samples shaken by hand vigorously for 1 minute; all samples were then centrifuged.

An aliquot of the acetonitrile extract (*ca.* 1 mL) was purified using an PSA-KIT-03 (Bekolut) containing PSA (primary secondary amine, 25 mg), C18 (25 mg) and magnesium sulfate (150 mg). The tubes were vortex mixed, shaken by hand for 30 seconds and centrifuged. Aliquots of the final extract were diluted with acetonitrile/0.1 percent formic acid (1:1).

The samples were analysed by LC-MS/MS in positive ion mode. Quantification was performed using external standards. The LOQ was determined to be 0.01 mg/kg for inpyrfluxam and 0.005 mg/kg for metabolites 1'-COOH-S-2840-A and -B and 1'-CH_2OH-S-2840-A and -B. A summary of method validation recovery data is presented in Table 96.

Table 96 Summary of method validation recovery data for inpyrfluxam and metabolites in animal matrices (n=5)

Matrix	Spiking level [mg/kg]	Primary Transition			Confirmation Transition			Reference
		Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
Inpyrfluxam								
Poultry egg	0.01	79-90	87	7.7	81-93	87	5.9	TPR-0015 (Van Middlesworth, 2017)
	0.5	93-98	95	2.7	92-97	94	2.9	
	0.01	91-97	94	2	90-92	91	1	TPA-0049 (Göcer, 2018)
	0.1	90-94	93	2	91-94	92	1	
Poultry muscle	0.01	85-89	86	1.9	86-92	89	2.4	TPR-0015 (VanMiddlesworth, 2017)
	0.5	100-108	102	4.5	96-106	102	4.3	
Poultry liver	0.01	76-80	77	2.1	75-81	78	3.6	
	0.5	70-83	75	7.4	69-82	74	7.3	
Poultry fat	0.01	81-95	87	6.5	76-99	85	10.1	
	0.5	89-105	98	6.0	93-105	100	4.4	
Bovine milk	0.01	76-80	77	2.2	74-80	78	3.2	TPR-0013 (Arnst, Van Middlesworth, 2016)
	0.50	77-85	83	2.9	77-85	82	4.0	
	0.01	84-96	90	6	85-94	90	4	TPA-0049 (Göcer, 2018)
	0.1	88-100	93	5	89-99	93	5	
Bovine muscle	0.01	71-111	85	18.1	63-115	84	22.5	TPR-0013 (Arnst, Van Middlesworth, 2016)
	0.50	101-105	102	1.5	99-103	100	1.7	
	0.01	89-98	93	4	90-97	94	3	TPA-0049 (Göcer, 2018)
	0.1	88-96	94	3	89-97	95	3	
Bovine liver	0.01	78-85	80	3.7	76-88	80	6.0	TPR-0013 (Arnst, Van
	0.50	84-86	85	1.0	83-88	85	2.1	

Matrix	Spiking level [mg/kg]	Primary Transition			Confirmation Transition			Reference
		Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
								Middlesworth, 2016)
	0.01	92-98	95	2	92-98	95	2	TPA-0049
	0.1	100-106	103	2	102-107	104	2	(Göcer, 2018)
	0.01	94-98	96	2	95-99	97	2	TPA-0061
	0.1	88-96	92	4	90-98	95	4	(Schmiedt, 2018)
Bovine kidney	0.01	81-85	82	2.1	81-89	83	4.0	TPR-0013
	0.50	83-94	88	4.5	81-90	86	3.8	(Arnst, Van
Bovine fat	0.01	100-114	105	5.3	93-108	98	6.4	Middlesworth, 2016)
	0.50	100-108	104	2.9	100-112	104	4.6	
	0.01	72-81	77	5	72-79	75	4	TPA-0049
	0.1	83-94	88	5	82-95	88	5	(Göcer, M.; 2018)
	0.01	102-107	104	2	103-110	106	3	TPA-0061
	0.1	97-102	100	2	97-102	100	2	(Schmiedt, S.; 2018)
Bovine blood	0.01	83-96	91	5	84-96	91	5	TPA-0049
	0.1	98-105	102	2	98-106	102	3	(Göcer, M.; 2018)
Bovine urine	0.01	99-103	100	2	98-103	101	2	
	0.1	99-105	101	3	98-105	101	3	
1'-COOH-S-2840A								
Poultry egg	0.005	63-77	68	8.8	63-77	69	9.3	TPR-0015
	0.25	78-80	79	1.1	78-82	80	2.1	(VanMiddlesworth, 2017)
Poultry muscle	0.005	72-80	76	3.9	59-78	69	10.7	
	0.25	82-92	88	4.3	84-92	89	3.7	
Poultry liver	0.005	87-96	91	4.0	78-108	94	12.8	
	0.25	80-94	85	7.0	80-95	86	7.4	
Poultry fat	0.005	62-84	71	13.0	55-89	75	17.5	
	0.25	70-86	80	8.0	72-88	81	7.7	
Bovine milk	0.005	75-80	77	2.5	67-78	71	6.4	TPR-0013
	0.25	84-92	89	3.7	86-91	89	2.6	(Arnst, VanMiddlesworth,
Bovine muscle	0.005	77-85	80	4.4	64-83	74	11.7	2016)
	0.25	105-110	108	1.7	103-109	106	2.2	
Bovine liver	0.005	87-93	90	3.1	76-101	90	12.5	
	0.25	100-102	101	0.7	100-104	102	1.6	
Bovine kidney	0.005	81-86	84	2.5	72-94	84	10.1	
	0.25	92-103	97	4.2	94-100	97	2.2	
Bovine fat	0.005	99-110	103	4.0	73-113	91	17.7	
	0.25	100-110	103	3.8	99-107	101	3.5	
1'-COOH-S-2840-B								
Poultry egg	0.005	73-83	78	4.9	62-98	86	16.5	TPR-0015
	0.25	87-92	90	2.1	87-90	89	1.9	(VanMiddlesworth, 2017)
Poultry muscle	0.005	83-94	88	4.5	80-102	90	9.7	
	0.25	97-107	104	3.8	96-109	103	4.8	
Poultry liver	0.005	86-93	90	3.1	92-99	95	3.1	
	0.25	80-95	86	7.7	82-94	87	5.5	
Poultry fat	0.005	80-107	95	10.8	66-95	83	13.8	
	0.25	83-100	96	7.7	81-97	92	7.0	
Bovine milk	0.005	7685	81	4.2	80-102	87	10.1	TPR-0013
	0.25	82-92	89	4.8	83-92	88	4.1	(Arnst, VanMiddlesworth,
Bovine muscle	0.005	82-88	85	3.3	79-104	91	12.3	2016)
	0.25	107-110	108	1.1	108-112	109	1.5	
Bovine liver	0.005	85-105	93	8.7	87-91	89	2.0	
	0.25	99-102	101	1.1	101-103	102	0.7	
Bovine	0.005	80-92	86	6.2	82-96	90	5.7	

Matrix	Spiking level [mg/kg]	Primary Transition			Confirmation Transition			Reference
		Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
kidney	0.25	93-100	97	2.7	93-101	96	3.1	
Bovine fat	0.005	93-105	100	5.5	80-115	91	15.2	
	0.25	99-105	101	2.5	99-110	103	4.2	
1'-CH_2OH-S-2840-A								
Poultry egg	0.005	70-83	77	7.6	67-79	73	6.2	TPR-0015 (VanMiddlesworth, 2017)
	0.25	67-88	78	9.6	67-87	77	9.3	
Poultry muscle	0.005	73-77	76	2.3	73-80	75	3.7	
	0.25	87-92	89	2.8	86-93	90	3.2	
Poultry liver	0.005	72-100	81	13.8	68-105	85	16.3	
	0.25	65-80	73	8.7	66-78	73	7.4	
Poultry fat	0.005	70-81	74	6.4	73-84	80	5.1	
	0.25	70-87	80	8.7	70-86	80	8.6	
Bovine milk	0.005	73-86	80	6.4	70-83	78	6.1	TPR-0013 (Arnst, VanMiddlesworth, 2016)
	0.25	85-92	90	3.4	85-92	90	3.2	
Bovine muscle	0.005	85-97	91	5.4	85-100	93	6.7	
	0.25	98-102	100	1.6	97-103	100	2.3	
Bovine liver	0.005	79-88	84	4.2	78-89	84	5.8	
	0.25	90-96	93	2.8	91-97	94	2.6	
Bovine kidney	0.005	79-84	82	2.5	79-93	86	6.7	
	0.25	88-98	93	4.3	88-98	93	4.3	
Bovine fat	0.005	71-81	76	5.9	66-79	75	6.9	
	0.25	82-103	92	7.8	82-103	91	8.3	
1'-CH_2OH-S-2840-B								
Poultry egg	0.005	72-78	75	3.6	69-80	75	6.6	TPR-0015 (VanMiddlesworth, 2017)
	0.25	70-89	79	8.7	73-87	79	6.6	
Poultry muscle	0.005	74-81	77	3.5	71-79	76	4.0	
	0.25	88-92	90	2.3	86-92	90	2.9	
Poultry liver	0.005	72-104	85	16.0	70-106	85	16.0	
	0.25	63-77	72	8.4	66-78	72	7.6	
Poultry fat	0.005	73-83	79	5.1	66-75	72	5.1	
	0.25	71-89	82	8.5	70-90	82	9.2	
Bovine milk	0.005	71-85	78	8.5	74-84	79	5.2	TPR-0013 (Arnst, VanMiddlesworth, 2016)
	0.25	83-91	88	3.5	83-91	89	3.8	
Bovine muscle	0.005	82-96	88	6.2	80-97	90	7.6	
	0.25	96-100	97	1.7	96-101	98	2.0	
Bovine liver	0.005	78-83	80	2.4	76-91	84	6.8	
	0.25	88-94	91	2.7	89-94	91	2.4	
Bovine kidney	0.005	75-85	81	4.5	73-82	79	4.3	
	0.25	84-96	90	5.0	84-96	90	5.0	
Bovine fat	0.005	70-80	75	4.8	62-79	71	8.9	
	0.25	82-104	92	8.7	83-105	92	8.7	

Soil

The analytical methods in studies TPA-0028, TPA-0043 and TPA-0070 are being proposed for analysis of inpyrfluxam and and its metabolites (3'-OH-S-2840, 1'-COOH-S-2840-A and 1'-COOH-S-2840-B) in soil for data generation.

In methods RM-50S (TPA-0028 and TPA-0070), soil samples (10.0 g) are weighted and 25 mL acetone/water (4:1) is added. The samples are shaken for 30 minutes and centrifuged for 5 minutes at 2000 rpm. The extraction step is repeated, combining the extracts in graduated cylinders (100 mL). 25 mL

acetone/0.5M HCl (4:1 v/v) is added and the samples shaken for 30 minutes and centrifuged for 5 minutes. The extracts are combined with the first two in the graduated cylinder, 0.5 M sodium acetate solution (2 mL) is added, and the samples mixed. The residues are partitioned into dichloromethane, the solvent evaporated and the residues dissolved in methanol/water (1:1). The samples are analysed by high performance liquid chromatography with tandem mass detection (HPLC-MS/MS) in both positive and negative ion modes using an C_8 column..

In method TPA-0043, soil samples (5.0 g) are weighed and acetone/water (80:20, 12.5 mL) is added. The samples are mechanically shaken for 10 minutes, centrifuged at 4000 rpm for 5 minutes and the supernatant is transferred to a fresh centrifuge tube (50 mL). The extraction process is repeated again and the supernatants combined. Acetone/0.5 M HCl (80:20, 12.5 mL) is added and the samples mechanically shaken for 10 minutes. 5 M sodium hydroxide (1mL) is added and the samples are shaken briefly by hand. The supernatants are filtered through filter paper covered in small amounts of celite and the combined supernatants adjusted to 50 mL with acetone/water (80:20). Aliquots (10 mL) are transferred to Schott bottles (100 mL) and ethyl acetate (32 mL) is added. The samples are agitated for 10 minutes on a horizontal flatbed shaker, before solid NaCl is added and the samples shaken for a further 2 minutes. Following addition of sodium sulphate, the samples are shaken further for 2 minutes. Aliquots of the supernatant (20 mL) are transferred to pear-shaped flasks and evaporated to dryness on a rotary evaporator at 40°C. The dried residues are reconstituted with methanol (0.125 mL) followed by water (0.375 mL) with the assistance of sonication. The samples are analysed by high performance liquid chromatography with tandem mass selective detection (HPLC-MS/MS) in positive and negative ionisation modes, using a C_{18} column.

A summary of the validation data for both methods are presented in Table 97.

Table 97 Summary of method validation recovery data in soil

Analyte	N	Level [mg/kg]	Primary Transition			Confirmation Transition			Reference
			Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
Inpyrfluxam	5	0.01	91.4- 99.2	93.3	3.9	NR	NR	NR	TPA-0028 (method RM-50S)
	5	0.1	101-105	104.2	1.9	NR	NR	NR	
	5	0.01	90.2-102,	96.6	4.6	NR	NR	NR	TPA-0070 (ILV of RM-50S)
	5	0.1	94.0-100	97.2	2.6	NR	NR	NR	
	5	0.002	97-111	102	5	95-114	103	7	TPA-0043 (Trial ATC F6)
	5	0.2	90-101	94	4	92-100	93	4	
	3	0.002	102-108	105	3	107-108	108	1	TPA-0043 (Trial TRI F5)
	3	0.2	88-96	93	4	88-96	93	5	
	3	0.002	87-112	103	13	90-112	104	12	TPA-0043 (Trial AGR F7)
	3	0.2	95-99	97	2	96-97	96	1	
	3	0.002	96-05	100	5	95-104,	99	5	TPA-0043 (Trial BIO F3)
	3	0.2	93- 96	94	2	93-96	94	2	
3'-OH-S-2840	5	0.01	86.5-108	95.4	8.8	NR	NR	NR	TPA-0028 (method RM-50S)
	5	0.1	98.8-106	104.2	4	NR	NR	NR	
	5	0.01	88.0-97.6,	92.0	4.6	NR	NR	NR	TPA-0070 (ILV of RM-50S)
	5	0.1	90.2- 98.6	94.9	3.7	NR	NR	NR	
	5	0.002	82-110	99	13	82-114	100	13	TPA-0043 (Trial ATC F6)
	5	0.2	102-108	104	2	102-108	105	2	
	3	0.002	100-101,	101	1	100	100	0	TPA-0043 (Trial TRI F5)
	3	0.2	87-93	90	3	86-92	91	5	
	3	0.002	82-108	99	15	80-111	99	17	TPA-0043 (Trial AGR F7)
	3	0.2	92-98	95	3	92-98,	95	3	
	3	0.002	101-108,	104	4	103-111	106	4	TPA-0043

Analyte	N	Level [mg/kg]	Primary Transition			Confirmation Transition			Reference
			Range [%]	Mean recovery [%]	RSD [%]	Range [%]	Mean recovery [%]	RSD [%]	
	3	0.2	102-106	103	2	99-102	101	2	(Trial BIO F3)
1'-COOH-S-2840A	5	0.01	71.0-89.1	83.4	8.6	NR	NR	NR	TPA-0028 (method RM-50S)
	5	0.1	77.0-88.4	82.4	5.3	NR	NR	NR	
	5	0.01	81.8-91.8	87.1	4.4	NR	NR	NR	TPA-0070 (ILV of RM-50S)
	5	0.1	86.2- 90.0	88.0	1.6	NR	NR	NR	
	3	0.001	88-92	88	3	81-90	86	5	TPA-0043 (Trial ATC F6)
	3	0.1	78-94	84	7	77-92	83	7	
	3	0.001	107-110	108	1	105-112	107	4	TPA-0043 (Trial TRI F5)
	3	0.1	86-95	90	5	92, 84, 87	88	5	
	3	0.001	90-102	98	7	96, 86, 92	91	6	TPA-0043 (Trial AGR F7)
	3	0.1	95-98	96	2	95, 99, 95	96	2	
	3	0.001	76-82	80	4	73, 81, 70	75	8	TPA-0043 (Trial BIO F3)
	3	0.1	74-77	76	2	76, 77, 78	77	1	
1'-COOH-S-2840B	7	0.01	70.8-93.6	78.5	10.1	NR	NR	NR	TPA-0028 (method RM-50S)
	5	0.1	72.1-77.3	73.8	2.7	NR	NR	NR	
	5	0.01	75.2- 83.4	77.6	4.3	NR	NR	NR	TPA-0070 (ILV of RM-50S)
	5	0.1	68.0-72.8	71.2	2.8	NR	NR	NR	
	5	0.001	89- 94	92	2	87-98	95	5	TPA-0043 (Trial ATC F6)
	5	0.1	85-99	91	6	83-101	90	8	
	3	0.001	100-107	104	3	106-113	109	3	TPA-0043 (Trial TRI F5)
	3	0.1	85-95	89	6	83-94	88	6	
	3	0.001	91-102	98	6	99-101	100	1	TPA-0043 (Trial AGR F7)
	3	0.1	92-97	94	3	94-98	96	2	
	3	0.001	87-88	87	1	83-94	89	6	TPA-0043 (Trial BIO F3)
	3	0.1	81-84	83	2	81-84	83	2	

Note:
NR: Not reported.

Extraction efficiency

A total of 4 studies investigating the extraction efficiency of the multiresidue method QuEChERS, used for enforcement, as presented in the study TPA-0027 for plant commodities and TPR-0013 for animal commodities. A summary of the studies is presented in Table 98.

Table 98 Summary of studies investigating the extraction efficient of the available analytical methods

Method Report no.	Analytes	Substrate	LOQ	Measurement principle	Comment
Plant Commodities					
TPA-0062	Inpyrfluxam	Rice grain Rice straw Soya bean pods Apple	0.002 mg/kg (Rice straw = 0.003 mg/kg)	HPLC-UV HPLC-RAD	Extraction in the metabolism studies (TPM-0013, TPM-0014 and TPM-0015) with the method TPA-0027 (QuEChERS) used for enforcement.
TPA-0030 201700063	Inpyrfluxam 1'-COOH-S-2840-A 1'-COOH-S-2840-B 1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B	Rice straw Radish tops	0.01 mg/kg	HPLC-MS/MS TLC-radio	Extraction in the metabolism studies (2509W and VP-38482) with method RM-50C-1 used for data generation.
Animal Commodities					
TPA-0054; 201700321	Inpyrfluxam 1'-COOH-S-2840-A	Hen muscle, liver,	0.010 mg/kg for inpyrfluxam	HPLC-MS/MS and HPLC-LSC	Extraction in metabolism studies (TPM-0024 and TPM-0025) with

Method Report no.	Analytes	Substrate	LOQ	Measurement principle	Comment
	1'-COOH-S-2840-B 1'-CH_2OH-S-2840-A 1'-CH_2OH-S-2840-B	eggs, fat Goat muscle, liver, milk, fat	0.005 mg/ for metabolites		methods TPR-0013 and TPR-0015 for data generation.
TPA-0063	Inpyrfluxam	Goat fat Goat milk Hen muscle Hen eggs	0.0005 mg/kg	HPLC-UV HPLC-RAD	Extraction in the metabolism studies (2452W and 2453W) with the method TPA-0063 (QuEChERS) used for enforcement.

In study TPA-0062, the extraction efficiency of radioactivity and inpyrfluxam from various crops (rice grain, rice straw, soya bean pods, apple) using a modified QuEChERS analytical method (TPA-0027) was compared to the results from the original analyses performed in the corresponding metabolism studies. The test systems used for the extraction efficiency experiment are rice samples from study TPM-0014, soya bean samples from study TPM-0015 and apples from study TPM-0013.

In the metabolism studies, soya bean pods and apple fruit were rinsed with acetonitrile. Rice grain and rice straw were not rinsed. A portion of rinsed apple fruit was further separated into peel and pulp. Rinsed matrices and unrinsed rice grain and rice straw were processed to a fine consistency in food processors in the presence of dry ice.

As all apples and mature soya bean pods were rinsed during the metabolism studies, rinsed whole apples and mature soya bean pods were used for the QuEChERS analytical method. Rinsed whole apples treated with [phenyl-U-^{14}C]inpyrfluxam were processed in a food processor to a fine consistency in the presence of dry ice. The dry ice was allowed to sublime overnight in a freezer. The rinses and extracts were radio-assayed directly by LSC in triplicate aliquots. The processed samples were combusted to determine the residue levels present.

In the original metabolism studies, aliquots of processed rice grain, rice straw, apple peel, apple fruit pulp (20–50 g) and mature soya bean pods (20–30 g) were extracted using a neutral solvent mixture of acetonitrile and water. Rice grain, rice straw and mature soya bean pod matrices were extracted twice with acetonitrile/water (1:1) and once with acetonitrile. Apple peel and pulp were extracted once with acetonitrile/water (1:1) and once with acetonitrile. For each extraction, the solution was mechanically shaken for *ca.* 30 minutes. The mixture was centrifuged and the supernatant was measured and aliquots were analysed by LSC. No further extractions were performed on apple pulp or peel. Soybean and rice matrices were further extracted with other solvents.

For the QuEChERS extraction method, aliquots of processed rice grain (*ca.* 5 g), rice straw (*ca.* 2 g), mature soya bean pod (*ca.* 2 g) or whole apple (*ca.* 10 g) were used and the amount of moisture in each aliquot was calculated. To rice grain, water was added to result in a total moisture content of 10 mL and acetonitrile (10 mL) was added to give a 1:1 solution. To rice straw (*ca.* 2 g), water was added to result in a total moisture content of 15 mL and acetonitrile (15 mL) was added to give a 1:1 solution. To mature soya bean pods (*ca.* 2 g) water was added to result in a total moisture content of 15 mL and acetonitrile (15 mL) was added to give a 1:1 solution. To whole processed apple, water was added to result in a total moisture content of 10 mL and acetonitrile (10 mL) was added to give a 1:1 solution.

Rice grain, rice straw and soya bean samples were left to soak for 20 minutes at room temperature between the addition of water and acetonitrile. Acetonitrile was added to apple samples

directly after the addition of water. Samples were shaken vigorously for 15 minutes. Samples were centrifuged, the supernatant volume was measured and aliquots were analysed by LSC.

The post-extraction solid residues (PES) from the modified QuEChERS method were analysed by combustion of the dried samples. The TRR for treated plant matrices was determined as the sum of QuEChERS extraction and PES. In the original studies, the TRR was determined by the sum of rinses (if any), acetonitrile/water extracts and PES.

In the original apple metabolism study, fruit peel and pulp were extracted separately. In order to compare the data obtained by modified QuEChERS extraction of whole fruit to the original data, the original peel and pulp TRR were determined by normalizing the weights of peel and pulp to the calculated weight of whole fruit. Analysis of the samples was performed by HPLC-UV. For apple, rice grain and soya bean the LOQ was determined as 0.002 mg/kg, while for rice straw the LOQ was determined to be 0.003 mg/kg.

For rice grain, the original metabolism method extracted somewhat more radioactivity than the QuEChERS method (95.9 and 74.5 percent of TRR, respectively). For rice straw, the multiple acetonitrile/water extractions removed more radioactivity than the modified QuEChERS method (81.4 and 56.1 percent of TRR, respectively). For mature soya bean pod, the combination of the acetonitrile rinse and acetonitrile/water extraction removed more radioactivity than the modified QuEChERS method (58.8 and 44.0 percent of TRR, respectively). For whole apple fruit, multiple acetonitrile/water extractions of peel and peeled fruit removed more radioactivity than the QuEChERS method applied to whole fruit (90.5 and 53.2 percent of TRR, respectively).

HPLC analysis of extracts showed similar profiles for the two methods. inpyrfluxam was the largest component and comparing the results from the original metabolism method to the modified QuEChERS method, inpyrfluxam was present in similar concentrations. The level of inpyrfluxam extracted (mg/kg and/or percent TRR) by the modified QuEChERS method was similar to the level extracted by the original metabolism method. A summary of the percent TRR and distribution of residues (mg/kg eq) is presented in Table 99.

Based on these results it was concluded that the extraction procedure based on the QuEChERS method is suitable for the extraction of a large fraction of the total radioactive residues and determination of residues of inpyrfluxam in foodstuffs of plant origin.

Table 99 Summary percent TRR and distribution of residues (mg/kg eq) between the QuEChERS method (TPA-0027) and the metabolism studies in apples (TPM-0013), rice (TPM-0014) and soybeans (TPM-0015)

TPA-0027 (QuEChERS method)	mg/kg eq	%TRR	Metabolism Method	mg/kg eq	%TRR
Rice Grain-Phenyl. Metabolism method: TPM-0014					
Extraction	0.038	74.5	Extraction	0.047	95.9
PES	0.013	25.5	PES	0.002	4.1
TRR	0.051	100.0	TRR	0.049	100.0
inpyrfluxam	0.037	72.5	inpyrfluxam	0.038	78.6
Rice Straw-Phenyl. Metabolism method: TPM-0014					
Extraction	0.510	56.1	Extraction	0.755	81.4
PES	0.399	43.9	PES	0.172	18.6
TRR	0.909	100.0	TRR	0.927	100.0
inpyrfluxam	0.447	49.2	inpyrfluxam	0.534[b]	57.6
Mature Soybean Pod-Phenyl (rinsed). Metabolism method: TPM-0015					
Extraction	0.404	44.0	Extraction	0.436	58.8
PES	0.514	56.0	PES	0.251	33.8
TRR	0.918	100.0	TRR	0.687	92.6

TPA-0027 (QuEChERS method)	mg/kg eq	%TRR	Metabolism Method	mg/kg eq	%TRR
inpyrfluxam	0.210	22.9	inpyrfluxam	0.170[c]	23.0
Whole Apple-Phenyl (rinsed). Metabolism method: TPM-0013					
Extraction	0.082	53.2	Extractions	0.094	90.4
PES	0.072	46.8	PES	0.010	9.6
TRR	0.154	100.0	TRR	0.104	100.0
inpyrfluxam	0.067	43.5	inpyrfluxam	0.060	57.7

Notes:
[a] TRR determined by sum of fractions.
[b] inpyrfluxam value from the extract stability analysis after 21 months storage.
[c] TRR in mature pods (including rinses) was 0.742 mg/kg. Rinses accounted for 0.055 mg/kg (7.4% TRR), of which 0.046 mg/kg (6.2% TRR) was inpyrfluxam. Extracts of rinsed pods accounted for 0.436 mg/kg (58.8% TRR), of which 0.170 mg/kg (23.0% TRR) was inpyrfluxam.
[d] Apple pulp and peel values were normalized to the weight of whole apple.
%TRR = mg/kg (Extraction or PES or inpyrfluxam) ÷ TRR * 100.

In study TPA-0030 the extraction efficiency of the method RM-50C-1 was evaluated. Rice straw and radish tops were extracted by two separate methods. In the case of rice straw, the 1st method, extraction was performed in the same manner as in the metabolism studies (TPM-0016 and TPM-0047) and the 2nd method extraction was performed in accordance with the residue method RM-50C-1 (201700135).

In the case of mature radish tops in the 1st method, extraction was performed in the same manner as in the confined rotational crop study (TPM-0047) while in the 2nd method, extraction was performed in accordance with the residue method RM-50C-1 (201700135).

Extraction efficiencies of inpyrfluxam and its metabolites in rice straw and radish tops were determined for the residue method RM-50C-1. The residue method extractions of rice straw and radish tops solubilized 69.5 and 88.4 percent TRR, respectively, compared to 64.6 percent and 90.4 percent of TRR in the metabolism method extracts. The metabolism and the residue method showed equivalent extraction efficiency and equivalent hydrolysis effectiveness for generating the aglycones. A summary of the percent TRR and distribution of residues (mg/kg eq) is presented in Table 100.

Table 100 Summary percent TRR and distribution of residues (mg/kg eq) between the RM-50C-1 method (201700135) and the metabolism studies in rice straw (TPM-0016 and TPM-0047) and mature radish tops (TPM-0047)

Rice Straw Metabolism Extract (TPM-0016 and TPM-0047)			Rice Straw Residue Extract of RM-50C-1 method		Difference	
Rice Straw–Pyrazolyl	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extracted Radioactivity	1.013	64.6	1.001	69.5	-0.012	4.9
1'-CH_2OH-S-2840-A (Conj)	0.112	7.1	0.115	8.0	0.003	0.8
1'-CH_2OH-S-2840-B (Conj)	0.232	14.8	0.244	16.9	0.012	2.2
1'-COOH-S-2840-A (Conj)	0.022	1.4	0.027	1.9	0.005	0.5
1'-COOH-S-2840-B (Conj)	0.022	1.4	0.029	2.0	0.006	0.6
Total as Aglycones	0.388	24.7	0.415	28.8		
Radish Tops Metabolism Extract (TPM-0047)			Radish Tops Residue Extract of RM-50C-1 method		Difference	
Radish tops–Pyrazolyl	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extracted Radioactivity	0.321	90.4	0.306	88.4	-0.015	-2.0
1'-CH_2OH-S-2840-A (Conj)	0.010	2.8	0.012	3.3	0.002	0.6
1'-CH_2OH-S-2840-B (Conj)	0.019	5.4	0.023	6.7	0.004	1.3
1'-COOH-S-2840-A (Conj)	0.012	3.5	0.014	3.9	0.001	0.4

Rice Straw Metabolism Extract (TPM-0016 and TPM-0047)			Rice Straw Residue Extract of RM-50C-1 method		Difference	
1'-COOH-S-2840-B (Conj)	0.006	1.8	0.006	1.8	0.000	0.0
Total as Aglycones	0.048	13.5	0.055	15.8		

In study TPA-0054 the extraction efficiency of the method TPR-0013 was investigated. The method was to determine the residues of metabolites in select tissues from TPM-0024 (lactating goat) and TPM-0025 (laying hens) metabolism studies.

Extraction efficiency was comparable between the metabolism study extraction and the residue method extraction based on total extracted residue or Percent TRR extracted. For all tissues, the percent relative recovery (percent TRR extracted using residue method / percent TRR extracted using metabolism method × 100) ranged from 84.2 to 105 percent. The results are shown in Table 101.

Table 101 Summary percent TRR and distribution of residues (mg/kg eq) between the TPR-0013 method and the metabolism studies TPM-0024 (lactating goat) and TPM-0025 (laying hens)

Sample Matrix	Metabolism Studies TPM-0024		Radio-validation (HPLC-LSC) of method TPR-0013		Percent Relative Recovery (%)
	mg/kg eq	% TRR	mg/kg eq	% TRR[c]	
Goat Milk (Day 2[a])	0.057	n/a	0.048	n/a	84.2
Goat Milk (Day 5[a])	0.057	n/a	0.051	n/a	89.5
Goat Muscle	0.014	93.3	0.014	85.7	91.9
Goat Liver	0.311	90.4	0.30	80.0	88.5
Goat Fat[b]	0.0295	90.0	0.031	87.1	96.8
Hen Egg (day 2)	0.018	90.0	0.088	94.3	105
Hen Egg (day 7)	0.018	90.0	0.024	91.7	102
Hen Muscle	0.012	92.3	0.010	93.0	101
Hen Liver	0.299	94.3	0.15	93.3	98.9
Hen Fat	0.091	96.8	0.098	89.1	92.0

Notes:
n/a: Not applicable.
[a] Combined mg/kg residues of skim milk and milk fat.
[b] Calculated values, based on ratios of omental, subcutaneous and renal fats.
[c] Extract mg/kg ÷ (extract mg/kg + combustion mg/kg).

Last, in study TPA-0063 the extraction efficiency of the method TPA-0049 (QuEChERS) for measurement for extractable radioactivity and determination of inpyrfluxam in animal matrices has been evaluated. The samples investigated were obtained from metabolism studies for goats (TPM-0024) and hens (TPM-0025). The test systems used for the extraction efficiency experiment were goat fat, goat liver and goat milk samples from study TPM-0024. Hen egg and hen muscle samples were from study TPM-0025.

Inpyrfluxam was detected in extracts from goat liver, hen muscle and hen egg. The levels of inpyrfluxam extracted (mg/kg and/or percent TRR) by the QuEChERS method was comparable to the levels extracted by the original metabolism method in goat liver, hen muscle and hen egg. Inpyrfluxam was not detected in extracts from goat fat or goat milk.

Based on these results, it was concluded that the extraction procedure based on the QuEChERS method is suitable for the extraction of a large fraction of the total radioactive residues and determination of residues of inpyrfluxam in goat liver, hen muscle and hen egg. These investigations are not conclusive for goat fat or milk as, whilst comparable amounts of the total radioactive residues were extracted by the QuEChERs method, the amount of inpyrfluxam in each matrix was below the detection limit of the

method. A summary of the percent TRR and distribution of residues (mg/kg eq) is presented in Table 102 and Table 103.

Table 102 Summary percent TRR and distribution of residues (mg/kg eq) between the TPR-0049 method and the metabolism studies TPM-0024 (lactating goat)

QuEChERS method (TPA-0049)	mg/kg eq	% TRR	Original Metabolism Method (TPM-0024)	mg/kg eq	% TRR
Goat Liver-Pyrazolyl			Goat Liver-Pyrazolyl		
Extraction	0.221	71.3	Extraction	0.285	91.1
PES	0.089	28.7	PES	0.028	8.9
TRR	0.310	100.0	TRR	0.313	100.0
Inpyrfluxam	0.006	1.9	Inpyrfluxam	0.019	5.94
Composite Goat Fat-Phenyl			Composite Goat Fat-Omental-Phenyl		
Extraction	0.027	77.1	Extraction	0.021	87.5
PES	0.008	22.9	PES	0.003	12.5
TRR	0.035	100.0	TRR	0.024	100.0
Inpyrfluxam	ND	ND	inpyrfluxam	0.004	15.8
Composite Goat Milk-Phenyl			Composite Goat Milk Fat - Phenyl		
Extraction	0.022	95.7	Extraction	0.017	94.4
PES	0.001	4.3	PES	0.001	5.6
TRR	0.023	100.0	TRR	0.018	100.0
Inpyrfluxam	ND	ND	Inpyrfluxam	0.002	9.1[c]
			Goat Fat-Subcutaneous-Phenyl		
			Extraction	0.028	96.6
			PES	0.001	3.4
			TRR	0.029	100.0
			Inpyrfluxam	0.002	6.4[a]
			Goat Fat-Renal-Phenyl	mg/kg	%TRR
			Extraction	0.037	90.2
			PES	0.004	9.8
			TRR	0.041	100.0
			Inpyrfluxam	0.004	8.2[b]

Notes:

ND: Not detected (≤0.001 mg/kg).

[a] %TRR and mg/kg for inpyrfluxam from the initial analysis (TPM-0024; goat metabolism study). Following storage for 14 months at -17 °C, inpyrfluxam accounted for 0.007 mg/kg (2.2 %TRR) which is comparable with the results from the present study based on analysis performed after 45-46 months of frozen storage.

[b] %TRR values were from original metabolism reports.

[c] %TRR for inpyrfluxam in milk fat from original study taken from original data.

Table 103 Summary % TRR and distribution of residues (mg/kg eq) between the TPR-0049 method and the metabolism studies TPM-0025 (laying hens)

QuEChERS method (TPA-0049)	mg/kg eq	% TRR	Original Metabolism Method (TPM-0025)	mg/kg eq	% TRR
Hen Composite Muscle-Pyrazolyl			Hen Breast Muscle-Pyrazolyl		
Extraction	0.009	69.2	Extraction	0.011	91.7
PES	0.004	30.8	PES	0.001	8.3
TRR	0.013	100.0	TRR	0.012	100.0
Inpyrfluxam	0.001	5.3	Inpyrfluxam	≤0.001	2.9
Hen Composite Eggs-Phenyl			Hen Composite Eggs - Phenyl		
Extraction	0.016	72.7	Extraction	0.018	90.0
PES	0.006	27.3	PES	0.002	10.0
TRR	0.022	100.0	TRR	0.020	100.0

QuEChERS method (TPA-0049)	mg/kg eq	% TRR	Original Metabolism Method (TPM-0025)	mg/kg eq	% TRR
Inpyrfluxam	0.006	25.7	Inpyrfluxam	0.002	10.9
			Hen Thigh Muscle-Pyrazolyl		
			Extraction	0.012	92.3
			PES	0.001	7.7
			TRR	0.013	100.0
			Inpyrfluxam	0.001	4.9

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

The Meeting received studies investigating the stability of residues of inpyrfluxam and its metabolites 3'-OH-S-2840, DFPA, DFPA-$CONH_2$, *N*-des-Me-DFPA, *N*-des-Me-S-2840, *N*-des-Me-1'-CH_2OH-S-2840 (determined separately as *N*-des-Me-1'-CH_2OH-S-2840A and B), 1'-COOH-S-2840 (determined separately as 1'-COOH-S-2840A and B) and 1'-CH_2OH-S-2840 (determined separately as 1'-CH_2OH-S-2840A and B) in various matrices (Arndt, 2016; TPR-0013) and soil (TPR-0064; TPR-0088). The maximum storage periods of inpyrfluxam and metabolites per commodity and per commodity groups are summarized in Table 104.

Table 104 Maximum storage stability periods of inpyrfluxam and metabolites per commodity

Group per content	Commodity	inpyrfluxam	3'-OH-S-2840	DFPA	DFPA-CONH2	N-des-Me-DFPA	1'-COOH-S-2840A	1'-COOH-S-2840B
high acid	Grape	679	679	679	679	679	679	679
high oil	Corn oil	115	115	-	115	-	115	115
	Soya bean seed (dry)	683	683	683	683	683	683	683
	Soya bean seed (dry)	594	594	-	514	-	-	-
high protein	Field bean	672	672	672	672	672	672	672
high starch	Corn starch	98	98	-	98	-	98	98
	Maize grain	630	630	-	630	-	616	616
	Polished rice	175	175	-	175	-	-	-
	Potato (starch)	425	425	425	425	425	425	425
	Potato tuber	623	623	-	514	-	610	610
	Wheat (bread)	519	519	519	519	519	519	519
	Wheat (flour)	518	518	518	518	518	518	518
	Wheat grain	679	679	679	679	679	679	679
high water	Apple	514	514	-	514	-	-	-
	Apple	142	142	-	142	227	-	-
	Apple	314	314	-	314	314	-	-
	Apple	327	327	-	327	327	-	-
	Apple	77	77	-	77	77	-	-
	Cucumber	681	681	681	681	681	681	681
	Maize forage	597	597	-	597	-	584	584
	Maize stover	591	591	-	584	-	584	584
no group	Apple pomace	155	155	-	155	-	-	-
	Peanut meal	119	119	-	119	-	119	119
	Potato (crisps)	428	428	428	428	428	428	428
	Potato chips	256	256	-	256	-	256	256
	Rice bran	174	174	-	174	-	-	-
	Rice hulls	175	175	-	175	-	-	-
	Soil	582	582	-	-	-	-	-
	Soil	725	725	-	-	-	-	-
	Soil	724	724	-	-	-	-	-
	Soil	582	582	-	-	-	-	-
	Soil	720	720	-	-	-	-	-

Group per content	Commodity	inpyrfluxam	3'-OH-S-2840	DFPA	DFPA-CONH2	N-des-Me-DFPA	1'-COOH-S-2840A	1'-COOH-S-2840B
	Soil	540	540	-	-	-	-	-

For all matrices, control samples were fortified with an individual standard of each analyte. Fortified samples were stored frozen (at -15 to -22 °C) and residues were analysed using either SUM-1701V or Method RM-50C-1 or JP2015C239. Inpyrfluxam and its metabolites was shown to be stable under frozen storage conditions as presented in Table 105 to Table 111.

Table 105 Storage stability results for inpyrfluxam and its metabolite 3'-OH-S-2840 in various matrices.

Commodity	Storage interval (days)	Inpyrfluxam			3'-OH-S-2840		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Fortification level: 0.1 mg/kg; Report TPR-0093							
Cucumber	0	-	108, 109, 104	107	-	101, 107, 113	107
	38	88	75, 98	87	96	75, 87	81
	86	107	100, 105	103	106	98, 94	96
	197	103	89, 95	92	99	101, 97	99
	399	106	92, 106	99	96	102, 102	102
	554	107	101, 100	101	105	99, 97	98
	681	102	99, 99	99	94	93, 100	97
Grape	0	-	108, 109 (2)	109	-	110, 109, 109	109
	43	108	109, 99	104	108	96, 93	95
	105	100	96, 90	93	99	83, 87	85
	201	106	94, 96	95	105	92, 88	90
	398	109	109, 103	106	105	98, 92	95
	679	94	97, 105	101	95	79, 81	80
Soya bean seed (dry)	0	-	79, 87, 83	83	-	87, 76, 81	81
	28	104	95, 91	93	99	96, 95	96
	98	91	76, 79	78	84	69, 72	71
	145	91	75, 72	74	90	79, 79	79
	385	87	90, 76	83	86	81, 86	84
	567	89	95, 93	94	89	98, 88	93
	683	91	94, 96	95	88	90, 87	89
Wheat grain	0	-	102, 106, 104	104	-	106, 113, 115	111
	42	61[†]	91, 94	93	74	98, 97	98
	85	89	90, 87	89	86	90, 92	91
	196	87	88, 82	85	109	105, 102	104
	395	70	81, 77	79	82	79, 79	79
	549	81	85, 80	83	95	87, 92	90
	679	82	88, 88	88	89	87, 78	83
Field bean	0	-	111, 112, 108	110	-	111, 109, 110	110
	27	84	81, 83	82	79	78, 80	79
	88	107	104, 97	101	106	95, 99	97
	167	98	88, 90	89	96	93, 92	93
	382	94	100, 93	97	97	99, 105	102
	550	88	80, 87	84	86	96, 93	95
	672	104	103, 103	103	102	106, 104	105
Fortification level: 0.1 mg/kg; Report TPR-0067							
Apple	0[a]	-	-	93	-	-	99
	126	76, 100	79, 72	75	87, 102	82	82

Commodity	Storage interval (days)	Inpyrfluxam			3'-OH-S-2840		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
	132 (P)						
	247	100, 105	95, 89	92	112, 113	107, 101	104
	253 (P)						
	371	83, 92	85, 82	83	84, 96	86, 86	86
	377 (P)						
	508	95, 92	93, 88	91	99, 94	91, 91	91
	514 (P)						
Soya bean seed (dry)	0[a]	-	-	87	-	-	93
	126	65, 70	69, 64	67	77, 80	72, 68	70
	132 (P)						
	269	93, 91	91, 95	93	106, 108	105, 120	113
	275 (P)						
	416	82, 87	77, 75	76	91, 92	79, 83	81
	422 (P)						
	434	88, 88	80, 83	81	88, 91	83, 90	87
	440 (P)						
	507	88, 100	80, 85	83	92, 100	83, 91	87
	513 (P)						
	588	94, 94	85, 88	86	97, 95	91, 90	91
	594 (P)						
Potato tuber	0[a]	-	-	91	-	-	98
	128	85, 82	79, 73	76	100, 101	70, 85	77
	135 (D)						
	141 (P)						
	262	95, 91	86, 88	87	101, 95	95, 90	93
	269 (D)						
	275 (P)						
	380	93, 94	84, 90	87	95, 98	88, 91	90
	387 (D)						
	393 (P)						
	500	95, 96	92, 88	90	102, 100	99, 91	95
	507 (D)						
	513 (P)						
	610	91, 91	88, 90	89	95, 94	88, 83	86
	617 (D)						
	623 (P)						
Maize grain	0[a]	-	-	91	-	-	94
	127	81, 87	77, 78	78	96, 98	84, 83	83
	135 (D)						
	141 (P)						
	280	87, 88	76, 83	80	92, 95	95, 98	96
	288 (D)						
	294 (P)						
	342	91, 89	84, 85	85	95, 89	89, 87	88
	350 (D)						
	356 (P)						
	406	91, 94	92, 86	89	91, 95	88, 92	90
	414 (D)						
	420 (P)						
	496	96, 97	90, 86	88	97, 96	91, 96	94

Commodity	Storage interval (days)	Inpyrfluxam			3'-OH-S-2840		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
	504 (D)						
	510 (P)						
	616						
	624 (D)	91, 94	93, 83	88	94, 91	89, 88	89
	630 (P)						
Maize forage	0[a]	-	-	88	-	-	90
	142						
	149 (D)	79, 84	86, 86	86	77, 88	73, 70	71
	155 (P)						
	240						
	247 (D)	90, 90	84, 87	85	92, 94	90, 98	94
	253 (P)						
	329						
	336 (D)	82, 85	81, 78	79	82, 86	81, 84	83
	342 (P)						
	416						
	423 (D)	92, 83	73, 77	75	91, 88	86, 84	85
	429 (P)						
	478						
	485 (D)	97, 96	81, 84	82	99, 97	87, 81	84
	491 (P)						
	584						
	591 (D)	93, 91	87, 83	85	92, 95	91, 89	90
	597 (P)						
Maize stover	0[a]	-	-	86	-	-	92
	142						
	145 (D)	64, 75	65, 66	65	96, 94	80, 78	79
	149 (P)						
	329						
	332 (D)	81, 84	80, 82	81	81, 87	80, 77	79
	336 (P)						
	416						
	419 (D)	83, 81	83, 83	83	91, 91	87, 86	87
	423 (P)						
	498						
	501 (D)	85, 86	86, 87	86	96, 96	89, 83	86
	505 (P)						
	525						
	528 (D)	92, 93	83, 87	85	86, 89	85, 86	86
	532 (P)						
	584						
	587 (D)	105, 100	88, 92	90	99, 100	86, 90	88
	591 (P)						
Fortification level: 0.1 mg/kg, TPR-0101							
Potato (starch)	0	-	102, 107, 109	106	-	107, 102, 110	106
	217	83	86, 79	83	89	97, 96	97
	425	108	100, 99	100	101	105, 101	103
Potato (crisps)	0	-	107, 110, 110	109	-	110, 110, 110	110
	217	82	87, 94	91	97	98, 103	101
	428	108	101, 99	100	104	109, 101	105

Commodity	Storage interval (days)	Inpyrfluxam			3'-OH-S-2840		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Wheat (flour)	0	-	81, 70, 71	74	-	91, 94, 95	93
	298	86	74, 91	83	99	86, 91	89
	518	106	93, 106	100	106	105, 101	103
Wheat (bread)	0	-	105, 107, 110	107	-	101, 99, 104	101
	220	109	100, 101	101	106	102, 102	102
	519	108	98, 96	97	107	99. 102	101
Soya bean hulls	0	83, 86, 89	88, 102	95	87, 93	89, 83	86
	45	63, 73	71, 76	74	76, 88	87, 88	88
	70	100, 97	100, 101	101	97, 90	93, 93	93
Rice hulls	0	86, 79	99, 78	89	86, 82	92, 66	79
	55	102, 99	105, 105	105	113, 117	110, 119	115
	104	98, 100	89, 95	92	92, 94	79, 92	86
	175	98, 100	93, 90	92	95, 95	84, 88	86
Rice bran	0	102	77, 89	83	108	88, 104	96
	70	86, 71	87, 82	85	104, 87	101, 106	104
	114	88, 87	91, 86	89	85, 86	82, 88	85
	174	90, 95	92, 92	92	90, 93	94, 91	93
Polished rice	0	87, 93	81, 93	87	86, 96	87, 89	88
	71	104, 91	95, 99	97	113, 106	101, 117	109
	115	98, 97	95, 94	95	100, 99	97, 95	96
	175	90, 92	92, 98	95	93, 92	94, 89	92
Apple pomace	0	90, 117	70, 92	81	91, 115	105, 76	91
	50	90, 91	87, 91	89	91, 91	96, 97	97
	113	93, 92	93, 88	91	97, 95	87, 86	87
	155	96, 89	78, 87	83	96, 96	93, 89	91
Corn starch	0	-	97	97	-	113, 91	102
	37	78, 86	78, 85	82	82, 87	82, 81	82
	65	91, 93	93, 96	95	82, 89	93, 95	94
	98	91, 91	85, 81	83	85, 84	86, 81	84
Corn oil	0	-	70, 73	72	-	81, 79	80
	49	74, 76	77, 71	74	79, 87	87, 82	85
	79	80, 90	92, 96	94	84, 87	85, 88	87
	115	97, 97	90, 103	97	97, 100	93, 88	91
Peanut meal	0	75, 77	86, 78	82	73, 70	86, 91	89
	44	68, 74	74, 80	77	84	90, 97	94
	80	96, 93	82, 93	88	96, 96	100, 97	99
	119	92, 94	92, 91	92	94, 97	88, 88	88
Wheat germ	0	89, 101	88, 99	94	97	114, 89	102
	97	85, 88	86, 91	89	84, 88	88, 85	87
	222	93, 106	86, 101	94	99, 103	97, 101	99
	313	98, 99	92, 89	91	99, 99	97, 97	97
Sugar beet dried pulp	0	78	80, 80	80	74	94, 82	88
	49	64, 66	60, 45	53	66, 68	59, 52	56
	84	78, 76	81, 75	78	76, 78	84, 64	74
	263	92, 93	89, 81	85	93, 90	78, 84	81
Sugar beet sugar	0	83	95, 89	92	86	90, 81	86
	49	88, 86	88, 93	91	92, 87	92, 96	94
	84	91, 89	89, 89	89	92, 86	96, 96	96
Sugar beet	0	93, 84	76, 67	72	92, 86	80, 81	81

Commodity	Storage interval (days)	Inpyrfluxam			3'-OH-S-2840		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
molasses	50	95, 98	90, 97	94	90, 93	84, 89	87
	85	82, 88	86, 75	81	82, 92	89, 73	81
Potato flakes	0	83, 82	78, 80	79	81, 85	85, 92	89
	119	82, 80	83, 83	83	77, 79	83, 83	83
	179	88, 90	88, 91	90	85, 89	93, 87	90
	253	90, 92	79, 85	82	94, 92	94, 93	94
Potato chips	0	-	86, 92	89	-	96, 97	97
	118	83, 82	88, 88	88	86, 86	86, 87	87
	178	88, 90	88, 87	88	89, 93	92, 94	93
	256	101, 89	86, 90	88	89, 98	95, 91	93
Fortification level: 0.5 mg/kg, Report TPR-0003[c]							
Apple	0	-	91[b]	91	-	93[b]	93
	131 (FS)	95	95, 93	94	99	98, 94	96;
	142 (AS)		95, 92	94		95, 94	94
	227	-	-	-	-	-	-
Fortification level: 0.5 mg/kg, Report TPR-0024[d]							
Apple	0	-	98[b]	98	-	93[b]	93
	308 (A)	91	92, 91	92	94	93, 92	92
	315 (I)		90, 88	89		96, 94	95
	332 (F)		97, 92	94		95, 90	92
	314 (N)		92, 91	92		94, 93	94
Apple pulp	0	-	95[‡]	95	-	93[‡]	93
	321 (A)	97	102, 100	101	96	100, 99	100
	328 (I)		101, 97	99		99, 99	99
	345 (F)		103, 102	102		100, 96	98
	327 (N)		103, 98	100		99, 96	98
Fortification level: 0.5 mg/kg, Report TPR-0029							
Apple	0	-	98[b]	98	-	93[b]	93
	77	86	97, 92, 98, 93	95	88	94, 89, 102, 95	95
Apple pulp	0	-	95[b]	95	-	93[b]	93
	77	93	96, 93, 98, 93	95	95	99, 97, 104, 102	101
Fortification level: 0.1 mg/kg, Report TPR-0064							
Soil (VP-38546)	0	95.2	102.9, 104.7	103.8	87.0	91.2, 93.1	92.2
	180	88.5	97.5, 111.0	104	N/A	-	
	197	91.4	86.3, 85.3	85.8	78.4	76.5, 78.8	77.7
	407	95.1	99.2, 108.3	103.8	98.7	106.4, 107.8	107.1
	585	111.0	117, 119	118	85.9	85.8, 89.0	87.4
Soil (VP-38553)	0	112.0	123, 120	121.5	91.1	82.7, 78.5	80.6
	176	97.7	93.6, 105.1	99.4	73.4	85.7, 84.8	85.3
	196	93.8	107.4, 101.4	104.4	72.0	70.5, 65.5	68
	409	94.1	89.7, 88.8	89.3	86.8	99.4, 87.6	93.5
	582	110.0	121, 120	120.5	98.1	96.6, 95.1	95.9
Soil (VP-38586)	0	96.7	108,6, 100,2	104.4	86.3	79,4, 84,4	81.9
	408	92.8	88,4, 92,5	90.5	92.9	99,3, 97,9	98.6
	582	102.0	103,4, 102,4	102.9	82.1	92,1, 94,5	93.3
	725	76.7	38, 75,8	56.9	84.0	86, 75,6	80.8
Soil (VP-38593)	0	96.2	100,4, 89,9	95.2	74.8	92,8, 89,2	91
	190	90.4	94,2, 110	102.1	69.6	82,4, 77,3	79.9
	406	97.1	96,5, 108,8	102.7	87.1	87, 97,4	92.2

Commodity	Storage interval (days)	Inpyrfluxam			3'-OH-S-2840		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
	582	109.0	112, 109,3	110.7	91.6	104,2, 102,2	103.2
	724	78.9	52, 46	49	89.3	82,9, 78.7	80.8
Soil (VP-38603)	0	108.8	105,1, 104,2	104.7	97.3	85,3, 87,2	86.3
	191	90.5	99,6, 102,7	101.2	79.5	90,4, 95,2	92.8
	405	96.5	101,1, 103,6	102.4	96.9	94,9, 109,9	102.4
	582	99.0	105,1, 117	111.1	93.8	105,9, 104,7	105.3
	727	73.7	74,1, 64,4	69.3	97.6	100,9, 98,9	99.9
Fortification level: 0.02 mg/kg, Report TPR-0088							
Soil (ATC F6)	0	97	95, 94, 98	96	101	90, 96, 97	94
	32	106	106, 101	104	105	97, 96	97
	95	93	81, 79	80	88	79, 76	78
	187	103	90, 89	90	103	89, 90	90
	273	103	91, 94	93	114	111, 111	111
	368	101	87, 88	88	103	91, 91	91
	550	105	92, 94	93	106	97, 97	97
	732	112	109, 115	112	111	98, 98	98

Notes:
P: Parent; D: DFPA
[a] Day 0 recoveries were not determined, the average of the freshly fortified sample recoveries across all intervals are reported as surrogates.
[b] Average of 6 replicates.
[c] The storage stability was investigated using each test site sample (FS: Fukushima-Shoku, AS: Aomori-Shoku test sites). When the storage intervals are different among the test sites, the test site names are presented for clarification.
[d] The storage stability was investigated using each test site sample (A: Aomori, I: Iwate, F: Fukushima and N: Nagano test sites). When the storage intervals are different among the test sites, the test site names are presented for clarification.

Table 106 Storage stability results for metabolites DFPA, DFPA-$CONH_2$ and N-des-Me-DFPA in various matrices

Commodity	Storage interval (days)[c]	DFPA			DFPA-$CONH_2$			*N*-des-Me-DFPA		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Fortification level: 0.1 mg/kg, Report TPR-0093										
Cucumber	0	-	112, 108,109	110	-	109, 115, 117	114	-	66, 77, 94	79
	38	104	102, 103	103	94	79, 91	85	85	89, 89	89
	92 (N)	-	-	-	-	-	-	-	-	-
	86	98	85, 88	87	106	100, 98	99	88	86, 87	87
	197	96	91, 87	89	106	101, 98	100	87	95, 97	96
	159 (D)	-	-	-	-	-	-	-	-	-
	399	97	96, 91	94	94	90, 88	89	80	84, 86	85
	407 (D)	-	-	-	-	-	-	-	-	-
	554	105	92, 90	91	104	95, 98	97	95	91, 95	93
	681	100	93, 95	94	96	99, 89	94	93	91, 88	90
Grape	0	-	117, 109, 108	111	-	111, 109, 108	109	-	91, 87, 85	88
	43	110	96, 95	96	107	104, 105	105	97	72, 80	76
	105	76	80, 79	80	102	100, 99	100	83	82, 75	79

Commodity	Storage interval (days)[c]	DFPA			DFPA-$CONH_2$			*N*-des-Me-DFPA		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
	88 (D, N)	-	-	-	-	-	-	-	-	-
	201	87	78, 65	72	109	105, 107	106	89	83, 86	85
	202 (N)									
	398	100	90, 85	88	100	105, 102	104	80	83, 80	82
	553	86	88, 83	86	-	-	-	-	-	-
	679	86	96, 91	94	94	98, 98	98	88	94, 88	91
Soya bean seed	0	-	87, 86, 87	87	-	89, 88, 83	87	-	79, 74, 70	74
	28	78	71, 71	71	94	88, 88	88	79	83, 80	82
	31 (D)									
	98	76	81, 82	82	90	83, 80	82	71	74, 75	75
	87 (D)									
	145	81	77, 74	76	97	83, 81	82	70	73, 75	74
	182 (D)									
	385	86	94, 92	93	88	84, 87	86	71	83, 74	79
	374 (D)									
	567	87	81, 82	82	92	85, 95	90	75	74, 75	75
	500 (D)									
	683	71	76, 72	74	92	82, 82	82	79	70, 75	73
	633 (D)									
Wheat grain	0	-	94, 103, 115	104	-	109, 106, 105	107	-	81, 82, 77	80
	42	93	90, 92	91	77	90, 111	101	86	93, 99	96
	85	97	80, 93	87	94	102, 94	98	78	77, 81	79
	196	82	76, 77	77	105	103, 103	103	76	82, 84	83
	395	73	80, 72	76	84	73, 90	82	110	108, 84	96
	549	107	75, 74	75	96	99, 96	98	91	97, 82	90
	679	102	84, 88	86	91	95, 87	91	78	71, 75	73
Field bean	0	-	88, 88, 84	87	-	106, 107, 101	105	-	84, 89, 90	88
	27	97	103, 104	104	93	93, 92	93	85	87, 79	83
	88	93	80, 85	83	107	84, 85	85	81	72, 81	77
	167	100	86, 82	84	97	91, 96	94	112	109, 85	97
	382	79	90, 92	91	95	108, 103	106	81	81, 89	85
	550	81	81, 74	78	88	77, 87	82	76	76, 80	78
	672	93	91, 88	90	97	107, 108	108	89	89, 91	90
Fortification level: 0.1 mg/kg, Report TPR-0067										
Apple	0[†]	-	-	-	-	-	98	-	-	-
	126	-	-	-	96, 90	87	87	-	-	-
	132 (P)	-	-	-				-	-	-
	247	-	-	-	94, 90	93, 97	95	-	-	-
	253 (P)	-	-	-				-	-	-
	371	-	-	-	89, 93	92, 97	95	-	-	-
	377 (P)	-	-	-				-	-	-
	508	-	-	-	123, 107	107, 111	109	-	-	-
	514 (P)	-	-	-				-	-	-
Soya bean seed	0[†]	-	-	-	-	-	93	-	-	-
	126	-	-	-	80, 78	81, 76	78	-	-	-
	132 (P)	-	-	-				-	-	-
	269	-	-	-	80, 83	79, 87	83	-	-	-
	275 (P)	-	-	-				-	-	-
	416	-	-	-	80, 78	84, 89	87	-	-	-
	422 (P)	-	-	-				-	-	-

Commodity	Storage interval (days)[c]	DFPA			DFPA-$CONH_2$			*N*-des-Me-DFPA		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
	434	-	-	-	84, 87	93, 90	92	-	-	-
	440 (P)	-	-	-				-	-	-
	507	-	-	-	110, 108	104, 97	101	-	-	-
	513 (P)	-	-	-				-	-	-
	588	-	-	-	119, 115	123, 118	121	-	-	-
	594 (P)	-	-	-				-	-	-
Potato	0†	-	-	-	-	-	96	-	-	-
	128	-	-	-	88, 81	64, 76	70	-	-	-
	135 (D)	-	-	-				-	-	-
	141 (P)	-	-	-				-	-	-
	262	-	-	-	90, 86	88, 90	89	-	-	-
	269 (D)	-	-	-				-	-	-
	275 (P)	-	-	-				-	-	-
	380	-	-	-	87, 94	90, 97	94	-	-	-
	387 (D)	-	-	-				-	-	-
	393 (P)	-	-	-				-	-	-
	500	-	-	-	128, 122	131, 124	128	-	-	-
	507 (D)	-	-	-				-	-	-
	513 (P)	-	-	-				-	-	-
	610	-	-	-	93, 95	99, 90	95	-	-	-
	617 (D)	-	-	-				-	-	-
	623 (P)	-	-	-				-	-	-
Corn grain	0†	-	-	-	-	-	85	-	-	-
	127	-	-	-	82, 80	81, 82	82	-	-	-
	135 (D)	-	-	-				-	-	-
	141 (P)	-	-	-				-	-	-
	280	-	-	-	84, 80	91, 96	93	-	-	-
	288 (D)	-	-	-				-	-	-
	294 (P)	-	-	-				-	-	-
	342	-	-	-	79, 81	89, 88	89	-	-	-
	350 (D)	-	-	-				-	-	-
	356 (P)	-	-	-				-	-	-
	406	-	-	-	84, 90	92, 86	89	-	-	-
	414 (D)	-	-	-				-	-	-
	420 (P)	-	-	-				-	-	-
	496	-	-	-	91, 89	94, 92	93	-	-	-
	504 (D)	-	-	-				-	-	-
	510 (P)	-	-	-				-	-	-
	616	-	-	-	89, 87	92, 89	91	-	-	-
	624 (D)	-	-	-				-	-	-
	630 (P)	-	-	-				-	-	-
Corn forage	0†	-	-	-	-	-	96	-	-	-
	142	-	-	-	87, 96	84, 84	84	-	-	-
	149 (D)	-	-	-				-	-	-
	155 (P)	-	-	-				-	-	-
	240	-	-	-	90, 93	91, 94	93	-	-	-
	247 (D)	-	-	-				-	-	-
	253 (P)	-	-	-				-	-	-
	329	-	-	-	84, 88	89, 87	88	-	-	-
	336 (D)	-	-	-				-	-	-
	342 (P)	-	-	-				-	-	-
	416	-	-	-	92, 86	80, 80	80	-	-	-

Commodity	Storage interval (days)[c]	DFPA			DFPA-$CONH_2$			*N*-des-Me-DFPA		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
	423 (D)	-	-	-				-	-	-
	429 (P)	-	-	-				-	-	-
	478	-	-	-				-	-	-
	485 (D)	-	-	-	99, 92	96, 90	93	-	-	-
	491 (P)	-	-	-				-	-	-
	584	-	-	-				-	-	-
	591 (D)	-	-	-	116, 122	127, 115	121	-	-	-
	597 (P)	-	-	-				-	-	-
Corn stover	0[†]	-	-	-	-	-	100	-	-	-
	142	-	-	-				-	-	-
	145 (D)	-	-	-	98, 95	91, 105	98	-	-	-
	149 (P)	-	-	-				-	-	-
	329	-	-	-				-	-	-
	332 (D)	-	-	-	78, 80	77, 89	83	-	-	-
	336 (P)	-	-	-				-	-	-
	416	-	-	-				-	-	-
	419 (D)	-	-	-	93, 96	95, 98	96	-	-	-
	423 (P)	-	-	-				-	-	-
	498	-	-	-				-	-	-
	501 (D)	-	-	-	126, 116	115, 126	120	-	-	-
	505 (P)	-	-	-				-	-	-
	525	-	-	-				-	-	-
	528 (D)	-	-	-	88, 83	88, 87	88	-	-	-
	532 (P)	-	-	-				-	-	-
	584	-	-	-				-	-	-
	587 (D)	-	-	-	132, 121	123, 131	127	-	-	-
	591 (P)	-	-	-				-	-	-
Fortification level: 0.1 mg/kg, Report TPR-0101										
Potato (starch)	0	-	105, 107, 110	107	-	106, 102, 108	105	-	83, 91, 96	90
	217	98	96, 93	95	79	88, 85	87	70	77, 71	74
	425	110	94, 103	99	108	99, 103	101	86	82, 70	76
Potato (crisps)	0	-	108, 107, 110	108	-	110, 105, 115	110	-	79, 72, 81	77
	217	122[a]	100, 99	100	106	99, 92	96	68	84, 80	82
	428	110	95, 96	96	109	106, 105	106	83	78, 83	81
Wheat (flour)	0	-	92, 92, 93	92	-	98, 108, 90	99	-	70, 71, 72	71
	298	101	90, 95	93	90	85, 95	90	75	64, 73	69
	518	109	97, 94	96	107	104, 106	105	85	78, 78	78
Wheat (bread)	0	-	84, 89, 84	86	-	95, 98, 102	98	-	80, 87, 84	84
	220	108	98, 96	97	107	106, 101	104	70	75, 76	76
	519	99	95, 92	94	108	105, 102	104	100	98, 93	96
Fortification level: 0.1 mg/kg, Report TPR-0065										
Soya bean hulls	0	-	-	-	74, 73, 79	71, 88	81	-	-	-
	45	-	-	-	72, 73	82, 80	81	-	-	-
	70	-	-	-	98	115, 93	104	-	-	-
Rice hulls	0	-	-	-	87, 80	74, 77	76	-	-	-
	55	-	-	-	97, 89	113, 113	113	-	-	-
	104	-	-	-	86, 87	94, 90	92	-	-	-
	175	-	-	-	93, 97	99, 100	100	-	-	-
Rice bran	0	-	-	-	89	72, 100	86	-	-	-
	70	-	-	-	97, 71	88, 100	94	-	-	-

Commodity	Storage interval (days)[c]	DFPA			DFPA-$CONH_2$			*N*-des-Me-DFPA		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
	114	-	-	-	85, 84	92, 86	89	-	-	-
	174	-	-	-	88, 89	98, 98	98	-	-	-
Polished rice	0	-	-	-	87, 86	90, 89	90	-	-	-
	71	-	-	-	83, 89	99, 124	112	-	-	-
		-	-	-				-	-	-
	115	-	-	-	93, 95	94, 98	96	-	-	-
	175	-	-	-	93, 87	94, 96	95	-	-	-
Apple pomace	0	-	-	-	78, 77	91, 91	91	-	-	-
	50	-	-	-	86, 96	102, 100	101	-	-	-
	113	-	-	-	83, 82	90, 104	97	-	-	-
	155	-	-	-	79, 89	100, 94	97	-	-	-
Corn starch	0	-	-	-	-	86, 84	85	-	-	-
	37	-	-	-	84, 85	83, 81	82	-	-	-
	65	-	-	-	73, 86	69, 101	85	-	-	-
	98	-	-	-	84, 86	99, 97	98	-	-	-
Corn oil	0	-	-	-	-	84, 85	85	-	-	-
	49	-	-	-	87, 88	96, 93	95	-	-	-
	79	-	-	-	91, 92	106, 97	102	-	-	-
	115	-	-	-	99, 92	100, 95	98	-	-	-
Peanut meal	0	-	-	-	70, 72	82, 79	81	-	-	-
	44	-	-	-	86, 85	101, 107	104	-	-	-
	80	-	-	-	112, 89	102, 104	103	-	-	-
	119	-	-	-	88, 96	86, 99	93	-	-	-
Wheat germ	0	-	-	-	94	95, 96	96	-	-	-
	97	-	-	-	85, 88	99, 91	95	-	-	-
	222	-	-	-	95, 93	98, 103	101	-	-	-
	313	-	-	-	93, 91	96, 96	96	-	-	-
Sugar beet dried pulp	0	-	-	-	80	94, 89	92	-	-	-
	49	-	-	-	72, 65	53, 58	56	-	-	-
	84	-	-	-	76, 77	88, 89	89	-	-	-
	263	-	-	-	87, 89	86, 91	89	-	-	-
Sugar beet sugar	0	-	-	-	78	89, 88	89	-	-	-
	49	-	-	-	87, 86	102, 87	95	-	-	-
	84	-	-	-	92, 90	108, 105	107	-	-	-
Sugar beet molasses	0	-	-	-	75, 87	96, 80	88	-	-	-
	50	-	-	-	92, 98	96, 104	100	-	-	-
	85	-	-	-	76, 90	84, 88	86	-	-	-
Potato flakes	0	-	-	-	88, 89	83, 89	86	-	-	-
	119	-	-	-	76, 76	98, 93	96	-	-	-
	179	-	-	-	84, 86	91, 94	93	-	-	-
	253	-	-	-	93, 94	103, 97	100	-	-	-
Potato chips	0	-	-	-	-	88, 115	102	-	-	-
	118	-	-	-	84, 87	93, 101	97	-	-	-
	178	-	-	-	86, 86	96, 96	96	-	-	-
	256	-	-	-	88, 88	94, 98	96	-	-	-
Fortification level: 0.5 mg/kg, Report TPR-0003										
Apple	0	-	-	-	-	97[b]	97	-	89[b]	89
	131 (FS)	-	-	-	-	104, 100;	102;	-	-	-
	142 (AS)	-	-	-	-	94, 87	90			
	227	-	-	-	-	-	-	75	83, 83,	82
		-	-	-	-				81, 81	

Commodity	Storage interval (days)[c]	DFPA			DFPA-$CONH_2$			*N*-des-Me-DFPA		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Fortification level: 0.5 mg/kg, Report TPR-0024										
Apple (whole fruit)	0	-	-	-	-	98[b]	98	-	85[b]	85
	308 (A)	-	-	-	-	95, 89	92	84	90, 84	87
	315 (I)	-	-	-	-	102, 95	98		85, 83	84
	332 (F)	-	-	-	-	96, 92	94		85, 80	82
	314 (N)	-	-	-	-	96, 91	94		86, 84	85
Apple (edible portion)	0	-	-	-	-	99[b]	99	-	87[b]	87
	321 (A)	-	-	-	-	107, 104	106	80	87, 84	86
	328 (I)	-	-	-	-	102, 98	100		89, 84	86
	345 (F)	-	-	-	-	102, 101	102		87, 84	86
	327 (N)	-	-	-	-	97, 96	96		86, 84	85
Fortification level: 0.5 mg/kg, Report TPR-0029										
Apple (whole fruit)	0	-	-	-	-	98[b]	98	-	85[b]	85
	77	-	-	-	-	103, 99, 109, 106	104	82	89, 88, 100, 92	92
Apple (edible portion)	0	-	-	-	-	99[b]	99	-	87[b]	87
	77	-	-	-	-	104, 101, 107, 105	104	83	96, 94, 99, 96	96

Notes:
D: DFPA; N: *N*-des-Me-DFPA.
a Considered an outlier since result at 3 month interval fell within the acceptance criteria.
b Average of 6 replicates.
c The storage stability was investigated using each test site sample (A: Aomori, AS: Aomori-Shoku, F: Fukushima, FS: Fukushima-Shoku, I: Iwate, N: Nagano, test sites). When the storage intervals are different among the test sites, the test site names are presented for clarification.

Table 107 Storage stability results for metabolite 1'-COOH-S-2840 (isomers A and B analysed separately) in various matrices

Commodity	Storage interval (days)	1'-COOH-S-2840A			1'-COOH-S-2840B		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Fortification level: 0.05 mg/kg, Report TPR-0093							
Cucumber	0	-	115, 100, 90	102	-	113, 98, 88	100
	38	97	102, 103	103	96	101, 100	101
	86	92	82, 82	82	98	77, 81	79
	197	90	87, 83	85	91	87, 85	86
	407	92	90, 90	90	88	84, 91	88
	554	94	86, 88	87	92	82, 90	86
	681	103	98, 100	99	108	96, 99	98
Grape	0	-	103, 109, 109	107	-	103, 107, 109	106
	43	93	85, 91	88	98	83, 89	86
	88	78	92, 84	88	81	93, 86	90
	201	84	80, 81	81	92	82, 83	83
	398	89	95, 86	91	87	91, 94	93
	553	78	88, 84	86	74	85, 83	84
	679	93	87, 92	90	96	80, 89	85

Commodity	Storage interval (days)	1'-COOH-S-2840A			1'-COOH-S-2840B		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Soya bean seed	0	-	87, 88, 89	88	-	81, 87, 83	84
	28	83	88, 90	89	87	86, 87	87
	98	84	77, 77	77	80	74, 77	76
	145	83	72, 72	72	80	68, 68	68
	385	75	82, 73	78	79	87, 75	81
	567	78	87, 76	82	90	90, 85	88
	683	99	87, 81	84	93	89, 84	87
Wheat grain	0	-	112, 102, 102	105	-	108, 102, 98	103
	42	77	83, 80	82	80	83, 81	82
	85	106	102, 100	101	107	95, 96	96
	196	84	70, 84	77	83	76, 80	78
	395	79	77, 77	77	76	75, 75	75
	549	87	85, 79	82	87	87, 78	83
	679	98	84, 72	78	106	82, 74	78
Field bean	0	-	101, 99, 99	100	-	98, 96, 99	98
	27	94	103, 104	104	97	103, 102	103
	88	91	77, 82	80	92	79, 82	81
	175	91	87, 82	85	94	89, 83	86
	382	77	90, 89	90	79	90, 89	90
	550	76	75, 79	77	75	75, 77	76
	672	94	90, 90	90	97	87, 88	88
Report TPR-0067		Fortification level: 0.129 mg/kg			0.1 mg/kg		
Potato	0	-	-	90	-	-	93
	128	93, 95	83, 87	85	91, 88	77, 81	79
	262	77, 72	78, 73	76	91, 87	81, 73	77
	380	92, 98	98, 107	103	89, 94	84, 91	88
	500	92, 94	95, 90	93	107, 107	99, 95	97
	610	92, 93	78, 73	76	88, 91	76, 73	74
Corn grain	0	-	-	89	-	-	87
	127	90, 85	83, 84	83	85, 84	83, 85	84
	280	85, 88	97, 103	100	81, 83	81, 86	84
	342	96, 87, 80, 81	96, 89	93	94, 83, 79, 81	84, 75	80
	406	88, 94	89, 98	94	85, 95	76, 84	80
	496	97, 93	94, 95	95	92, 88	82, 86	84
	616	90, 85	81, 77	79	91, 89	76, 77	77
Corn forage	0[†]	-	-	81	-	-	88
	142	85, 88	82, 84	83	83, 87	85, 86	85
	240	88, 85	93, 92	92	87, 86	86, 81	84
	329	64, 66	68, 70	69	81, 82	75, 76	76
	416	66, 65	71, 71	71	83, 82	77, 70	73
	478	97, 93	88, 87	87	95, 90	75, 74	74
	584	88, 87	89, 87	88	95, 101	87, 90	89
Corn stover	0	-	-	85	-	-	90
	142	85, 89	85, 87	86	87, 85	87, 82	85
	329	78, 81	83, 87	85	81, 82	74, 78	76
	416	84, 82	90, 90	90	83, 86	81, 80	81
	498	89, 84	85, 83	84	101, 93	84, 81	83
	525	84, 85	79, 81	80	83, 88	76, 73	75
	584	88, 90	91, 81	86	104, 102	89, 89	89
Fortification level: 0.05 mg/kg, Report TPR-0101							

Commodity	Storage interval (days)	1'-COOH-S-2840A			1'-COOH-S-2840B		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Potato (starch)	0	-	94, 94, 95	94	-	93, 93, 95	94
	217	81	79, 77	78	85	81, 79	80
	425	94	81, 76	79	96	88, 82	85
Potato (crisps)	0	-	98, 98, 97	98	-	99, 100, 100	100
	217	87	87, 77	82	89	99, 86	93
	428	99	89, 88	89	99	90, 92	91
Wheat (flour)	0	-	94, 88, 85	89	-	91, 85, 88	88
	298	76	71, 77	74	86	75, 74	75
	518	85	81, 84	83	88	84, 86	85
Wheat (bread)	0	-	95, 97, 95	96	-	99, 101, 97	99
	220	99	94, 81	88	99	99, 90	95
	519	93	87, 85	86	97	89, 86	88
TPR-0065		Fortification level: 0.129 mg/kg			0.1 mg/kg		
Corn starch	0	84, 88	92	92	72, 75	82	82
	37	76, 78	93, 81	82	73, 77	86, 81	84
	65	90, 87	100, 96	98	79, 90	79, 82	81
	98	82, 84	92, 94	93	84, 92	86, 86	86
Corn oil	0	88, 87	96, 93	95	84, 88	89, 91	90
	49	88, 92	102, 99	101	88, 85	85, 85	85
	79	79, 90	98, 98	98	77, 89	83, 85	84
	115	89, 93	100, 84	92	93, 91	84, 75	80
Peanut meal	0	77, 86	81, 86	84	76, 82	79, 82	81
	44	93, 83	90, 91	91	87, 74	78, 84	81
	80	122, 87	105, 101	103	115, 95	95, 88	92
	119	88, 92	96, 98	97	89, 93	88, 86	87
Wheat germ	0	96	91, 98	95	83	83, 86	85
	97	85, 81	92, 90	91	82, 87	81, 79	80
	222	89, 86	93, 92	93	90, 84	82, 79	81
	313	95, 95	94, 100	97	92, 90	81, 87	84
Sugar beet dried pulp	0	87	86, 88	87	78	77, 81	79
	49	79, 68	76, 50	63	73, 69	67, 44	56
	84	73, 74	88, 76	82	73, 76	77, 66	72
	263	87, 86	91, 91	91	86, 86	79, 78	79
Sugar beet sugar	0	101	107, 100	104	85	92, 87	90
	49	93, 90	106, 111	109	95, 84	88, 97	93
	84	91, 90	108, 91	100	91, 88	98, 82	90
Sugar beet molasses	0	77, 97	89, 94	92	80, 93	85, 93	89
	50	93, 95	97, 99	98	93, 100	90, 86	88
	85	90, 93	95, 100	98	90, 95	82, 85	84
Potato flakes	0	94, 96	100, 98	99	99, 99	104, 105	105
	119	75, 74	89, 93	91	75, 74	76, 78	77
	179	79, 82	93, 99	96	84, 88	82, 81	82
	253	81, 86	97, 97	97	84, 88	85, 82	84
Potato chips	0	-	74, 91	83	-	72, 79	76
	118	83, 83	90, 100	95	84, 83	80, 80	80
	178	82, 85	98, 96	97	83, 88	87, 78	83
	256	84, 87	96, 94	95	84, 90	84, 81	83
Fortification level: 0.1 mg/kg, Report TPR-0064							
Soil (VP-38546)	0	98.7	106, 102.4	102.4	90.5	95, 92.1	92.5
	180	77.3	89.3, 89.5	116	-	-	-
	197	90.3	86.1, 92	98.6	81.9	62.8, 61	75.6

Commodity	Storage interval (days)	1'-COOH-S-2840A			1'-COOH-S-2840B		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
	407	107.5	107.4, 104.6	98.6	99.2	85.7, 82.9	85
Soil (VP-38546)	0	104.9	99.7, 95	99.9	91.1	92.7, 78.5	84.1
	176	74.9	92, 90.9	122	73.4	85.7, 84.8	116
	196	87.5	93.5, 91.3	105.6	72	70.5, 65.5	94.4
	409	101.8	106.5, 99.3	99.6	86.8	99.4, 87.6	107.7
	582	95	125, 115	126	98.1	96.6, 95.1	97.7
Soil (VP-38586)	0	99.4	89.4, 94.1	94.3	86.3	79.4, 84.4	83.4
	408	90.1	106.5, 105.6	118	92.9	99.3, 97.9	106.1
	582	107.4	109.9, 109.8	102.3	82.1	92.1, 94.5	114
	725	95.2	88.7, 96.9	97.5	84	86, 75.6	96.2
Soil (VP-38593)	0	90.1	107.8, 113	103.6	98.4	103.6, 107.4	103.1
	190	83.2	95.1, 102.5	119	73.2	72.9, 75.8	101.6
	406	98.9	94.1, 112	104.2	88.2	73, 80.8	87.2
	582	104.1	109.6, 109.5	105.2	99.4	88.2, 88.6	88.9
	724	111	106.3, 79.1	83.5	104.3	81.2, 73.6	74.2
Soil (VP-38603)	0	98.6	98.6, 87.5	85.2	84.6	79.9, 66.2	76.9
	191	N/A	-	N/A	N/A	-	N/A
	405	74.5	74.5, 91.1	126	65	55.1, 68.2	94.8
	582	94.6	94.6, 107.6	111	84.9	79.7, 77.6	92.6
	727	96.7	96.7, 94.9	94.8	76.6	68.7, 76.9	95
Soil	0	104	103, 103, 102	103	106	104, 106, 105	105
	32	102	93, 90	92	98	101, 99	100
	95	100	88, 87	88	98	88, 88	88
	187	98	89, 87	88	98	96, 93	95
	275	116	101, 100	101	100	99, 98	99
	368	103	87, 87	87	100	86, 90	88
	550	105	89, 92	91	105	87, 89	88
	732	104	95, 86	91	102	99 91	95

Table 108 Storage stability results for metabolite 1'-CH_2OH-S-2840 (isomers A and B analysed separately) in various matrices

Commodity	Storage interval (days)	1'-CH_2OH-S-2840A			1'-CH_2OH-S-2840B		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Fortification level: 0.05 mg/kg, Report TPR-0093							
Cucumber	0	-	105, 103, 106	105	-	107, 103, 105	105
	38	98	102, 102	102	96	101, 99	100
	86	101	95, 93	94	102	95, 93	94
	197	86	77, 77	77	87	79, 79	79
	407	100	92, 101	97	95	95, 96	96
	554	92	85, 84	85	94	85, 86	86
	681	94	96, 96	96	95	102, 103	103
Grape	0	-	106, 102, 105	104	-	105, 100, 100	102
	43	101	89, 91	90	97	88, 87	88
	88	82	90, 88	89	80	88, 89	89
	201	74	83, 80	82	85	83, 81	82

Commodity	Storage interval (days)	1'-CH_2OH-S-2840A			1'-CH_2OH-S-2840B		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
	398	95	94, 92	93	96	100, 105	103
	553	89	95, 93	94	92	98, 91	95
	679	86	91, 88	90	89	95, 95	95
Soya bean seed	0	-	80, 77, 80	79	-	79, 72, 79	77
	28	88	89, 89	89	89	92, 92	92
	98	84	80, 79	80	81	79, 77	78
	145	74	66, 67	67	76	66, 66	66
	385	80	82, 83	83	82	84, 83	84
	567	80	72, 71	72	92	78, 72	75
	683	93	78, 81	80	93	77, 81	79
Wheat grain	0	-	113, 109, 110	111	-	112, 108, 112	111
	42	78	84, 78	81	77	84, 77	81
	85	104	104, 111	108	102	100, 112	106
	196	87	77, 75	76	85	78, 79	79
	395	78	80, 90	85	77	89, 88	89
	549	86	78, 79	79	91	82, 87	85
	679	102	78, 84	81	106	78, 86	82
Field bean	0	-	101, 99, 102	101	-	101, 94, 97	97
	27	96	101, 107	104	96	102, 104	103
	88	94	89, 85	87	97	93, 90	92
	175	93	85, 86	86	89	85, 85	85
	382	83	91, 96	94	80	92, 99	96
	550	73	71, 72	72	75	71, 73	72
	672	82	82, 78	80	84	86, 84	85
Fortification level: 0.1 mg/kg, Report TPR-0067							
Apple	0[a]	-	-	95	-	-	95
	126	97, 99	83	83	102, 104	87	87
	247	92, 94	88, 88	88	94, 91	92, 90	91
	371	88, 95	89, 90	90	90, 90	102, 101	102
	508	100, 99	101, 100	100	96, 93	98, 94	96
Soya bean seed	0[a]	-	-	91	-	-	88
	126	87, 90	75, 72	73	87, 83	71, 66	68
	269	91, 88	89, 101	95	85, 87	95, 93	94
	416	87, 87	83, 83	83	87, 88	87, 87	87
	434	84, 86	79, 83	81	87, 87	87, 93	90
	507	94, 100	87, 98	92	87, 95	84, 95	90
	588	96, 100	96, 97	96	90, 89	92, 93	92
Potato	0[a]	-	-	93	-	-	94
	128	89, 84	73, 82	78	95, 92	71, 86	79
	262	94, 90	93, 88	90	97, 90	96, 93	95
	380	93, 94	90, 95	93	91, 92	93, 97	95
	500	103, 103	104, 95	100	98, 99	102, 97	100
	610	90, 91	100, 93	96	91, 93	99, 94	97
Corn grain	0[a]	-	-	91	-	-	91
	127	86, 89	80, 80	80	94, 95	86, 81	84
	280	89, 88	91, 93	92	91, 90	101, 101	101
	342	93, 87	86, 77	81	92, 88	85, 76	81
	406	93, 94	89, 95	92	92, 91	90, 93	91
	496	95, 94	93, 98	95	88, 88	94, 98	96
	616	92, 88	98, 99	99	90, 90	102, 101	102

Commodity	Storage interval (days)	1'-CH_2OH-S-2840A			1'-CH_2OH-S-2840B		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Corn forage	0[a]	-	-	92	-	-	89
	142	82, 84	82, 84	83	82, 85	71, 74	73
	240	93, 94	94, 101	98	89, 91	97, 106	101
	329	84, 82	78, 83	81	81, 85	86, 90	88
	416	92, 85	82, 86	84	93, 83	88, 89	89
	478	101, 98	87, 85	86	98, 93	91, 87	89
	584	97, 98	98, 94	96	93, 90	94, 97	95
Corn stover	0[a]	-	-	95	-	-	93
	142	95, 85	85, 81	83	90, 91	76, 76	76
	329	80, 86	83, 82	82	80, 85	86, 86	86
	416	90, 97	93, 94	94	93, 96	101, 102	101
	498	105, 103	102, 92	97	99, 101	103, 97	100
	525	87, 92	84, 85	85	88, 93	92, 92	92
	584	110, 106	95, 100	98	96, 97	96, 93	94
Fortification level: 0.05 mg/kg, Report TPR-0101							
Potato (starch)	0	-	103, 100, 99	101	-	91, 91, 89	90
	217	90	81, 77	79	86	76, 75	76
	425	95	87, 87	87	95	77, 78	78
Potato (crisps)	0	-	99, 104, 104	102	-	101, 95, 101	99
	217	88	76, 83	80	86	78, 78	78
	428	95	85, 89	87	95	88, 85	87
Wheat (flour)	0	-	87, 83, 92	87	-	80, 86, 84	83
	298	83	81, 94	88	84	81, 88	85
	518	85	88, 89	89	87	92, 90	91
Wheat (bread)	0	-	96, 96, 94	95	-	101, 99, 98	99
	220	103	97, 87	92	102	96, 91	94
	519	99	94, 91	93	95	94, 93	94
Fortification level: 0.1 mg/kg, Report TPR-0065							
Soya bean hulls	0	84, 88	85, 78	82	85, 90	87, 81	84
	45	69, 77	95, 84	90	65, 73	84, 78	81
	70	96, 94	93, 98	96	95, 91	101, 100	101
Rice hulls	0	87, 85	89, 68	79	89, 84	90, 68	79
	55	88, 89	94, 98	96	93, 93	96, 107	102
	104	92, 87	82, 94	88	94, 91	87, 101	94
	175	90, 94	89, 95	92	94, 98	92, 100	96
Rice bran	0	105	94, 106	100	99	88, 99	94
	70	94, 72	86, 98	92	90, 76	84, 90	87
	114	86, 89	89, 93	91	88, 90	95, 100	98
	174	91, 95	90, 95	93	87, 96	97, 96	97
Polished rice	0	87, 94	88, 86	87	90, 93	90, 89	90
	71	94, 91	89, 103	96	88, 96	92, 106	99
	115	99, 94	98, 97	98	99, 97	106, 102	104
	175	89, 88	97, 93	95	91, 88	95, 96	96
Apple pomace	0	85, 114	100, 75	88	94, 115	104, 78	91
	50	90, 91	99, 98	99	88, 91	97, 105	101
	113	95, 92	93, 90	92	97, 97	100, 96	98
	155	94, 88	95, 85	90	98, 92	101, 93	97
Corn starch	0	-	88, 93	91	-	90, 98	94
	37	83, 85	83, 81	82	81, 87	78, 78	78
	65	91, 93	94, 98	96	90, 88	100, 99	100

Commodity	Storage interval (days)	1'-CH_2OH-S-2840A			1'-CH_2OH-S-2840B		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
	98	88, 88	91, 93	92	91, 89	98, 97	98
Corn oil	0	-	110, 105	108	-	91, 91	91
	49	91, 93	98, 93	96	92, 92	106, 95	101
	79	84, 89	95, 94	95	83, 87	97, 94	96
	115	94, 101	99, 93	96	96, 100	102, 94	98
Peanut meal	0	71, 76	80, 84	82	79, 77	80, 85	83
	44	88, 92	94, 93	94	88, 95	102, 102	102
	80	92, 91	100, 95	98	96, 93	107, 101	104
	119	95, 100	91, 91	91	90, 95	97, 94	96
Wheat germ	0	97	111, 91	104	102	118, 95	107
	97	88, 92	91, 87	89	88, 93	97, 92	95
	222	96, 98	97, 100	99	94, 101	100, 104	102
	313	96, 97	99, 95	97	98, 101	101, 97	99
Sugar beet dried pulp	0	73	87, 79	83	127	98, 100	99
	49	66, 66	68, 58	63	71, 68	78, 63	71
	84	77, 82	94, 70	82	82, 79	91, 74	83
	263	92, 91	82, 89	86	90, 90	84, 92	88
Sugar beet sugar	0	83	87, 82	85	96	98, 89	94
	49	88, 85	98, 97	98	88, 89	105, 109	107
	84	97, 90	95, 99	97	95, 89	105, 104	105
Sugar beet molasses	0	87, 86	78, 80	79	91, 88	81, 82	82
	50	93, 94	95, 98	97	97, 98	99, 106	103
	85	92, 87	98, 83	91	91, 93	104, 93	99
Potato flakes	0	80, 86	87, 86	87	84, 90	88, 89	89
	119	79, 81	88, 89	89	83, 84	94, 99	97
	179	87, 85	91, 86	89	87, 87	102, 93	98
	253	94, 96	99, 99	99	95, 95	104, 100	102
Potato chips	0	-	92, 104	98	-	94, 106	100
	118	86, 87	93, 90	92	87, 89	96, 97	97
	178	85, 92	94, 95	95	86, 91	104, 103	104
	256	84, 88	86, 86	86	97, 94	95, 94	95
Fortification level: 0.5 mg/kg, Report TPR-0003							
Apple	0	-	99‡	99	-	97‡	97
	131 (AS)	97	97, 96;	96;	97	99, 96;	98;
	142 (FS)		96, 93	94		93, 93	93
Fortification level: 0.5 mg/kg, Report TPR-0024							
Apple (whole fruit)	0	-	84‡	84	-	85‡	85
	447 (A)	79	73, 70	72	85	82, 81	82
	454 (I)		74, 72	73		81, 76	78
	471 (F)		74, 71	72		78, 78	78
	453 (N)		74, 72	73		83, 80	82
Apple (edible portion)	0	-	86‡	86	-	86‡	86
	447 (A)	75	72, 70	71	86	81, 78	80
	454 (I)		74, 73	74		79, 75	77
	471 (F)		74, 71	72		80, 79	80
	453 (N)		75, 72	74		85, 83	84
Fortification level: 0.5 mg/kg, Report TPR-0029							
Apple (whole fruit)	0	-	84‡	84	-	85‡	85
	77	79	82, 70, 94, 87	83	78	83, 71, 89, 84	82
Apple (edible	0	-	86‡	86	-	86‡	86

Commodity	Storage interval (days)	1'-CH_2OH-S-2840A			1'-CH_2OH-S-2840B		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
portion)	77	82	86, 83, 88, 87	86	82	87, 87, 91, 83	87

Table 109 Storage stability results for metabolites N-des-Me-S-2840 and N-des-Me-1'-CH_2OH-S-2840 (isomers A and B) in various matrices (Report TPR-0075)

Commodity	Storage interval (days)	*N*-des-Me-S-2840			*N*-des-Me-1'-CH_2OH-S-2840A			*N*-des-1'-CH_2OH-Me-S-2840B		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Fortification level		0.1 mg/kg			0.05 mg/kg			0.05 mg/kg		
Cucumber	0	-	106, 108, 101	105	-	84, 95, 91	87	-	82, 86, 88	85
	35	103	101, 98	100	99	95, 94	95	100	94, 93	94
	91	108	93. 98	96	89	80, 79	80	81	77, 77	77
	182	105	102, 97	100	96	86, 82	84	95	82, 79	81
	379	99	89, 91	90	90	82, 85	84	90	84, 85	85
Grape	0	-	101, 102, 102	102	-	81, 87, 86	85	-	80, 86, 85	84
	35	106	100, 100	100	76	76, 80	78	77	79, 82	81
	91	107	96, 104	100	81	74, 76	75	84	76, 79	78
	182	103	94, 93	94	94	88, 85	87	91	94, 85	90
	379	102	99, 96	98	87	85, 91	88	85	81, 92	87
Soya bean seed	0	-	94, 93, 94	94	-	71, 71, 70	71	-	72, 73, 70	72
	34	90	91, 96	94	91	87, 81	84	97	88, 86	87
	90	102	92, 96	94	79	77, 76	77	83	76, 77	77
	182	99	93, 88	91	89	82, 72	77	95	83, 77	80
	378	95	98, 99	99	81	78, 79	79	82	78, 79	79
Wheat grain	0	-	98, 95, 95	96	-	80, 82, 78	80	-	79, 80, 77	79
	35 (50)[a]	71, (82)[a]	64, 63 (63, 75)[a]	66[†]	96	91, 92	92	99	95, 99	97
	91	96	81, 86	84	87	79, 74	77	91	82, 75	79
	182	82	84, 80	82	87	81, 83	82	86	84, 80	82
	377	84	78, 79	79	93	88, 83	86	96	91, 86	89
Field bean	0	-	90, 89, 90	90	-	92, 93, 104	96	-	94, 92, 104	97
	34	99	102, 107	105	95	96, 98	97	95	95, 97	96
	90	107	103, 106	105	94	89, 88	89	93	84, 87	86
	181	108	104, 99	102	93	92, 86	89	94	92, 80	86
	376	100	104, 101	103	90	87, 86	87	91	89, 86	88

Notes:

[a] Samples were re-analysed after 50 days of storage; results are displayed in brackets. Therefore, an additional procedural recovery experiment was conducted. The average recovery takes into account all four recovery values.

Table 110 Storage stability results for inpyrfluxam and its metabolite 3'-OH-S-2840 in various processed commodities (TPR 0087; fortification level of 0.1 mg/kg))

Commodity	Storage interval (days)[a]	Inpyrfluxam			3'-OH-S-2840		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Potato (starch)	0	-	102, 107, 109	106	-	107, 102, 110	106
	217	83	86, 79	83	89	97, 96	97
	425	108	100, 99	100	101	105, 101	103
Potato (crisps)	0	-	107, 110, 110	109	-	110, 110, 110	110
	217	82	87, 94	91	97	98, 103	101
	428	108	101, 99	100	104	109, 101	105
Wheat (flour)	0	-	81, 70, 71	74	-	91, 94, 95	93
	298	86	74, 91	83	99	86, 91	89
	518	106	93, 106	100	106	105, 101	103
Wheat (bread)	0	-	105, 107, 110	107	-	101, 99, 104	101
	220	109	100, 101	101	106	102, 102	102
	519	108	98, 96	97	107	99. 102	101

Note:
[a] Day 0 results taken from Lebrun, F. (2018; TPR-0076).

Table 111 Storage stability results for DFPA, DFPA-$CONH_2$ and *N*-des-Me-DFPA in various processed commodities (Report TPR 0087; fortification level of 0.1 mg/kg)

Commodity	Interval (days)	DFPA			DFPA-$CONH_2$			*N*-des-Me-DFPA		
		Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining	Fresh recovery (%)	% remaining	Mean % remaining
Potato (starch)	0	-	105, 107, 110	107	-	106, 102, 108	105	-	83, 91, 96	90
	217	98	96, 93	95	79	88, 85	87	70	77, 71	74
	425	110	94, 103	99	108	99, 103	101	86	82, 70	76
Potato (crisps)	0	-	108, 107, 110	108	-	110, 105, 115	110	-	79, 72, 81	77
	217	122[a]	100, 99	100	106	99, 92	96	68	84, 80	82
	428	110	95, 96	96	109	106, 105	106	83	78, 83	81
Wheat (flour)	0	-	92, 92, 93	92	-	98, 108, 90	99	-	70, 71, 72	71
	298[b]	101	90, 95	93	90	85, 95	90	75	64, 73	69
	518	109	97, 94	96	107	104, 106	105	85	78, 78	78
Wheat (bread)	0	-	84, 89, 84	86	-	95, 98, 102	98	-	80, 87, 84	84
	220	108	98, 96	97	107	106, 101	104	70	75, 76	76
	519	99	95, 92	94	108	105, 102	104	100	98, 93	96

Notes:
[a] Recovery of the confirmation method = 110%, therefore recovery of 122% was deemed acceptable.
[b] 294 days for DFPA-$CONH_2$.

Table 112 Storage stability results for inpyrfluxam, 1'-COOH-S-2840A, 1'-COOH-S-2840B, 1'-CH2OH-S-2840A and 1'-CH_2OH-S-2840B in animal commodities (Report TPR 013)

Matrix	Storage interval (days)	Inpyrfluxam		1'-COOH-S-2840A		1'-COOH-S-2840B		1'-CH_2OH-S-2840A		1'-CH_2OH-S-2840B	
		Fresh rec. (%)	% remaining (mean)	Fresh rec. (%)	% remaining (mean)	Fresh rec. (%)	% remaining (mean)	Fresh rec. (%)	% remaining (mean)	Fresh rec. (%)	% remaining (mean)
Fortification level		0.1 mg/kg		0.05 mg/kg		0.05 mg/kg		0.05 mg/kg		0.05 mg/kg	
Milk	0	-	74, 85 (80)	-	87, 96 (92)	-	86, 99 (93)	-	95, 96 (96)	-	92, 94 (93)
	29	78, 83 (81)	78, 82 (80)	91, 94 (93)	91, 93 (92)	93, 97 (95)	93, 93 (93)	89, 90 (90)	83, 87 (85)	87, 88 (88)	81, 85 (83)
	75	100, 101 (101)	82, 83 (83)	109, 114 (112)	88, 92 (90)	112, 112 (112)	90, 90 (90)	99, 86 (93)	82, 89 (86)	93, 77 (85)	74, 81 (78)
Muscle	0	-	72, 76 (74)	-	80, 87 (84)	-	81, 88 (85)	-	95, 100 (98)	-	93, 96 (95)
	29	94, 89 (92)	87, 83 (85)	103, 95 (99)	95, 87 (91)	99, 96 (98)	97, 87 (92)	87, 91 (89)	85, 86 (86)	83, 88 (86)	84, 83 (84)
Liver	0	-	81, 80 (81)	-	92, 94 (93)	-	93, 88 (91)	-	93, 90 (92)	-	86, 85 (86)
	29	88, 79 (84)	77, 78 (78)	101, 90 (96)	85, 85 (85)	99, 91 (95)	89, 89 (89)	84, 77 (81)	77, 77 (77)	80, 71 (76)	74, 73 (74)
Kidney	0	-	69, 73 (71)	-	84, 86 (85)	-	81, 87 (84)	-	90, 92 (91)	-	87, 86 (87)
	29	91, 85 (88)	81, 75 (78)	103, 97 (100)	87, 85 (86)	100, 97 (99)	91, 88 (90)	88, 86 (87)	77, 77 (77)	86, 79 (83)	71, 72 (72)
Fat	0	-	83, 72 (78)	-	94, 82 (88)	-	91, 83 (87)	-	87, 79 (83)	-	80, 74 (77)
	31	98, 100 (99)	94, 89 (92)	99, 103 (101)	95, 91 (93)	98, 101 (100)	95, 88 (92)	87, 86 (87)	85, 77 (81)	87, 85 (86)	84, 75 (80)

USE PATTERN

Inpyrfluxam is registered for use on numerous crops in multiple countries. Inpyrfluxam can be used a seed treatment with a flowable concentrate (FS) formulation, or foliar application with a suspension concentrate (SC) formulation. Information on registered uses that was provided to the meeting is summarized in Table 113.

Table 113 Summary of registered use patterns for inpyrfluxam. For all uses, application timing, coincides with threshold pest pressure Adjuvants permitted to be used for foliar applications unless specified

Crop	Country	Formulation		Application						PHI (days)	Remarks
		Name	Active ingredient (%)	Method	No.	Rate (g ai/ha)	Timing	Interval (days)	Seasonal max. (g ai/ha)		
Apple	United States	2.84 SC	31	Foliar	2	100	Between green tip and petal fall (BBCH 69)	10	200	n.a.	Do not use adjuvants
Apple	Japan	40 SC	37	Foliar	3	700 (9.25 g ai/hL)	n.s.	n.s.	2100 (27.75 g ai/hL)	1	Do not use adjuvants

Crop	Country	Formulation		Application						PHI (days)	Remarks
		Name	Active ingredient (%)	Method	No.	Rate (g ai/ha)	Timing	Interval (days)	Seasonal max. (g ai/ha)		
Soya bean	United States	3.2 FS	34.05	Seed treatment	1	2.5-5 g ai/100 kg seeds (0.004-0.008 mg ai/seed) 25 g ai/100 kg seed (0.04 mg ai/seed)	n.s.	n.a.	200 g ai/ha (from any treatment)	n.a.	Combined seed and foliar treatments possible do not graze treated fields or feed treated hay to livestock Rotation interval: 9 months
		2.84 SC	31	Foliar	2	50	Prior to disease development but not prior to V3 (third node BBCH 14) or after R5 (beginning seed; approximate BBCH 75-79)	14	100	n.a.	
Sugar beet	United States	3.2 FS	34.05	Seed treatment	1	0.05-0.1 g ai/ /100,000 seeds	n.a.	n.a.	0.05-0.1/100,000 seeds	n.a.	Combined seed and foliar treatments possible.
		2.84 SC	31	Foliar broadcast application	2	50	2-8 leaf beets	21	100	50	Rotation interval: 9 months
				banded application	1	50			50		
Rice	United States	2.84 SC	31	Foliar	1	50-100	Prior to disease development, approximately 25-30 days after the permanent flood has been established	n.a.	100	Early boot (BBCH 41)	Rotation interval: 9 months
		3.2 FS	34.05	Seed treatment	1	5-10 g ai/100 kg seed	n.a.	n.a.	10 g ai/100 kg seed	n.a.	Rotation interval: 9 months
Corn (field, sweet, pop	United States	2.84 SC	31	In furrow	1	50	In furrow, at planting	N/A	50	n.s.	Rotation interval: 9 months
		3.2 FS	34.05	Seed treatment	1	0.014 mg ai/seed	n.a.	n.a.	0.014 mg ai/seed	n.a.	Rotation interval: 9 months
Peanut	United States	2.84 SC	31	Foliar	4	50-100	Prior to disease development but no earlier than 30 days	14-28	200	40	Rotation interval: 9 months

Crop	Country	Formulation		Application						PHI (days)	Remarks
		Name	Active ingredient (%)	Method	No.	Rate (g ai/ha)	Timing	Interval (days)	Seasonal max. (g ai/ha)		
							after planting				

Notes:
n.s. = Not specified.
n.a. = Not applicable.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received data from supervised residue trials on apples, soya beans, sugar beets, rice corn and peanuts.

The field trial reports included method validation data, as recoveries from spiked samples at levels reflecting those observed in the field trial samples, dates from critical events during the study, including application, harvest, storage, and analysis; as well as detailed information on the field site and treatment parameters. Analytical reports were sufficiently detailed and included example chromatograms and example calculations. Samples were analysed by the methods described above. Unless otherwise noted in the tables below, harvested commodities were maintained whole in the field and not cut or homogenized until they reached the analytical laboratory.

The field trial study designs included control plots. For most crops measured residues from control plots were < 0.01 mg/kg (i< LOQ) and are not included in the summary tables in this evaluation. Cases where quantifiable residues were found in control matrices are noted in the Table 114 to Table 123.

In the summary tables, values used for making maximum residue level recommendations are underlined and highest individual values for estimating dietary intake are bolded. Trial locations that the Meeting has determined are not independent are grouped by a heavy cell border in the tables. A summary of supervised trials for inpyrfluxam per crop group and commodity is shown below

Commodity	Study No	Table No
Apple (FP 0226)	201700036, 201700086, JP2014C288, JP2015C239, JP2016C334	Table 114
Soya beans, dry (VD 0541)	201700156	Table 115
Sugar beet (VR 0596)	201700065, 201700098	Table 116
Rice (GC 0649)	201700341	Table 117
Sweet corn (GC 0447)	201700217	Table 118
Maize (GC 0645)	201700217	Table 119
Maize forage (AF 0645	201700217	Table 120
Maize Stover (AS 3558)	201700217	Table 121
Peanut (SO 0697)	201700318	Table 122
Peanut hay (AL 3352)	201700318	Table 123

Apple

Nineteen field trials were conducted in Canada and the United States during 2014 -2015 growing seasons (201700036 and 201700086), inpyrfluxam was applied as a SC formulation, twice as a foliar spray, at full bloom and petal fall growth stages, at a rate of 100 g ai/ha per treatment and with a PHI of 111–185 days. In trial V-38516-Q a second plot was treated with two applications of 500 g ai/ha (5× rate) as to obtain apples for processing and trials V-38516-F, V-38516-L in which sampling dates were varied to evaluate the effect of harvest timing.

Fruits in the US and Canadian trials were analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (analysed separately as 1'-CH_2OH-S-2840A and 1'-CH_2OH-S-2840B) and DFPA-$CONH_2$ using validated method RM-50C-1, with a LOQ of 0.01 mg/kg for each analyte. Overall, concurrent recovery samples for inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840A, 1'-CH_2OH-S-2840B and DFPA-$CONH_2$ in apples, gave results ranging from 67 to 108 percent recovery, with relative standard deviations up to 4 percent. Results from the trials are summarized in Table 114.

In addition eight trials were conducted in Japan during 2014, 2015 and 2016 growing seasons. Inpyrfluxam, was applied as an SC formulation, three times (6–7 days interval) as a foliar spray to apple trees between the fruit colouring stage to the beginning of harvest, at a nominal rate of 429 and 450 g ai/ha and with a PHI of 1 days. Apples were harvested at 1, 3, 7, 14 and 21 days after the last application.

Fruits in the Japanese trials (JP2014C288, JP2015C239 and JP2016C334) were analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840A, 1'-CH_2OH-S-2840B, *N*-des-Me-DFPA and DFPA-$CONH_2$ using a validated LC-MS/MS method (JP2015C239) with an LOQ of 0.01 mg/kg for each analyte. Overall, concurrent recovery samples for inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840A, 1'-CH_2OH-S-2840B, DFPA-$CONH_2$ and *N*-des-Me-DFPA in apples, gave results ranging from 67 to 107 percent recovery, with RSD up to 10 percent.

Samples were stored for a maximum of 505 days prior to extraction and analysis. This period is covered by the available storage stability study (514 days in apple). Results from the trials are summarized in Table 114.

Table 114 inpyrfluxam residues in apples resulting from supervised trials in the Unites States of American, Canada and Japan conducted with 2 foliar applications of SC formulation

APPLE Country, Year (Variety)	Application		DALA	Commodity	Residues, mg/kg [mean]					Reference
	Rate, g ai/ha	Growth stage			Inpyrfluxam	3'-OH-S-2840	1'-CH2OH-S-2840[a]	DFPA-CONH2	N-des-Me-DFPA	
Canada Branchton, Ontario, 2014 (Ida Red)	96 94	BBCH 65 BBCH 67	120	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	201700086 Trial: V-38564-A,
Canada Simcoe, Ontario, 2014 (Mutsu)	100 100	BBCH 65 BBCH 67	110	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38564-B
Canada Simcoe, Ontario, 2014 (Macintosh)	101 100	BBCH 65 BBCH 67	110	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38564-C
United States Alton, New York, 2014 (Rome)	101 100	BBCH 65 BBCH 69	132	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-A,

APPLE Country, Year (Variety)	Application		DALA	Commodity	Residues, mg/kg [mean]					Reference
	Rate, g ai/ha	Growth stage			Inpyrfluxam	3'-OH-S-2840	1'-CH2OH-S-2840[a]	DFPA-CONH2	N-des-Me-DFPA	
United States North Rose, New York, 2014 (Ida Red)	100 100	BBCH 65 BBCH 69	121	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-B,
United States Lula, Georgia, 2015 (Arkansas Black)	101 102	BBCH 63-65 BBCH 66-68	185	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-C
United States Dix, Illinois, 2014 (Chieftain)	103 102	Full bloom; 80% petal fall	145	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-D
United States Hotchkiss, Colorado, 2014 (Jonathan)	99 99	BBCH 65 BBCH 67	138	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-E
United States Granger, Washington, 2014 (Granny Smith)	95 99	Full bloom; Petal fall	126	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	
			131		<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-F
			136		<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	
			141		<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	
United States Hood River, Oregon, 2014 (Jonagold)	100 100	BBCH 65 BBCH 67	140	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-G
United States Oregon, Wisconsin 2014 (Cortland)	99 99	Full flower; Petal fall	111	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-H
United States North Rose, New York, 2015 (Cortland)	100 100	BBCH 65 BBCH 69	122	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-I
United Statees Phelps, New York, 2015 (Macoun)	101 100	BBCH 65 BBCH 69	115	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	
United States Cana, Virginia, 2015 (Rome)	102 103	BBCH 65 BBCH 67	129	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-J
Canada,Cambridge, Ontario, 2015 (Ida Red)	95 101	BBCH 65-67 BBCH 69	136	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-K
			141		<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-L
			146		<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	
			151		<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	
United States Linden, California, 2015 (Granny Smith)	103 103	BBCH 65 BBCH 67	132	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	

APPLE Country, Year (Variety)	Application		DALA	Commodity	Residues, mg/kg [mean]					Reference
	Rate, g ai/ha	Growth stage			Inpyrfluxam	3'-OH-S-2840	1'-CH2OH-S-2840[a]	DFPA-CONH2	N-des-Me-DFPA	
United States Zillah, Washington, 2015 (Gala Buckeye)	97 102	BBCH 65 Petal fall	119	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-N
United States Parkdale, Oregon, 2015 (Gala)	99 101	BBCH 65 BBCH 69	129	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-O
United States Hood River, Oregon, 2015 (Jonagold)	100 98	BBCH 65 BBCH 69	140	Fruit	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	Trial: V-38516-P Trial: V-38516-
Aomori-Shoku, Japan, 2014 (Fuji)	450	Fruit thickening/colouring stage to beginning of harvest time	1	Fruit	1.23, 1.22 [1.22]	<0.01 (2)	<0.01	<0.01 (2)	<0.01 (2)	JP2014C288
			3	Fruit	1.23 (2)	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			7	Fruit	1.10, 1.09 [1.10]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			14	Fruit	1.04, 1.03 [1.04]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			21	Fruit	1.19, 1.13 [1.16]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
Fukushima-Shoku, Japan, 2014 (Fuji)	430	Fruit colouring stage to beginning of harvest time	1	Fruit	1.44, 1.39 [1.42]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			3	Fruit	1.36 (2)	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			7	Fruit	1.15, 1.14 [1.14]	0.05, 0.04 [0.05]	<0.01	<0.01 (2)	<0.01 (2)	
			14	Fruit	0.76; 0.73 [0.74]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			21	Fruit	0.83, 0.80 [0.82]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
Aomori-Shoku, Japan, 2015 (Fuji)	450	Fruit thickening/colouring stage to beginning of harvest time	1	Fruit	0.90, 0.88 [0.89]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	JP2015C239
			3	Fruit	0.99, 0.96 [0.98]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	

APPLE Country, Year (Variety)	Application		DALA	Commodity	Residues, mg/kg [mean]					Reference
	Rate, g ai/ha	Growth stage			Inpyrfluxam	3'-OH-S-2840	1'-CH2OH-S-2840[a]	DFPA-CONH2	N-des-Me-DFPA	
			7	Fruit	0.54, 0.53 [0.54]	0.03, 0.02 [0.02]	<0.01	<0.01 (2)	<0.01 (2)	
			14	Fruit	0.63, 0.61 [0.62]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			21	Fruit	0.49, 0.49 [0.49]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			28	Fruit	0.56, 0.52 [0.54]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			35	Fruit	0.48, 0.46 [0.47]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			1	Pulp	0.80, 0.77 [0.78]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			3	Pulp	1.02, 1.02 [1.02]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			7	Pulp	0.51, 0.49 [0.50]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			14	Pulp	0.65, 0.64 [0.64]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			21	Pulp	0.56, 0.54 [0.55]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			28	Pulp	0.54, 0.52 [0.53]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			35	Pulp	0.53, 0.53 [0.53]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
Iwate-Shoku, Japan, 2015 (Fuji)	450	Fruit colouring stage to harvest time	1	Fruit	0.72, 0.71 [0.72]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			3	Fruit	0.59, 0.59 [0.59]	0.02, 0.01 [0.02]	<0.01	<0.01 (2)	<0.01 (2)	
			7	Fruit	0.53, 0.52 [0.52]	0.02, 0.01 [0.02]	<0.01	<0.01 (2)	<0.01 (2)	
			14	Fruit	0.47, 0.44 [0.46]	0.01 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			1	Pulp	0.75, 0.74 [0.74]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	

APPLE Country, Year (Variety)	Application		DALA	Commodity	Residues, mg/kg [mean]					Reference
	Rate, g ai/ha	Growth stage			Inpyrfluxam	3'-OH-S-2840	1'-CH2OH-S-2840[a]	DFPA-CONH2	N-des-Me-DFPA	
			3	Pulp	0.59, 0.57 [0.58]	0.01 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			7	Pulp	0.57, 0.57 [0.57]	0.01 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			14	Pulp	0.46, 0.44 [0.45]	0.01 (2)	<0.01	<0.01 (2)	<0.01 (2)	
	450	Fruit thickening stage to fruit colouring stage	21	Fruit	0.40, 0.39 [0.40]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			28	Fruit	0.34, 0.32 [0.33]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			35	Fruit	0.32, 0.31 [0.32]	0.03, 0.02 [0.02]	<0.01	<0.01 (2)	<0.01 (2)	
			21	Pulp	0.37, 0.36 [0.36]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			28	Pulp	0.35, 0.33 [0.34]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			35	Pulp	0.32, 0.30 [0.31]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
Fukushima-Shoku, Japan, 2015 (Fuji)	470	Fruit colouring stage to harvest time	1	Fruit	0.77, 0.75 [0.76]	0.04 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			3	Fruit	0.85, 0.81 [0.83]	0.06 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			7	Fruit	0.85, 0.83 [0.84]	0.08 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			14	Fruit	0.70, 0.65 [0.68]	0.09 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			21	Fruit	0.73, 0.71 [0.72]	0.12, 0.11 [0.12]	<0.01	<0.01 (2)	<0.01 (2)	
			28	Fruit	0.66, 0.63 [0.64]	0.10 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			35	Fruit	0.53, 0.51 [0.52]	0.08 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			1	Pulp	0.59, 0.59 [0.59]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	

APPLE Country, Year (Variety)	Application		DALA	Commodity	Residues, mg/kg [mean]					Reference
	Rate, g ai/ha	Growth stage			Inpyrfluxam	3'-OH-S-2840	1'-CH2OH-S-2840[a]	DFPA-CONH2	N-des-Me-DFPA	
			3	Pulp	0.60, 0.59 [0.60]	0.05 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			7	Pulp	0.68, 0.67 [0.68]	0.07 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			14	Pulp	0.58, 0.56 [0.57]	0.08 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			21	Pulp	0.65, 0.61 [0.63]	0.12, 0.11 [0.12]	<0.01	<0.01 (2)	<0.01 (2)	
			28	Pulp	0.60, 0.60 [0.60]	0.09 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			35	Pulp	0.49, 0.49 [0.49]	0.08, 0.07 [0.08]	<0.01	<0.01 (2)	<0.01 (2)	
Nagano-Shoku, Japan, 2015 (Fuji)	470	Fruit colouring stage to harvest time	1	Fruit	0.78, 0.77 [0.78]	0.05 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			3	Fruit	0.75, 0.70 [0.72]	0.06 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			7	Fruit	0.79, 0.77 [0.78]	0.07, 0.06 [0.07]	<0.01	<0.01 (2)	<0.01 (2)	
			14	Fruit	0.77, 0.76 [0.76]	0.05 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			21	Fruit	0.58, 0.58 [0.58]	0.05 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			28	Fruit	0.54, 0.54 [0.54]	0.05 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			35	Fruit	0.48, 0.45 [0.46]	0.04 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			1	Pulp	0.73, 0.67 [0.70]	0.05 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			3	Pulp	0.69, 0.68 [0.68]	0.06, 0.05 [0.06]	<0.01	<0.01 (2)	<0.01 (2)	
			7	Pulp	0.79, 0.77 [0.78]	0.06 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			14	Pulp	0.77, 0.74 [0.76]	0.06, 0.05 [0.06]	<0.01	<0.01 (2)	<0.01 (2)	

APPLE Country, Year (Variety)	Application		DALA	Commodity	Residues, mg/kg [mean]					Reference
	Rate, g ai/ha	Growth stage			Inpyrfluxam	3'-OH-S-2840	1'-CH2OH-S-2840[a]	DFPA-CONH2	N-des-Me-DFPA	
			21	Pulp	0.57, 0.57 [0.57]	0.05 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			28	Pulp	0.52, 0.50 [0.51]	0.05, 0.04 [0.05]	<0.01	<0.01 (2)	<0.01 (2)	
			35	Pulp	0.48, 0.47 [0.48]	0.04 (2)	<0.01	<0.01 (2)	<0.01 (2)	
Fukushima-Shoku, Japan, 2016 (Fuji)	440	Fruit maturity	1	Fruit	1.65, 1.60 [1.62]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	JP2016C334
			3	Fruit	1.89, 1.88 [1.88]	0.03; 0.02 [0.02]	<0.01	<0.01 (2)	<0.01 (2)	
			7	Fruit	1.74, 1.58 [1.66]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			1	Pulp	1.98, 1.96 [1.97]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			3	Pulp	1.98, 1.84 [1.91]	0.03, 0.02 [0.02]	<0.01	<0.01 (2)	<0.01 (2)	
			7	Pulp	1.70, 1.68 [1.69]	0.03 (2)	<0.01	<0.01 (2)	<0.01 (2)	
Yamanashi, Japan, 2016 (Fuji)	420	Fruit maturity	1	Fruit	0.53 0.51 [0.52]	<0.01 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			3	Fruit	0.35, 0.32 [0.34]	<0.01 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			7	Fruit	0.46, 0.44 [0.45]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			1	Pulp	0.47, 0.46 [0.46]	<0.01 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			3	Pulp	0.39, 0.36 [0.38]	<0.01 (2)	<0.01	<0.01 (2)	<0.01 (2)	
			7	Pulp	0.40, 0.39 [0.40]	0.02 (2)	<0.01	<0.01 (2)	<0.01 (2)	

Notes:
DALA: days of the last application
[a] Free and conjugated, sum of A and B isomers

Soya bean, dry

Twenty one residue trials were conducted on soya bean in the United States in 2014–2015 growing season (Foster, 2017; 201700156). Soya bean seeds were treated with inpyrfluxam as a FS formulation, at a rate of 10 g ai/100 kg seed, 14 days prior to the R5 growth stage (visible seed in pod of upper four nodes) and with two foliar application as an SC formulation at a rate of 100 g ai/ha (on a 10–21 interval) at the BBCH 75. Adjuvant (non-ionic surfactant (NIS), crop oil concentrate (COC) or silicone) was added. Soil type was not reported.

In trial V-38537-O the sampling dates varied as to evaluate the effect of harvest timing in trial. In V-38537-H an additional plot was treated at an exaggerated rate of 2× the GAP rate for each formulation, in trials V-38537-I, R and U an additional plot was treated at 5× the GAP rate, and in trial V-38537-U soya bean seeds were processed into hulls, meal and refined oil.

Dry seed samples were collected at normal commercial harvest, 26–84 days after the last application for harvest trials and 31–43 days after last application for the decline trials.

Samples were analysed for inpyrfluxam and its major metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (analysed as 1'-CH_2OH-S-2840A and B), DFPA-$CONH_2$ and conjugated forms of metabolites analysed as aglycones (1'-COOH-S-2840 and 1'-CH_2OH-S-2840) using the validated method RM-50C-1. The LOQ was 0.01 mg/kg for each analyte. The combined LOQ for l'-CH_2OH-S-2840 (A and B) or 1'-COOH-S-2840 (A and B) was 0.02 mg/kg (sum of the individual LOQs). For the determination of *N*-des-Me-DFPA, method RM-50C-2a was used, with a LOQ of 0.02 mg/kg. Overall, concurrent recovery samples for inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840A, 1'-CH_2OH-S-2840B, 1'-COOH-S-2840 and *N*-des-Me-DFPA in seeds, gave results ranging from 67 to 115 percent recovery, with RSD up to 11 percent.

Samples of dry soya bean seed were stored frozen for up to 587 days prior to the extraction and analysis. This period is covered by the available storage stability study (683 days in dry for soya bean seed). Results from the trials are summarized in Table 115.

Table 115 Inpyrfluxam residues in dry soya bean seeds resulting from a seed treatment followed by two foliar treatments in supervised trials in the United States of America (Study 201700156)

SOYA BEAN Location Year (Variety)	Application			Growth stage[a]	DALA	Residues, mg/kg				Trial
	Formulation	g ai/100 kg seed g ai/ha	No.			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[b]	*N*-des-Me-DFPA	
Elko, South Carolina, 2014 (AG7231)	3.2 FS 2.84 SC	10 99 102	1 2	BBCH 73 (R5)	69	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.026, 0.022	V-38537-A
Chula, Georgia, 2, 2014 (AG7231)	3.2 FS 2.84 SC	10 102 104	1 2	BBCH 78 (R5)	48	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.036, 0.038	V-38537-B
Leland, Mississippi, 2014 (P50T94)	3.2 FS 2.84 SC	10 100 106	1 2	BBCH 72-74 (R5)	64	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.052, 0.049	V-38537-C
Procter, Arkansas, 2014 (P50T64R)	3.2 FS 2.84 SC	10 100 98	1 2	BBCH 76	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.066, 0.058	V-38537-D
Cheneyville, Louisiana, 2014 (P50T64R)	3.2 FS 2.84 SC	10 102 100	1 2	BBCH 74-75	84	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.025, 0.027	V-38537-E

SOYA BEAN Location Year (Variety)	Application			Growth stage[a]	DALA	Residues, mg/kg				Trial
	Formulation	g ai/100 kg seed g ai/ha	No.			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[b]	*N*-des-Me-DFPA	
Northwood, North Dakota, 2014 (H09Y11)	3.2 FS 2.84 SC	10 100 99	1 2	BBCH 75	53	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-F
St. Cloud, Minnesota, 2014 (H20Y1R)	3.2 FS 2.84 SC	10 100 100	1 2	BBCH 67-74	56	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-G
York, Nebraska, 2014 (H29Y12)	3.2 FS 2.84 SC	10 102 102	1 2	BBCH 75	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-H
	3.2 FS 2.84 SC	20 205 202	1 2	BBCH 75	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.094, 0.096; (0.095)	
Jefferson, Iowa, 2014 (H23R3)	3.2 FS 2.84 SC	10 99 99	1 2	R5	76	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	V-38537-I
	3.2 FS 2.84 SC	50 504 499	1 2	R5	76	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.153, 0.161	
Mason, Illinois, 2014 (Hughes 777RR)	3.2 FS 2.84 SC	10 98 99	1 2	BBCH 75	60	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-J
Troy, Ohio, 2015 (Sty3702R2)	3.2 FS 2.84 SC	10 101 103	1 2	R5	51	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.021, 0.024	V-38537-K
Marysville, Ohio, 2015 (Steyer 3702R2)	3.2 FS 2.84 SC	10 101 103	1 2	BBCH 77	53	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-L
Manilla, Indiana, 2015 (3702R2)	3.2 FS 2.84 SC	10 102 101	1 2	R5	49	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.032 (2)	V-38537-M
Farson, Iowa, 2015 (Styr 2702R2)	3.2 FS 2.84 SC	10 99 101	1 2	BBCH 77 (R5)	33	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-N
York, Nebraska, 2015 (Steyer 2702R2)	3.2 FS 2.84 SC	10 101 100	1 2	BBCH 75 (R5)	31	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-O
					35	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	
					39	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.027, 0.028	
					43	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	
Lenexa, Kansas, 2015 (Styr3702R2)	3.2 FS 2.84 SC	10 104 103	1 2	BBCH 79	36	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-P
Sheridan, Indiana, 2015 (Styer 3103R2)	3.2 FS 2.84 SC	10 101 103	1 2	R5	31	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-Q
Edgewood, Illinois, 2015 (Styr3702R2)	3.2 FS 2.84 SC	10 102 102	1 2	R5	26	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-R

SOYA BEAN Location Year (Variety)	Application			Growth stage[a]	DALA	Residues, mg/kg				Trial
	Formulation	g ai/100 kg seed g ai/ha	No.			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[b]	*N*-des-Me-DFPA	
	3.2 FS 2.84 SC	50 495 512	1 2	R5	26	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.034, 0.037	
Brookings, South Dakota, 2015 (H20R2)	3.2 FS 2.84 SC	10 102 100	1 2	BBCH 79	44	<u><0.01</u> (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-S
Geneva, Minnesota, 2015 (Hefty H23Y10)	3.2 FS 2.84 SC	10 100 98	1 2	R5	62	<u><0.01</u> (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	V-38537-T
Richland, Iowa, 2015 (Steyer 2702R2)	3.2 FS 2.84 SC	10 97 101	1 2	BBCH 75-77	41	<u><0.01</u> (2)	<0.01 (2)	<0.02 (2)	0.044, 0.046	V-38537-U
	3.2 FS 2.84 SC	50 497 506	1 2	BBCH 75-77	41	<0.01 (2)	<0.01 (2)	<0.02 (2)	0.196, 0.184	

Notes:
DALA: days of the last application.
[a] At the last treatment.
[b] Free and conjugated, sum of A and B isomers.
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Sugar beet

Twenty residue trials were conducted on sugar beets in Canada and the United States in 2014 and 2015 growing seasons (Bitter, 2016; 201700065, Bitter, 2017; 201700098), from which fifteen are considered independent. In each trial, sugar beet seeds were treated with a FS formulation, at a rate of 0.1 g ai/100,000 seeds. Following planting at 71 and 50 days before harvest, two foliar applications of inpyrfluxam as an SC were performed at a rate of 100 g ai/ha per treatment. Adjuvant (non-ionic surfactant (NIS), crop oil concentrate (COC) or silicone) was added. Sugar beet roots were collected at normal commercial harvest, 50–51 days after the last application. Residues in sugar beet tops were not investigated. Soil type was not reported.

In trials V-38533-E and L an additional plot was treated at an exaggerated (5×) rate. In trial V-38533-N, sugar beet roots were processed into sugar, dried pulp and molasses. Seeds were treated at nominal rate of 0.5 g ai/100,000 seeds. Application intervals were the same as those in the associated 1× plot. In trial V-38533-B and -K the sampling dates varied as to evaluate the effect of harvest timing.

Samples of roots were analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (analysed separately as 1'-CH_2OH-S-2840A and B), DFPA-$CONH_2$ and conjugated forms of metabolites analysed as aglycones (1'-COOH-S-2840 and 1'-CH_2OH-S-2840) using the validated method RM-50C-1. The LOQ was 0.01 mg/kg for each analyte. The combined LOQ for l'-CH_2OH-S-2840 (A and B) or 1'-COOH-S-2840 (A and B) was 0.02 mg/kg or 0.023 mg/kg respectively (sum of the individual LOQs). Samples were not analysed for N-des-Me-DFPA. Overall, concurrent recovery samples for inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840A, 1'-CH_2OH-S-2840B, DFPA-$CONH_2$, 1'-COOH-S-2840A, 1'-COOH-S-2840B, and *N*-des-Me-DFPA in roots, gave results ranging from 70 to 121 percent recovery, with RSD up to 20 percent. The samples of sugar beet obtained at harvest were frozen for a maximum of 607 days before extraction and analysis. This period is covered by the available storage stability study (610 days in potatoes). Results from the trials are summarized in Table 116.

Table 116 Inpyrfluxam residues in sugar beet roots resulting from a seed treatment followed by two foliar treatments in supervised trials from Canada and the United States of America

SUGAR BEET Country, Location Year (Variety)	Application			Growth stage[a]	DALA	Residues, mg/kg							Study
	Formulation	g ai/100,000 seeds g ai/ha	No.			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[c]	DFPA-$CONH_2$	1'-COOH-S-2840[b]	1'-CH_2OH-S-2840[c]	*N*-des-Me-DFPA	
Canada Purple Springs, Alberta, 2015 (SX1212RR)	3.2 FS 2.84 SC	0.18 103 104	1 2	BBCH 38-39 BBCH 39	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	20170006 5 Trial: V-38580-C
Canada Taber, Alberta, 2015 (SX1521N)	3.2 FS 2.84 SC	0.2 101 103	1 2	BBCH 39 BBCH 39	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38580-D
Canada Taber, Alberta, 2015 (SV36902)	3.2 FS 2.84 SC	0.2 101 102	1 2	BCH 37-38 BBCH 38-39	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)		Trial: V-38580-E
Canada Taber, Alberta, 2015 (SX1212RR)	3.2 FS 2.84 SC	0.19 100 105	1 2	BBCH 37-38 BBCH 39	51	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38580-F
Canada Taber, Alberta, 2015 (SX1521N)	3.2 FS 2.84 SC	0.2 99 101	1 2	BBCH 38-39 BBCH 39	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38580-G
Canada Purple Springs Alberta, 2015 (SV36902)	3.2 FS 2.84 SC	0.18 101 101	1 2	BBCH 35-36 BBCH 72-74	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38580-H
United States Fitchburg, Wisconsin, 2014 (SV36902 RR)	3.2 FS 2.84 SC	0.2 101 101	1 2	NR	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	20170009 8 Trial: V-38533-A
United States St. Cloud, Minnesota, 2014 (SV36918 RR)	3.2 FS 2.84 SC	0.18 99 101	1 2	BBCH 37 BBCH 39	45	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-B
					50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	
					55	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	
					59	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	
United States Northwood, North Dakota, 2014 (SVRR245N)	3.2 FS 2.84 SC	0.18 99 101	1 2	BBCH 15 BBCH 39	49	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-C
United States, Grand Junction, Colorado2014 (SX1521N RR)	3.2 FS 2.84 SC	0.58 105 107	1 2	BBCH 35 BBCH 37-38	51	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-D

SUGAR BEET Country, Location Year (Variety)	Application			Growth stage[a]	DALA	Residues, mg/kg							Study
	Formulation	g ai/100,000 seeds g ai/ha	No.			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[c]	DFPA-$CONH_2$	1'-COOH-S-2840[b]	1'-CH_2OH-S-2840[c]	*N*-des-Me-DFPA	
United States Yuba City, California,2014 (SX1521N RR)	3.2 FS 2.84 SC	0.24 103 99	1 2	BBCH 15 BBCH 45	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-E
	3.2 FS 2.84 SC	1.2 511 496	1 2	BBCH 15 BBCH 45	50	0.014, 0.013	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	<0.01 (2)	
United States Rupert, Idaho, 2014 (SX1521N RR)	3.2 FS 2.84 SC	0.26 101 102	1 2	BBCH 39 BBCH 39	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial V-38533-F
United States Geneva, Minnesota, 2015 (SX1212)	3.2 FS 2.84 SC	0.18 100 99	1 2	Vegetative Vegetative	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-G
United States Claude, Texas, 2015 (SX1211N)	3.2 FS 2.84 SC	0.08 101 101	1 2	BBCH 37 BBCH 47	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-H
United States Paso Robles, California, 2015 (SX1211N)	3.2 FS 2.84 SC	0.16 101 101	1 2	BBCH 39 BBCH 49	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-I
United States Ephrata, Washington, 2015 (SX1211N)	3.2 FS 2.84 SC	0.11 99 100	1 2	BBCH 39 BBCH 41	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-J
United States Sykeston, North Dakota, 2015 (SX1212RR)	3.2 FS 2.84 SC	0.18 101 105	1 2	BBCH 37 BBCH 49	35	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-K
					42	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	
					50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	
					56	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	
United States St. Cloud, Minnesota, 2015 (SX1521N RR)	3.2 FS 2.84 SC	0.16 100 100	1 2	BBCH 38 BBCH 39	51	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-L
	3.2 FS 2.84 SC	0.73 505 499	1 2	BBCH 38 BBCH 39	51	0.013, 0.010	<0.01 (2)	<0.02 (2)	<0.01 (2)	0.033, <0.023;	<0.02 (2)	<0.01 (2)	
United States Toronto, South Dakota, 2015 (SX1212RR)	3.2 FS 2.84 SC	0.1 99 99	1 2	BBCH 37 BBCH 39	50	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	-	Trial: V-38533-M

Notes:
DALA: days after the last application.
[a] At the last application.
[b] sum of isomers.
[c] free and conjugated.

Rice

Seventeen residue trials were conducted on rice (Irrigated paddies) in the United States in 2014, 2015 and 2017 growing season [Bitter, 2017; 201700341), from which fourteen are considered independent. In each trial, rice grains were treated with inpyrfluxam as a FS formulation, at a rate of 10 g ai/100 kg seed. Treated seeds were planted 0–55 days after receipt and we stored at approximately 14 ºC prior to planting.

At the late boot stage, a single foliar application of 96.5–106.7 g ai/ha was made using inpyrfluxam as a formulation. Adjuvant (non-ionic surfactant (NIS), crop oil concentrate (COC) or silicone) added. Rice was collected at normal commercial harvest, 35-71 days after the last application for harvest trials and 35–50 days after last application for the decline trial (V-38528-N). Soil type was not reported.

In trial V-38528-N, seeds in the second plot were treated at 5× rate (50 g ai/100 kg seed), in three trials (V-38528-K, V-38528-N and V-38528-M), an additional plot was also treated at 5× rate (500–501 g ai/ha) as to investigate the effect of exaggerated application rates on residues. In trial V-38528-N the sampling dates varied as to evaluate the effect of harvest timing and in trial V-38528-L grains were processed into hulls, bran and polished rice.

Grain samples were analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (analysed separately as 1'-CH_2OH-S-2840A and B), DFPA-$CONH_2$ and conjugated forms of metabolites analysed as aglycones (1'-COOH-S-2840 and 1'-CH_2OH-S-2840) using the validated method RM-50C-1. The LOQ was 0.01 mg/kg for each analyte. The combined LOQ for l'-CH_2OH-S-2840 (A and B) or 1'-COOH-S-2840 (A and B) was 0.02 mg/kg (sum of the individual LOQs). Overall, concurrent recovery samples for inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840A, 1'-CH_2OH-S-2840B, DFPA-$CONH_2$, 1'-COOH-S-2840A (agly), 1'-COOH-S-2840B (agly) in seeds, gave results ranging from 70 to 121 percent recovery, with RSD up to 20 percent.

The samples of grain (husked rice) obtained at harvest were frozen for a maximum of 609 days before extraction and analysis. This period is covered by the available storage stability study (616 days in rice grain). Results from the trials are summarized in Table 117.

Table 117 Inpyrfluxam residues in husked rice resulting from a seed treatment followed by two foliar treatments in supervised trials in the United States of America (Report 201700341)

RICE Location, Year (Variety)	Application			Growth state	DALA	Residues, mg/kg			Trial
	Formulation	g ai/100 kg seed[a] g ai/ha	No.			inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[a]	
Proctor, Arkansas, 2014 (Cheniere)	3.2 FS 2.84 SC	10.1 101	1 1	BBCH 45	51	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-38528-A
Bayou Meto, Arkansas, 2014 (CL 152)	3.2 FS 2.84 SC	10.1 100	1 1	BBCH 45	54	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-38528-D
Greenville, Mississippi, 2014 (Cocodrie)	3.2 FS 2.84 SC	10.1 97	1 1	BBCH 45	53	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-38528-E
Morrow, Louisiana, 2014 (Mermentau)	3.2 FS 2.84 SC	10.1 107	1 1	BBCH 45	42	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-38528-F
Winchester, Tillar, 2014 (CL 152)	3.2 FS 2.84 SC	10 100	1 1	BBCH 45	47	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-38528-G
Stuttgart, Arkansas, 2014 (CL 152)	3.2 FS 2.84 SC	10 104	1 1	BBCH 45	48	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-38528-H

RICE Location, Year (Variety)	Application			Growth state	DALA	Residues, mg/kg			Trial
	Formulation	g ai/100 kg seed[a] g ai/ha	No.			inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[a]	
Stuttgart, Arkansas, 2014 (CL 152)	3.2 FS 2.84 SC	10 105	1 1	BBCH 45	48	<0.01 (2)	<0.01 (2)	<0.02 (2)	: V-38528-J
Cheneyville, Louisiana, 2014 (Cheniere)	3.2 FS 2.84 SC	10.1 101	1 1	BBCH 41	48	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-38528-I
East Bernard, Texas, 2014 (Cheniere)	3.2 FS 2.84 SC	10.1 100	1 1	BBCH 45	71	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-38528-K
	3.2 FS 2.84 SC	10.1 501	1 1	BBCH 45	71	<0.01 (2)	<0.01 (2)	<0.02 (2)	
Greenville, Mississippi, 2015 (CL 153)	3.2 FS 2.84 SC	10 103	1 1	BBCH 45	41	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-38528-M
	3.2 FS 2.84 SC	10 499	1 1	BBCH 45	41	0.030, 0.020	<0.01 (2)	<0.02 (2)	
East Bernard, Texas, 2015 (CL 163)	3.2 FS 2.84 SC	10 99	1 1	BBCH 45	35	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-38528-N
					40	<0.01 (2)	<0.01 (2)	<0.02 (2)	
					44	<0.01 (2)	<0.01 (2)	<0.02 (2)	
					50	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	3.2 FS 2.84 SC	50 500	1 1	BBCH 45	40	0.020, 0.030	<0.01 (2)	0.03, 0.02	
Yuba City, California, 2017 (M-104)	3.2 FS 2.84 SC	10 105	1 1	BBCH 45	46	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-39339-A
Cheneyville, Louisiana, 2017 (Cheniere)	3.2 FS 2.84 SC	10 101	1 1	BBCH 45	56	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-39339-C
Fisk, Missouri, 2017 (CL XP4534)	3.2 FS 2.84 SC	10 101	1 1	BBCH 45	35	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-39339-D
Leland, Mississippi, 2017 (Cheniere)	3.2 FS 2.84 SC	10 86	1 1	BBCH 45	42	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-39339-E
Yuba City, California, 2017 (M-105)	3.2 FS 2.84 SC	10 103	1 1	BBCH 45	46	<0.01 (2)	<0.01 (2)	<0.02 (2)	V-39339-F

Notes:
DALA: Days after the last application.
[a] Free and conjugated, sum of A and B isomers.

Sweet corn

Seventeen residue trials were conducted on sweet corn in the United States in 2015 and 2016 growing season (Fosster, 2017; 201700217). In each trial, seeds were treated with inpyrfluxam as a FS formulation containing at a rate of 0.014 mg ai/seed inpyrfluxam, planted at a seeding rate and row spacing typical for the selected locations followed by an in furrow (spray treatment) as a SC formulation at a rate of 50 g ai/ha. (at BBCH 00). In addition, in trials V-38939-AF and -AG, an additional plot was treated at an exaggerated rate (5× the GAP) at nominally 0.070 mg ai/seed and 250 g ai/ha, respectively. In trial V-38939-AD, and –AE, the sampling dates varied as to evaluate the effect of harvest timing.

The raw agricultural commodity (RAC) samples of sweet corn were harvested at the milk stage (BBCH 75). Samples were analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (analysed separately as 1'-CH_2OH-S-2840A and B), DFPA-$CONH_2$ and conjugated forms of metabolites analysed as aglycones (1'-COOH-S-2840 and 1'-CH_2OH-S-2840) using the validated method RM-50C-1. LOQs were 0.01 mg/kg for each analyte. The combined LOQ for l'-CH_2OH-S-2840 (A and B) or 1'-COOH-S-2840 (A and B) was 0.02 mg/kg (sum of the individual LOQs). The metabolite, *N*-des-Me-DFPA was also analysed using method RM-50C-2a, with a LOQ of 0.02 mg/kg. Overall, concurrent recovery samples for inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840A, 1'-CH_2OH-S-2840B, DFPA-$CONH_2$, 1'-COOH-S-2840A, 1'-COOH-S-2840B and *N*-des-Me-DFPA in sweet corn gave mean recoveries ranging from 79.7-93.4 percent, with RSD up to 13 percent. Samples of sweet corn were stored for up to 642 days prior to analysis, whici is covered by the storage stability study (681 days in wheat grain). Results from the trials are summarized in Table 118.

Table 118 Inpyrfluxam residues in sweet corn resulting from supervised trials in the United States using seed treatment (FS formulation) followed by in furrow treatment at BBCH 00 (SC formulation) (Study 20170021)

SWEET CORN Location Year (Variety)	Application			DALA	Residues, mg/kg							Trial
	Formulation	mg ai/seed g ai/ha	No.		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[a]	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)[b]	1'-CH_2OH-S-2840 (agly)[b]	*N*-des-Me-DFPA	
Waterloo, New York, 2015 (TA 290-18)	3.2 FS 2.84 SC	0.014 50	1 1	82	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-A
North Rose, New York, 2015 (TA 290-18)	3.2 FS 2.84 SC	0.014 49	1 1	83	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-B
Elko, South Carolina, 2015 (Becks 6575HR)	3.2 FS 2.84 SC	0.014 50	1 1	70	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-C
HighSprings, Florida, 2015 (Becks 6575HR)	3.2 FS 2.84 SC	0.014 49	1 1	70	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-D
Manilla, Indiana, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	84	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-F
Delavan, Wisconsin, 2015 (TA 566-13)	3.2 FS 2.84 SC	0.014 50	1 1	93	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-G
Carlyle, Illinois, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	82	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-H
Mason, Illinois, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 49	1 1	83	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-I
Richland, Iowa, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	82	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-J
Farson, Iowa, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	86	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-K
Paso Robles, California, 2015 (TA255-18, sweet)	3.2 FS 2.84 SC	0.014 55	1 1	84	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-X

SWEET CORN Location Year (Variety)	Application			DALA	Residues, mg/kg							Trial
	Formulation	mg ai/seed g ai/ha	No.		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[a]	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)[b]	*N*-des-Me-DFPA	
Oregon City, Oregon, 2015 (TA255-18)	3.2 FS 2.84 SC	0.014 48	1 1	95	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-Z
Yakima, Washington, 2015 (TA255-18-1x)	3.2 FS 2.84 SC	0.014 50	1 1	80	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-AC
Troy, Ohio, 2016 (TA625-30)	3.2 FS 2.84 SC	0.014 50	1 1	77	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-AD
Taber, Alberta, 2016 (GS274A)	3.2 FS 2.84 SC	0.014 100	1 1	97	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-AE
Procter, Arkansas, 2016 (7400VT24F)	3.2 FS 2.84 SC	0.070 248	1 1	69	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	<0.02 (2)	V-38939-AF
Chula, Georgia, 2016 (TA790-31)	3.2 FS 2.84 SC	0.070 260	1 1	73	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	<0.02 (2)	V-38939-AG

Notes:
[a] Free and conjugated, sum of A and B isomers.
[b] Sum of isomers.

Maize

Twenty-nine residue trials were conducted on maize in the United States in 2015 and 2016 growing season (201700217). In each trial, seeds were treated with inpyrfluxam as a FS formulation containing at a rate of 0.014 mg ai/seed inpyrfluxam, planted at a seeding rate and row spacing typical for the selected locations followed by an in furrow (spray treatment) as a SC formulation at a rate of 50 g ai/ha. (at BBCH 00). In trial V-38939-V an adjuvant (crop oil concentrate (COC)) was used. In addition, in trials V-38939-V, -AF and -AG, an additional plot was treated at an exaggerated rate (5× or 10× the GAP) for a sample processing trial at nominally 0.070 mg ai/seed and 250 or 500 g ai/ha, respectively. In trial V-38939-B, and -E, the sampling dates of forage varied as to evaluate the effect of harvest timing.

The samples of maize grains were harvested at the milk stage (BBCH 75). Cut samples of the whole aerial portion of the plant (forage) were harvested at the late dough/early dent stage (BBCH 85–86). Mature corn kernels removed from cob, with a maximum of 25 percent moisture (grain) and mature dried stalks from which the whole ear (cob and grain) had been removed, containing 80–85 percent dry matter (stover) were harvested after the BBCH 95 growth stage.

Samples were analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (analysed separately as 1'-CH_2OH-S-2840A and B), DFPA-$CONH_2$ and conjugated forms of metabolites analysed as aglycones (1'-COOH-S-2840 and 1'-CH_2OH-S-2840) using the validated RM-50C-1. The LOQs were 0.01 mg/kg for each analyte. For the method RM-50C-1 for forage and stover, the LOQ was 0.02 mg/kg. The combined LOQ for l'-CH_2OH-S-2840 (A and B) or 1'-COOH-S-2840 (A and B) was 0.02 mg/kg (sum of the individual LOQs) in all matrices. The metabolite, *N*-des-Me-DFPA was also analysed using method RM-50C-2a, with a LOQ of 0.02 mg/kg in all matrices. Overall, concurrent recovery samples for inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840A, 1'-CH_2OH-S-2840B, DFPA-

$CONH_2$, 1'-COOH-S-2840A, 1'-COOH-S-2840B and *N*-des-Me-DFPA in grain, forage and stover g and RSD up to 18 percent.

Samples of corn were stored for up to 642 days prior to analysis. This period is covered by the available storage stability study (679 days in wheat grain). Samples of forage and stover were stored for up to 563 and 560 days prior to analysis, respectively, with the except of one sample of forage (B-17, analysed by method 2a) which was stored for 591 days. This period is covered by the available storage stability study (591 days in forage and 587 in stover). Results from the trials are summarized in Table 119 to Table 121.

Table 119 Inpyrfluxam residues in maize grain resulting from supervised trials in the United States of America using seed treatment (FS formulation) followed by in furrow treatment at BBCH 00 (SC formulation) (Study 201700217).

MAIZE Location, Year (Variety)	Application			DALA	Residues, mg/kg (mean)							Trial
	Formulation	mg ai/seed g ai/ha	No.		inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
Waterloo, New York, 2015 (TA 290-18)	3.2 FS 2.84 SC	0.014 50	1 1	138	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-A
North Rose, New York, 2015 (TA 290-18)	3.2 FS 2.84 SC	0.014 49	1 1	134	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-
Elko, South Carolina, 2015 (Becks 6575HR)	3.2 FS 2.84 SC	0.014 50	1 1	124	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-C
HighSprings, Florida, 2015 (Becks 6575HR)	3.2 FS 2.84 SC	0.014 49	1 1	112	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-D
Manilla, Indiana, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	133	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-F
Delavan, Wisconsin, 2015 (TA 566-13)	3.2 FS 2.84 SC	0.014 50	1 1	155	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-G
Carlyle, Illinois, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	150	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-H
Mason, Illinois, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 49	1 1	125	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-I
Richland, Iowa, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	136	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-J
Farson, Iowa, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	135	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-K
Toronto, South Dakota, 2015 (TA477-18)	3.2 FS 2.84 SC	0.014 51	1 1	147	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-L
Brookings, South Dakota, 2015 (TA475-18)	3.2 FS 2.84 SC	0.014 51	1 1	153	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-M

MAIZE Location, Year (Variety)	Application			DALA	Residues, mg/kg (mean)							Trial
	Formulation	mg ai/seed g ai/ha	No.		inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
Geneva, Minnesota, 2015 (TA477-18)	3.2 FS 2.84 SC	0.014 49	1 1	162	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-N
St. Cloud, Minnesota, 2015 (TA371-18)	3.2 FS 2.84 SC	0.014 51	1 1	145	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-O
Northwood, North Dakota, 2015 (TA080-00)	3.2 FS 2.84 SC	0.014 49	1 1	150	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-P
Louisville, Nebraska, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 51	1 1	153	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-Q
Lenexa, Kansas, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 51	1 1	152	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-R
Wyoming, Illinois, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 52	1 1	152	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-S
Milo, Illinois, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 49	1 1	152	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-T
Camp Grove, Illinois, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 50	1 1	152	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-U
Hinton, Oklahoma, 2015 (Burrus 5241GT)	3.2 FS 2.84 SC	0.014 52	1 1	127	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-V
	3.2 FS 2.84 SC	0.070 512	1 1	84	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	
Paso Robles, California, 2015 (TA255-18, sweet)	3.2 FS 2.84 SC	0.014 55	1 1	114	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-X
Oregon City, Oregon, 2015 (TA255-18)	3.2 FS 2.84 SC	0.014 48	1 1	130	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-Z
Glenboro, Manitoba, 2015 (TA255-18)	3.2 FS 2.84 SC	0.014 50	1 1	124	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-AA
Hinton, Oklahoma, 2015 (Burrus 5241GT)	3.2 FS 2.84 SC	0.070 512	1 1	84	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.02 (2)	<0.02	<0.02 (2)	V-38939-AB
Yakima, Washington, 2015 (TA255-18-1x)	3.2 FS 2.84 SC	0.014 50	1 1	179	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-AC
Troy, Ohio, 2016 (TA625-30)	3.2 FS 2.84 SC	0.014 50	1 1	143	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	-	-	<0.02 (2)	V-38939-AD
Taber, Alberta, 2016 (GS274A)	3.2 FS 2.84 SC	0.014 100	1 1	165	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	-	-	<0.02 (2)	V-38939-AE

MAIZE Location, Year (Variety)	Application			DALA	Residues, mg/kg (mean)							Trial
	Formulation	mg ai/seed g ai/ha	No.		inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
Procter, Arkansas, , 2016 (7400VT24F)	3.2 FS 2.84 SC	0.070 248	1 1	109	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	<0.02 (2)	V-38939-AF
Chula, Georgia, 2016 (TA790-31)	3.2 FS 2.84 SC	0.070 260	1 1	125	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	<0.02 (2)	V-38939-AG

Notes:
DALA: Days of the last application.

Table 120 Inpyrfluxam residues in maize forage resulting from supervised trials in the United States of America with seed treatment (FS) followed by in-furrow at BBCH00 (SC) (Study 201700217)

MAIZE FORAGE Location, Year (Variety)	Application			DALA	Residues, mg/kg (mean)							Trial
	Formulation	mg ai/seed g ai/ha	No.		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
Waterloo, New York, 2015 (TA 290-18)	3.2 FS 2.84 SC	0.014 50	1 1	113	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-A
North Rose, New York, 2015 (TA 290-18)	3.2 FS 2.84 SC	0.014 49	1 1	106	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-B
				110	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	
				114	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	
				118	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	
Elko, South Carolina, 2015 (Becks 6575HR)	3.2 FS 2.84 SC	0.014 50	1 1	80	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-C
HighSprings, Florida, 2015 (Becks 6575HR)	3.2 FS 2.84 SC	0.014 49	1 1	89	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-D
Manilla, Indiana, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	91	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-F
Delavan, Wisconsin, 2015 (TA 566-13)	3.2 FS 2.84 SC	0.014 50	1 1	113	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-G
				117	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	
				121	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	
				125	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	
Carlyle, Illinois, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	97	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-H

MAIZE FORAGE Location, Year (Variety)	Application			DALA	Residues, mg/kg (mean)							Trial
	Formulation	mg ai/seed g ai/ha	No.		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
, Mason, Illinois, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 49	1 1	92	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-I
Richland, Iowa, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	97	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-J
Farson, Iowa, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	100	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-K
Toronto, South Dakota, 2015 (TA477-18)	3.2 FS 2.84 SC	0.014 51	1 1	112	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-L
Brookings, South Dakota, 2015 (TA475-18)	3.2 FS 2.84 SC	0.014 51	1 1	124	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-M
Geneva, Minnesota, 2015 (TA477-18)	3.2 FS 2.84 SC	0.014 49	1 1	114	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-N
St. Cloud, Minnesota, , 2015 (TA371-18)	3.2 FS 2.84 SC	0.014 51	1 1	94	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-O
Northwood, North Dakota, 2015 (TA080-00)	3.2 FS 2.84 SC	0.014 49	1 1	109	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-P
Louisville, Nebraska, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 51	1 1	100	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-Q
Lenexa, Kansas, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 51	1 1	116	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-R
Wyoming, Illinois, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 52	1 1	99	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-S
Milo, Illinois, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 49	1 1	99	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-T
Camp Grove, Illinois, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 50	1 1	99	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-U
Hinton, Oklahoma, 2015 (Burrus 5241GT)	3.2 FS 2.84 SC	0.014 52	1 1	86	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-V
Paso Robles, California, 2015 (TA255-18, sweet)	3.2 FS 2.84 SC	0.014 55	1 1	93	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-X
Oregon City, Oregon, 2015 (TA255-18)	3.2 FS 2.84 SC	0.014 48	1 1	115	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-Z

MAIZE FORAGE Location, Year (Variety)	Application			DALA	Residues, mg/kg (mean)							Trial
	Formulation	mg ai/seed g ai/ha	No.		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
Glenboro, Manitoba, 2015 (TA255-18)	3.2 FS 2.84 SC	0.014 50	1 1	84	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AA
Yakima, Washington, 2015 (TA255-18-1x)	3.2 FS 2.84 SC	0.014 50	1 1	93	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AC
Troy, Ohio, 2016 (TA625-30)	3.2 FS 2.84 SC	0.014 50	1 1	102	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AD
Taber, Alberta, 2016 (GS274A)	3.2 FS 2.84 SC	0.014 100	1 1	148	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AE
Procter, Arkansas, 2016 (7400VT24F)	3.2 FS 2.84 SC	0.070 248	1 1	79	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AF
Chula, Georgia, 2016 (TA790-31)	3.2 FS 2.84 SC	0.070 260	1 1	82	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AG

Notes:
DALA: days after the last application

Table 121 Inpyrfluxam residues in maize stover resulting from supervised trials in the United States of America conducted with seed treatment followed by in-furrow at BBCH00 (Report 201700217)

MAIZE STOVER Location Year (Variety)	Application[a]			DALA	Residues, mg/kg (mean)							Trial
	Formulation	mg ai/seed g ai/ha	No.		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
New York, 2015 (TA 290-18)	3.2 FS 2.84 SC	0.014 50	1 1	138	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-A
North Rose, New York, , 2015 (TA 290-18)	3.2 FS 2.84 SC	0.014 49	1 1	134	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-B
Elko, South Carolina, 2015 (Becks 6575HR)	3.2 FS 2.84 SC	0.014 50	1 1	124	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-C
HighSprings, Florida, 2015 (Becks 6575HR)	3.2 FS 2.84 SC	0.014 49	1 1	112	<0.01 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-D
Manilla, Indiana, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	133	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-F
Delavan, Wisconsin, 5, 2015 (TA 566-13)	3.2 FS 2.84 SC	0.014 50	1 1	155	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-G

MAIZE STOVER Location Year (Variety)	Application[a]			DALA	Residues, mg/kg (mean)							Trial
	Formulation	mg ai/seed g ai/ha	No.		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
Carlyle, Illinois, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	150	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-H
Mason, Illinois, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 49	1 1	125	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-I
Richland, Iowa, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	136	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-J
Farson, Iowa, 2015 (Burrus 5Z41GT)	3.2 FS 2.84 SC	0.014 50	1 1	135	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-K
Toronto, South Dakota, 2015 (TA477-18)	3.2 FS 2.84 SC	0.014 51	1 1	147	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-L
Brookings, South Dakota, 2015 (TA475-18)	3.2 FS 2.84 SC	0.014 51	1 1	153	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-M
Geneva, Minnesota, 2015 (TA477-18)	3.2 FS 2.84 SC	0.014 49	1 1	162	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-N
St. Cloud, Minnesota, 2015 (TA371-18)	3.2 FS 2.84 SC	0.014 51	1 1	145	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-O
Northwood, North Dakota, 2015 (TA080-00)	3.2 FS 2.84 SC	0.014 49	1 1	150	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-P
Louisville, Nebraska, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 51	1 1	153	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-Q
Lenexa, Kansas, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 51	1 1	152	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-R
Wyoming, Illinois, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 52	1 1	152	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-S
Milo, Illinois, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 49	1 1	152	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-T
Camp Grove, Illinois, 2015 (TA566-31)	3.2 FS 2.84 SC	0.014 50	1 1	152	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-U
Hinton, Oklahoma, 2015 (Burrus 5241GT)	3.2 FS 2.84 SC	0.014 52	1 1	127	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-V
Paso Robles, California, 2015 (TA255-18, sweet)	3.2 FS 2.84 SC	0.014 55	1 1	114	<0.02	<0.02	<0.04	<0.02	<0.04	<0.04	0.027	V-38939-X

MAIZE STOVER Location Year (Variety)	Application[a]			DALA	Residues, mg/kg (mean)							Trial
	Formulation	mg ai/seed g ai/ha	No.		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
Oregon City, Oregon, 2015 (TA255-18)	3.2 FS 2.84 SC	0.014 48	1 1	121	<u><0.02</u> (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-Z
Glenboro, Manitoba, 2015 (TA255-18)	3.2 FS 2.84 SC	0.014 50	1 1	124	<u><0.02</u> (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AA
Yakima, Washington, 2015 (TA255-18-1x)	3.2 FS 2.84 SC	0.014 50	1 1	179	<u><0.02</u> (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AC
Troy, Ohio, 2016 (TA625-30)	3.2 FS 2.84 SC	0.014 50	1 1	143	<u><0.02</u> (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AD
Taber, Alberta 2016 (GS274A)	3.2 FS 2.84 SC	0.014 100	1 1	165	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AE
Corn Procter, Arkansas, 2016 (7400VT24F)	3.2 FS 2.84 SC	0.070 248	1 1	109	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AF
Chula, Georgia, 2016 (TA790-31)	3.2 FS 2.84 SC	0.070 260	1 1	125	<0.02 (2)	<0.02 (2)	<0.04 (2)	<0.02 (2)	<0.04 (2)	<0.04 (2)	<0.02 (2)	V-38939-AG

Notes:
DALA: Days of the last application.

Peanut

Thirteen residue trials were conducted on peanuts in the United States in 2015 growing season (Bietter, 2017: 201700318) In each trial, peanuts were treated with two foliar applications of inpyrfluxam as a SC formulation at a rate of 99–104 g ai/ha per application. The first application was made at approximately 61 days before normal harvest (DBH) and the second at 40 DBH. Adjuvant (non-ionic surfactant (NIS), crop oil concentrate (COC) or silicone) added at nominally 0.063–0.50 percent.

In trials TPR-0066-D and -L two additional plots were treated at 5× and 7.5× the GAP rate. In trials TPR-0066-C and -H, the sampling dates varied as to evaluate the effect of harvest timing.

Peanut samples were collected at normal commercial harvest, 21–42 days after the last application for harvest trials and 29–46 days after last application for the decline trial. Peanuts (nutmeat and hulls) were dug and hay was cut at normal harvest (39–42 days after the last application), and allowed to dry in the field for 0–16 days, as needed, before sample collection. The peanuts (nutmeat and hulls) were collected from at least 24 plants and allowed to dry in the field until commercially acceptable (BBCH 29). The hulls (shell) were removed from the nutmeat (nutmeat), and only the nutmeat was collected for analysis. Samples were analysed for inpyrfluxam and its major metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (analysed as 1'-CH_2OH-S-2840A and B), DFPA-$CONH_2$ and conjugated forms of metabolites analysed as the aglycones of 1'-COOH-S-2840 and 1'-CH_2OH-S-2840 using the validated method RM-50C-1. The LOQ was 0.01 mg/kg for each analyte except for 1'-COOH-S-2840-A (for which LOQ was 0.013 mg/kg). The combined LOQ for l'-CH_2OH-S-2840 (A and B) or 1'-COOH-S-2840 (A and B) was 0.02 mg/kg or 0.023 mg/kg respectively (sum of the individual LOQs). For the determination of *N*-des-Me-

DFPA, method RM-50C-2a was used, with a LOQ of 0.02 mg/kg. Overall, concurrent recovery samples for inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840A, 1'-CH_2OH-S-2840B, DFPA-$CONH_2$, 1'-COOH-S-2840A (agly), 1'-COOH-S-2840B (agly) and *N*-des-Me-DFPA in nutmeat and hay gave results ranging from 64 to 117 percent recovery, with relative standard deviations up to 17 percent. Only for the metabolite 1'-CH_2OH-S-2840A recoveries ranged from 99 to 136 percent, however mean recovery of 13 samples was 114 percent with RSD of 8 percent, thus the results can be considered valid.

Samples of peanut nutmeat and hay were stored for up to 465 days prior to analysis. This period is covered by the available storage stability study (683 days in soya bean). Results from the trials are summarized in Table 122 and Table 123.

Table 122 inpyrfluxam residues in peanut nutmeat resulting from supervised trials in the United States of America in 2015 using 2.84 SC formulation (Study 201700318)

PEANUT Location (Variety)	Application		Growth stage[a]	DALA	Residues, mg/kg (mean)							Trial
	g ai/ha	No.			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
Abbeville, Georgia, (Georgia 06G)	103 101	2	BBCH 77	39	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-A
Lenox, Georgia, (Georgia 06G)	104 102	2	BBCH 79	39	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-B
Blackville, South Carolina, (Georgia 06G)	101 100	2	BBCH 75	29	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-C
				35	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	
				40	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	
				46	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	
Elko, South Carolina, (Bailey) (10 Km from trial C)	100 100	2	BBCH 75	40	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-D
	500 497	2	BBCH 75	40	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	<0.01 (2)	
	377 376 376 372	4	BBCH 77	21	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	<0.01 (2)	
Suffolk, Virginia, (Bailey)	101 100	2	BBCH 81	38	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-E
Windsor, Virginia, (Bailey)	102 101	2	BBCH 79	39	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-F
Norman Park, Georgia, (Georgia 06G)	99 101	2	BBCH 77	39	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-G
Chula, Georgia, (Georgia 06G)	99 99	2	BBCH 77	31	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-H
				35	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	
				40	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	
				45	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	

PEANUT Location (Variety)	Application		Growth stage[a]	DALA	Residues, mg/kg (mean)							Trial
	g ai/ha	No.			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
Lee, Florida, (Florida 09B)	101 101	2	BBCH 77	42	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-I
Hinton, Oklahoma, (Tamnut 0L06)	102 99		BBCH 82	39	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-J
Dill City, Oklahoma (Tamnut 0L06)	99 103	2	BBCH 81	41	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-K
Peanut Wellington, Texas, (Tamrun)	100 101	2	BBCH 83	40	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	<0.023 (2)	<0.02 (2)	-	TPR-0066-L
	509 505	2	BBCH 83	40	0.020, 0.019 (0.020)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	<0.02 (2)	
	383 374 374 376	4	BBCH 87	21	0.012, 0.035; (0.024)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.023 (2)	<0.02 (2)	<0.02 (2)	
Wellington, Texas, (Tamrun)	383 374 374 376	4	-	21	<0.01 (3)	<0.01 3)	<0.02 (3)	<0.01 (3)	<0.023 (3)	<0.02 (3)	<0.02 (3)	TPR-0066-M[b]

Notes:
DALA: Days of the last application.
[a] At the last application.

Table 123 Inpyrfluxam residues in peanut hay resulting from supervised trials in the United States of America in 2015 using 2.84 SC formulation (Study 201700318)

PEANUT HAY Location (Variety)	Application		Growth state[a]	DALA	Residues, mg/kg (mean)							Trial
	g ai/ha	No.			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[a]	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
Abbeville, Georgia, (Georgia 06G)	103 101	2	BBCH 77	39	1.41, 1.05 (1.23)	0.102, 0.077 (0.090)	0.879, 0.519 (0.699)	<0.01, <0.01 (<0.01)	0.150, 0.109 (0.129)	0.914, 0.678 (0.796)	0.745, 0.507 (0.626)	TPR-0066-A
Lenox, Georgia, (Georgia 06G)	104 102	2	BBCH 79	39	0.198, 0.235 (0.217)	0.058, 0.064 (0.061)	0.018, 0.019 (0.019)	<0.01, <0.01 (<0.01)	0.024, 0.172 (0.098)	0.763, 0.831 (0.797)	0.806, 0.629 (0.718)	TPR-0066-B
Blackville, South Carolina, (Georgia 06G)	101 100	2	BBCH 75	29	0.218, 0.247 (0.233)	0.053, 0.062 (0.058)	0.024, 0.033 (0.029)	<0.01, <0.01 (<0.01)	0.028, 0.030 (0.029)	0.352, 0.446 (0.339)	0.319, 0.270 (0.295)	TPR-0066-C
				35	0.216, 0.245 (0.231)	0.053, 0.060 (0.057)	0.015, 0.020 (0.017)	<0.01, <0.01 (<0.01)	0.024, 0.034 (0.029)	0.299, 0.339 (0.319)	0.234, 0.268 (0.251)	

PEANUT HAY Location (Variety)	Application		Growth state[a]	DALA	Residues, mg/kg (mean)							Trial
	g ai/ha	No.			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[a]	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
				40	0.239, 0.197 (0.218)	0.055, 0.048 (0.052)	0.032, 0.029 (0.031)	<0.01, <0.01 (<0.01)	0.042, 0.039 (0.040)	0.274, 0.230 (0.252)	0.202, 0.174 (0.188)	
				46	0.170, 0.206 (0.188)	0.044, 0.051 (0.048)	0.073, 0.088 (0.081)	<0.01, <0.01 (<0.01)	0.052, 0.060 (0.056)	0.198, 0.216 (0.207)	0.160, 0.191 (0.176)	
Elko, South Carolina, , (Bailey)	100 100	2	BBCH 75	40	0.433, 0.391 (0.412)	0.042. 0.044 (0.043)	0.019, 0.024 (0.022)	<0.01, <0.01 (<0.01)	0.024, 0.028 (0.026)	0.462, 0.483 (0.473)	0.481, 0.442 (0.462)	TPR-0066-D
	500 497	2	BBCH 75	40	1.17, 0.939 (1.05)	0.214, 0.183 (0.199)	0.065, 0.057 (0.061)	<0.01, <0.01 (<0.01)	0.087, 0.096 (0.092)	1.43, 1.55 (1.49)	0.972, 1.07 (1.02)	
Suffolk, Virginia, (Bailey)	101 100	2	BBCH 81	38	0.148, 0.120 (0.134)	0.032, 0.026 (0.029)	0.068, 0.057 (0.063)	<0.01, <0.01 (<0.01)	0.077, 0.058 (0.068)	0.245, 0.213 (0.229)	0.246, 0.157 (0.202)	TPR-0066-E
Windsor, Virginia, (Bailey)	102 101	2	BBCH 79	39	0.445, 0.607 (0.526)	0.150, 0.200 (0.175)	0.024, 0.031 (0.028)	<0.01, <0.01 (<0.01)	0.021, 0.025 (0.023)	0.211, 0.330 (0.271)	0.163, 0.314 (0.239)	TPR-0066-F
Norman Park, Georgia, (Georgia 06G)	99 101	2	BBCH 77	39	0.684, 0.777 (0.731)	0.060, 0.069 (0.065)	0.173, 0.206 (0.190)	<0.01, <0.01 (<0.01)	0.047, 0.049 (0.048)	1.46, 1.74 (1.60)	0.320, 0.463 (0.392)	TPR-0066-G
Chula, Georgia, (Georgia 06G)	99 99	2	BBCH 77	31	0.194, 0.269 (0.232)	0.040, 0.048 (0.044)	0.203, 0.173 (0.188)	<0.01, <0.01 (<0.01)	0.016, 0.027 (0.021)	0.152, 0.337 (0.2454)	0.174, 0.254 (0.214)	TPR-0066-H
				35	0.257, 0.269 (0.263)	0.048, 0.051 (0.050)	0.080, 0.096 (0.088)	<0.01, <0.01 (<0.01)	0.042, 0.035 (0.038)	0.432, 0.461 (0.447)	0.250, 0.260 (0.255)	
				40	0.159, 0.163 (0.161)	0.025, 0.029 (0.027)	0.091, 0.146 (0.119)	<0.01, <0.01 (<0.01)	0.038, 0.039 (0.039)	0.108, 0.108 (0.108)	0.079, 0.130 (0.105)	
				45	0.225, 0.278 (0.252)	0.041, 0.050 (0.046)	0.060, 0.063 (0.062)	<0.01, <0.01 (<0.01)	0.096, 0.074 (0.085)	0.322, 0.401 (0.362)	0.237, 0.179 (0.208)	
Lee, Florida, (Florida 09B)	101 101	2	BBCH 77	42	0.110, 0.082 (0.096)	0.017, 0.017 (0.017)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	0.067, 0.042 (0.055)	0.143, 0.091 (0.117)	TPR-0066-I
Hinton, Oklahoma, (Tamnut 0L06)	102 99	2	BBCH 82	39	0.314, 0.361 (0.338)	0.048, 0.053 (0.051)	0.028, 0.050 (0.039)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	0.777, 0.937 (0.857)	0.305, 0.328 (0.317)	TPR-0066-J
Dill City, Oklahoma, (Tamnut 0L06)	99 103	2	BBCH 81	41	0.083, 0.082 (0.083)	0.023, 0.024 (0.024)	<0.02, <0.02 (<0.02	<0.01, <0.01 (<0.01)	0.023, <0.023 (0.023)	0.160, 0.157 (0.159)	0.145, 0.202 (0.174)	TPR-0066-K
Wellington, Texas, (Tamrun)	100 101	2	BBCH 83	40	0.435, 0.409 (0.422)	0.048, 0.046 (0.047)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)	0.029, 0.025 (0.027)	0.033, 0.032 (0.033)	0.039, 0.046 (0.043)	TPR-0066-L

PEANUT HAY Location (Variety)	Application		Growth state[a]	DALA	Residues, mg/kg (mean)							Trial
	g ai/ha	No.			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[a]	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA	
	509 505	2	BBCH 83	40	0.560, 0.554 (0.557)	0.512, 0.484 (0.498)	0.224, 0.234 (0.229)	0.036, 0.036 (0.036)	0.221, 0.241 (0.231)	0.487, 0.567 (0.527)	0.303, 0.383 (0.343)	

Notes:
DALA: Days of the last application.
[a] At the last application.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Storage

Inpyrfluxam is not intended for use in stored products.

Nature of the residue during processing

The Meeting received three high temperature hydrolysis studies of inpyrfluxam and its metabolites 3'-OH-S-2840 and 1'-CH_2OH-S-2840. In all studies the spiked buffer solutions were put into conditions simulating pasteurisation (90 °C, pH 4, 20 minutes); baking, brewing, boiling (100 °C, pH 5, 60 minutes); and sterilisation (120 °C, pH 6, 20 minutes). Prior to and after processing, an aliquot from each sample was collected and analysed by LSC for total radioactivity, by radio-HPLC for determination of hydrolysis products and chiral chromatography for the determination of R/S isomers.

High-temperature hydrolysis of inpyrfluxam was investigated by Freelander (2016, TPM-0022). In the study, [Pyrazolyl-4-^{14}C]inpyrfluxam was spiked, in duplicate, into buffered solutions at a target concentration of 1 mg/L. Mass balance of radioactivity after processing was 100.5, 103.2, and 100.7 percent for 90 °C/pH 4, 100 °C/pH 5, and 120 °C/pH 6, respectively. [^{14}C]inpyrfluxam did not degrade over the incubation period thus, inpyrfluxam is considered to be stable under high-temperature hydrolysis conditions. Chiral analysis showed the *R*-isomer to account for 100 percent of the residue at all time points (Table 124).

Table 124 High-temperature hydrolysis radio-HPLC results for inpyrfluxam

Process represented	Test conditions	Analyte	Total Recovery[a] (% of applied dose)		Mass balance (%)[b]
			Before incubation	After incubation	
Pasteurisation	pH 4, 90 °C, 20 minutes	inpyrfluxam (*R*-isomer)	97.1, 97.7 (97.4)	97.5, 98.2 (97.9)	100.5
		S-2940 (*S*-isomer)	-	-	n.a.
Baking/brewing/boiling	pH 5, 100 °C, 60 minutes	inpyrfluxam (*R*-isomer)	93.9, 95.9 (94.9)	97.1, 98.7 (97.9)	103.2
		S-2940 (*S*-isomer)	-	-	n.a.
Sterilisation	pH 6, 120 °C, 20 minutes	inpyrfluxam (*R*-isomer)	94.9, 98.9 (96.9)	97.4, 97.7 (97.6)	100.7
		S-2940 (*S*-isomer)	-	-	n.a.

Notes:
Value in parentheses = average of two determinations.

n.a. = Not applicable.
[a] %AR (all residues comprised the *R*-isomer of inpyrfluxam).
[b] Compared to 'time zero'.

High-temperature hydrolysis of 3'-OH-S-2840 was investigated by Lamond (2018, TPM-0054). In the study, [Pyrazolyl-4-^{14}C] 3'-OH-S-2840 was spiked, in duplicate, into buffered solutions at a target concentration of 1 mg/L. 3'-OH-S-2840 degraded to form 3'-OH-S-2840 dehydrate at an average of 13.0 percent TRR (0.133 mg/kg) under pasteurization, 9.0 percent TRR (0.092 mg/kg) under brewing, baking and boiling and 1.7 percent TRR (0.017 mg/kg) under sterilisation. In all processed a small number of minor degradation products were also observed in HPLC and TLC analyses (Table 125).

Table 125 High-temperature hydrolysis radio-HPLC results for 3'-OH-S-2840

Process represented	Test conditions	Analyte	Total Recovery [a] % of applied dose (mg/L)	
			Before incubation	After incubation
Pasteurisation	pH 4, 90 °C, 20 minutes	3'-OH-S-2840	97.8 (0.999)	82.8 (0.846)
		3'-OH-S-2840 dehydrate	ND	13.0 (0.133)
		Unknown [c]	1.4 (0.015)	2.9 (0.029)
Baking/brewing/boiling	pH 5, 100 °C, 60 minutes	3'-OH-S-2840	98.5 (1.005)	87.6 (0.895)
		3'-OH-S-2840 dehydrate	ND	9.0 (0.092)
		Unknown [d]	1.0 (0.005)	2.9 (0.030)
Sterilisation	pH 6, 120 °C, 20 minutes	3'-OH-S-2840	99.0 (1.011)	96.7 (0.987)
		3'-OH-S-2840 dehydrate	ND	1.7 (0.017)
		Unknown [e]	0.2 (0.002)	1.0 (0.010)

Notes:
ND: Not detected (<0.21% TRR).
[a] Average of duplicate samples.
[c] Unknown degradants were maximally comprised of 8 components in which the largest peak was quantified to be 1.9% TRR (0.019 mg/L).
[d] Unknown degradants were maximally comprised of 6 components in which the largest peak was quantified to be 2.8% TRR (0.029 mg/L).
[e] Unknown degradants were maximally comprised of 2 components in which the largest peak was quantified to be 1.0% TRR (0.010 mg/L).

High-temperature hydrolysis of 1'-CH_2OH-S-2840 was investigated by Gilbert (2017, TPM-0055). In the study, [Pyrazolyl-4-^{14}C]1'-CH_2OH-S-2840 (supplied as isomers A and B) was spiked, in duplicate, into buffered solutions at a target concentration of 1 mg/L. Degradation products were not detected in any buffer samples, thus 1'-CH_2OH-S-2840 (isomers A and B) are considered to be stable under high-temperature hydrolysis conditions (Table 126).

Table 126 High-temperature hydrolysis radio-HPLC results for 1'-CH_2OH-S-2840

Process represented	Test conditions	Analyte	Total Recovery [a] % of applied dose (mg/L)	
			Before incubation	After incubation
Pasteurisation	pH 4, 90 °C, 20 minutes	1'-CH_2OH-S-2840A	48.7 (0.498)	48.0 (0.491)
		1'-CH_2OH-S-2840B	51.0 (0.522)	51.8 (0.530)
		Total 1'-CH_2OH-S-2840	99.7 (1.02)	99.8 (1.02)
Baking/brewing/boiling	pH 5, 100 °C, 60 minutes	1'-CH_2OH-S-2840A	49.8 (0.51)	49.3 (0.505)
		1'-CH_2OH-S-2840B	49.9 (0.51)	50.4 (0.516)
		Total 1'-CH_2OH-S-2840	99.7 (1.02)	99.8 (1.02)
Sterilisation	pH 6, 120 °C, 20	1'-CH_2OH-S-2840A	49.2 (0.50)	48.8 (0.50)

Process represented	Test conditions	Analyte	Total Recovery [a] % of applied dose (mg/L)	
			Before incubation	After incubation
Pasteurisation	pH 4, 90 °C, 20 minutes minutes	1'-CH_2OH-S-2840A	48.7 (0.498)	48.0 (0.491)
		1'-CH_2OH-S-2840B	51.0 (0.522)	51.8 (0.530)
		Total 1'-CH_2OH-S-2840	99.7 (1.02)	99.8 (1.02)
		1'-CH_2OH-S-2840B	50.6 (0.518)	50.9 (0.521)
		Total 1'-CH_2OH-S-2840	99.8 (1.02)	99.8 (1.02)

Note:
[a] Average of duplicate samples.

Residues after processing

Apple

A single sample of apples from a residue trial (V-38516-R) conducted at an exaggerated rate and described above was used to investigate the effect of juicing process on inpyrfluxam residues. In the study by Bitter (2017, 201700086 or TPR-0022) apple samples were washed and reduced to apple pulp using a fruit press. The pulp was then transferred to a steam-jacketed kettle and heated with low pressure steam to approximately 50 °C before pectic enzyme was added. After 2 hours, the treated pulp was pressed, and the wet pomace and juice were collected. The juice was filtered to remove solids. A pasteurization step was not included.

Samples were homogenized in the presence of dry ice and analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (analysed separately as 1'-CH_2OH-S-2840A and 1'-CH_2OH-S-2840B) and DFPA-$CONH_2$ using Method RM-50C-1. Concurrent recoveries ranged from 68 to 95 percent with a maximum RSD of 14 percent. Samples were stored frozen (-20 °C) for up to 155 days before analysis, which is covered by the storage stability study (514 days in apples).

Results from the trials are summarized in Table 127. Residues of inpyrfluxam, concentrated in wet pomace (PF = 2.7) but were diluted in fresh juice (PF = 0.125).

Table 127 Inpyrfluxam residues in apple and processed commodities resulting from trial V-38516-R in the United States of America (Report 201700086)

Trial information (variety)	DALA	Commodity	Residues, mg/kg [mean]			
			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[a]	DFPA-$CONH_2$
2 applications of SC formulation: 490/493 g ai/ha, BBCH 69/79 (Jonagold)	35	Fruit (27.14 kg)	0.078, 0.081 [0.080]	<0.01 (2)	<0.02 (2)	<0.01 (2)
	18	Juice (19.32 kg)	0.01, 0.009, 0.009 [0.010]	<0.01 (2)	<0.02 (2)	<0.01 (2)
		PF	**0.125**			
	18	Wet pomace (5.16 kg)	0.24, 0.23, 0.18 [0.22]	0.012, 0.011, <0.01 [0.011]	<0.02 (3)	<0.01 (3)
		PF	**2.7**			

Soya bean

A single sample of soya bean from a residue trial (V-38537-V) conducted at an exaggerated rate was used to investigate the effect of oil production process on inpyrfluxam residues. In the study by Foster (2017,

201700156), soya bean seeds processed into hulls, meal and refined oil in a manner reflecting commercial production. For the production of hulls, whole soybeans were fed into a roller mill and were separated using an aspirator and screen cleaner into hulls and kernels. For meal production, kernel material was heated and flaked using a flaking roll. The flakes were extruded and converted into collets by direct steam injection and compression. The collets were then ground in a disc mill and dried before being placed in a batch extractor for repeated extraction with hexane at 49–60 °C. The extracted ground collets were toasted by direct steam injection and the meal was collected after sieving.

For the refined oil production, crude oil and hexane (collected from the extrusion and extraction procedures) were separated *in vacuo.* The crude oil fraction was filtered and neutralised with 14° Baumé sodium hydroxide. The neutralised oil was centrifuged and alkaline refined oil removed and filtered. The refined oil was then bleached before deodorisation under vacuum at 220–230 °C in a steam bath. On cooling, a 0.5 percent citric acid solution was added and the deodorised oil (refined oil) was collected for analysis.

Samples were homogenised in the presence of dry ice and analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840 and 1'-CH_2OH-S-2840 (analysed separately as 1'-CH_2OH-S-2840A and 1'-CH_2OH-S-2840B) using Method RM-50C-1 and *N*-des-Me-DFPA using Method RM-50C-2a. Concurrent recoveries across all matrices (meal, hulls, oil), analytes and fortification levels ranged from 67 to 126 percent with a maximum relative standard deviation of 9 percent. Samples were stored at -20 °C for up to 303 days before analysis. This period is covered by the available storage stability study (683 days in soya bean).

Results from the trials are summarized in Table 128. Residues in in refined oil were 0.012 mg/kg, but as no residues of inpyrfluxam was detected in the seed (< 0.01 mg/kg), no processing factors can be derived.

Table 128 Inpyrfluxam residues in soya bean and processed commodities resulting from trial V-38537-V in the United States of America (Report 2017000156)

Trial information (variety)	Commodity	Residues, mg/kg						
		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA
1 seed treatment (50 g ai/100 kg seed) *plus* 2 foliar applications (497/506 g ai/ha) 41 DALA (Steyer 2702R2)	Seed (26.5 kg)	<0.01 (2)	<0.01 (2)	<0.02 (2)	<0.01 (2)	<0.02 (2)	<0.02 (2)	0.13 (3)
	Meal (19.6 kg)	<0.01 (2)	<0.01 (2))	<0.02 (2)	-	-	-	0.11, 0.12, 0.15
	Hulls	<0.01 (2)	<0.01 (2))	<0.02 (2)	-	-	-	0.07, 0.06, 0.06
	Refined oil (1.79 kg)	0.011, 0.012, 0.012	<0.01 (3)	<0.02 (3)	-	-	-	<0.01 (3)

Sugar beet

As single sample of sugar beets from a residue trial (V-38533-N) conducted at an exaggerated rate was used to investigate the effect of sugar and molasses production process on inpyrfluxam residues. In the study by Bitter (2017, 201700098) beets were sliced and sugar was extracted in steam-jacketed kettles using a mixture of water and pulp press water at 60–65 °C. The extracted pulp was pressed to recover the sugar solution and a portion of the solution was returned to the kettles. The pressed pulp was dried and milled before storage (dried pulp). Raw juice was purified in a steam-jacketed kettle using lime and

carbon dioxide and the precipitates were coagulated using settling aid. Following filtration, the clarified thin juice was concentrated to thick juice and filtered before returning to the kettles, filtering again and storing the molasses for analysis. The washed sugar was removed from the filter and dried using hot air before storage.

Samples were homogenised in the presence of dry ice and analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (analysed separately as 1'-CH_2OH-S-2840A and B), DFPA-$CONH_2$ and conjugated forms of metabolites analysed as aglycones (1'-COOH-S-2840 and 1'-CH_2OH-S-2840) using the validated LC-MS/MS method RM-50C-1. The LOQ was 0.01 mg/kg for each analyte. Concurrent recoveries across all matrices (sugar, dried pulp, molasses), analytes and fortification levels ranged from 73 to 126 percent with a maximum relative standard deviation of 13 percent. Samples were stored frozen (-20 °C) for up to 361 days before analysis. This period is covered by the available storage stability study (681 days in cucumbers).

Results from the trials are summarized in Table 129. Residues of inpyrfluxam were detected in dried pulp and molasses (0.02-0.03 mg/kg), but as residues in the roots were < 0.01 mg/kg, no processing factors can be derived.

Table 129 Inpyrfluxam residues in sugar beet and processed commodities resulting from trial V-38533-N in the United States of America (Report 201700098)

Application (variety)	DALA	Commodity	Residues, mg/kg						
			Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[b]	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)[b]	1'-CH_2OH-S-2840 (agly)[b]	*N*-des-Me-DFPA
1 seed treatment (0.5 g ai/100 kg seed) *plus* 2 foliar applications (505/409 g ai/ha) (SX1521NRR)	51	Root (128.4 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	<0.01 (3)	<0.023 (3)	<0.02 (3)	<0.01 (3)
	51	Sugar (1.8 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	<0.01 (3)	<0.023 (3)	<0.02 (3)	<0.01 (3)
	51	Dried pulp (1.53 kg)	0.03 (3)	<0.01 (3)	<0.02 (3)	<0.01 (3)	<0.023 (3)	0.024, 0.022 (2)	<0.01 (3)
	51	Molasses (5.92 kg)	0.02 (2), 0.03	<0.01 (3)	<0.02 (3)	<0.01 (3)	0.140, 0.143, 0.142	0.069, 0.074, 0.069	<0.01 (3)

Maize

A single sample of maize grain from the residue trial (V-15-38939) conducted at an exaggerated rate was used to investigate the effect of flour, grits, meal, starch and oil (wet and dry milled) production process on inpyrfluxam residues.

In the study by Foster (2017, TPR-0059) corn grains were cleaned by aspiration and screening and went through a dry and wet milling process.

In the dry milling process, grain was fed into a disc mill to crack the kernel. The corn stock was dried and screened to separate the bran, germ and large grits from the grits, meal and flour. These three fractions were further screened by sieving. The bran, germ and large grits were screened again and material above the screen was aspirated to separate the hull from germ with attached hull and endosperm. The germ fraction was milled and screened and the material above the screen was aspirated to remove hull from germ. The material that passed through the screen (large grits and detached germ) was passed over a gravity separator to separate the germ from large grits. The grits, meal and flour fractions were collected for frozen storage and the germ material was flaked in a flaking roll. Flaked kernel material was extracted three times with hexane in a batch extractor at 49–60 °C. After each extraction, the crude oil/hexane and germ flakes were collected. The oil and hexane were separated *in vacuo* and the oil was filtered before alkaline refining with 16° Baumé sodium hydroxide. The neutralised

oil was isolated via centrifugation and filtered before bleaching under vacuum at 85–100 °C and further filtration. The bleached oil was deodorised in a steam bath at 220–230 °C under vacuum and cooled, with addition of 0.5 percent citric acid. The refined oil was then collected for analysis.

In the wet milling process, the grain was steeped in water containing 0.1–0.2 percent sulphur dioxide at 49–54 °C for 22–48 hours. The whole corn was milled and the germ and hull removed using a hydroclone. The germ and hull were then dried and separated using aspiration and screening. The cornstock remaining after milling was screened and the process water was separated into starch and gluten via centrifugation. The starch fraction was dried prior to collection for analysis and the germ samples were heated, flaked and pressed in an expeller to generate presscake (containing residual crude oil) and crude oil. The presscake was extracted three times with hexane in the same manner as for the dry milling process to generate crude oil/hexane and germ cake fractions. The crude oil was collected by vacuum evaporation and heated to evaporate the remaining hexane. The crude oil fractions were filtered, alkali refined, bleached and deodorised using the same method as used for the dry milling procedure.

Samples were homogenised in the presence of dry ice and analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH2OH-S-2840 (analysed separately as 1'-CH2OH-S-2840A and B), DFPA-CONH2 and conjugated forms of metabolites analysed as aglycones (1'-COOH-S-2840 and 1'-CH2OH-S-2840) using the validated LC-MS/MS method RM-50C-1. The LOQ was 0.01 mg/kg for each analyte. Concurrent recoveries across all matrices (flour, grits, meal, starch and oil), analytes and fortification levels ranged from 72 to 123 percent with a maximum RSD of 13 percent. Samples were stored frozen (-20 °C) for up to 319 days before analysis. This period is covered by the available storage stability study in maize grain.

Results are summarized in Table 130. As residues of parent were not detected in the RAC and all processed samples of corn, no processing factors can be derived.

Table 130 Inpyrfluxam residues in maize grain and processed commodities resulting from trial V-38939-AB in the United States of America (Study 201700217)

Application (Variety)	Commodity	Residues, mg/kg						
		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)	1'-CH_2OH-S-2840 (agly)	*N*-des-Me-DFPA
One seed treatment (0.070 mg ai/seed) Plus One foliar application (512 g ai/ha) 84 DALA (Burrus 5241GT)	Grain (265.1 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	<0.01 (3)	<0.02 (3)	<0.02	<0.02, (3)
	Flour (2.08 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	-	-	-	<0.02 (3)
	Grits (8.6 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	-	-	-	<0.02 (3)
	Meal (9.4 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	-	-	-	<0.02 (3)
	Oil, dry milled (0.77 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	-	-	-	<0.02 (3)
	Oil, wet milled (0.78 kg)	<0.01 (2)	<0.01 (2)	<0.02 (2)	-	-	-	<0.02 (3)
	Starch (53.6 kg)	<0.01 (3)	<0.01 (2)	<0.02 (2)	-	-	-	<0.02 (3)

Rice

As single sample of rice from a residue trial (V-38528) conducted at an exaggerated rate was used to investigate the effect of polished rice production process on inpyrfluxam residues. In the study by Bitter

(2017, 201700341), rough rice grain samples were cleaned by aspiration and screening. The rough rice was milled, grains were hulled and the hulls were separated from the brown rice by aspiration before being placed into frozen storage. The brown rice was then milled into white milled rice (polished rice) and bran by friction. The bran was then separated from the milled rice using air injected into the milling chamber and was sieved to remove rice and hull particulates.

Samples were homogenised in the presence of dry ice and analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840 and 1'-CH_2OH-S-2840 (analysed separately as 1'-CH_2OH-S-2840A and B), using the validated Valent LC-MS/MS method RM-50C-1. Concurrent recoveries across all matrices (sugar, dried pulp, molasses), analytes and fortification levels ranged from 73 to 124 percent with a maximum relative standard deviation of 21 percent. Samples were stored frozen (-20 °C) for up to 174 days before analysis. This period is covered by the available storage stability study (679 days in wheat grain).

Results from the trials are summarized in Table 131. Residues of inpyrfluxam were found in hussl and bran, but residues in grain were <LOQ, no processing factors can be derived.

Table 131 Inpyrfluxam residues in rice grain and processed commodities resulting from trial V-38528-Q in the United States of America (Study 201700341)

Variety Application	Commodity	Residues, mg/kg					
		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[b]	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)[b]	1'-CH_2OH-S-2840 (agly)[b]
CL 163 One seed treatment (50 g ai/100 seed) *plus* One foliar application (500 g ai/ha) DALA=36	Grain (105.8 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	<0.01 (3)	<0.02 (3)	<0.02 (3)
	Hulls (3.5 kg)	0.047, 0.047, 0.034	<0.01 (3)	0.067, 0.068, 0.062;	-	-	-
	Bran (1.7 kg)	0.012, 0.014, 0.014	<0.01 (3)	<0.02 (3)	-	-	-
	Polished rice (6.9 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	-	-	-

Peanut

A single sample of peanuts from a residue trial (V-38942-M) conducted in an exaggerated rate was used to investigate the effect of oil production process on inpyrfluxam residues. In the study by Bitter (2017, 201700318) nutmeats were heated and pressed to liberate a portion of crude oil from the presscake. The presscake was then ground in a disc mill to remove the remaining oil before being extracted three times with hexane in a batch extractor at 49–60 °C. Following extraction, the meal was collected for analysis and the crude oil and hexane fraction separated *in vacuo*. Crude oil fractions were filtered and combined before alkali refinement using 16° Baumé sodium hydroxide. The homogenized oil was then filtered and bleached under vacuum before further filtration. The bleached oil was homogenize in a steam bath at 220–230 °C under vacuum before citric acid solution was added on cooling. The resulting oil was collected for analysis.

Samples were homogenized in the presence of dry ice and analysed for residues of inpyrfluxam and its metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (analysed as 1'-CH_2OH-S-2840A and B) and DFPA-$CONH_2$ using the validated LC-MS/MS method RM-50C-1. For the determination of *N*-des-Me-DFPA, Valent method RM-50C-2a was used. The LOQ was 0.01 mg/kg for each analyte. Concurrent recoveries across all matrices (peanuts. Meal and refined oil), analytes and fortification levels ranged from 73 to 126 percent with a maximum RSD of 13 percent. Samples were stored frozen (-20 °C) for up to 361 days before analysis. This period is covered by the available storage stability study (683 days in soya bean).

Results from the trials are summarized in Table 132. Residues of inpyrfluxam are <LOQ in all samples thus no processing factors can be derived.

Table 132 inpyrfluxam residues in peanut and processed commodities resulting from trial V-38942-M in the United States of America (Study 201700318)

Variety Application rate	Commodity	Residues, mg/kg						
		Inpyrfluxam	3'-OH-S-2840	1'-CH_2OH-S-2840[a]	DFPA-$CONH_2$	1'-COOH-S-2840 (free/agly)[a]	1'-CH_2OH-S-2840 (agly)[a]	*N*-des-Me-DFPA
Tamrun 4 foliar applications at 374-383 g ai/ha DALA=21	Nutmeat (23.7 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	<0.01 (3)	<0.02 (3)	<0.02 (3)	<0.02 (3)
	Meal (2.9 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	-	-	-	<0.02 (3)
	Refined oil (1.97 kg)	<0.01 (3)	<0.01 (3)	<0.02 (3)	<0.01 (3)	-	-	<0.02 (3)

RESIDUES IN ANIMAL COMMODITIES

The Meeting received a feeding study conducted in lactating cows and in laying hens.

Ruminants

In the cow feeding study (Arndt, and Van Middlesworth, 2016, TPR-0013) lactating Holstein cows were dosed for 29 consecutive days via gelatine capsule using a balling gun at levels equivalent to ca. 2 (3 cows), 6 (3 cows) and 20 (6 cows) ppm in their feed (dry-weight basis) per day for 28 consecutive days (mean mg/kg bw: 0.07, 0.20 and 0.61, respectively). Milk was collected twice daily and composited (evening milking with the next morning milking). Extra milk from days 14 and 28 was separated into cream and skimmed milk for cows from each group. Milk samples were held frozen prior to transport to the analytical facility.

After administration of the final dose, the animals were slaughtered (within 24h) and samples of liver, kidneys, fat and muscle were collected and weighed. After collection, the liver, kidney, and muscle samples were cubed and frozen. Samples were stored for the following a maximum period prior to analysis: milk: 75 days, cream: 67 days, skim milk: 16 days, liver and kidney: 19 days, muscle: 21 days, fat: 30 days. With the exception of cream, all samples were analysed within the periods where storage stability was demonstrated (milk: at least 75 days, meat, liver and kidney 29 days, fat: 31 days).

Tissue and milk samples were analysed for inpyrfluxam and metabolites 1'-COOH-S-2840 (A and B) and 1'-CH2OH-S-2840 (A and B) by method TPR-0013. Concurrent recoveries across all analytes and matrices ranged from 71 to 114 percent with a maximum RSD of 18.1 percent. Residues in milk, muscle and fat were <LOQ in all sampling periods and for all dosing levels. Residues were only detected in liver and kidney at levels above the LOQ and the results are summarized in Table 133.

Residues detected at or above the LOQ were of the metabolite 1'-CH2OH-S-2840 in day 28 liver at the 20 ppm diet dose level and kidney samples at the 6 mg/kg diet and 20 ppm diet dose levels. The average residue level of 1'-CH2OH-S-2840 in liver was 0.014 mg/kg at the 20 ppm diet dose level, in kidney was 0.013 mg/kg for the 6 ppm diet dose level and 0.022 mg/kg for the 20 ppm diet dose level on the first day after cessation of dosing. Residue levels returned to below the LOQ within 3 days. All other matrices (milk, skimmed milk, cream, muscle and fat) had no residues at or above the LOQ for parent, 1'-COOH-S-2840 and 1'-CH2OH-S-2840 throughout the dosing period and for the two week depuration phase.

The results of this study indicate that there is no transfer of residues of inpyrfluxam or its main metabolites (at ≥ 0.01 mg/kg) to milk, skimmed milk or cream during or up to two weeks after 28 days of consecutive dosing. There were no appreciable residues found even at an exaggerated rate of dosing. Similarly, there was no appreciable residue transfer (≥ 0.01 mg/kg) or preferential accumulation to bovine muscle or fat. For liver and kidney, the only residues detected were of 1'-CH2OH-S-2840 and were found only on the first day after cessation of dosing and only from cows administered at the 6 mg/kg diet and 20 mg/kg diet dosing levels for kidney and at the 20 ppm diet dosing level for liver, demonstrating rapid withdrawal from tissue.

The results are summarized in Table 133 and Table 134.

Table 133 Residues (mg/kg) of inpyrfluxam and its metabolites in milk from dairy cows

Day	2 ppm			6 ppm			20 ppm		
	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840
Whole milk									
-1	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (6)	<0.01 (6)	<0.01 (6)
1	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (6)	<0.01 (6)	<0.01 (6)
3	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (6)	<0.01 (6)	<0.01 (6)
7	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (6)	<0.01 (6)	<0.01 (6)
10	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (6)	<0.01 (6)	<0.01 (6)
14	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (6)	<0.01 (6)	<0.01 (6)
17	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (6)	<0.01 (6)	<0.01 (6)
21	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (6)	<0.01 (6)	<0.01 (6)
24	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (6)	<0.01 (6)	<0.01 (6)
28	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (6)	<0.01 (6)	<0.01 (6)
+3	-	-	-	-	-	-	<0.01 (3)	<0.01 (3)	<0.01 (3)
+7	-	-	-	-	-	-	<0.01 (2)	<0.01 (2)	<0.01 (2)
+14	-	-	-	-	-	-	<0.01	<0.01	<0.01

Table 134 Residues of inpyrfluxam and its metabolites in tissues from the 2, 6 and 20 ppm dose groups after 28 days of dosing and up to 14 days post dose (at 20 ppm)

Tissue	2 ppm			6 ppm			20 ppm		
	Inpyrfluxam	1'-COOH-S-2840	1'-CH_2OH-S-2840	Inpyrfluxam	1'-COOH-S-2840	1'-CH_2OH-S-2840	Inpyrfluxam	1'-COOH-S-2840	1'-CH_2OH-S-2840
Day 28									
Liver	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01, 0.017, 0.016
Kidney	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01, 0.014 (2)	<0.01 (3)	<0.01 (3)	0.015, 0.018, 0.033

20 ppm									
	Inpyrfluxam			1'-COOH-S2-840			1'-CH_2OH-S-2840		
Tissue	+3 days	+7 days	+14 days	+3 days	+7 days	+14 days	+3 days	+7 days	+14 days
Liver	<0.01	<0.01	<0.01 (2)	<0.01	<0.01	<0.01 (2)	<0.01	<0.01	<0.01 (2)
Kidney	<0.01	<0.01	<0.01 (2)	<0.01	<0.01	<0.01 (2)	<0.01	<0.01	<0.01 (2)

Poultry

In the poultry feeding study (Van Middlesworth, 2017, TPR-0015) ISA Brown laying hens were dosed for 28 consecutive days *via* gelatine capsules at levels equivalent to at 1 (12 hens), 3 (12 hens) and 10 (24 hens) ppm feed (DM) per day (mean: 0.058, 0.164 or 0.547 mg/kg bw, respectively). Eggs were collected twice daily (morning and evening) and pooled. Hens were sacrificed on 28th day within 6 hours of the receipt of the final dose. After dosing was stopped, selected hens from the high dose group were kept alive for further 3–14 days in order to investigate the depuration of inpyrfluxam and metabolites in eggs and tissues after the end of application. Samples of muscle (thigh and breast combined), liver and fat (subcutaneous and abdominal combined) were taken and stored frozen prior to analysis. Samples were stored prior to analysis for up to: eggs: 85 days, egg yolk: 44 days, egg white: 24 days, liver: 37 days, muscle: 35 days, fat: 45 days. Stability was demonstrated for at least: eggs: 90 days, liver and muscle: 40 days, fat: 49 days.

Tissue and egg samples were analysed for inpyrfluxam and metabolites 1'-COOH-S-2840 (A and B) and 1'-CH_2OH-S-2840 (A and B) by method TPR-0015. Concurrent recoveries across all analytes and matrices ranged from 70 to 108 percent with a RSD of 7.7 percent.

Residues detected at or above the LOQ were inpyrfluxam and 1'- CH_2OH -S-2840 in day 28 fat and liver from the 10 ppm, respectively. 1'-CH_2OH-S-2840 was identified in day 28 liver at the 3 ppm and 10 ppm dose levels. The average concentration of inpyrfluxam in fat for the 10 ppm was 0.017 mg/kg. In liver, 1'-CH_2OH-S-2840 averaged 0.017 mg/kg at the 10 ppm less than a day after cessation of dosing. However, each of these residues in the liver and fat were <LOQ within 3 days.

No residues of inpyrfluxam or its metabolites were found in whole egg and egg white. Egg yolk contained residues of 1'-CH_2OH-S-2840 in the 10 ppm samples on day 28. The average total of 1'-CH_2OH-S-2840 in egg yolk at the 10 ppm on day 28 was 0.012 mg/kg (Table 135). No discernible plateau of residues was observed because of the low levels of residues in all samples.

Table 135 Residues (mg/kg) of inpyrfluxam in egg yolk from the 1, 3 and 10 ppm dose group during the 28-day hen study

Day	1 ppm			3 ppm			10 ppm		
	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840
14	<0.01 (3)	<0.01 (3)	<0.01 (3)	-	-	-	<0.01 (3)	<0.01 (3)	<0.01 (3)
28	<0.01 (3)	<0.01 (3)	<0.01 (3)	-	-	-	<0.01 (3)	<0.01 (3)	0.011, 0.012, 0.012

Samples of muscle had no residues at or above the LOQ throughout the dosing period. No residues were found in muscle, liver or fat in the 10 ppm group up two weeks after cessation of dosing, suggesting limited transfer from feed to tissues. The results for liver and fat are summarized in Table 136.

Table 136 Residues of inpyrfluxam in in liver and fat of hen from the 1, 3 and 10 ppm dose group after 28 days of dosing

Tissue	1 ppm			3 ppm			10 ppm		
	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840
Liver	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (2), 0.010	<0.01 (3)	<0.01 (2), 0.010	0.013, 0.018,

Tissue	1 ppm			3 ppm			10 ppm		
	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840	Inpyrfluxam	1'-COOH-S2840	1'-CH_2OH-S-2840
									0.019
Fat	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	<0.01 (3)	0.015, 0.018 (2)	<0.01 (3)	<0.01 (3)

APPRAISAL

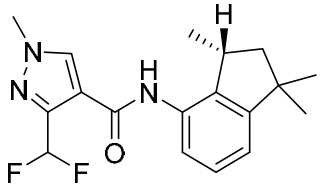
Inpyrfluxam is a broad spectrum fungicide belonging to the succinate dehydrogenase inhibitor (SDHI) group of fungicides, which mode of action involves inhibition of energy production processes in pathogenic fungi.

Inpyrfluxam has not previously been evaluated by JMPR and was scheduled at the Fifty-first Session of the CCPR (2019) for toxicology and residue evaluation as a new compound by the 2022 JMPR.

The Meeting received information on identity, physical-chemical properties, plant and animal metabolism, analytical methods, storage stability, use patterns, residues resulting from supervised trials, fate of residues in succeeding crops, fate of residues during processing and livestock feeding studies.

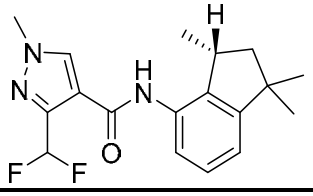
All critical studies contained statements of compliance with GLP and were conducted in accordance with relevant national or international test guidelines, unless otherwise specified.

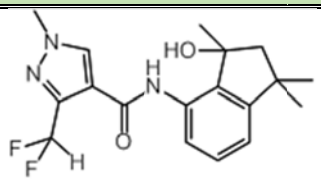
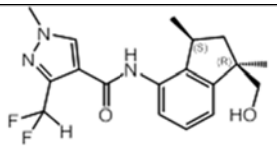
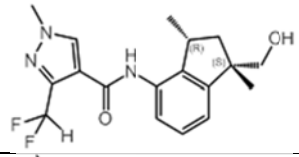
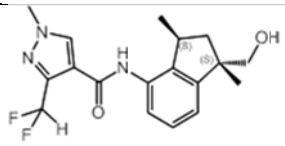
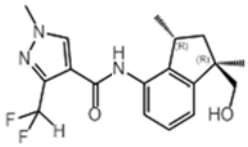
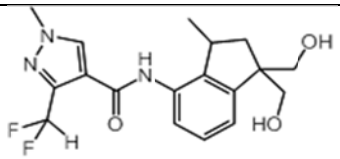
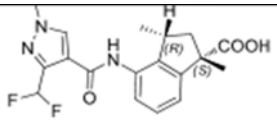
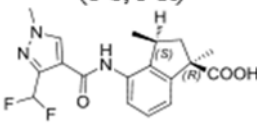
Inpyrfluxam (S-2399) is the ISO-approved common name for 3-(Difluoromethyl)-1-methyl-*N*-[(3*R*)-1,1,3-trimethyl-2,3-dihydro-1*H*-inden-4-yl]-1*H*-pyrazole-4-carboxamide with the CAS number 1352994-67-2.

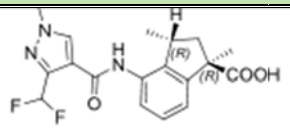
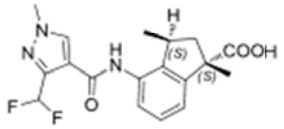
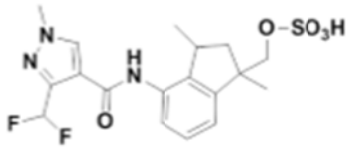
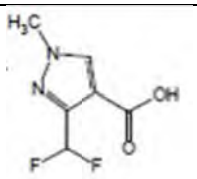
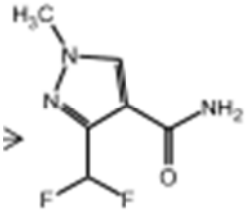
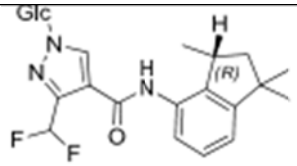
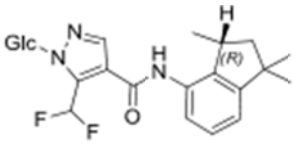
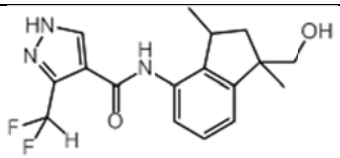


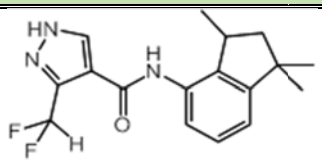
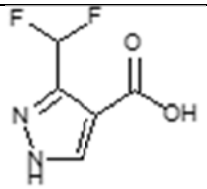
The abbreviations, chemical names, and structures discussed in the appraisal are summarized in Table 137.

Table 137 Abbreviations for the relevant compounds referred to in this document

Compound code (other names)	Name and matrix	Structure
Parent MW: 333.38	3-(Difluoromethyl)-1-methyl-*N*-[(3*R*)-1,1,3-trimethyl-2,3-dihydro-1*H*-inden-4-yl]-1*H*-pyrazole-4-carboxamide Found in: plants (apple, soya , rice, potato, lettuce (RC), radish (RC), sorghum (RC)), animals (milk, goat tissues, eggs, hen tissues), environment (soil) MW: 333.38	

Compound code (other names)	Name and matrix	Structure
3'-OH-S-2840 MW: 349.4	3-(Difluoromethyl)-N-[3 ' -hydroxy-l ', 1 ',3 '-trim ethyl- 2 · ,3 '-dihydro-1 · H-inden-4 · -yl]-1-methyl-lH-pyrazole-4- carboxamide Found in: plants (Apple, Soya forage, Soya hay, Soya pods, Soy beans, Immature rice, Rice straw, Rice hulls, Rice grain, Potato, Lettuce (RC), Radish (RC), Sorghum (RC)), animals (Hen eggs, Hen fat, Ruminant kidney, Ruminant fat), environment (Soil, Water)	
1'-CH_2OH-S-2840 MW: 366.4	Found in: plants (Potato), animals (Hen eggs, Hen liver, Hen muscle, Hen fat, Ruminant skimmed milk, Milk fat, Ruminant liver, Ruminant kidney, Ruminant muscle, Ruminant fat), environment (soil)	See below
1'-CH_2OH-S-2840A MW: 366.4	Found in: plants (Rice, Potato, Lettuce (RC), Radish (RC), Sorghum (RC)), animals (Goat tissues, Eggs, Hen tissues), environment (soil)	 
1'-CH_2OH-S-2840B MW: 366.4	Found in: plants (Apple, Soya, Rice, Potato, Lettuce (RC), Radish (RC), Sorghum (RC)), animals (Milk, Goat tissues, Eggs, Hen tissues), environment (soil)	 
1',1'-bis (CH_2OH)-S-2840 MW: 351.35	Found in: animals (Goat tissues)	
1'-COOH-S-2840 A MW: 363.4	Found in: plants (Potato, Lettuce (RC), Radish (RC), Sorghum (RC)), animals (Milk, Goat tissues, Eggs, Hen tissues), environment (soil)	 (1'S, 3'R)  (1'R, 3'S)

Compound code (other names)	Name and matrix	Structure
1'-COOH-S-2840B MW: 363.4	Found in: plants (Potato, Lettuce (RC), Radish (RC), Sorghum (RC)), animals (Milk, Goat tissues, Eggs, Hen tissues), environment (soil)	 (1'R, 3'R)  (1'S, 3'S)
1'-CH_2OH-S-2840-sulfate (sum of isomers) MW: 429.44	Found in: animals (Eggs, Hen tissues),	
DFPA MW: 176.12	3-(Difluoromethyl)-1-methyl- lH-pyrazole-4-carboxylic acid Found in: plants (Potato, Lettuce (RC), Radish (RC), Sorghum (RC)), animals (Goat tissues), environment (Soil, Water)	
DFPA-$CONH_2$ MW: 175.1	3-(Difluoromethyl)-1-methyl-lH-pyrazole-4-carboxamide. Found in: plants (Rice, Lettuce (RC), Radish (RC), Sorghum (RC)), animals (Milk, Goat tissues, Eggs, Hen tissues), environment Water)	
Glc-NDM-inpyrfluxam (sum of isome) MW: 481.20	Found in: plants (Soya)	 
Gly- 1'-CH_2OH-S-2840	Found in: animals (Immature rice, rice straw, rice hulls, rice grain)	Structure not elucidated.
N-des-Me-1'-CH_2OH-S-2840 MW: 335.35	Found in: Lettuce (RC), Radish (RC), Sorghum (RC)	

Compound code (other names)	Name and matrix	Structure
N-des-Me-S-2840 MW: 319.36	Found in: plants (Soya, Rice, Lettuce (RC), Radish (RC), Sorghum (RC)), animals (Eggs, hen tissues), environment (Soil)	
N-DesMet-pyrazole carboxylic acid (*N*-des-Me-DFPA) MW: 162.1	3 -(Difluoromethyl)-lH-pyrazole-4-carboxylic acid AS238la Found in: plants (Soya, Rice, Potato, Lettuce (RC), Radish (RC), Sorghum (RC)), environment (Soil)	

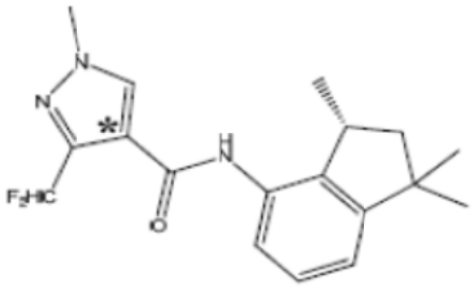
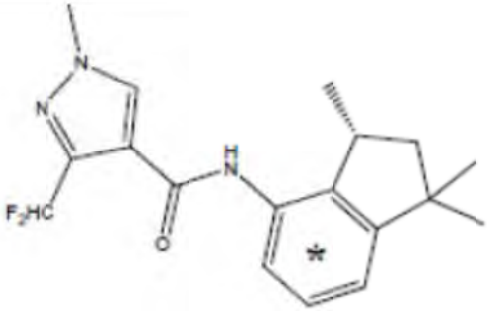
Physical chemical properties

Inpyrfluxam is not soluble in water (0.0164 g/L at 20 °C), but is soluble in acetone (621 g/L), methanol (368 g/L) and ethyl acetate 396 g/L). Inpyrfluxam is hydrolytically and photolytically stable. The compound has a LogPow of 3.65 suggesting potential accumulation in fat and a vapour pressure of 1.2×10^{-7} Pa at 25 °C suggesting that the compound in not volatile.

Plant metabolism

The Meeting received plant metabolism studies for inpyrfluxam in apple (fruit), soya bean and rapeseed (pulse and oilseed crops), maize, sorghum and rice (cereal crops) and potato (root crop). Inpyrfluxam was applied using either [pyrazolyl-4-^{14}C]-inpyrfluxam or [phenyl-U-^{14}C]-inpyrfluxam (Table 138). In metabolism studies, total radioactive residues (TRR) are expressed in mg inpyrfluxam equivalents/kg.

Table 138 Labels used in the plant metabolism studies

	
[Pyrazolyl-4-^{14}C]-inpyrfluxam used in all primary metabolism studies.	[Phenyl-U-^{14}C]-inpyrfluxam used in all primary metabolism studies.

The use patterns received include foliar application (apples, soya, sugar beet, rice, peanut), broadcast granular application (rice) and seed treatment (soya, sugar beet, rice, maize).

Apple

The metabolic fate of [pyrazolyl-4-^{14}C]-inpyrfluxam and [phenyl-U-^{14}C]-inpyrfluxam was investigated in apples following three foliar applications with approximate 10-day intervals 35, 24 and 14 days before harvest (BBCH stage not specified in the report) at an actual rate of 214–221 g ai/ha each.

Samples of apple fruits were collected 14 days after the final application.. Fruits were rinsed with acetonitrile and separated into peel and flesh before homogenisation. Processed samples were extracted twice with acetonitrile:water (1:1) and once with acetonitrile.

Extracted radioactivity was similar in both labels ranging between 95.9–96.3 percent TRR (0.25–0.3 mg eq/kg) in whole fruit (including apple rinse). The PES accounted for 3.6–4 percent TRR (0.01–0.011 mg eq/kg) and were not further analysed. The majority of radioactivity was recovered in the fruit rinse (58–64 percent TRR, (0.14–0.19 mg eq/kg) and peel (17–22 percent TRR, 0.052–0.055 mg eq/kg). Radioactivity in the flesh was markedly lower than in the peel for both treatment groups.

The major component identified was inpyrfluxam, representing 78–79 percent of the TRR (0.19–0.24 mg /kg). Minor metabolites identified were 3'-OH-S-2840 (11.5 percent TRR, 0.035 mg eq/kg) and 1-CH_2OH-S-2840 (5.6 percent TRR, 0.014 mg eq/kg).

Soya bean

The metabolic fate of [pyrazolyl-4-14C]-inpyrfluxam and [phenyl-U-14C]-inpyrfluxam was investigated in outdoor soya beans following two foliar applications with a 36-day interval (at BBCH 60 and 75) at actual rates 107–113 g ai/ha each.

Samples of soya bean forage and hay were collected 20 and 33 days after the final application as follows:

Forage was harvested at BBCH 75 and left to dry to soya bean hay, immature pods were taken at BBCH 77 and mature pods were taken at BBCH 89. Samples were separated into pods and seeds and a portion of mature bean pods were rinsed (with acetonitrile) before homogenisation.

Homogenized samples were extracted twice with acetonitrile:water, and once with acetonitrile. The post-extraction solids (PES) were further submitted to either acidic or alkaline hydrolysis to release plant natural components (e.g. pectin (lignin, hemicellulose and cellulose).

Extracted radioactivity was similar in both labels being 100 percent TRR (1.71–2.37 mg eq/kg) in hay ranging between 99.8–99.9 percent TRR (1.39–1.5 mg eq/kg) in forage, 72.7–96.35 percent TRR (0.016–0.1 mg eq/kg) in immature seed, 100 percent TRR (0.61–0.71 mg eq/kg) in immature seed, 65.8–95.4 percent TRR (0.023–0.2 mg eq/kg) in mature pods and 92.5–94.6 percent TRR (0.59–1.04 mg eq/kg) in mature seeds.

Unextracted residues after hydrolysis accounted for 4.1–36.8 percent TRR (0.009–0.014 mg eq/kg) only for mature seeds and were not further characterized.

Parent inpyrfluxam was extensively metabolised in soya beans accounting for the major part of the residue in forage samples (40.3–50.5 percent TRR, 0.56–0.79 mg /kg) but declined as the plant matured. Residues of inpyrfluxam in hay were between 0.42–0.49 mg/kg (17.8–22.1 percent TRR), in immature pods levels ranged from 0.24–0.41 mg/kg (34–65.2 percent TRR) and in mature pods levels ranged from 0.13–0.22 mg/kg (10.9–29.2 percent TRR), including the surface rinse fraction. Only trace levels of inpyrfluxam residues were detected in the soya bean seeds.

In <u>forage</u>, 3'-OH-S-2840 was present at the highest levels (15.3–22.1 percent TRR, 0.24–0.31 mg eq/kg), whilst 1'-CH_2OH-S-2840 was identified but in low levels below 3.7 percent TRR (0.058 mg eq/kg).

In <u>hay</u>, 3'-OH-S-2840 was present at the highest levels (14.3–14.7 percent TRR, 0.32–0.35 mg eq/kg), whilst *N*-des-Me-S-2840 was detected at low levels (≤ 2.4 percent TRR, 0.05 mg eq/kg).

Also the metabolites 1'-CH_2OH-S-2840 (free and conjugated) and Glc-NDM-inpyrfluxam, were detected at minor levels.

In immature pods, metabolites 3'-OH-S-2840, *N*-des-Me-S-2840 and 1'-CH_2OH-S-2840 were found below 10 percent TRR but at levels up to 0.065 mg eq/kg, 0.042 mg eq/kg and 0.026 mg eq/kg respectively.

In immature seeds, N-DesMet-pyrazole carboxylic acid and *N*-des-Me-S-2840 were detected at levels below 10 percent TRR (< 0.01 mg eq/kg). The majority of the residue (61.6 percent TRR, 0.067 mg eq/kg) was characterised as multiple polar components with a single component representing 4.6 percent TRR (0.005 mg eq/kg).

In mature pods, 3'-OH-S-2840 was identified as the most dominant metabolite at 11.6 percent TRR (0.086 mg eq/kg), whilst *N*-des-Me-S-2840 (3.9 percent TRR; up to 0.029 mg eq/kg) and 1'-CH_2OH-S-2840 (2.8 percent TRR; up to 0.021 mg eq/kg) were also characterized at lower levels. In the [pyrazolyl-^{14}C] label, polar components were present at high levels (48.9 percent TRR, 0.59 mg eq/kg), with the highest component being no greater than 2.1 percent TRR (up to 0.032 mg eq/kg).

In mature seed, N-DesMet-pyrazole carboxylic acid (conjugated), was detected (17.5 percent TRR, 0.038 mg eq/kg). Metabolites 3'-OH-S-2840, Glc-NDM-inpyrfluxam and 1'-CH_2OH-S-2840 were detected at low levels (< 10 percent TRR; ≤ 0.001 mg eq/kg). The major fraction contained unretained polar components (11.7–63.8 percent TRR, 0.004–0.140 mg eq/kg).

Rice–foliar treatment

The metabolic fate of [pyrazolyl-4-^{14}C] inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam was investigated in outdoor rice following one foliar applications at actual rates 95–108.1 g ai/ha, 28 days (at BBCH 77) before normal commercial harvest.

Samples of the immature whole plant (BBCH not specified) were taken for analysis 14 days after application. Samples of rice heads and straw were collected 28 days after application (at normal commercial harvest). Rice heads were separated into husked rice and hulls.

Homogenised samples were extracted twice with acetonitrile:water and once with acetonitrile. The PES were further submitted to either acidic or alkaline hydrolysis.

Extracted radioactivity was similar in both labels, ranging between 99.3–99.7 percent TRR (0.29–0.37 mg eq/kg) in immature rice, 97.4–99.3 percent TRR (0.84–1.44 mg eq/kg) in straw, 96.9–98 percent TRR (1.4–1.5 mg eq/kg) in hulls and 95.3–95.9 percent TRR (0.063–0.047 mg eq/kg) in husked rice.

Unextracted residues after hydrolysis accounted for 0.6–3.2 percent TRR (0.002–0.054 mg eq/kg).

In immature rice plants, the only major component for both radiolabels detected in the neutral extract was parent inpyrfluxam, present at 81.2–86.7 percent TRR (0.25–0.31 mg/kg). The metabolite, 3'-OH-S2480 was detected between 5.6–7.1 percent TRR (0.016–0.027 mg eq/kg), with trace levels of *N*-des-Me-S-2840 and 1'-CH_2OH-S-2840 (two isomers) also detected in both labelled extracts. The acidic acetonitrile extracts for both labels contained predominantly parent (2.5–3.4 percent TRR, 0.007–0.013 mg/kg).

In husked rice, inpyrfluxam accounted for 60.6–78.6 percent TRR (0.038–0.039 mg/kg). Metabolites 3'-OH-S-2840 and Gly-1'-CH_2OH-S-2840 were also detected but at ≤ 0.01 mg eq/kg (up to 16 percent TRR).

In straw, inpyrfluxam was present as the major residue (67.7–77.8 percent TRR, 0.58–0.72 mg/kg). Metabolites 3'-OH-S-2840 (up to 12 percent TRR; 0.102 mg eq/kg), Gly-1'-CH_2OH-S-2840 (5.2 percent TRR; 0.040 mg eq/kg) and DFPA-$CONH_2$ (4.6 percent TRR; 0.039 mg eq/kg) were also present.

In rice hulls, residues were characterized as a mixture of inpyrfluxam at 52.5–41.8 percent TRR (0.64–0.88 mg/kg), 1'-CH_2OH-S-2840 at 18–33.9 percent TRR (0.52–0.28 mg eq/kg) and 3'-OH-S-2840 at 6–12 percent TRR (0.055–0.1 mg eq/kg) respectively.

Rice–granular treatment

The metabolic fate of [pyrazolyl-4-^{14}C] inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam was also investigated in rice grown in trays and transplanted at the 4 leaf stage of growth in outdoor plots. Inpyrfluxam was applied following one granular treatment at BBCH 13-14 at actual rate 400 g ai/ha.

Immature rice plants were harvested 30 days after treatment (BBCH 30) and mature rice plants were harvested 132 days after treatment (BBCH 89) and were separated into straw, hulls and husked rice.

Homogenised samples were extracted twice with acetonitrile:water and once with acetonitrileand PES were further submitted to either acidic or alkaline hydrolysis.

Extracted radioactivity was similar in both labels, being 100 percent TRR (1.9–3.9 mg eq/kg) in immature rice, 100 percent TRR (1.07–1.58 mg eq/kg) in straw, ranging from 91.1–93.2 percent TRR (0.14–0.16 mg eq/kg) in hulls and 40.4–55.6 percent TRR (0,005–0.006 mg eq/kg). Unextracted residues after hydrolysis accounted for 6.9 percent TRR (0.012 mg eq/kg) in hulls and 60 percent TRR (0.009 mg eq/kg) in husked rice but were not further identified.

In immature rice plants, parent inpyrfluxam accounted for a large proportion of the residues at 20–38.2 percent TRR (0.72–0.78 mg/kg). In addition, 1'-CH_2OH-S-2480 (5.8-6.2 percent TRR, 0.11–0.24 mg eq/kg), 3'-OH-S-2840 (1.2–3.6 percent TRR, 0.023-0.14 mg eq/kg) and DFPA-$CONH_2$ (2.2 percent TRR, 0.086 mg eq/kg) were identified. Glycosidic derivative of 1'-CH_2OH-S-2840, was present at 16.8–26.0 percent TRR (0.32–1.01 mg eq/kg).

In straw, parent inpyrfluxam accounted for 1.9–2.8 percent TRR (0.03 mg eq/kg). The major part of the residue was metabolite 1'-CH_2OH-S-2840 and its glycosidic derivatives (57.4–61.6 percent TRR (0.77–0.66 mg/kg), whilst metabolites *N*-des-Me-S-2840 (1.6 percent TRR, 0.025 mg eq/kg) and DFPA-$CONH_2$ (2.1 percent TRR, 0.034 mg eq/kg) in lower levels.

In rice hulls, parent inpyrfluxam was not detectable. Metabolite 1'-CH_2OH-S-2840 and its glycosidic conjugates (40.1–60.2 percent TRR, 0.031–0.085 mg eq/kg) were the main residues, followed by DFPA-$CONH_2$ (17.5 percent TRR, 0.031 mg eq/kg) and N-DesMet-pyrazole carboxylic acid (5.3 percent TRR, 0.009 mg eq/kg).

In husked rice, parent inpyrfluxam was not detectable. Metabolites DFPA, N-DesMet-pyrazole carboxylic acid and 1'-CH_2OH-S-2840 were detected at ≤ 0.002 mg eq/kg (up to 23.1 percent TRR).

Seed treatment

Maize

The metabolic fate of [pyrazolyl-4-^{14}C] inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam was investigated in outdoor maize following seed treatment at actual rates 22.1 g ai/tonne seeds and planted after 3–4 days.

Maize forage was sampled at late dough/early dent stage, sweet corn (kernels plus cob with husks removed at the milk/succulent stage, approximately 95 days after planting) whilst stover and

grains were harvested at maturity. Grain was separated from the cob (approximately 126 days after planting) and grain-free mature cobs and stalks were processed as maize stover.

In all samples from both labels, TRR were not found above the LOQ (0.005 mg/kg), as a result no metabolite identification was attempted.

Sorghum

The metabolic fate of [pyrazolyl-4-^{14}C] inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam was investigated in outdoor sorghum following seed treatment at actual rates 50 g ai/tonne seeds.

Sorghum forage samples were collected at the soft dough to hard dough stage. The remaining plants were harvested at maturity and separated into grain and stover. In all samples from both labels, TRR were not found above the LOQ (0.005 mg/kg), thus no metabolite identification was attempted.

Rapeseed

The metabolic fate of [pyrazolyl-4-^{14}C] inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam was investigated in outdoor rapeseed following seed treatment at actual rates 5 g ai/tonne seeds and planted after 7 days.

Samples of mature seeds of rapeseed, were harvested after 4 months from individual plots at BBCH 97–99 (approximately 161 days after planting).

In all samples from both labels, TRR were not found above the LOQ (0.005 mg/kg), thus no metabolite identification was attempted.

Potatoes

The metabolic fate of [pyrazolyl-4-^{14}C] inpyrfluxam and [phenyl-U-^{14}C]inpyrfluxam was investigated in potato following seed piece treatment at actual rates 50 g ai/tonne seeds and planted in outdoor plots at the same day.

Samples of the mature tubers were harvested at the appropriate growth stage (BBCH 49; 84–85 days from treatment and planting) and samples of foliage (collected at BBCH 48; 71–72 days from treatment and planting) were also taken from the plots.

Surface radioactivity was extracted into acetone and homogenised tuber samples were extracted twice with acetone and twice with acetone:water. To identify conjugates in tuber samples, the acetone extract was evaporated to dryness and dissolved in acetonitrile:water before being subjected to acid hydrolysis.

Extracted radioactivity was similar in both labels ranging between 87.9–93.4 percent TRR (0.011–0.039 mg eq/kg) in potato tubers. The PES accounted for 6.6–12.1 percent TRR (0.001–0.003 mg eq/kg).

In tubers, parent accounted for 5.8–15 percent TRR (0.002 mg eq/kg). Metabolite 1'-COOH-S-2840 accounted for 14.5–22.3 percent TRR (0.006–0.009 mg eq kg). Metabolites 1'-CH_2OH-S-2840, 3'-OH-S-2840, DFPA and N-DesMet-pyrazole carboxylic acid were also detected but individually accounted for ≤ 0.004 mg eq/kg (≤ 10.2 percent TRR).

Low TRR values in tubers, in both labels, indicated that the uptake of inpyrfluxam from the treated seed pieces was low. Inpyrfluxam metabolised into a number of metabolites, none of which were present at levels above ≥ 0.01 mg/kg in the tuber tissue. Inpyrfluxam was shown to metabolise via the routes of oxidation, amide bond cleavage and conjugation in potato tubers.

Summary of plant metabolism

Plant metabolism studies were conducted in apple and soya bean (foliar spray), rice (foliar and granular treatments) and maize, sorghum rapeseed and potato (seed treatment) at rates that accommodate the anticipated maximum total seasonal GAP application rates.

Uptake and transport of inpyrfluxam in the maize, sorghum, oilseed and potato studies, after seed treatment was low. Metabolism in soya bean, rice and apple proceeds via oxidation to form the hydroxylated components, 3'-OH-S-2840 and 1'-CH_2OH-S-2840 the latter forming multiple glycoside conjugates. The glycoside conjugates of 1'-CH_2OH-S-2840 are further transformed into plant constituents. DFPA-$CONH_2$ can be also formed from the degradation of 3'-OH-S-2840 and 1'-CH_2OH-S-2840, which is further metabolised into plant components. Additional minor pathways include the demethylation of inpyrfluxam or cleavage of the amide bond to DFPA which is rapidly demethylated to N-DesMet-pyrazole carboxylic acid followed by sugar conjugation and metabolism into multiple high polarity components.

Environmental fate

The Meeting received information on hydrolysis, aqueous photolysis, aerobic degradation in soil under laboratory conditions, soil field dissipation, confined and field rotational crops.

Hydrolysis

[Pyrazolyl-4-^{14}C] inpyrfluxam incubated in the dark in sterile aqueous buffered solutions at pH 4, 7 and 9 for 5 days at 50 °C remained stable. No degradation products were detected and no change in ratio between the R- and S-isomers was observed. The results indicate that inpyrfluxam is hydrolytically stable at environmental conditions and hydrolysis is not considered a significant route of degradation.

Photochemical degradation

In an aqueous photolysis study, inpyrfluxam was minimally degraded under simulated sunlight in sterilized aqueous phosphate buffer pH 7 and in sterilized natural water. The formation of metabolites 3'-OH-S-2840 and DFPA-$CONH_2$ was observed below < 10 percent AR (applied radioactivity. The DT_{50} values ranged from 36–88 days (69–579 sunlight days).

In a soil photodegradation study, the main degradation product was 3-'OH-S-2840 (mean maximum of 8.3 percent AR at day 13) and soil bound residues represented less than 3 percent AR. Isomerization from inpyrfluxam R-isomer to its S-isomer was not observed.

Based on the above, photolysis is not considered a significant route of degradation in water or the soil surface.

Soil metabolism

DT_{50} values for inpyrfluxam in aerobic soil under laboratory conditions ranged from 101–1720 days with a geomean DT_{50} of 348 days, indicating moderate persistence to persistence in soil resulting in the formation of two major metabolites (3'-OH-S-2840 and 1'-COOH-S-2840) and many minor metabolites.

Using the best fit kinetics for the parent molecule, calculated DT_{50} values for 3'-OH-S-2840 ranged from 78–843 (> 10,000 at Atwater soil) days and for 1'-COOH-S-2840 ranged from 34.2–669 (> 10,000 at Penn soil) days. However since the duration of the studies was short (120–182 days) and metabolites did not show significant decline during the incubation, there is significant uncertainty in the study, thus the Meeting concluded that the calculated DT_{50} are not reliable.

Soil field dissipation studies

The field soil dissipation of inpyrfluxam has been studied in Europe and the United States with DT_{50} values ranging from 78.1–419 days and 14.6–113 days, respectively, with an overall geomean DT_{50} of 117 days (n = 9). The DT_{50} values of the metabolites were assessed only in Europe, however residues below the LOQ were observed thus the Meeting concluded that the reliable DT_{50} could not be calculated.

Water/sediment degradation in the field

Inpyrfluxam degraded slowly under anaerobic (DT_{50} of 3537 and 3498 days) or aerobic (DT_{50} of 154 to 704 days depending on the study) aquatic conditions. Degradation was primarily to form 1′-COOH-S-2840 (0.9–13.1 percent AR) and 3′-OH-S-2840 (2.9–6.8 percent AR). In natural surface water, no significant degradation of inpyrfluxam was observed (DT_{50} ≥ 1.540 days).

The dissipation, mobility and degradation of inpyrfluxam and its transformation products was investigated in an aquatic field dissipation study following planting of treated rice seed. Inpyrfluxam was applied at 10 g ai/100 kg seed or at 10 g ai/100 kg seed followed by foliar application 77 days after sowing at 100 g ai/ha. In the seed treatment only, inpyrfluxam was observed in the sediment and soil phases at very low concentrations up to 0.027 mg/kg, with no transformation product residues observed (<LOQ) at any sampling event. Formation of 1'-COOH-S-2840 in the water phase was observed at low concentrations up to 5. 1µg/L. In the seed treatment/foliar application study, inpyrfluxam was observed in the sediment and soil phases at very low concentrations up to 0.027 mg/kg, and no degradants were observed in the water, sediment or soil. Inpyrfluxam, dissipated rapidly from the system as a whole (paddy water and sediment) with a calculated DT_{50} of 0.064–0.87 days.

Confined rotational crop studies

[Phenyl-^{14}C] inpyrfluxam and [pyrazolyl-^{14}C] inpyrfluxam were applied to bare soil at a rate of approximately 235 g ai/ha. Lettuce, radish and sorghum were grown as rotational crops 30, 120 and 365 days after treatment, i.e., plant back intervals (PBI), and harvested according to normal agricultural practice.

TRR levels were lower in lettuce and radish samples at the 365 days PBI compared to the levels at 30 and 120 days PBI. The highest AR was observed in sorghum stover samples, with TRR of 0.69–0.7 mg eq/kg at 30 days PBI and 0.94–1.07 mg eq/kg at 120 days PBI, decreasing to 0.13–0.24 mg eq/kg at 365 days PBI. The sorghum forage contained considerably lower residue levels than the stover samples at all planting intervals.

Inpyrfluxam and its primary oxidation product 3'-OH-S-2840 (free and conjugated) was found in all crops except for sorghum grain. Inpyrfluxam was found in lettuce (up to 17.4 percent TRR; 0.011 mg/kg), radish tops (up to 12.3 percent TRR; 0.025 mg/kg), radish roots (up to 57.8 percent TRR; 0.045 mg/kg), sorghum forage (up to 3.4 percent TRR; 0.007 mg/kg) and sorghum stover (up to 1.8 percent TRR; 0.02 mg/kg). The major portion of metabolite 3'-OH-S-2840 was present in conjugated form accounting for up to 17.6 percent in lettuce (0.012 mg eq/kg), for up to 13.3 percent TRR in radish tops (0.017 mg eq/kg) and for up to 12.1 percent TRR sorghum stover (0.017 mg eq/kg). 1'-COOH-S-284, mostly conjugated, was found in lettuce (up to 14.6 percent TRR; 0.045 mg eq/kg), radish tops (up to 25.6 percent; 0.021 mg eq/kg), radish roots (up to 25.7 percent TRR; 0.06 mg eq/kg) and potatoes (up to 19.1 percent TRR; 0.005 mg eq/kg). 1'-CH_2OH-S-2840, mostly conjugated, was found in lettuce (up to 24.8 percent TRR; 0.024 mg eq/kg) and sorghum stover (15.5 percent TRR; 0.008 mg eq/kg). N-des-Me-S-2840, mostly in free form, was found in radish immature (up to 15.1 percent TRR; 0.027 mg eq/kg) and mature tops (up to 12.8 percent TRR; 0.039 mg eq/kg). N-des-Me-1'-CH_2OH-S-2840, mostly conjugated,

was found in radish immature (14.7 percent TRR; 0.017 mg eq/kg) and mature tops (15 percent TRR; 0.056 mg eq/kg).

Field rotational crop studies

Inpyrfluxam and its metabolites are persistent in the environment and may contribute to residues in follow/rotational crops through uptake from soil. In assessing the potential uptake of residues, excluding apples, the maximal season rate was 200 g ai/ha for peanut, i.e., the highest seasonal application rate of the uses evaluated by the Meeting. The use pattern of inpyrfluxam in the United States of America for apple, maize, peanut, rice, soya bean and sugar beet includes a rotational interval of 9 months (120 days) and a restriction for livestock grazing (soya bean use). Five field rotational crop studies were conducted in Canada (1), Europe (1), and the United States (3).

In the European study inpyrfluxam was applied at a single rate of 240 g ai/ha on winter and spring barley. The crops were destroyed and incorporated in the soil whilst preparing the soil for rotational crops within 13–14 days of application. Follow-up crops lettuce, carrot and wheat or barley were planted at PBIs of 28, 120 and 350 days. Residues of inpyrfluxam and its metabolites were < 0.01 mg/kg in all rotated crop matrices at all PBIs except for cereal straw on 30 days PBI, residues were up to 0.017 mg eq/kg for 3'-OH-S-2840, up to 0.019 mg eq/kg for 1'-COOH-S-2840, up to 0.023 mg eq/kg for 1'-CH_2OH-S-2840 and up to 0.1 mg eq/kg for DFPA.

In the Canadian and one US study, inpyrfluxam was applied at a single rate of 100–120 g ai/ha on wheat. The crops, after growth to maturity, the wheat was harvested and either destroyed (Canadian study) or tilled into the ground near the treated plot (US study). In the Canadian study, wheat, field peas and rapeseed were planted at PBIs of 328, 328 and 339 days and in the US study, wheat and rapeseed were planted at PBIs of 328 and 312 days. In two additional US studies inpyrfluxam was applied twice at a rate of 105–110 g ai/ha on soya bean. After growth to maturity the crops were tilled into the ground near the treated plot and sorghum and cotton planted at PBIs of 273–267 days. Residues of inpyrfluxam and its metabolites were < 0.01 mg/kg in all rotated crop matrices at all PBIs.

Summary of environmental fate

Inpyrfluxam is hydrolytically stable, does not photodegrade in aqueous buffered solutions or on the surface of soil. Aerobic degradation studies under laboratory conditions indicated a DT_{50} of 101–331days of inpyrfluxam in various soils, indicating moderate persistence to persistence in soil. Field dissipation studies confirmed the results, with DT_{50} for inpyrfluxam residues ranging from 41.6 to 419 days (geometric mean of 117 days) in European Union and United States soils. 3'-OH-S-2840 and 1'-COOH-S-2840 were identified in soil, but reliable DT_{50} values could not be calculated.

In the aquatic field dissipation studies, inpyrfluxam was observed in the sediment and soil phases at very low concentrations, as well as 1'-COOH-S-2840 in the water phase. Decline in the total aquatic system was very quick (<1 day DT_{50}). Based on the available studies, a soil plateau level estimation is not required as to address the residues in rotational crops.

Confined rotational crop studies indicate that inpyrfluxam was extensively metabolized into a large number of metabolites, but the residues were < 0.01 mg/kg in all rotated crop matrices at all PBI, except wheat straw were 3'-OH-S-2840, 1'-COOH-S-2840, 1'-CO_2OH-S-2840 and DFPA were detected at quantitative levels.

In conclusion, no residues of inpyrfluxam are expected in rotated crops under the conditions investigated in the studies provided to the Meeting. Should a more cGAP or additional uses received in the future, the expectation of residues of inpyrfluxam in rotational crops may need to be re-evaluated.

Animal metabolism

The meeting received information on the fate of orally-dosed inpyrfluxam in rat, lactating goats and laying hens.

Laboratory animals

Metabolism in laboratory animals was summarized and evaluated by the WHO panel of the current Meeting.

Lactating goats

The metabolic fate of inpyrfluxam was investigated in lactating goats using [pyrazolyl-^{14}C] inpyrfluxam and [phenyl-^{14}C] inpyrfluxam. The compound was administered orally once daily (after morning milking) for five consecutive days at 0.51 mg/kg body weight/day (13.74 ppm feed per day) for [pyrazolyl-^{14}C] inpyrfluxam and at 0.64 mg/kg body weight/day (15.74 ppm feed per day) for the [phenyl-^{14}C]inpyrfluxam. Milk was collected twice daily during the dosing period and the goat was sacrificed ca. 6-7 hours after the final dose.

Muscle, liver and kidney samples were extracted twice with acetonitrile:water, once with acetonitrile and for liver and kidney, extracts were further characterized by enzyme hydrolysis using β-glucuronidase. Milk fat was extracted twice with hexane:acetone and once with acetone, while skimmed milk was extracted once with acetone. Fat samples were extracted once with hexane:acetone and twice with acetone.

The majority of the radioactive dose (≥ 76.5 percent of the administered dose (AD)) was found in excreta whilst the highest tissue radioactivity was in liver (up to 0.26 percent AD; 0.35 mg eq/kg) and kidney (up to 0.02 percent AD; 0.17 mg eq/kg). Radioactivity was qualitatively lower in the muscle (up to ≤ 0.01 percent AD; up to 0.024 mg eq/kg) and fat (up to ≤ 0.01 percent AD; up to 0.040 mg eq/kg). In whole milk, the radioactive residues ranged between 0.09–0.12 percent AD for both labels. Residue levels reached a plateau after the first dose (1st day) and very low levels (0.09–0.12 percent AD) were excreted in whole milk. Residues in milk fat were found at 0.011–0.042 mg eq/kg and in skimmed milk at 0.014–0.041 mg eq/kg.

The extraction efficiency was high in liver (up to 91.1 percent TRR; 0.31 mg eq/kg), kidney (up to 98.2 percent TRR; 0.17 mg eq/kg), muscle (up to 100 percent TRR; up to 0.021 mg eq/kg) and fat (up to 96.6 percent TRR ; up to 0.041 mg eq/kg). The PES accounted between 1.9–28.6 percent TRR (≤ 0.001–0.033 mg eq/kg) and were further characterized by β-glucuronidase.

Inpyrfluxam was only quantified at low levels in liver (up to 5.9 percent TRR; 0.019 mg/kg), milk fat (9.1 percent TRR; 0.002 mg/kg) and fat (up to 15.8 percent TRR; 0.004 mg/kg). The major metabolites in the tissue samples were 1'-COOH-S-2840 (up to 49.7 percent TRR; 0.13 mg eq/kg) and 1'-CH_2OH-S-2840 (up to 45.8 percent TRR; 0.079 mg eq/kg) As minor metabolites 3'-OH-S-2840 (up to 3.1 percent TRR; 0.005 mg eq/kg) and DFPA-$CONH_2$ (up to 11.2 percent TRR; 0.008 mg eq/kg) were also detected.

Residues of 1'-COOH-S-2840 were found in fat (up to 46.4 percent TRR; 0.018 mg eq/kg) and skimmed milk (up to 15.9 percent TRR; 0.006 mg eq/kg) and 1'-CH_2OH-S-2840 in fat (up to 35.6 percent TRR; 0.005 mg eq/kg).

Laying hens

The metabolic fate of inpyrfluxam was investigated in laying hens using [pyrazolyl-^{14}C] inpyrfluxam and [phenyl-^{14}C] inpyrfluxam. The compound was administered orally once daily for seven consecutive days at

12.44 ppm feed per day for [pyrazolyl-^{14}C] inpyrfluxam and at 13.13 ppm feed per day for the [phenyl-^{14}C]inpyrfluxam. Eggs and excreta were collected twice daily. The hens were sacrificed approximately 6 hours after the last dose administration.

Excreta, egg, liver, thigh and breast muscle samples were extracted twice with acetonitrile:water once with acetonitrile. Residues in liver extracts were further subjected to enzyme or chemical hydrolysis. Fat samples were extracted once with hexane:acetone and twice with acetone.

The majority of the radioactivity (80.3–81.7 percent AD) was found in excreta whilst the highest tissue radioactivity was found in liver (up to 0.22 percent AD; 0.526 mg eq/kg), in the gastrointestinal tract (up to 1.1 percent AD; 2.48 mg eq/kg) and less than 0.1 percent AD (up to 0.11 mg eq/kg) in the other tissues and eggs. In eggs, levels increased over the 7 day dosing period and plateau levels reached for both radiolabels by the end of day 7. The maximum radioactivity in egg was 0.033 mg/kg (0.01 percent AD).

TRR in eggs were 90–91.3 percent (0.023–0.02 mg eq/kg), with major residues being inpyrfluxam (11.5–11.9 percent TRR, 0.002 mg/kg) and 1'-CH_2OH-S-2840 (29.8–31.6 percent TRR, 0.006–0.008 mg eq/kg). Sulfate conjugates of 1'-CH_2OH-S-2840, 1'-COOH-S-2840, 3'-OH-S-2840 and *N*-des-Me-S-2840 were present at ≤ 9.2 percent TRR (≤ 0.002 mg/kg).

TRR in liver was between 91.4–94.3 percent (0.25–0.32 mg eq/kg), with sulphate conjugates of 1'-CH_2OH-S-2840) the major residue (up to 51.7 percent TRR; up to 0.16 mg eq/kg. Other metabolites identified (free and conjugated) were *N*-des-Me-S-2840 (up to 9.5 percent TRR; up to 0.024 mg eq/kg) and 1'-COOH-S-2840 (up to 11 percent TRR; up to 0.028 mg eq/kg).

TRR in fat (abdominal and subcutaneous) ranged between 96.8–99 percent (0.064–0.102 mg eq/kg), with inpyrfluxam accounting for up to 80.7 percent TRR (0.045–0.075 mg/kg), and sulphate conjugates of 1'-CH_2OH-S-2840 with up to 16.9 percent TRR (up to 0.014 mg eq/kg).

TRR in muscle (thigh and breast) were between 80–92.3 percent (0.012–0.023 mg/kg), with several metabolites identified, with sulphate conjugates of 1'-CH_2OH-S-2840 accounting for 47.7 percent TRR, but at low levels (≤ 0.011 mg/kg).

Summary of animal metabolism

The metabolism of inpyrfluxam in poultry and ruminants demonstrates a comparable metabolite profile. The majority of the administered dose was rapidly excreted and parent was extensively metabolised in several metabolites, proceeding via two main pathways: (a) Oxidation to form 1'-CH_2OH-S-2840 isomers, which is further transformed by conjugation to sulphate or glucuronic acid, or by oxidation to form 1'-COOH-S-2840 isomers and to 1',1'-bis-(CH_2OH)-S-2840. (b) *N*-demethylation to form *N*-des-Me-S-2840, amide cleavage to form DFPA-$CONH_2$ or oxidation to 3'-OH-S-2840.

Methods of analysis

Several analytical methods with minor modifications were available for the determination of inpyrfluxam and its metabolites (3'-OH-S-2840, 1'-CH_2OH-S-2840-A and B, DFPA-$CONH_2$ and 1'-COOH-S-2840-A and B, and N-DesMet-pyrazole carboxylic acid) in plant commodities (apple, maize grain, maize stover, maize forage, soya bean, wheat plant and grain, potato tubers, grapes, soya bean seeds, lettuce, carrot roots and tops). The methods (including RM-50C-1) involve extraction with acetonitrile/water, the extracts partitioned into hexane/ethyl acetate and purified with SPE. The extract was hydrolysed with HCl to release the conjugates before analysis. In method SUM-1701V used only for the determination of *N*-des-Me-S-2840, acetonitrile/water was used for the extraction and no clean-up step was performed. In all

methods, residues were determined by LC-MS/MS with LOQs ranging from 0.005 to 0.02 mg/kg depending on the analyte and the matrix.

For animal commodities, the analytes were extracted with hexane/acetone (1:1) from egg/white/yolk, with hexane/acetone (4:1) from fat and with acetonitrile/water from liver/muscle. In muscle and liver, conjugates were hydrolyzed with HCl and cleaned up with SPE (methods TPR-013 and TPR-015). Final quantification is by LC-MS/MS, with LOQs of 0.01 mg/kg for inpyrfluxam and 0.005 mg/kg for metabolites 1'-COOH-S-2840-A and -B and 1'-CH2OH-S-2840 (A and B).

The extraction efficient of the enforcement QuEChERS method for the analysis of inpyrfluxam in plant and animal commodities, and methods RM-50C-1 and TPR-0013 used for data generation was evaluated. The QuEChERS method was investigated in rice grain, rice straw, soya bean pods and apples, muscle, liver, fat and milk, the RM-50C-1 in rice straw and radish tops and the TPR-0013 in milk, egg, muscle, liver and fat. The level of inpyrfluxam and metabolites extracted with the methods was similar to the level extracted by the original metabolism methods in all matrices.

In conclusion, the provided analytical methods are suitable for the analysis of inpyrfluxam and metabolites 3'-OH-S-2840, 1'-CH_2OH-S-2840 (A and B isomers), DFPA-$CONH_2$, N-DesMet-pyrazole carboxylic acid and 1'-COOH-S-2840 (A and B isomers) in plants and/or animal commodities.

Stability of pesticide residues in stored analytical samples

The Meeting received freezer storage stability data for inpyrfluxam and its metabolites (3'-OH-S-2840, DFPA, DFPA-CONH2, N-DesMet-pyrazole carboxylic acid, N-des-Me-S-2840, N-des-Me-1'-CH2OH-S-2840 (determined separately as A and B isomers), 1'-COOH-S-2840 (determined separately as A and B isomers) and 1'-CH2OH-S-2840 (determined separately as A and B isomers)) in various plant and processing commodities. Samples were fortified with each analyte at 0.05 to 0.5 mg/kg levels.

Residues of inpyrfluxam and metabolites were stable for at least 679 days in high acid content (grapes), 683 days in high oil content (soya bean, maize oil), 672 days in high protein content (field bean), 681 days in high water content (cucumber, apple, maize forage and stover) and 679 days in high starch content (maize grain, corn starch, polished rice, potato starch, potato tuber, wheat grain and flour) crops.

This period covers the storage period of the samples from the supervised and processing studies.

Residues of inpyrfluxam, 1'-COOH-S-2840 (determined separately as A and B isomers), 1'-CH_2OH-S-2840 (determined separately as A and B isomers) in animal matrices (milk, muscle, liver, kidney and fat), with samples fortified with each analyte at 0.1 or 0.5 mg/kg levels. The analytes were shown to be stable for at least 75 day in milk, 29 days in muscle, liver and kidney and 31 days in fat, when stored under frozen conditions. This period covers the storage period of the samples from the feeding studies.

Definition of the residue

Plant commodities

The metabolism of inpyrfluxam was assessed in apple, potatoes, soya bean and rice and found to be similar in all crops. The metabolism in rotational crops was similar to the metabolism observed in primary crops and the processing of inpyrfluxam is not expected to modify the nature of residues.

Inpyrfluxam was the predominant residue in apple (up to 79 percent TRR; 0.24 mg/kg), mature rice grain (up to 78.6 percent TRR; 0.039 mg /kg) and potato tubers (up to 15 percent TRR; 0.002 mg/kg), but was only found at very low concentrations in mature soya seed (up to 2 percent TRR; < 0.001 mg/kg). In feed commodities, parent was detected in soya bean forage (up to 50.5 percent TRR; 0.79 mg /kg),

soya bean hay (up to 22.1 percent TRR; 0.5 mg/kg), immature pods (up to 65.2 percent TRR; 0.41 mg/kg), rice straw (up to 77.8 percent TRR; 0.72 mg/kg) and rice hulls (up to 52.5 percent TRR; 0.88 mg/kg). In the confined rotational crops study, parent was detected in mature lettuce (up to 26.9 percent; 0.027 mg/kg) and radish immature and mature roots (up to 58.9 percent; 0.045 mg/kg) at 30 to 365 days PBI.

Suitable analytical methods for enforcement are available for inpyrfluxam in plant matrices. The Meeting concluded that inpyrfluxam only should be considered as a suitable marker compound for enforcement purposes.

In deciding which compounds should be included in the residue definition for dietary risk assessment, the Meeting considered the likely occurrence of the compounds and the toxicological properties of the candidates 3'-OH-S-2840, 1'-CH_2OH-S-2840, 1'-COOH-S-2840, DFPA, N-DesMet-pyrazole carboxylic acid, DFPA-$CONH_2$ and *N*-des-Me-1'-CH_2OH-S-2840.

3'-OH-S-2840 was not found at significant levels in primary or rotational crop metabolism studies or field rotational crop studies. In residue trials, 3'-OH-S-2840 was detected in apple fruits at a maximum concentration of 0.08 mg/kg, In most trials, parent residues were at least 10–90 times higher compared to 3'-OH-S-2840.

1'-CH_2OH-S-2840 (free or conjugated), was not found in any food commodities at significant levels in primary crop metabolism studies, residue trials or field rotation crops studies. In the confined rotational crops study 1'-CH_2OH-S-2840 (free or conjugated) was detected only in lettuce (24.8 percent TRR; 0.024 mg/kg).

1'-COOH-S-2840 (free or conjugated) was not found in any food commodities at significant levels in primary crop metabolism studies or field rotation crops studies. In the confined rotational crops study, 1'-COOH-S-2840 (free or conjugated) was detected in lettuce (14.6 percent TRR; 0.045 mg/kg) and radish immature or mature tops (22.1 percent TRR; 0.026 mg/kg). In residue trials, the metabolite was found only in sugar beets root at a maximum of 0.028 mg/kg.

The toxicity of these metabolites is covered by the toxicological properties of the parent compound. The Meeting concluded that residues are low compared to inpyrfluxam, do not contribute significantly to the consumer exposure based on parent residues and decided that an inclusion into the residue definition for exposure purposes is unnecessary.

Metabolites DFPA (free and conjugated) and N-DesMet-pyrazole carboxylic acid (free and conjugated) are not covered by the health based reference values for inpyrfluxam, thus the Meeting assessed the relevance of these metabolites against the TTC Cramer Class III (0.0015 mg/kg bw per day).

N-DesMet-pyrazole carboxylic acid (free and conjugated) was found in the metabolism study in soya bean seeds (17.5 percent TRR; 0.038 mg eq/kg) and in residue trials was the only compound detected in soyabean seeds at levels < 0.02 to 0.19 mg/kg (median residue: 0.026 mg/kg). In the confined rotational crops study, the metabolite was detected in radish tops (13.6 percent TRR; 0.015 mg/kg) but not detected in the field rotation crop studies. This metabolite is also a metabolite formed after use of other active substances, such as bixafen, fluxapyroxad, benzovindiflupyr, and fluindapyr. In the absence of overall information on the uses of all active substances and considering the lack of a specific health based reference value, the Meeting decided there was insufficient information to perform a combined risk assessment for residues resulting from use with all active substances leading to formation of N-DesMet-pyrazole carboxylic acid. The Meeting concluded that N-DesMet-pyrazole carboxylic acid could be assessed by TTC approach against the Cramer Class III and that the exposure should be based on the anticipated residues following use of each active substance, separately.

DFPA (free and conjugated) was not found in any food commodities at significant levels in primary crop metabolism studies, residue trials or field rotational crops studies. In the confined rotational crops study, DFPA (free or conjugated) was detected in immature lettuce and mature lettuce (29.1 percent TRR; 0.028 mg/kg).

In summary, the Meeting agreed that the residue definition for dietary risk assessment should be *inpyrfluxam*.

Animal commodities

Inpyrfluxam was observed in poultry fat (up to 81 percent TRR; 0.075 mg eq/kg), goat fat (up to 15.8 percent TRR; 0.004 mg eq/kg) and eggs (up to 11 percent TRR; 0.002 mg eq/kg). In the feeding studies, parent was only present at 0.017 mg/kg in poultry fat from the highest dose group but not in any other animal tissue, milk or eggs.

DFPA-$CONH_2$ was observed in poultry muscle (up to 15 percent TRR; 0.001 mg eq/kg) but was not found in the feeding studies.

1'-COOH-S-2840 (free and conjugates) was observed in poultry liver (up to 11 percent TRR; 0.028 mg eq/kg), poultry muscle (up to 14 percent TRR; 0.003 mg eq/kg), skimmed milk (up to 16 percent TRR; 0.006 mg eq/kg), goat liver (up to 42 percent TRR; 0.13 mg eq/kg), goat kidney (up to 50 percent TRR; 0.08 mg eq/kg), goat muscle (up to 32 percent TRR; 0.007 mg eq/kg) and goat fat (up to 39.7 percent TRR; 0.018 mg eq/kg). In the feeding studies, residues were present only at 0.01 mg/kg in poultry liver.

1'-CH_2OH-S-2840 (free or conjugated) was observed in poultry liver (up to 52 percent TRR; 0.164 mg eq/kg), poultry muscle (up to 51 percent TRR; 0.012 mg eq/kg), fat (up to 17 percent TRR; 0.014 mg eq/kg), eggs (up to 39 percent TRR; 0.009 mg eq/kg), goat liver (up to 25 percent TRR; 0.088 mg eq/kg), goat kidney (up to 37 percent TRR; 0.063 mg eq/kg) and goat muscle (up to 32 percent TRR; 0.007 mg eq/kg). In the feeding studies, residues were present at 0.012 mg/kg in egg yolk, 0.017 mg/kg in poultry liver, 0.014 mg/kg in goat liver and at 0.022 mg/kg in goat kidney.

3'-OH-S-2840 and *N*-des-Me- S-2840 were not found in any food commodities at levels > LOQ nor were residues found in the feeding studies.

Besides parent inpyrfluxam, 1'-CH_2OH-S-2840 (free or conjugated) was a major residue in most animal matrices and the predominant residue found in the livestock feeding studies. Suitable analytical methods for enforcement are available for inpyrfluxam and 1'-CH_2OH-S-2840 (free or conjugated) in animal matrices. The Meeting decided that the sum of both compounds represents a suitable marker for enforcement purposes in animal matrices.

For dietary exposure purposes, the only other metabolite found in feeding studies at quantified levels was 1'-COOH-S-2840 (free and conjugates) in poultry liver (0.01 mg/kg). Given its low occurrence and that its toxicity is covered by the health-based guidance values for parent inpyrfluxam, the Meeting decided that no inclusion into the residue definition is necessary.

Parent inpyrfluxam has an octanol-water partition coefficient of 3.65, suggesting potential accumulation in fat.

Parent and 1'-CH_2OH-S-2840 residues were predominantly found in liver and kidney commodities. In goats, concentrations between fat and muscle were close to the LOQ without clear tendency for accumulation in the fat. In poultry metabolism studies, fat contained approximately 30x higher residue concentrations compared to muscle. However, no accumulation was observed in milk fat or egg yolk, In

the feeding studies, residues were generally low, not allowing estimation of ratios between fatty and non-fatty tissues. The Meeting decided that the residue is not fat-soluble

In summary, the Meeting agreed that the residue definition for compliance with the MRL and dietary risk assessment for animal commodities should be: *inpyrfluxam and 1'-CH_2OH-S-2840 (free or conjugated) expressed as inpyrfluxam*.

The residue is not fat-soluble.

In deciding which compounds should be taken into consideration for estimation of livestock dietary burdens, the Meeting decided that inpyrfluxam and 1'-CH2OH-S-2840 (free or conjugated) expressed as inpyrfluxam, should be taken into consideration for estimation of livestock dietary burden calculations since they are included in the residue definition for animal commodities and are found in feed commodities in metabolism (soya bean forage, soya bean hay, rice straw, rice hulls, sorghum forage, sorghum stover) and field studies (wheat straw, peanut hay) in primary and rotational crops.

Conclusion

Based on the above the Meeting recommended the following residue definitions:

Residue definition for compliance with the MRL and dietary exposure for plant commodities: *inpyrfluxam*.

Residue definition for compliance with the MRL and dietary exposure for animal commodities is *inpyrfluxam and 1'-CH_2OH-S-2840 (free or conjugated) expressed as inpyrfluxam*.

Residues to be included in the livestock dietary burden calculations: *inpyrfluxam and 1'-CH_2OH-S-2840 (free or conjugated) expressed as inpyrfluxam*.

Results of supervised residue trials on crops

The Meeting received supervised residue trial data for seed treatments and foliar applications of inpyrfluxam on apples, soya beans, maize, peanuts, sugar beets and rice.

In this appraisal, the following residue summaries are given:

- *Inpyrfluxam:* For maximum residue level estimation in plant commodities and dietary exposure calculations.
- *Inpyrfluxam and 1'-CH_2OH-S-2840 (free or conjugated) expressed as inpyrfluxam*: For dietary burden calculations. When the residues of the metabolite was <LOQ (0.02 mg/kg) then it was not taken into consideration in the animal dietary burden calculations
- *N-DesMet-pyrazole carboxylic acid*: For dietary exposure calculations based on TTC approach.

Apples

The critical GAP for inpyrfluxam on apples is from Japan, and consists of a maximum of three foliar applications at a rate of 9.25 g ai/hL with a re-treatment interval of 7 days and a PHI of 1 day.

Trials performed on apples from Japan matching this GAP were available. Residue levels in fruits in ranked order were (n = 8): 0.52, 0.72, 0.78, 0.84, 0.98, 1.23, 1.42, 1.88 mg/kg.

The Meeting estimated a maximum residue level of 4 mg/kg, an STMR of 0.91 mg/kg and an HR of 1.88 mg/kg for apples. Residues of metabolite 1'-CH_2OH-S-2840 were < 0.02 mg/kg in all trials.

Soya bean

The critical GAP for inpyrfluxam on soya beans is from the United States and consists of a seed treatment up to 5 g ai/100 kg seeds and two foliar application at a rate of 75 g ai/ha (not before BBCH 14 or after BBCH 75–76) with an retreatment interval of 14 days and a PHI covered by the growth stage of the crop with a livestock grazing restriction: do not graze treated fields or feed treated hay to livestock.

In trials performed on soya beans in the United States, the seeds were treated at 10 g ai/100 kg, followed by two applications at 100 g ai/ha (one at 200 g ai/ha) at BBCH above 75 and, in most cases. Residue levels in dry seeds were (n = 21): < 0.01 (21) mg/kg.

Since residues of inpyrfluxam were not detected in any overdosed trials, the Meeting estimated a maximum residue level of 0.01(*) mg/kg and an STMR of 0 mg/kg for soya bean (dry).

Residue levels of metabolite N-des-Me-DFPA in dry seeds were (n = 21): < 0.02 (7), 0.02, 0.023, 0.024, 0.026, 0.028, 0.032, 0.036, 0.037, 0.051, 0.062, 0.095, 0.13, 0.16, 0.19 mg/kg. Residues of metabolite 1'-CH_2OH-S-2840 were < 0.02 mg/kg in all trials.

Sugar beet

The critical GAP for inpyrfluxam on sugar beet is in the United States and consists of a seed treatment up to 0.1 g ai/100,000 seeds and up to two foliar applications at a rate of 50g ai/ha with a maximum seasonal rate of 100 g ai/ha (BBCH 12–18), an retreatment interval of 21 days and a PHI of 50 days.

In trials performed on sugar beets, the plant received two foliar application at 100 g ai/ha after the seed treatment. Residue levels in roots were (n = 15): < 0.01 (15) mg/kg.

Since residues of inpyrfluxam were not detected in any overdosed trials, the Meeting estimated a maximum residue level of 0.01(*) mg/kg, an STMR and HR of 0 for beet root. Residues of metabolite 1'-CH_2OH-S-2840 were < 0.02 mg/kg in all trials.

Rice

The critical GAP for inpyrfluxam on rice is from the United States and consists of a seed treatment up to 10 g ai/100 kg seeds and one foliar application at a rate of 100 g ai/ha with a maximum seasonal rate of 100 g ai/ha (approximately 25–30 days after the permanent flood has been established) and PHI covered by the growth stage.

Trials performed on rice, according to this GAP, gave residue levels in husked rice of (n = 14): < 0.01 (15) mg/kg. Three trials conducted at approximately 500 g ai/ha gave the same results.

Since residues of inpyrfluxam were not detected in any trial, including the overdosed trials, the Meeting estimated a maximum residue level of 0.01(*) mg/kg, an STMR of 0 for husked rice. Residues of metabolite 1'-CH_2OH-S-2840 were < 0.02 mg/kg in all trials.

Sweet corn (Corn-on-the-cob)

The critical GAP for inpyrfluxam on maize is from the United States and consists of a seed treatment up to 0.014 mg ai/seed and one in furrow application at a rate of 50 g ai/ha at planting and PHI covered by the growth stage.

Fourteen trials performed on maize in the United States matching this GAP and three additional trials at exaggerated rates of 100–260 g ai/ha were also available. Residue levels in kernels plus corn without husks in all trials were (n = 17):< 0.01 (17) mg/kg.

Since residues of inpyrfluxam were not detected in the trials including the overdosed trials and in primary plant metabolism studies, uptake and transport of inpyrfluxam in the maize, sorghum, oilseed and potato studies, where the seed was treated, was low, the Meeting estimated a maximum residue level of 0.01(*) mg/kg, an STMR and HR of 0 mg/kg for sweet corn. Residues of metabolite 1'-CH_2OH-S-2840 were < 0.02 mg/kg in all trials.

Peanut

The critical GAP for inpyrfluxam on peanuts is in the United States and consists of a maximum of four foliar applications at a rate of 100 g ai/ha (no earlier than 30 days after planting) with a maximum seasonal rate of 200 g ai/ha, an retreatment interval of 14–28 days and a PHI of 40 days.

Trials performed on peanuts from the United States according to GAP (2 × 100 g ai/ha) were available. Residue levels in nutmeal were (n = 13): < 0.01 (13) mg/kg.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg, an STMR and HR of 0.01 mg/kg for peanuts. Residues of metabolite 1'-CH_2OH-S-2840 were < 0.02 mg/kg in all trials.

Maize

The critical GAP for inpyrfluxam on maize is from the United States and consists of a seed treatment up to 0.014 mg ai/seed and one in furrow application at a rate of 50 g ai/ha at planting and PHI covered by the growth stage.

Fourteen trials performed on maize from the United States matching the GAP and three trials at exaggerated rates of 100–260 g ai/ha were also available. Residue levels in all trials were (n = 27): < 0.01 (27) mg/kg.

Since residues of inpyrfluxam or the metabolites were not detected in the trials including the overdosed trials and in primary plant metabolism studies, uptake and transport of inpyrfluxam in the maize, sorghum, oilseed and potato studies, where the seed was treated, was low, the Meeting estimated a maximum residue level of 0.01(*) mg/kg, an STMR of 0 mg/kg for maize and popcorn, Residues of metabolite 1'-CH_2OH-S-2840 were < 0.02 mg/kg in all trials.

Animal Feed commodities

The total residue for estimating the median and highest residues in feed was inpyrfluxam **+** 1'-CH_2OH-S-2840 (free or conjugated)**,** expressed as inpyrfluxam**.**

When the metabolite concentration was < 0.04 mg/kg, it was considered to be 0 in the calculation.

Maize forage-40 percent dry matter

The critical GAP for inpyrfluxam on maize is from the United States and consists of a seed treatment up to 0.014 mg ai/seed and one in furrow application at a rate of 50 g ai/ha at planting and PHI covered by the growth stage.

Trials performed on maize from the United States matching the US GAP were available. Residue levels in forage were (n = 27): < 0.01 (4), < 0.02 (23) mg/kg.

Residues of metabolite *1'-CH_2OH-S-2840,* were < 0.04 mg/kg in all trials.

The Meeting estimated a highest residue of 0.02 mg/kg (as received) for inpyrfluxam in maize forage.

Maize stover–83 percent dry matter

The critical GAP for inpyrfluxam on maize is from the United States and consists of a seed treatment up to 0.014 mg ai/seed and one in furrow application at a rate of 50 g ai/ha at planting and PHI covered by the growth stage.

Trials performed on maize from the United States matching the US GAP were available. Residue levels in fodder were (n = 27): < 0.01 (4),< 0.02 (23) mg/kg. Residues of metabolite 1'-CH_2OH-S-2840 were < 0.04 mg/kg in all trials.

The Meeting estimated a maximum residue level of 0.02(*) mg/kg (based on a dry matter content of 83 percent), a median and highest residue of 0.02 mg/kg for inpyrfluxam in maize fodder (as received).

Peanut hay

The critical GAP for inpyrfluxam on peanuts is in the United States and consists of a maximum of four foliar applications at a rate of 100 g ai/ha (no earlier than 30 days after planting) with a maximum seasonal rate of 200 g ai/ha, an retreatment interval of 14–28 days and a PHI of 40 days.

Trials performed on peanuts from the United States matching GAP were available. Residue levels in hay were (n = 12): 0.083, 0.096, 0.134, 0.217, 0.218, 0.252, 0.338, 0.412, 0.422, 0.526, 0.731, and1.23 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg (as received), an median of 0.35 mg/kg and highest residue of 2 mg/kg for peanut hay (as received).

Levels of metabolite 1'-CH_2OH-S-2840 were (n = 12): 0.1, 0.12, 0.20, 0.24, 0.25, 0.32, 0.38, 0.44, 0.57, 0.94, 1.12 and 2 mg/kg.

Since metabolite 1'-CH2OH-S-2840 is included in the residue definition for dietary risk assessment in animal commodities, residues of this metabolite (CF to parent = 1.1) were also taken into consideration as to estimate the median and highest residue for inpyrfluxam and 1'-CH2OH-S-2840 (, free or conjugated) expressed as inpyrfluxam in peanut hay. Total residue levels were (n = 12): 0.1, 0.12, 0.20, 0.24, 0.25, 0.32, 0.38, 0.44, 0.57, 0.94, 1.12, 2 mg/kg.

The Meeting also estimated a median of 0.35 mg/kg and highest residue of 2 mg/kg for peanut hay (as received).

Fates of residues during processing

High temperature hydrolysis

The degradation of [Pyrazolyl-4-^{14}C] inpyrfluxam and its metabolites [Pyrazolyl-4-^{14}C] 3'-OH-S-2840 and [Pyrazolyl-4-^{14}C] 1'-CH_2OH-S-2840 were studied under hydrolytic conditions at high temperatures in sterile aqueous buffers at pH 4, 5 and 6 for periods up to 60 minutes (20 minutes for pH 4 and 6) so as to simulate common processing practice (pasteurisation, baking/brewing/boiling and sterilisation). Degradation of inpyrfluxam, 3'-OH-S-2840 and 1'-CH_2OH-S-2840 was not observed at any investigated condition. Chiral analysis showed the *R*-isomer of inpyrfluxam to account for 100 percent of the residue at all time points confirming no isomerization occurred. The Meeting concluded that inpyrfluxam, is stable under hydrolytic conditions.

Residues in processed commodities

The fate of inpyrfluxam residues after processing has been examined in apple, soya bean, sugar beet, rice, maize and peanut.

For soya bean, sugar beet, rice, maize and peanut processed fractions no reliable PFs can be calculated, since the RAC and the processed commodities contained residues <LOQ. In rice, residues in RAC were also <LOQ however quantitative residues were observed in hulls and bran indicating that residues concentrated in the final processed commodities.

One study was conducted in rice and one study in apple. Maximum residue levels in processed commodities are only estimated when processing factor was higher than 1. The Meeting concluded that the processing factors based on apple data can be extrapolated to other processed pome commodities (pome fruit juice and wet pomace). The results are shown in Tables 140 and 141.

Table 140 Processing factors and median and highest residue values for inpyrfluxam used for estimation of maximum residue levels including livestock dietary burdens

Processed commodity	Raw commodity [median] mg/kg	Individual processing factors	Median residue-P (mg/kg)
Apple wet pomace	0.91	2.7	2.4
Rice Hulls	0	4.3	0
Rice bran (husked)	0	1.3	0

Table 141 Processing factors and STMR value for inpyrfluxam in apple juice

Processed commodity	Raw commodity [STMR]	Individual processing factors	Median or best estimate processing factor	STMR-P = STMRRAC × PF (mg/kg)
Apple juice	0.91	0.125	0.125	0.114

The Meeting estimated a maximum residue level of 0.01(*) mg/kg for inpyrfluxam in rice hulls

Residues in animal commodities

In a feeding study in lactating cows, inpyrfluxam was fed via the diet, to three to six cows per dose group, for 29 consecutive days. The animals received equivalents of 2, 6, or 20 ppm of inpyrfluxam in the diet (DM). Residues of inpyrfluxam and metabolites 1'-COOH-S-2840 (A and B) and 1'-CH_2OH-S-2840 (A and B) were determined.

There was no transfer of residues of inpyrfluxam or its main metabolites (1'-COOH-S-2840 and 1'-CH_2OH-S-2840) at levels above the LOQ (0.01 mg/kg) in milk, skimmed milk or cream during or up to two weeks after 28 days at any dose. Similarly, there were no residues above the LOQ (0.01 mg/kg) or preferential accumulation to bovine muscle or fat. Only residues of metabolite 1'-CH_2OH-S-2840 in liver at the 20 ppm DM (0.014 mg/kg) and in kidney at the 6 ppm DM (0.013 mg/kg) and 20 ppm DM (0.022 mg/kg) were detected.

In a feeding study in laying hens twelve to twenty-four hens/treatment group were dosed with inpyrfluxam for 28 days, at feeding levels equivalent to 1, 3, and 10 ppm of inpyrfluxam in the diet (DM). Residues of inpyrfluxam and metabolites 1'-COOH-S-2840 (A and B) and 1'-CH_2OH-S-2840 (A and B) were determined.

Inpyrfluxam and 1'-CH_2OH-S-2840 were found in liver at 0.01/0.017 mg/kg at the 3/10 ppm levels and in fat and egg yolk at 0.017 mg/kg 0.012 mg/kg, respectively, at the 10 ppm. No discernible plateau of residues was observed because of the low levels of residues in all samples. Samples of muscle had no residues at or above the LOQ throughout the dosing period and for two weeks after cessation of dosing, suggesting limited transfer from feed to tissues.

Farm animal dietary burden

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the JMPR by the current Meeting including processed and forage commodities. Those commodities are included in Table 142.

Table 142 Processed and forage commodities used in estimating livestock dietary burdens

Codex classification	Commodity	Median residue (-P) (mg/kg)	Highest residue (-P) (mg/kg)
AL 3352	Peanut hay	0.35 [a]	2 [a]
GC 0649	Rice grain	0	n.a
AB 1230	Apple pomace, wet	2.4	n.a
AM3599	Sugar beet, pulp, dry	0	n.a
DM 0596	Sugar beet molasses	0	n.a
AS 3570	Rice, hulls	0	n.a
CF 0649	Rice bran, processed	0	n.a
GC 0645	Maize	0	n.a
AS 0645	Maize, forage (40% DM)	0.02	n.a
GC 0656	Popcorn	0.02	n.a
AS 3558	Maize stover (83% DM)	0.02	0.02
VD 0541	Soya bean	0	n.a

Note:
a residues are calculated as inpyrfluxam and 1'-CH_2OH-S-2840 (free or conjugated) expressed as inpyrfluxam.

The dietary burdens, estimated using the 2018 OECD Feed diets listed in Appendix XIV Electronic attachments to the 2016 edition of the FAO manual[1], are presented in Annex 6 and summarized below in Table 143.

Table 6143 Estimated maximum and minimum dietary burdens of farm animals

	Animal dietary burden: parent ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0.008	0.008	1.24	1.24	2.62❶	1.46❷	0	0
Dairy cattle	0.98	0.68	0.63	0.63	2.03	0.86	0.025	0.025
Poultry–broiler	0	0	0	0	0	0	0	0
Poultry–layer	0	0	0	0	0	0	0	0

Notes:
❶Highest maximum beef or dairy cattle dietary burden suitable for maximum residue level estimates for mammalian tissues
❷Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues and milk.

The Meeting used the calculated beef and dairy cattle maximum and mean dry weight dietary burdens of 2.62 ppm and 1.46 ppm for estimating residue levels in milk and ruminant tissues.

For poultry commodities, no feed items were applicable thus the calculated dry weight maximum and mean dietary burden and is 0 ppm dry weight in feed.

[1] http://www.fao.org/agriculture/crops/thematic-sitemap/theme/pests/jmpr/jmpr-docs/en/

Animal commodity maximum residue levels

Ruminants

The calculations used to estimate maximum residue levels, STMR and HR values for cattle matrices are shown in Table 144.

Table 144 Anticipated residues of inpyrfluxam and 1'-CH_2OH-S-2840 (free or conjugated) expressed as inpyrfluxam in cattle commodities

	Feed Level (ppm) for milk residues	Total residues (mg eq/kg) in milk	Feed Level (ppm) for tissue residues	Total residues (mg eq/kg)			
				Muscle	Liver	Kidney	Fat
HR Determination (beef or dairy cattle)–Parent							
Feeding Study	2	0.02*	2	0.02*	0.02*	0.02*	0.02*
	6	0.02*	6	0.02*	0.02*	0.02*	0.02*
Dietary burden and estimate of highest residue	2.6	0.02*	2.6	0.02*	0.02*	0.02*	0.02*
STMR Determination (beef or dairy cattle)–Parent							
Feeding Study	2	0.02*	2	0.02*	0.02*	0.02*	0.02*
	6	0.02*	6	0.02*	0.02*	0.02*	0.02*
Dietary burden and estimate of highest residue	1.46	0.02*	1.46	0.02*	0.02*	0.02*	0.02*
MRL Determination (beef or dairy cattle)–Parent							
Feeding Study	2	0.01*	2	0.02*	0.02*	0.02*	0.02*
	6	0.01*	6	0.02*	0.02*	0.02*	0.02*
Dietary burden and estimate of highest residue	2.6	0.01*	2.6	0.02*	0.02*	0.02*	0.02*

The Meeting estimated maximum residue levels of 0.02(*) mg/kg in milk, meat (mammalian except marine mammals), mammalian fats, and edible offal.

The Meeting also estimated a STMR of 0 mg/kg for edible offal, muscle, kidney, fat and milk, and a HR of 0 mg/kg for edible offal, muscle, edible offal and fat.

Poultry

As the mean and maximum dietary burden for poultry is 0, no residues are expected in poultry commodities.

The Meeting estimated maximum residue levels of 0.02(*) mg/kg in poultry meat (muscle), poultry fat, poultry edible offal, and eggs. The Meeting also estimated a HR and STMR of 0 mg/kg for poultry edible offal, muscle, fat and eggs.

Recommendations

On the basis of the data from supervised trials, processing studies, storage stability studies and feeding studies the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

The residue definition for compliance with the MRL for plant commodities is *inpyrfluxam*.

The residue definition for dietary exposure for plant commodities is *inpyrfluxam.*

The residue definition for compliance with the MRL and dietary exposure for animal commodities is *inpyrfluxam and 1'-CH_2OH-S-2840 (free or conjugated) expressed as inpyrfluxam*.

The residue is not fat soluble.

CCN	Commodity name	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FP 0226	Apples	4	-	0.91	1.88
JF 0226	Apple, juice		-	0.114	
MO 0105	Edible offal (mammalian)	0.02*	-	0	0
PE 0112	Eggs	0.02*	-	0	0
MF 0100	Mammalian fats	0.02*	-	0	0
AS 3558	Maize stover	0.02* (dw)	-	Median: 0.02 (ar)	Highest: 0.02 (ar)
GC 0645	Maize grain	0.01*	-	0	-
MM 0095	Meat from mammals other than marine mammals	0.02*	-	0	0
ML 0106	Milks	0.02*	-	0	
SO 0697	Peanut	0.01*	-	0.01	
AL 0697	Peanut, hay and/or straw	3	-	0.35	2
GC 0656	Popcorn	0.01*	-	0	-
PO 0111	Poultry edible offal	0.02*	-	0	0
PM 0110	Poultry meat	0.02*	-	0	0
PF 0111	Poultry fat	0.02*	-	0	0
GC 0649	Rice, husked	0.01*	-	0	
AS 3570	Rice, hulls	0.01*			
VD 0541	Soya bean (dry)	0.01*	-	0	-
VR 0596	Sugar beet roots	0.01*	-	0	0
GC 0447	Sweet corn (Corn-on-the-cob) (kernels plus cob with husk removed)	0.01*	-	0	0

Note:
dw= dry weight basis; as= as received

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for inpyrfluxam is 0–0.06 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for fenpicoxamid were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 0–5 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of inpyrfluxam from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for inpyrfluxam is 0.3 mg/kg bw The International Estimate of Short Term Intakes (IESTIs) for spiropidion were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs varied from 0–40 percent of the ARfD for children and 0–10 percent of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of inpyrfluxam from uses considered by the present Meeting is unlikely to present a public health concern.

Threshold of toxicological concern (TTC) consideration for metabolites

The metabolite N-DesMet-pyrazole carboxylic acid found in soya bean seeds (STMR of 0.026 mg/kg) was assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day). The estimated a dietary exposure for metabolite N-DesMet-pyrazole carboxylic acid of 0.096 µg/kg bw for the uses of bixafen, fluxapyoxad, fluindapyr and benzovindiflupyr (Report 2022–fluindapyr).

The Meeting concluded that the estimated dietary exposure to residues of N-DesMet-pyrazole carboxylic acid from uses considered by the current JMPR is below the TTC for Cramer Class III compounds and is unlikely to present a public health concern. Should further uses be considered in the future, these conclusions may need to be re-evaluated.

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ISOFLUCYPRAM (330)

First draft prepared by J Giordano, Environmental Protection Agency, United States of America

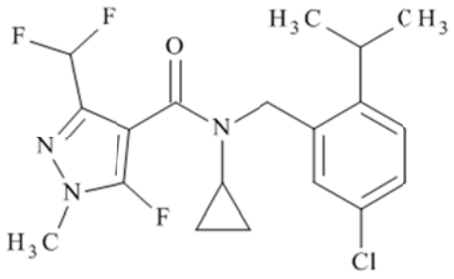
EXPLANATION

Isoflucypram is a broad-spectrum fungicide of the chemical class of N-cyclopropyl-N-benzyl-pyrazole-carboxamides. Isoflucypram is a succinate dehydrogenase (SDH) inhibitor assigned to the Fungicide Resistance Action Committee (FRAC) resistance Group 7.

Isoflucypram was scheduled at the Fifty-first Session of the CCPR for evaluation as a new compound in 2020 and rescheduled to the 2022 JMPR. All relevant information in terms of chemical identity, physical and chemical properties, metabolism and environmental fate, methods of residue analysis, storage stability, intended use patterns, supervised residue trials, fate of residues upon processing, and farm animal feeding studies were submitted for evaluation by the 2022 JMPR.

IDENTITY

Information on the chemical identity of isoflucypram is summarised below.

ISO common name:	Isoflucypram
IUPAC name:	N-(5-chloro-2-isopropylbenzyl)-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide
CA name:	N-[[5-chloro-2-(1-methylethyl)phenyl]methyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide
CAS No.:	1255734-28-1
CIPAC No.:	Not allocated
Manufacturer's experimental name:	BCS-CN88460
Structural formula:	
Molecular formula:	$C_{19}H_{21}ClF_3N_3O$
Molecular weight:	399.84 g/mol

Physical and Chemical Properties

Property	Results	Test material purity and specification	Reference
Appearance	White powder	Pure (99.1 %)	PA14/031
	Light beige powder	Technical (98.6 %)	PA17/054
Odour	Odourless	Pure (99.1 %)	PA14/031
	Weak odour, not characteristic	Pure (98.6 %)	PA17/054
Melting point	108.8 °C	Pure (99.1 %)	20140107.01
Boiling point	No endothermic effect was observed between the melting of the test item and its decomposition starting at 215 °C (thermal stability). Therefore the test item has no boiling point at atmospheric conditions.	Pure (99.1 %)	20140107.01

Property	Results	Test material purity and specification	Reference
Thermal Stability	Pure: The test item showed an endothermic effect in the temperature range 105-130 °C. The endothermic effect was followed by an exothermic effect in the temperature range 215-395 °C with an energy of 1239 J/g and 1040 J/g, respectively. No further endothermic or exothermic effects were observed up to the final temperature (400°C), with a decomposition energy greater than -380 J/g. Technical compound: The test item showed an endothermic effect in the temperature range 100-120 °C. The endothermic effect was followed by an exothermic effect in the temperature range 360-440 °C, with a decomposition energy greater than -1110 J/g.	Pure (99.1 %) Technical (98.6 %)	20140107.01, CSL-14-0428.01, CSL-17-1436.01
Flammability	No flammability properties	Technical (98.6 %)	PS20170460-2
Auto-flammability	No self-heating properties	Technical (98.6 %)	PS20170460-3
Explosive properties	No explosive properties	Technical (98.6 %)	CSL-17-1436.01
Oxidising properties	No oxidising properties	Technical (98.6 %)	M-612014-01-1
Surface tension	68.2 mN/m at 20 °C	Pure (99.1 %)	PA14/059
Relative density	D_4^{20} = 1.22	Pure (99.1 %)	PA14/035
	D_4^{20} = 1.31	Technical (98.6 %)	PA17/051
Vapour pressure	Vapour pressure values extrapolated (vapour pressure balance method): 1.2×10^{-7} Pa at 20 °C 2.8×10^{-7} Pa at 25 °C 1.5×10^{-5} Pa at 50 °C	Pure (99.1 %)	CSL-14-0428.01
Henry's law constant	2.7×10^{-5} Pa m^3 mol^{-1} at 20 °C	Pure (99.1 %)	AF14/022
Solubility in water	1.8 mg/L at 20 °C	Pure (99.1 %)	PA14/030
Solubility in organic solvents	At 20 °C: Heptane 1.2 g/L Toluene > 260 g/L Dichloromethane > 260 g/L Methanol 97 g/L Acetone > 260 g/L Ethyl acetate > 260 g/L Dimethyl sulfoxide > 260 g/L	Pure (99.1 %)	PA14/060
Octanol/water partition coefficient	At 25 °C Pow log Pow pH 4 10000 4.0 pH 7 10000 4.0 pH 9 10000 4.0	Pure (99.1 %)	PA14/029
Hydrolysis rate at pH 4, 7,and 9	At 70 °C: stable at all pH values, therefore no DT_{50} and DT_{90} values were calculated.	Radiochemical purity: >98 % Chemical purity: >98 %	EnSa-14-1032
Direct photo-transformation	At 24.5 °C and pH 7: Experimental DT_{50}: 150 days Environmental DT_{50}: -484 solar summer days at Phoenix (Arizona, USA) -750 solar summer days at Athens (Greece)	Radiochemical purity: >98 % Chemical purity: >99 %	EnSa-14-1033

Property	Results	Test material purity and specification	Reference
Quantum yield of direct transformation	Mean Φ = 0.00077	Radiochemical purity: >99 % Chemical purity: >99 %	EnSa-13-0236
Dissociation constant	No dissociation constant (pKa) was determined in aqueous solution of isoflucypram in the pH range of 1<pH<12. Isoflucypram shows no acidic or basic properties in the range of 1<pKa<12.	Pure (99.1 %)	PA14/048

METABOLISM AND ENVIRONMENTAL FATE

The studies for plant metabolism, animal metabolism, rotational crops, and environmental degradation were conducted with isoflucypram radiolabelled in either the pyrazole or phenyl moiety, as shown below. (*) indicates the position of ^{14}C-radiolabel. The metabolites or degradates found in metabolism and environmental fate studies are shown in Table 1.

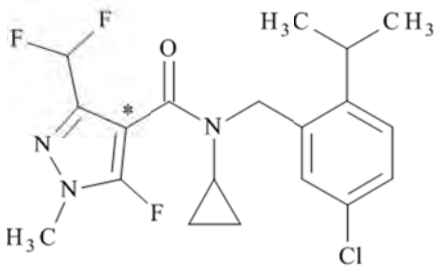
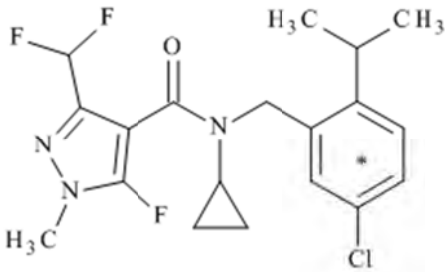
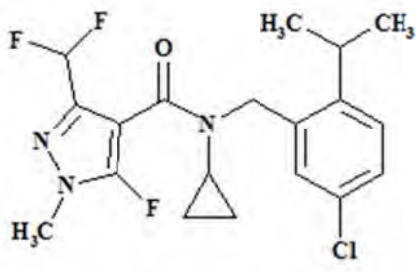
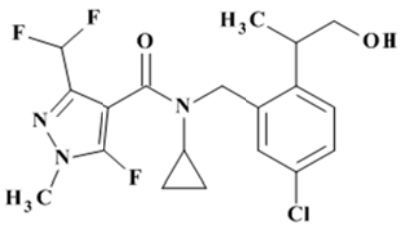
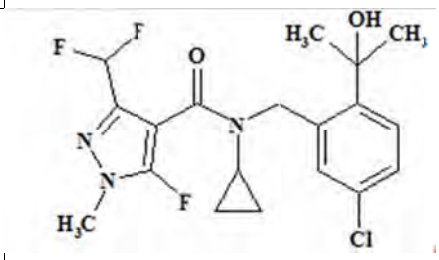
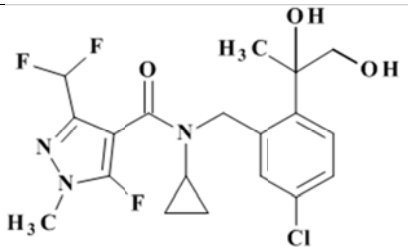
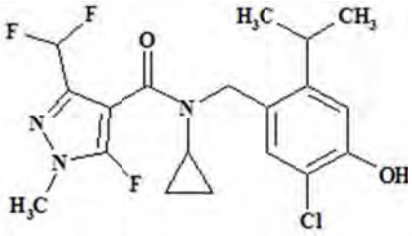
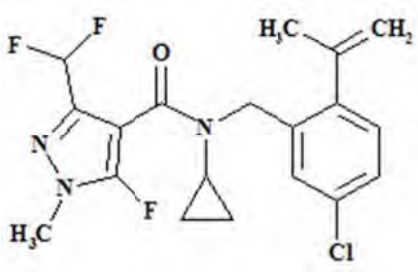
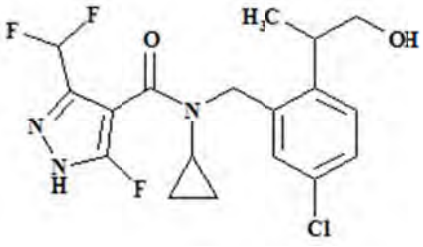
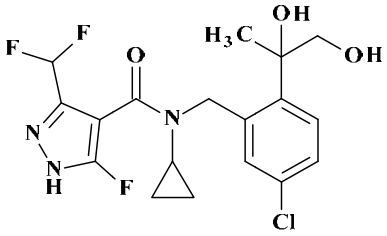
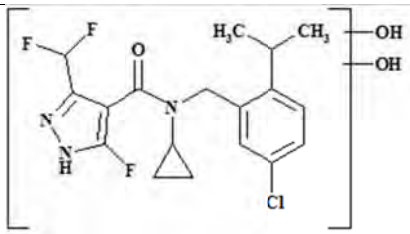
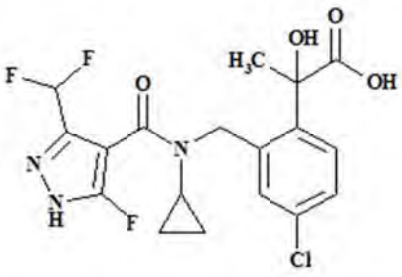
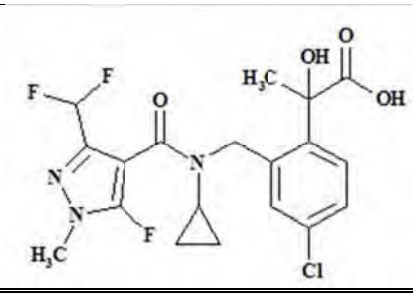
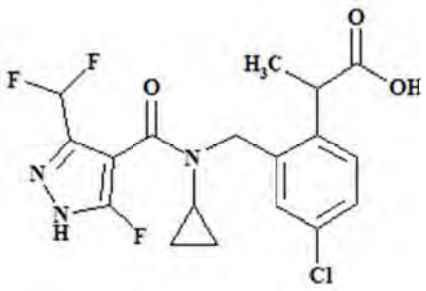
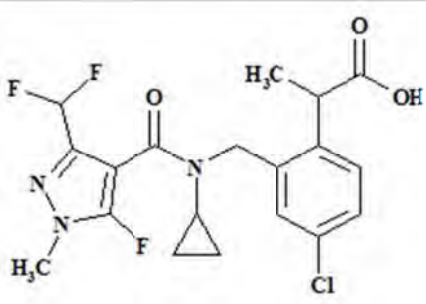
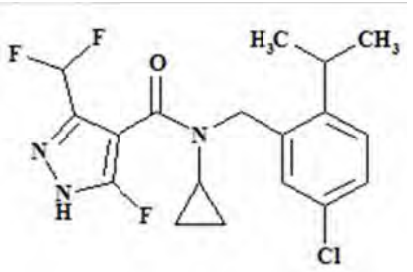
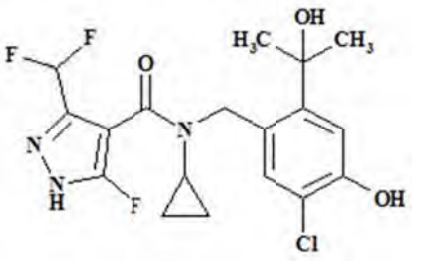
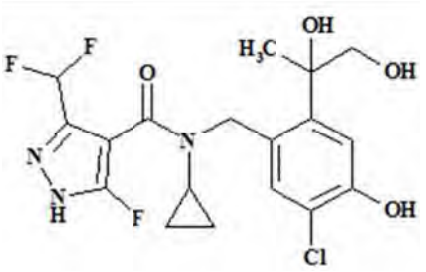
Pyrazole label	Phenyl label
	
[pyrazole-4-^{14}C]isoflucypram	[phenyl-UL-^{14}C]isoflucypram

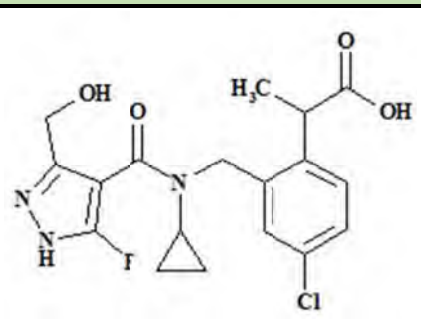
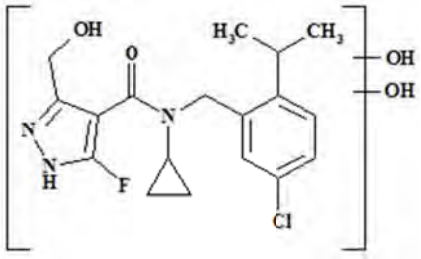
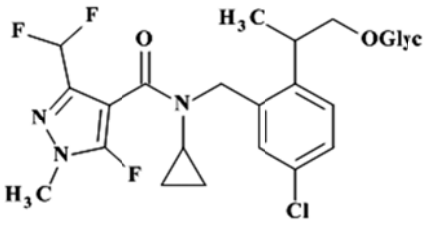
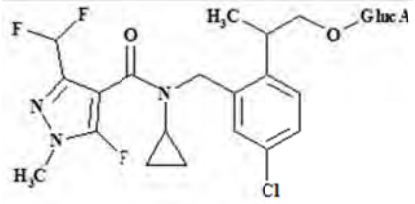
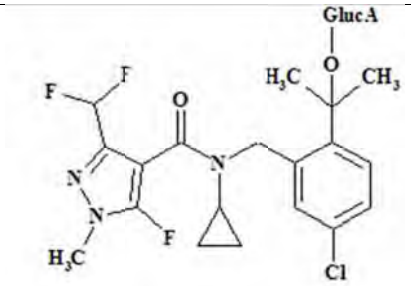
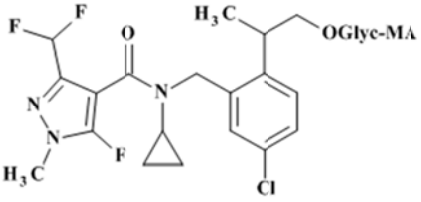
Table 1 Isoflucypram and metabolites/degradates found in metabolism and environmental fate studies

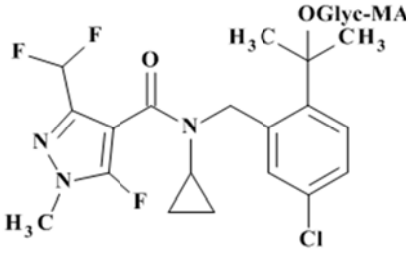
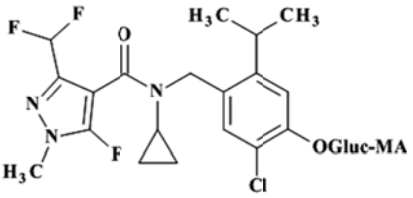
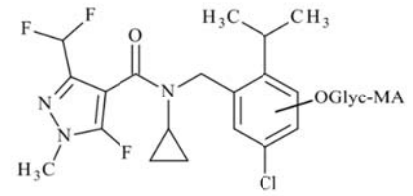
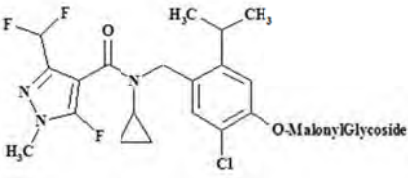
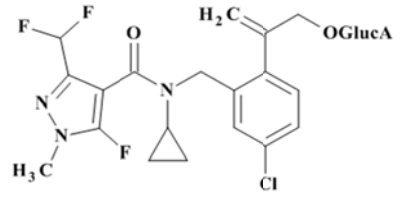
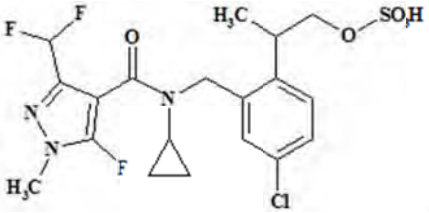
Number Name Identity (IUPAC)	Structure	Identified in:
Isoflucypram BCS-CN88460 CAS: 1255734-28-1 N-(5-chloro-2-isopropylbenzyl)-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide		Animal: Rat (faeces, liver, kidney); hen (eggs, muscle leg, fat); goat (milk, muscle, fat, liver, kidney, faeces) sunfish (edible parts, viscera) Plant: Soya bean (forage, hay, straw, seed); wheat (hay, straw, grain); CRC (wheat forage, Swiss chard, turnip leaves); oilseed rape (intermediate harvest, mature plants, seeds), tomatoes, potato (tubers, leaves) Soil: Aerobic & anaerobic, field dissipation, photolysis Water: Hydrolysis, photolysis, water-sediment

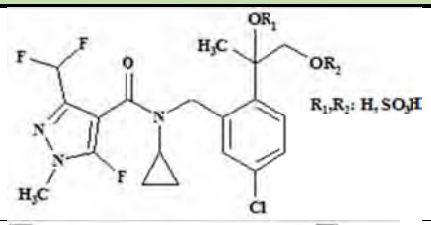
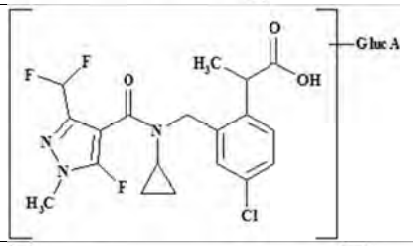
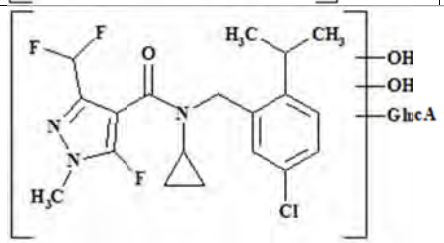
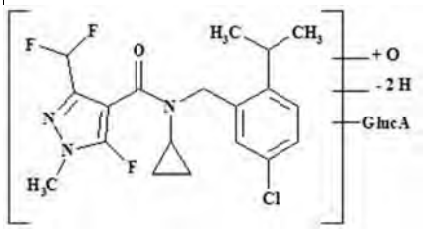
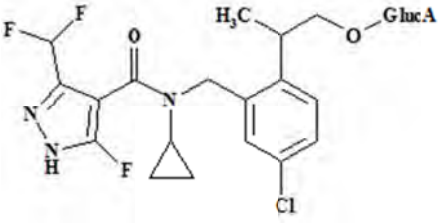
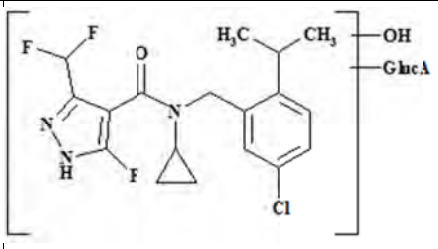
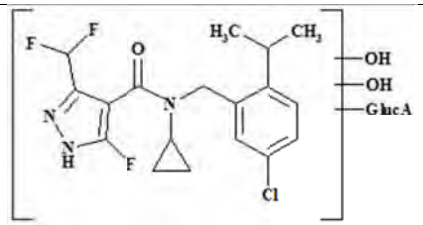
Number Name Identity (IUPAC)	Structure	Identified in:
M01 Isoflucypram-propanol N-[5-chloro-2-(1-hydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide		Animal: Rat (faeces); hen (eggs, muscle, fat, liver, excreta); goat (muscle, fat, liver, kidney, faeces, urine); sunfish (edible parts, viscera) Plant: Wheat (hay, straw)
M02 Isoflucypram-2-propanol 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)-methyl]phenyl}propan-2-yl		Animal: Rat (faeces); goat (milk, muscle, fat, liver, kidney, faeces, urine)
M03 Isoflucypram-1,2-propandiol N-[5-chloro-2-(1,2-dihydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoro-methyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide		Animal: Hen (excreta)
M04 Isoflucypram-hydroxyphenyl N-(5-chloro-4-hydroxy-2-isopropylbenzyl)-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide		Animal: Goat (urine)
M05 Isoflucypram-olefine 1H-Pyrazole-4-carboxamide, N-[[5-chloro-2-(1-methylethenyl)phenyl]methyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl- [CA] N-[5-chloro-2-(prop-1-en-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide		Animal: Rat (faeces, bile)

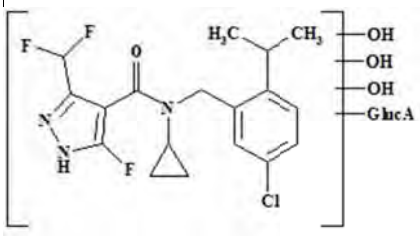
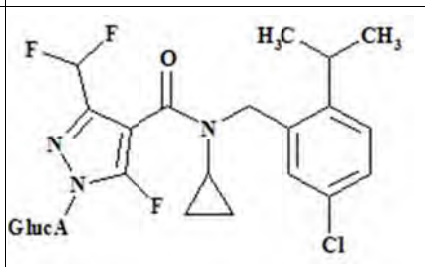
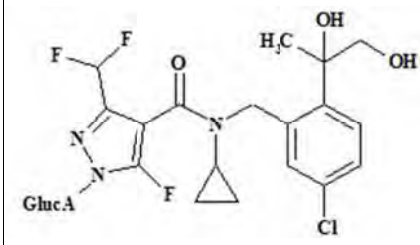
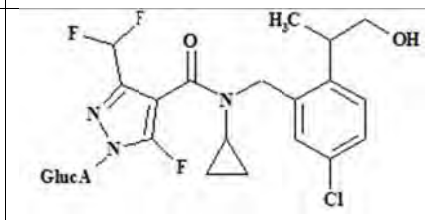
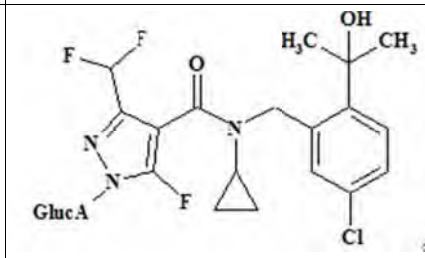
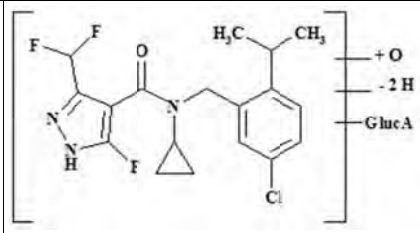
Number Name Identity (IUPAC)	Structure	Identified in:
M06 Isoflucypram-desmethyl-propanol N-[5-chloro-2-(1-hydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxamide		Animal: Rat (faeces, bile); hen (eggs, muscle, fat, liver, excreta); goat (milk, muscle, fat, liver, kidney, faeces, urine); sunfish (edible parts, viscera) Plant: Wheat (straw)
M07 Isoflucypram-desmethyl-1,2-propandiol N-[5-chloro-2-(1,2-dihydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxamide		Animal: Hen (eggs, muscle, fat, liver, excreta)
M08 Isoflucypram-desmethyl-diOH (isomers)		Animal: Rat (faeces)
M09 Isoflucypram-desmethyl-lactic acid 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}-2-hydroxypropanoic acid		Animal: Rat (faeces, plasma, liver, kidney, bile)
M10 Isoflucypram-lactic acid 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}-2-hydroxypropanoic acid		Animal: Rat (faeces, plasma, liver, kidney, bile); goat (liver, kidney, faeces, urine) Plant: - Soil: met., aerobic

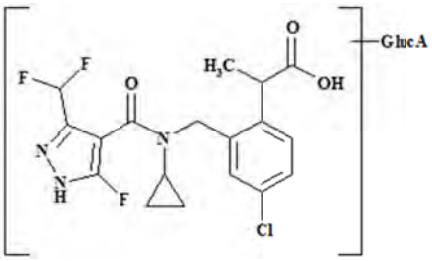
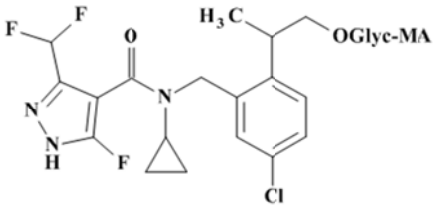
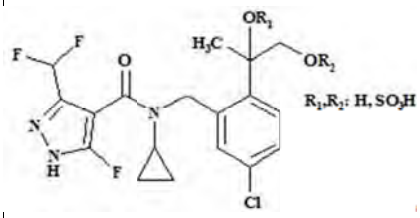
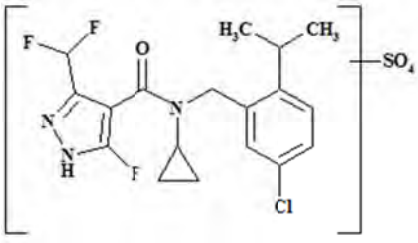
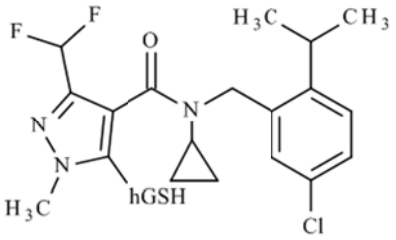
Number Name Identity (IUPAC)	Structure	Identified in:
M11 Isoflucypram-desmethyl-carboxylic acid 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}propanoic acid		Animal: Rat (urine, faeces, plasma, liver, kidney, bile); hen (muscle, fat, liver, excreta); goat (liver, kidney, faeces, urine) Soil: Met., aerobic
M12 Isoflucypram-carboxylic acid 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)-methyl]phenyl}-propanoic acid		Animal: Rat (urine, faeces, plasma, liver, kidney, bile); hen (eggs, muscle, fat, liver, excreta); goat (muscle, fat, liver, kidney, faeces, urine) Soil: met., aerobic Water: met., aerobic
M13 Isoflucypram-desmethyl N-(5-chloro-2-isopropylbenzyl)-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxamide		Animal: Rat (faeces, plasma, liver, kidney); sunfish (edible parts, viscera)
M14 Isoflucypram-desmethyl-hydroxyphenyl-2-propanol N-[5-chloro-4-hydroxy-2-(2-hydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxamide		Animal: Rat (urine, faeces, liver, bile)
M15 Isoflucypram-desmethyl-hydroxyphenyl-1,2-propandiol N-[5-chloro-2-(1,2-dihydroxypropan-2-yl)-4-hydroxybenzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxamide		Animal: Rat (faeces)

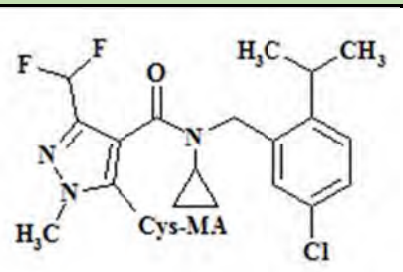
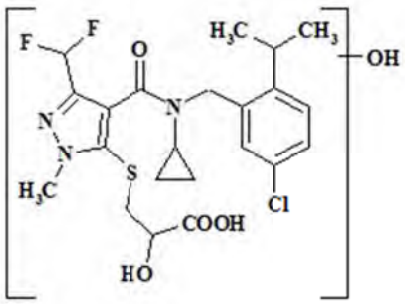
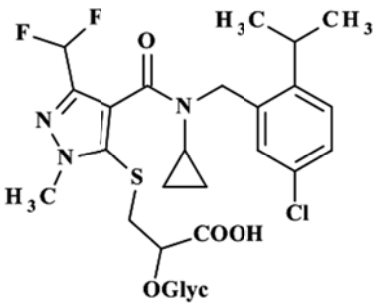
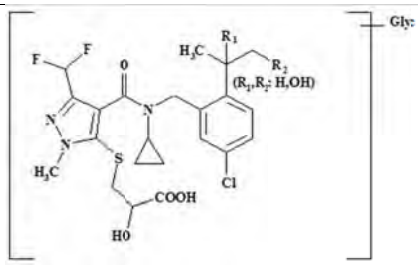
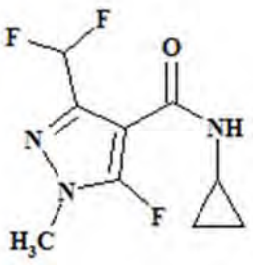
Number Name Identity (IUPAC)	Structure	Identified in:
M16 Isoflucypram-desmethyl-hydroxymethyl-carboxylic acid 2-{4-chloro-2-[(cyclopropyl{[5-fluoro-3-(hydroxymethyl)-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}propanoic acid		Animal: Rat (urine, faeces)
M17 Isoflucypram-desmethyl-hydroxymethyl-diOH		Animal: Rat (faeces, bile)
M18 Isoflucypram-propanol-Glyc N-{5-chloro-2-[1-(hexopyranosyloxy)propan-2-yl]benzyl}-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide		Plant: Wheat (hay, straw)
M19 Isoflucypram-propanol-GlucA (isomer 1 and 2) 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)methyl]phenyl}propyl glucopyranosiduronic acid		Animal: Rat (faeces, bile); goat (milk, muscle, liver, kidney, faeces, urine); sunfish (edible parts, viscera)
M20 Isoflucypram-2-propanol-GlucA 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}propan-2-yl beta-D-glucopyranosiduronic acid		Animal: Goat (liver, kidney, faeces, urine)
M21 Isoflucypram-propanol-Glyc-MA 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)methyl]phenyl}propyl 6-O-(carboxyacetyl)hexopyranoside		Plant: Wheat (hay, straw); oilseed rape (intermediate harvest, mature plants)

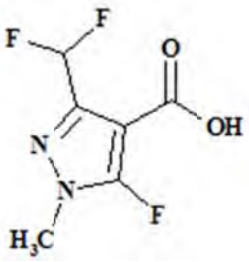
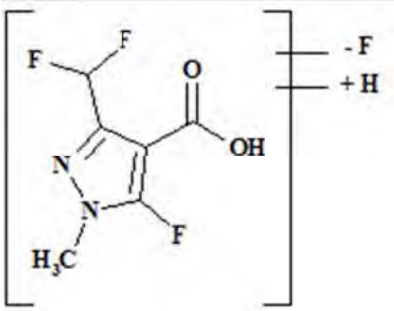
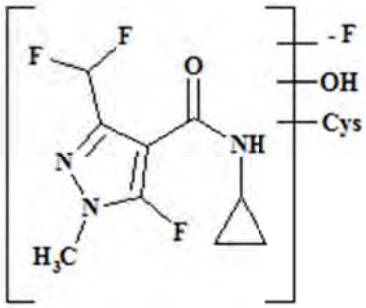
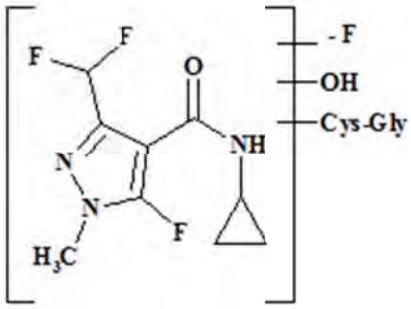
Number Name Identity (IUPAC)	Structure	Identified in:
M22 Isoflucypram-2-propanol-Glyc-MA 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)-methyl]phenyl}propan-2-yl 6-O-(carboxy-acetyl)hexopyranoside		Plant: Oilseed rape (intermediate harvest, mature plants); potato (leaves)
M23 Isoflucypram-hydroxyphenyl-Gluc-MA 2-chloro-4-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)-methyl]-5-isopropyl-phenyl 6-O-(carboxyacetyl)-beta-D-glucopyranoside		Plant: Oilseed rape (intermediate harvest, mature plants)
M23a Isoflucypram-OH-phenyl-Glyc-MA		Plant: Potato (leaves)
M24 Isoflucypram-hydroxyphenyl-Glyc-MA 2-chloro-4-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)-methyl]-5-isopropyl-phenyl 6-O-(carboxyacetyl)-hexopyranoside		Plant: Oilseed rape (intermediate harvest, mature plants)
M25 Isoflucypram-propenol-GlucA 2-{4-chloro-2-[(cycloprop yl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}prop-2-en-1-yl beta-D-glucopyranosiduronic acid		Animal: Goat (kidney, urine)
M26 Isoflucypram-propanol-SA 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}propyl hydrogen sulfate		Animal: Hen (liver, excreta)

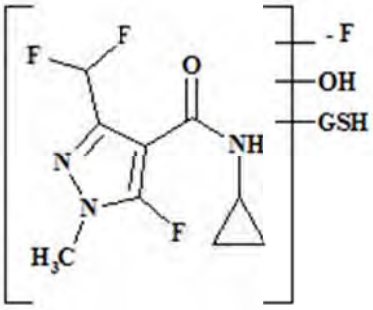
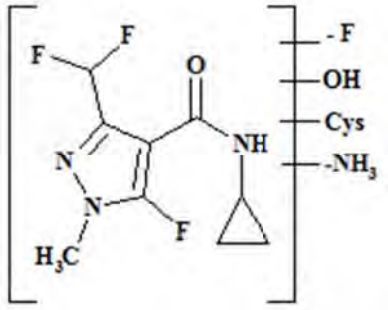
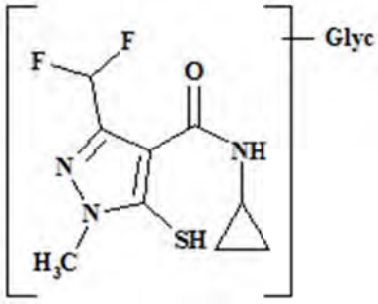
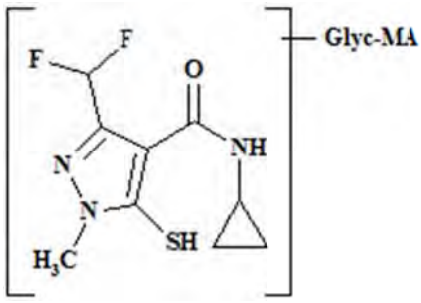
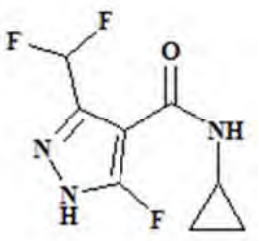
Number Name Identity (IUPAC)	Structure	Identified in:
M27 Isoflucypram-1,2-propandiol-SA		Animal: Hen (excreta)
M28 Isoflucypram-carboxylic acid-GlucA		Animal: Rat (bile)
M29 Isoflucypram-diOH-GlucA (isomer 1 and 2)		Animal: Rat (urine, faeces, bile)
M30 Isoflucypram-oxo-GlucA		Animal: Rat (faeces, bile)
M31 Isoflucypram-desmethyl-propanol-GlucA (isomer 1 and 2) 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}propyl glucopyranosiduronic acid		Animal: Rat (faeces, bile); sunfish (edible parts, viscera)
M32 Isoflucypram-desmethyl-OH-GlucA (isomer 1 to 2)		Animal: Rat (urine, faeces, bile)
M33 Isoflucypram-desmethyl-diOH-GlucA (isomer 1 to 6)		Animal: Rat (urine, faeces, bile)

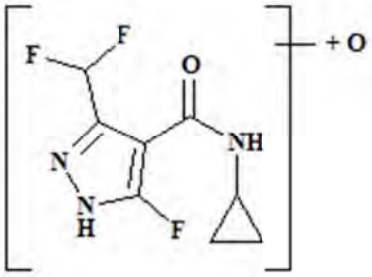
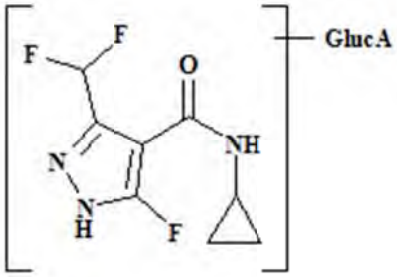
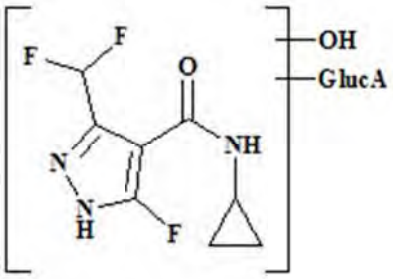
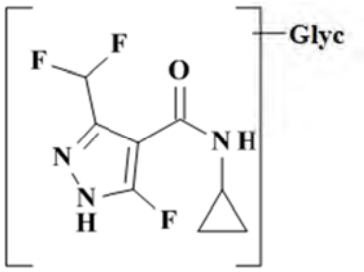
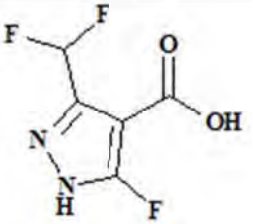
Number Name Identity (IUPAC)	Structure	Identified in:
M34 Isoflucypram-desmethyl-triOH-GlucA		Animal: Rat (faeces, bile)
M35 Isoflucypram-desmethyl-GlucA (isomer 1 and 2) N-(5-chloro-2-isopropylbenzyl)-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-(beta-D-glucopyranuronosyl)-1H-pyrazole-4-carboxamide		Animal: Rat (bile); sunfish (edible parts, viscera)
M36 Isoflucypram-desmethyl-1,2-propandiol-N-GlucA N-[5-chloro-2-(1,2-dihydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-(gluco-pyranuronosyl)-1H-pyrazole-4-carboxamide		Animal: Hen (muscle leg, liver, excreta)
M37 Isoflucypram-desmethyl-propanol-N-GlucA N-[5-chloro-2-(1-hydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-(gluco-pyranuronosyl)-1H-pyrazole-4-carboxamide		Animal: Hen (eggs, muscle leg, liver, excreta)
M38 Isoflucypram-desmethyl-2-propanol-N-GlucA N-[5-chloro-2-(2-hydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoro-methyl)-5-fluoro-1-(gluco-pyranuronosyl)-1H-pyrazole-4-carboxamide		Animal: Hen (liver, excreta)
M39 Isoflucypram-desmethyl-oxo-GlucA		Animal: Rat (bile)

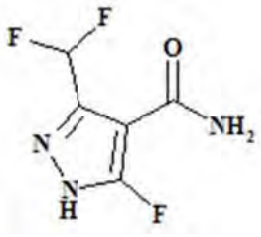
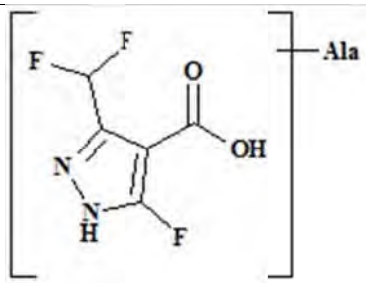
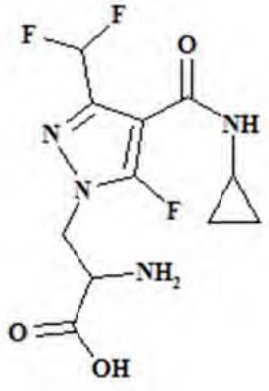
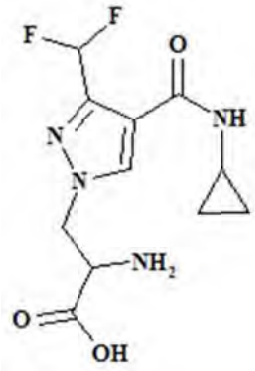
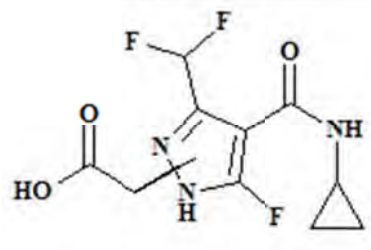
Number Name Identity (IUPAC)	Structure	Identified in:
M40 Isoflucypram-desmethyl-carboxylic acid-GlucA (isomer 1 and 2)		Animal: Rat (urine, faeces, bile)
M41 Isoflucypram-desmethyl-propanol-Glyc-MA 2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}propyl 6-O-(carboxyacetyl)hexopyranoside		Plant: Wheat (hay, straw)
M42 Isoflucypram-desmethyl-1,2-propandiol-SA		Animal: Hen (excreta)
M43 Isoflucypram-desmethyl-SA		Animal: Rat (faeces, bile)
M44 Isoflucypram-desfluoro-homoGSH gamma-glutamyl-S-{4-[(5-chloro-2-isopropylbenzyl)(cyclopropyl)carbamoyl]-3-(difluoromethyl)-1-methyl-1H-pyrazol-5-yl}cysteinyl-beta-alanine		Plant: Soya bean (forage, hay, straw)

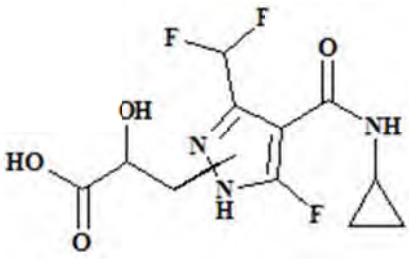
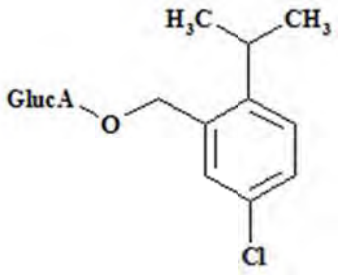
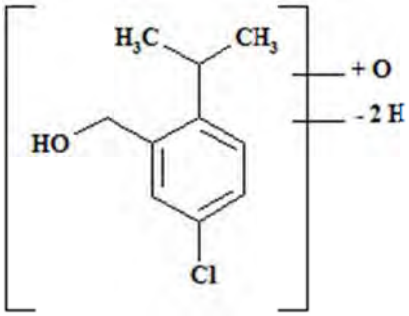
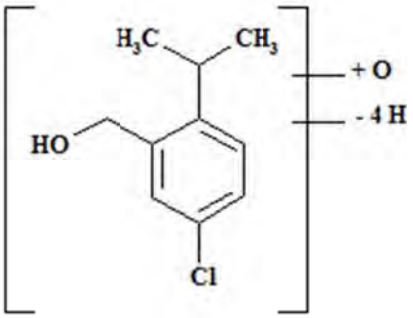
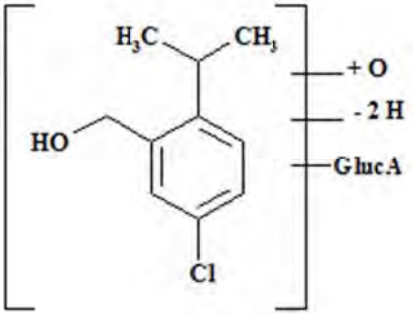
Number Name Identity (IUPAC)	Structure	Identified in:
M45 Isoflucypram-desfluoro-Cys-MA N-(carboxyacetyl)-S-{4-[(5-chloro-2-isopropylbenzyl)(cyclopropyl)carbamoyl]-3-(difluoromethyl)-1-methyl-1H-pyrazol-5-yl}cysteine		Plant: Soya bean (forage, hay, straw)
M46 Isoflucypram-desfluoro-mercapto-lactic acid-OH		Plant: Soya bean (forage, hay, straw)
M47 Isoflucypram-desfluoro-mercapto-lactic acid-Glyc 3-({4-[(5-chloro-2-isopropylbenzyl)(cyclopropyl)carbamoyl]-3-(difluoromethyl)-1-methyl-1H-pyrazol-5-yl}sulfanyl)-2-(hexo-pyranosyloxy)propanoic acid		Plant: Soya bean (forage, hay, straw)
M48 Isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc		Plant: Soya bean (forage, hay, straw)
M49 BCS-CR60082 Isoflucypram-N-methyl-cyclopropyl-pyrazole-carboxamide N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide		Plant: CRC (wheat forage, wheat hay, wheat straw, Swiss chard immature, Swiss chard at maturity, turnip leaves)

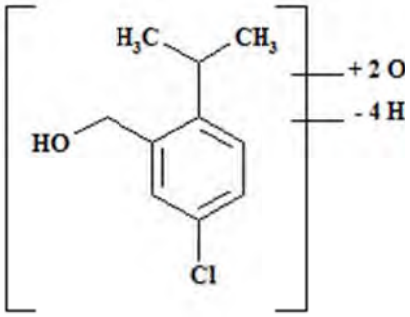
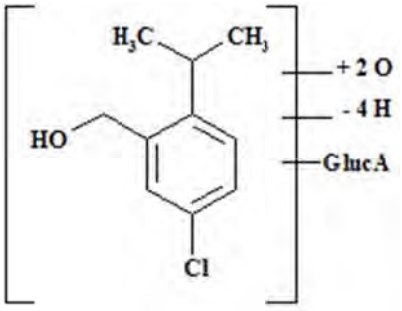
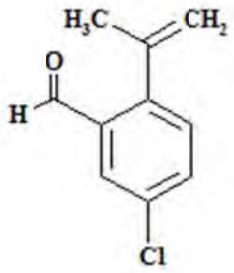
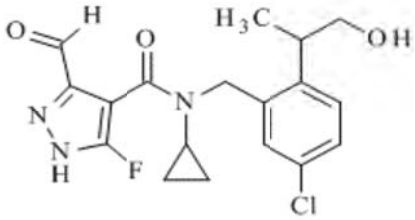
Number Name Identity (IUPAC)	Structure	Identified in:
M50 Isoflucypram-N-methyl-pyrazole-carboxylic acid CAS: 1255735-09-1 3-(difluoromethyl)-5-fluoro-1-methyl-pyrazole-4-carboxylic acid [IUPAC] 1H-Pyrazole-4-carboxylic acid, 3-(difluoromethyl)-5-fluoro-1-methyl- [CAS]		Animal: Rat (urine), goat (urine, kidney); sunfish (edible parts, viscera)
M51 Isoflucypram-desfluoro-N-methyl-pyrazole-carboxylic acid		Animal: Rat (urine)
M52 Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys		Animal: Rat (bile) Plant: CRC (wheat forage, wheat hay, wheat straw, Swiss chard immature, Swiss chard at maturity, turnip leaves)
M53 Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys-Gly		Animal: Rat (bile)

Number Name Identity (IUPAC)	Structure	Identified in:
M54 Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH		Animal: Rat (bile) plant: CRC (wheat forage, wheat hay, wheat straw, Swiss chard immature, Swiss chard at maturity, turnip leaves)
M55 Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-desamino-Cys		Plant: CRC (wheat forage, wheat hay, wheat straw, Swiss chard immature, turnip leaves)
M56 Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc		Plant: CRC (wheat forage, wheat hay, wheat straw, turnip leaves)
M57 Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc-MA		Plant: CRC (wheat forage, wheat hay, wheat straw, turnip leaves)
M58 Isoflucypram-cyclopropyl-pyrazole-carboxamide N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxamide		Animal: Rat (urine, plasma, liver, kidney); sunfish (edible parts, viscera) Plant: Potato (leaves, tubers)

Number Name Identity (IUPAC)	Structure	Identified in:
M59 Isoflucypram-cyclopropyl-oxy-pyrazole-carboxamide		Animal: Rat (urine)
M60 Isoflucypram-cyclopropyl-pyrazole-carboxamide-GlucA (isomer 1 and 2)		Animal: Rat (urine, bile, kidney)
M61 Isoflucypram-cyclopropyl-pyrazole-carboxamide-OH-GlucA		Animal: Rat (urine, bile)
M62 Isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 1 and 2)		Plant: CRC (wheat forage, wheat hay, wheat straw, Swiss chard immature, Swiss chard at maturity, turnip leaves)
M63 Isoflucypram-pyrazole-carboxylic acid 3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxylic acid		Animal: Rat (urine, faeces, kidney, bile)

Number Name Identity (IUPAC)	Structure	Identified in:
M64 Isoflucypram-pyrazole-amide 3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxamide		Animal: Rat (urine)
M65 Isoflucypram-pyrazole-carboxylic acid-Ala		Animal: Rat (urine, bile)
M66 Isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala 3-[4-(cyclopropylcarbamoyl)-3-(difluoromethyl)-5-fluoro-1H-pyrazol-1-yl]alanine		Plant: CRC (wheat forage, wheat hay, wheat straw, wheat grain, Swiss chard immature, Swiss chard at maturity, turnip leaves)
M67 Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala 3-[4-(cyclopropylcarbamoyl)-3-(difluoromethyl)-1H-pyrazol-1-yl]alanine		Plant: CRC (wheat forage, wheat hay, wheat straw, wheat grain, Swiss chard immature, Swiss chard at maturity, turnip leaves)
M68 Isoflucypram-cyclopropyl-pyrazole-carboxamide-acetic acid		Plant: CRC (wheat hay, wheat straw)

Number Name Identity (IUPAC)	Structure	Identified in:
M69 Isoflucypram-cyclopropyl-pyrazole-carboxamide-OH-lactic acid (isomer 1 and 2)		Plant: CRC (wheat forage, wheat hay, wheat straw, Swiss chard immature, Swiss chard at maturity)
M70 Isoflucypram-benzylalcohol-GlucA 5-chloro-2-isopropylbenzyl hexo-pyranosiduronic acid		Animal: Rat (urine)
M71 Isoflucypram-benzylalcohol-oxo		Animal: Rat (urine)
M72 Isoflucypram-benzylalcohol-oxo-desdihydro (isomer 1 and 2)		Animal: Rat (urine)
M73 Isoflucypram-benzylalcohol-oxo-GlucA (isomer 1 and 2)		Animal: Rat (urine)

Number Name Identity (IUPAC)	Structure	Identified in:
M74 Isoflucypram-benzylalcohol-dioxo (isomer 1 and 2)		Animal: Rat (urine)
M75 Isoflucypram-benzylalcohol-dioxo-GlucA (isomer 1 and 2)		Animal: Rat (urine)
M76 Isoflucypram-phenyl-formyl-olefine CAS: 1006685-15-9 5-chloro-2-(prop-1-en-2-yl)benzaldehyde [IUPAC] benzaldehyde, 5-chloro-2-(1-methylethenyl)- [CA]		Animal: Rat (urine)
M77 Isoflucypram-desmethyl-propanol-aldehyde N-[5-chloro-2-(1-hydroxypropan-2-yl)benzyl]-N-cyclopropyl-5-fluoro-3-formyl-1H-pyrazole-4-carboxamide		plant: Processing (hydrolysis)

PLANT METABOLISM

The Meeting received studies describing the metabolism of isoflucypram in tomatoes, wheat, soya bean plants and oilseed rape following foliar application and in potatoes following seed treatment.

Tomato

Report Nos. EnSa-16-0959 and EnSa-16-0960.

The metabolism of isoflucypram in tomato fruits was investigated following foliar application of either [pyrazole-4-^{14}C] isoflucypram or [phenyl-UL-^{14}C] isoflucypram (Lamshoeft, M.; 2017). For both tests, radiolabelled isoflucypram was formulated as an emulsifiable concentrate (EC) 200. For the pyrazole study, one foliar application was made at growth stage BBCH 14–15 at 79 g ai/ha and a second foliar application was made at growth stage BBCH 85–86 at 89 g ai/ha, corresponding with the end of fruit ripening. The timing between applications was 99 days. The total application rate was 168 g ai/ha based on a plant density of 12,000 tomato plants/ha. For the phenyl study, one foliar application was made at growth stage BBCH 14–15 at 78 g ai/ha and a second foliar application was made at growth stage BBCH 85–87 at 78 g ai/ha, corresponding with the end of fruit ripening. The timing between applications was 97 days. The total application rate was 156 g ai/ha based on a plant density of 12,000 tomato plants/ha.

Tomato fruits were harvested 14 days after the last application. After surface washing with DCM, tomatoes were conventionally extracted three times with acetonitrile (ACN)/water (8:2). The combined extracts were subjected to solid-phase extraction (SPE) followed by rinsing with ACN/water (8:2) for both labels and either methanol/dichloromethane (DCM) (1:1) for the pyrazole label or tetrahydrofuran (THF)/methanol (1:1) for the phenyl label. The surface wash solution and the purified SPE fractions were analysed by high performance liquid chromatography (HPLC).

The radioactivity in the extracts and the post extraction solids (PES) was determined by combustion/liquid scintillation counting (LSC). Total radioactive residue (TRR) was calculated by summing the radioactivity in the surface wash, the extract, and the PES. Parent compound was identified by spectroscopic methods and co-chromatography with reference compound. Extraction and quantification of four minor metabolites and parent compound was completed within six months (storage temperature ≤-18 °C); no storage stability investigations were conducted.

TRR was 0.170 mg eq/kg for the pyrazole label and 0.095 mg eq/kg for the phenyl label. The main portion of the radioactivity was recovered in the surface wash, comprising 73.6 percent TRR (0.125 mg eq/kg) for the pyrazole label and 74.6 percent TRR (0.071 mg eq/kg) for the phenyl label. Residues in tomato fruits were efficiently extracted using ACN/water (8:2) and amounted to 26.1 percent TRR (0.045 mg eq/kg) in the pyrazole study and 25.1 percent TRR (0.024 mg eq/kg) in the phenyl study. The PES amounted to 0.2 percent TRR (<0.001 mg eq/kg) in both studies. There were no losses during the sample preparation and no radioactivity was observed in the distillate of the concentration procedures.

Isoflucypram was the only metabolite observed, comprising 96.7 percent TRR (0.165 mg/kg) in the pyrazole study and 98.2 percent TRR (0.094 mg/kg) in the phenyl study. Up to four minor unknown peaks were isolated, each comprising ≤ 1.6 percent TRR (≤ 0.003 mg eq/kg). The results are shown in Tables 2 and 3.

Table 2 Distribution of radioactivity in tomatoes following two foliar applications of [Pyrazole-4-^{14}C] Isoflucypram and [Phenyl-UL-^{14}C] Isoflucypram

	[Pyrazole-4-^{14}C][1] Isoflucypram. TRR = 0.170 mg eq/kg		Tom [Phenyl-UL-^{14}C][2] Isoflucypram. TRR = 0.095 mg eq/kg	
Component/Sample	% TRR	mg eq/kg	% TRR	mg eq/kg
Surface wash solution	73.6	0.125	74.6	0.071
Conventional extract	26.1	0.045	25.1	0.024
Losses (distillate)	-	-	-	-
Total extracted	99.8	0.170	99.8	0.095
PES	0.2	<0.001	0.2	< 0.001

Notes:
[1] Reference: EnSa-16-0959.
[2] Reference: EnSa-16-0960.

Table 3 Composition of radioactivity in tomato extracts following two foliar applications of [Pyrazole-4-^{14}C] Isoflucypram and [Phenyl-UL-^{14}C] Isoflucypram

	[Pyrazole-4-^{14}C][1] Isoflucypram. TRR = 0.170 mg eq/kg		[Phenyl-UL-^{14}C][2] Isoflucypram. TRR = 0.095 mg eq/kg	
Component/Sample	% TRR	mg eq/kg	% TRR	mg eq/kg
Total extracted (surface wash + extract)	99.8	0.170	99.8	0.095
Isoflucypram	96.7	0.165	98.2	0.094
Total identified	96.7	0.165	98.2	0.094
Total characterised	3.1	0.007	1.5	0.002
PES	0.2	<0.001	0.2	<0.001
Accountability	100.0	0.170	100.0	0.095

Notes:
[1] Reference: EnSa-16-0959.
[2] Reference: EnSa-16-0960.

Wheat

Report Nos. S14-01087 and S14-01086.

The metabolism of isoflucypram in wheat commodities was investigated following foliar application of [pyrazole-4-^{14}C] isoflucypram or [phenyl-UL-^{14}C] isoflucypram (Traub, M.; 2018). Isoflucypram was formulated as an EC 50 and applied at BBCH growth stage 30 at 64–69 g ai/ha and reapplied at BBCH growth stage 69 at 66–67 g ai/ha for a total application rate of 130–136 g ai/ha. The interval between applications was 28 days or 33 days for the pyrazole and phenyl studies, respectively.

Wheat hay was harvested 1 day prior to the second application (BBCH 69) and allowed to dry for four days. Wheat straw and grain were harvested at maturity (BBCH 89) corresponding to 17–18 days between application and harvest. Samples were subjected to conventional and exhaustive extraction and acid hydrolysis.

Conventional extraction and clean-up

Homogenised samples of wheat hay, straw, and grain were conventionally extracted three times with ACN/water (8:2). Individual extracts were filtered and the solids were rinsed with the solvent mixture used for extraction. The solids were dried and subjected to combustion.

The extracts were combined and cleaned-up by SPE which was rinsed with methanol and water and conditioned with ACN/water (8:2). The percolate was collected and the cartridge was rinsed with ACN/water (8:2). The percolate and rinse fractions were combined. Less polar fractions on the cartridge were eluted by rinsing the cartridge with methanol/THF (1:1). Each combined percolate/rinse solution was mixed with emulsifier and evaporated to the aqueous remainder. The purified conventional extracts were then analysed by HPLC.

Exhaustive extraction and clean-up

In the pyrazole study, solids from the conventional extraction of wheat straw were exhaustively extracted twice with ACN/water/formic acid (50:50:1) with microwave assistance. The microwave extracts were cooled down at room temperature and combined by rotary evaporation. Aliquots of the extracts were

centrifuged. The pellet was dissolved in ACN/water. Both the supernatant and extract were analysed HPLC.

Hydrolysis of conventional extracts

For both labels, hydrolysis experiments in acidic medium were conducted with conventional extract of wheat hay and straw to further characterise the residues. Aliquots of the purified conventional extract of wheat hay and straw were incubated with 1 mol/L hydrochloric acid (HCl) and 5 percent ACN at 100 °C for 1 hour prior to centrifugation. For wheat straw, the supernatant was removed and the pellet was dissolved in ACN/water (1:1) whereas no pellet was formed during processing of wheat hay. Aliquots of the extracts were analysed by HPLC.

The radioactivity in extracts and the PES was determined by combustion/LSC. The TRR was calculated by summing the radioactivity of the combined extracts and the PES.Parent compound and major metabolites were identified by spectroscopic methods and co-chromatography with reference compounds.

For the pyrazole study, grain and hay were stored for a maximum period of one month and wheat straw was stored for a maximum of 13 months (≤-18 °C). A reanalysis of extract after 14 months demonstrated a stable metabolite profile.

For the phenyl study, samples of wheat hay were extracted within one month of being placed in frozen storage (-18 °C). Samples of wheat grain and straw were extracted within 30 months of frozen storage (-18 °C). Stored extract of wheat straw and grain were analysed after 28 and 15 months of storage, respectively, demonstrating stability of the metabolite profile. The results are showing in Tables 4 and 5.

Table 4 Distribution of radioactivity in the extracts of wheat commodities following foliar application of [Pyrazole-4-^{14}C] Isoflucypram (S14-01087)

	Hay TRR = 4.032 mg eq/kg		Straw TRR = 15.536 mg eq/kg		Grain TRR = 0.385 mg eq/kg	
Component/Sample	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	95.8	3.864	94.0	14.604	93.6	0.360
Analysed extracts	95.4	3.846	93.5	14.521	92.0	0.354
Losses (not analysed)[1]	0.4	0.018	0.5	0.083	1.6	0.006
Microwave extract	-	-	4.7	0.727	-	-
Analysed extracts	-	-	4.7	0.727	-	-
Total extracted	95.8	3.864	98.7	15.330	93.6	0.360
PES	4.2	0.168	1.3	0.206	6.4	0.025
Accountability	100.0	4.032	100.0	15.536	100.0	0.385

Notes:

[1] Losses during clean up, concentration, centrifugation, etc.

Table 5 Distribution of Radioactivity in the Extracts of Wheat Commodities Following Foliar Application of [Phenyl-UL-^{14}C] Isoflucypram (S14-01086)

Component/Sample	Hay TRR = 3.040 mg eq/kg		Straw TRR = 16.031 mg eq/kg		Grain TRR = 0.284 mg eq/kg	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	96.7	2.940	95.2	15.264	93.5	0.266
Analysed extracts	96.2	2.925	93.1	14.922	92.7	0.264
Losses (not analysed)[1]	0.5	0.015	2.1	0.342	0.8	0.002
Total extracted	96.7	2.940	95.2	15.264	93.5	0.266

Component/Sample	Hay TRR = 3.040 mg eq/kg		Straw TRR = 16.031 mg eq/kg		Grain TRR = 0.284 mg eq/kg	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
PES	3.3	0.099	4.8	0.767	6.5	0.019
Accountability	100.0	3.040	100.0	16.031	100.0	0.284

Notes:
[1] Losses during clean up, concentration, centrifugation, etc.

In the conventional extract from wheat hay, 66.0 percent TRR (2.659 mg eq/kg) [pyrazole] and 66.3 percent TRR (2.015 mg eq/kg) [phenyl] was identified. Parent isoflucypram was the major component representing 50.0 percent (2.016 mg/kg) [pyrazole] and 54.7 percent TRR (1.661 mg/kg) [phenyl]. Remaining major metabolites included isoflucypram-propanol-Glyc accounting for 0.096 mg eq/kg (2.4 percent TRR) [pyrazole] and 0.023 mg eq/kg (0.8 percent TRR) [phenyl], isoflucyrpam-desmethyl-propanol-Glyc-MA accounting for 0.103 mg eq/kg (2.5 percent TRR) [pyrazole] and 0.081 mg eq/kg (2.7 percent TRR) [phenyl], isoflucypram-propanol-Glyc-MA accounting for 0.414 mg eq/kg (10.3 percent TRR) [pyrazole] and 0.229 mg eq/kg (7.5 percent TRR) [phenyl], and isoflucypram-propanol accounting for 0.029 mg eq/kg (0.7 percent TRR) [pyrazole] and 0.021 mg eq/kg (0.7 percent TRR) [phenyl]. In wheat hay, up to 23 unknown metabolites were characterised in the extracts by their chromatographic behaviour, individually accounting for ≤3.1 percent TRR (≤ 0.127 mg eq/kg).

In the conventional extract from wheat straw, 77.1 percent TRR (11.969 mg eq/kg) [pyrazole] and 72.6 percent TRR (11.640 mg eq/kg) [phenyl] was identified. Parent isoflucypram was the major component representing 62.9 percent TRR (9.761 mg/kg) [pyrazole] and 62.1 percent TRR (9.954 mg/kg) [phenyl]. Other major metabolites included isoflucypram-propanol-Glyc accounting for 0.383 mg eq/kg (2.5 percent TRR) [pyrazole] and 0.373 mg eq/kg (2.3 percent TRR) [phenyl], isoflucypram-desmethyl-propanol-Glyc-MA accounting for 0.448 mg eq/kg (2.9 percent TRR) [pyrazole] and 0.306 mg eq/kg (1.9 percent TRR) [phenyl], isoflucypram-propanol-Glyc-MA accounting for 1.042 mg eq/kg (6.7 percent TRR) [pyrazole] and 0.808 mg eq/kg (5.0 percent TRR) [phenyl], isoflucypram-desmethyl-propanol accounting for 0.116 mg eq/kg (0.7 percent TRR) [pyrazole] and 0.052 mg eq/kg (0.3 percent TRR) [phenyl], and isoflucypram-propanol accounting for 0.209 mg eq/kg (1.4 percent TRR) [pyrazole] and 0.147 mg eq/kg (0.9 percent TRR) [phenyl].

In the exhaustive extract of pyrazole-labelled wheat straw, 3.0 percent TRR (0.453 mg eq/kg) were further identified, consisting of parent isoflucypram, isoflucypram-propanol-Glyc, isoflucypram-desmethyl-propanol, and isoflucypram-propanol, representing 1.1, 1.2, 0.4, and 0.3 percent TRR, corresponding to 0.172, 0.178, 0.055, and 0.048 mg eq/kg, respectively. In total, 80.0 percent (12.422 mg eq/kg) of the TRR was identified in the conventional and exhaustive extracts of pyrazole-labelled wheat straw. Between both studies, up to 39 unknown metabolites were characterised in the extracts by their chromatographic behaviour, individually accounting for ≤ 2.1 percent TRR (≤ 0.345 mg eq/kg).

Conventional extract from wheat grain contained only parent isoflucypram, representing 92.0 percent TRR (0.354 mg/kg) [pyrazole] and 92.7 percent TRR (0.264 mg/kg) [phenyl]. The results are shown in Tables 6 to 8.

Table 6 Distribution of isoflucypram and metabolites in the extracts of wheat commodities following foliar application of [Pyrazole-4-^{14}C] Isoflucypram (S14-01087)

	Hay TRR = 4.032 mg eq/kg		Straw TRR = 15.536 mg eq/kg		Grain TRR = 0.385 mg eq/kg	
Component/Sample	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	95.8	3.864	94	14.604	93.6	0.36
Isoflucypram	50	2.016	62.9	9.761	92	0.354
Isoflucypram-propanol-Glyc	2.4	0.096	2.5	0.383	ND	ND
Isoflucypram-desmethyl-propanol-Glyc-MA	2.5	0.103	2.9	0.448	ND	ND
Isoflucypram-propanol-Glyc-MA	10.3	0.414	6.7	1.042	ND	ND
Isoflucypram-propanol	0.7	0.029	1.4	0.219	ND	ND
Isoflucypram-desmethyl-propanol	ND	ND	0.7	0.116	ND	ND
Subtotal identified	66	2.659	77.1	11.969	92	0.354
Subtotal characterised	29.5	1.187	16.4	2.552	ND	ND
Exhaustive extract[1]	-	-	4.7	0.727	-	-
Isoflucypram	-	-	1.1	0.172	-	-
Isoflucypram-propanol-Glyc	-	-	1.2	0.178	-	-
Isoflucypram-desmethyl-propanol	-	-	0.4	0.055	-	-
Isoflucypram-propanol	-	-	0.3	0.048	-	-
Subtotal identified	-	-	3	0.453	-	-
Subtotal characterised	-	-	1.8	0.276	-	-
Total identified	66	2.659	80	12.422	92	0.354
Total characterised	29.5	1.187	18.2	2.827	-	-
Analysed extract(s)	95.4	3.846	98.2	15.248	92	0.354
Not analysed / Losses	0.4	0.018	0.5	0.083	1.6	0.006
Total extracted	95.8	3.864	98.7	15.33	93.6	0.36
PES	4.2	0.168	1.3	0.206	6.4	0.025
Accountability	100	4.032	100	15.536	100	0.385

Notes:

ND: Not detected.

[1] Given as sum of supernatant and dissolved pellet.

Table 7 Summary of characterisation and identification of TRR in wheat commodities following foliar application of [Pyrazole-4-^{14}C] Isoflucypram (S14-01087)

Component/Sample	Hay TRR = 4.032 mg eq/kg		Straw TRR = 15.536 mg eq/kg		Grain TRR= 0.385 mg eq/kg	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total extracted	95.8	3.864	98.7	15.330	93.6	0.360
Isoflucypram	50.0	2.016	64.0	9.933	92.0	0.354
Isoflucypram-propanol-Glyc	2.4	0.096	3.7	0.561	ND	ND
Isoflucypram-desmethyl- propanol-Glyc-MA	2.5	0.103	2.9	0.448	ND	ND
Isoflucypram-propanol-Glyc-MA	10.3	0.414	6.7	1.042	ND	ND
Isoflucypram-propanol	0.7	0.029	1.7	0.267	ND	ND
Isoflucypram-desmethyl- propanol	ND	ND	1.1	0.171	ND	ND
Total identified	66.0	2.659	80.0	12.422	92.0	0.354
Number of unknown peaks	21		32		-	
Largest unknown peak	3.1	0.119	1.5	0.239	-	-
Total characterised	29.5	1.187	18.2	2.827	-	-
Analysed extract(s)	95.4	3.846	98.2	15.248	92.0	0.354

Component/Sample	Hay TRR = 4.032 mg eq/kg		Straw TRR = 15.536 mg eq/kg		Grain TRR= 0.385 mg eq/kg	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Not analysed / Losses	0.4	0.018	0.5	0.083	1.6	0.006
PES	4.2	0.168	1.3	0.206	6.4	0.025
Accountability	100.0	4.032	100.0	15.536	100.0	0.385

Table 8 Summary of characterisation and identification of TRR in wheat commodities following foliar application of [Phenyl-UL-^{14}C] Isoflucypram (S14-01086)

Component/Sample	Hay TRR = 3.040 mg eq/kg		Straw TRR = 16.031 mg eq/kg		Grain TRR = 0.284 mg eq/kg	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	96.7	2.940	95.2	15.264	93.5	0.266
Isoflucypram	54.7	1.661	62.1	9.954	92.7	0.264
Isoflucypram-propanol-Glyc	0.8	0.023	2.3	0.373	ND	ND
Isoflucypram-desmethyl- propanol-Glyc-MA	2.7	0.081	1.9	0.306	ND	ND
Isoflucypram-propanol-Glyc-MA	7.5	0.229	5.0	0.808	ND	ND
Isoflucypram-propanol	0.7	0.021	0.9	0.147	ND	ND
Isoflucypram-desmethyl- propanol	ND	ND	0.3	0.052	ND	ND
Total identified	66.3	2.015	72.6	11.640	92.7	0.264
Number of unknown peaks	23		39		-	
Largest unknown peak	3.1	0.095	2.1	0.345	-	-
Total characterised	30.0	0.910	20.5	3.281	-	-
Analysed extract(s)	96.2	2.925	93.1	14.922	92.7	0.264
Not analysed / Losses	0.5	0.015	2.1	0.342	0.8	0.002
PES	3.3	0.099	4.8	0.767	6.5	0.019
Accountability	100.0	3.040	100.0	16.031	100.0	0.284

Notes:
ND: Not detected.

Conventional extracts of wheat hay and straw were subjected to acid hydrolysis in an attempt to release possible hydrolysable conjugates.

In hydrolysed extract of wheat hay, 90.3 percent TRR (3.640 mg eq/kg) [pyrazole] and 95.2 percent TRR (2.893 mg eq/kg) [phenyl] was analysed. Parent isoflucypram was the major component representing 44.4 percent TRR (1.791 mg/kg) [pyrazole] and 49.6 percent TRR (1.508 mg/kg) [phenyl]. Other major metabolites included isoflucypram-propanol-Glyc accounting for 0.031 mg eq/kg (0.8 percent TRR) [pyrazole] and 0.019 mg eq/kg (0.6 percent TRR) [phenyl], isoflucypram-propanol accounting for 0.901 mg eq/kg (22.3 percent TRR) [pyrazole] and 0.642 mg eq/kg (21.1 percent TRR) [phenyl], isoflucypram-desmethyl-propanol accounting for 0.277 mg eq/kg (6.9 percent TRR) [pyrazole] and 0.204 mg eq/kg (6.7 percent TRR) [phenyl], and isoflucypram-propanol-Glyc-MA accounting for 0.036 mg eq/kg (0.9 percent TRR) [pyrazole only].

In the hydrolysed extract of wheat straw, 92.5 percent TRR (14.372 mg eq/kg) [pyrazole] and 92.5 percent TRR (14.832 mg eq/kg) [phenyl] were analysed. Parent isoflucypram was the major component, representing 67.0 percent TRR (10.397 mg/kg) [pyrazole] and 60.8 percent TRR (9.751 mg/kg) [phenyl]. Other major metabolites included isoflucypram-propanol-Glyc accounting for 0.2 percent TRR (0.024 mg eq/kg) [pyrazole] and 0.3 percent TRR (0.053 mg eq/kg) [phenyl], isoflucypram-propanol-Glyc-MA accounting for 0.3 percent TRR (0.054 mg eq/kg) [pyrazole] and 0.1 percent TRR (0.022 mg eq/kg) [phenyl], isoflucypram-propanol accounting for 10.5 percent TRR (1.625 mg eq/kg) [pyrazole] and

12.7 percent TRR (2.046 mg eq/kg), isoflucypram-desmethyl-propanol accounting for 3.6 percent TRR (0.564 mg eq/kg) [pyrazole] and 4.0 percent TRR (0.644 mg eq/kg) [phenyl], and isoflucypram-desmethyl-propanol-Glyc-MA accounting for 0.3 percent TRR (0.054 mg eq/kg) [phenyl only]. In contrast to hydrolysis of the hay extract, a pellet was formed during hydrolysis of the straw extract. Therefore values of analysed residues are given as sum of supernatant and dissolved pellet.

In the pyrazole study, identification rates after hydrolytic treatment increased from 66.0 percent TRR (2.659 mg eq/kg) before hydrolysis to 75.3 percent TRR (3.035 mg eq/kg) after hydrolysis for wheat hay and from 77.1 percent TRR (11.969 mg eq/kg) before hydrolysis to 81.6 percent TRR (12.666 mg eq/kg) after hydrolysis for wheat straw. In the phenyl study, identification rates after hydrolytic treatment increased from 66.3 percent TRR (2.015 mg eq/kg) before hydrolysis to 78.0 percent TRR (2.373 mg eq/kg) for wheat hay after hydrolysis and from 72.6 percent TRR (11.640 mg eq/kg) before hydrolysis to 78.2 percent TRR (12.570 mg eq/kg) for wheat straw after hydrolysis. Two major metabolites were formed after acidic hydrolysis as a result of de-conjugation of residues: isoflucypram-desmethyl-propanol and isoflucypram-propanol. Isoflucypram-desmethyl-propanol accounted for 6.9 percent TRR (0.277 mg eq/kg) [pyrazole] and 6.7 percent TRR (0.204 mg eq/kg) [phenyl] in wheat hay and 3.6 percent TRR (0.564 mg eq/kg) [pyrazole] and 4.0 percent TRR (0.644 mg eq/kg) in wheat straw. Isoflucypram-propanol was detected in hydrolysed extract accounting for 22.3 percent TRR (0.901 mg eq/kg) [pyrazole] and 21.1 percent TRR (0.642 mg eq/kg) [phenyl] in wheat hay and 10.5 percent TRR (1.625 mg eq/kg) [pyrazole] and 12.7 percent TRR (2.046 mg eq/kg) [phenyl] in wheat straw. The results are shown in Tables 9 and 10.

A significant amount of conjugated residues in the conventional extracts of hay and straw hydrolyse to isoflucypram-desmethyl-propanol and isoflucypram-propanol.

Table 9 Distribution of [Pyrazole-4-^{14}C] isoflucypram and metabolites in the conventional extracts of wheat commodities following acid hydrolysis (S14-01087)

Sample	Hay TRR = 4.032 mg eq/kg				Straw TRR = 15.536 mg eq/kg			
	Before hydrolysis		After Hydrolysis[1]		Before hydrolysis		After Hydrolysis[1]	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Isoflucypram	50.0	2.016	44.4	1.791	62.9	9.761	67.0	10.397
Isoflucypram-propanol-Glyc	2.4	0.096	0.8	0.031	2.5	0.383	0.2	0.024
Isoflucypram-desmethyl-propanol-Glyc-MA	2.5	0.103	ND	ND	2.9	0.448	ND	ND
Isoflucypram-propanol-Glyc-MA	10.3	0.414	0.9	0.036	6.7	1.042	0.3	0.056
Isoflucypram-propanol	0.7	0.029	22.3	0.901	1.4	0.219	10.5	1.625
Isoflucypram-desmethyl-propanol	ND	ND	6.9	0.277	0.7	0.116	3.6	0.564
Total identified	66.0	2.659	75.3	3.035	77.1	11.969	81.6	12.666
Total characterised	29.5	1.187	15.0	0.605	16.4	2.552	11.0	1.706

Notes:

ND: Not detected.

[1] Values are given as sum of supernatant and dissolved pellet; during hydrolysis of wheat hay extract, no pellet was formed.

Table 10 Distribution of [Phenyl-UL-^{14}C] isoflucypram and metabolites in the conventional extracts of wheat commodities following acid hydrolysis (S14-01086)

Sample	Hay, TRR = 3.040 mg eq/ha				Straw, TRR = 16.031 mg eq/ha			
	Before hydrolysis		After Hydrolysis[1]		Before hydrolysis		After Hydrolysis[1]	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Isoflucypram	54.7	1.661	49.6	1.508	62.1	9.954	60.8	9.751
Isoflucypram-propanol-Glyc	0.8	0.023	0.6	0.019	2.3	0.373	0.3	0.053

Sample	Hay, TRR = 3.040 mg eq/ha				Straw, TRR = 16.031 mg eq/ha			
	Before hydrolysis		After Hydrolysis[1]		Before hydrolysis		After Hydrolysis[1]	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Isoflucypram-desmethyl-propanol-Glyc-MA	2.7	0.081	ND	ND	1.9	0.306	0.3	0.054
Isoflucypram-propanol-Glyc-MA	7.5	0.229	ND	ND	5.0	0.808	0.1	0.022
Isoflucypram-propanol	0.7	0.021	21.1	0.642	0.9	0.147	12.7	2.046
Isoflucypram-desmethyl-propanol	ND	ND	6.7	0.204	0.3	0.052	4.0	0.644
Total identified	66.3	2.015	78.0	2.373	72.6	11.640	78.2	12.570
Total characterised	30.0	0.910	17.2	0.520	20.5	3.281	14.3	2.262

Notes:
ND: Not detected.
[1] Values are given as sum of supernatant and dissolved pellet; during hydrolysis of wheat hay extract, no pellet was formed.

Figure 1 shows the proposed metabolic pathway of isoflucypram in wheat

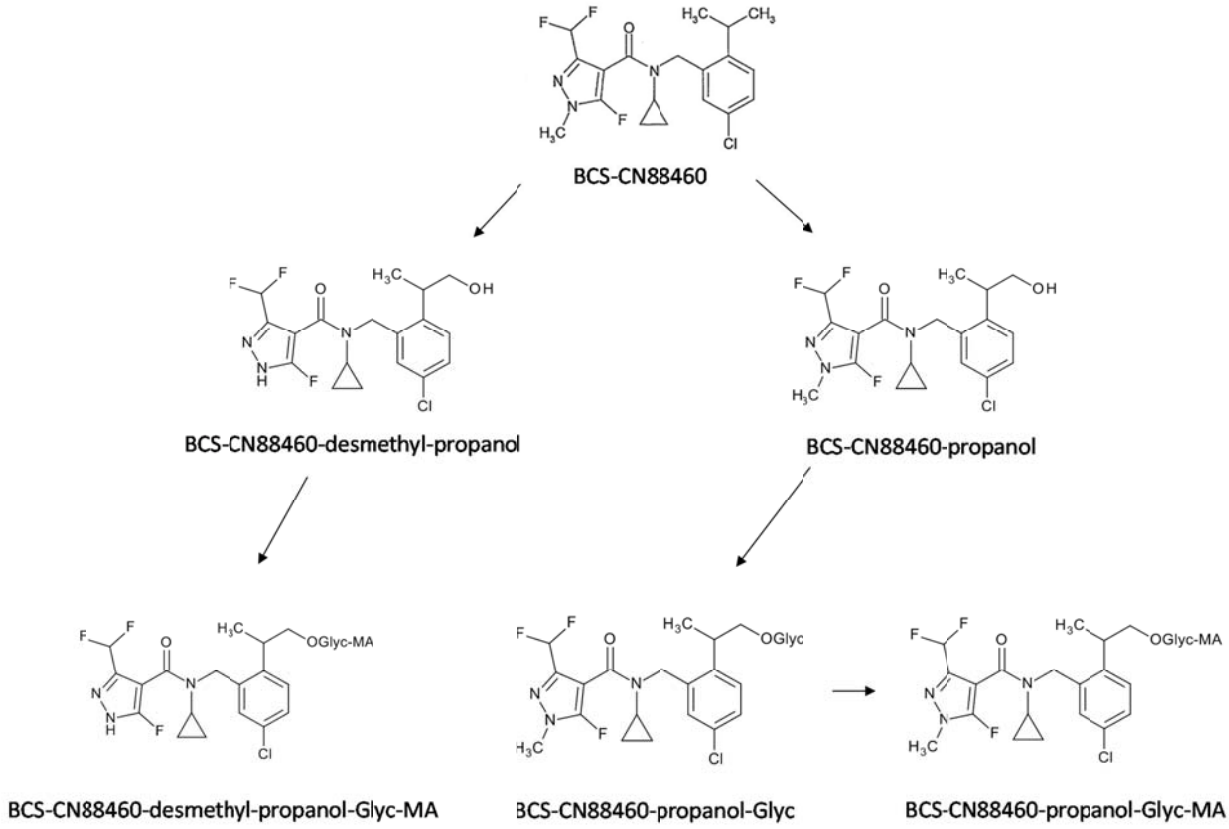


Figure 1 Proposed metabolic pathway of isoflucypram (BCS-CN88460) in wheat

Soya bean

Report Nos. S14-01089 and S14-01090.

The metabolism of isoflucypram was investigated in soya bean commodities after three foliar-directed post-emergent spray applications of [pyrazole-4-^{14}C] and [phenyl-UL-^{14}C] isoflucypram (Traub, M.; 2017). Isoflucypram was formulated as an EC 50. The first application was made at BBCH growth stage 14 at

54–59 g ai/ha, the second application was made at BBCH 51 at 56–57 g ai/ha, and the third application was made at BBCH growth stage 84–85 at 65–66 g ai/ha, for a total application rate of 176–181 g ai/ha. Application timing was 6–8 days between applications one and two, and 62–69 days between applications two and three.

Soya bean forage was harvested at BBCH 49, corresponding to 5–6 days after the first application. Hay was harvested at BBCH 77, corresponding to 38–39 days after the second application, and allowed to dry for four days. Straw and seed were harvested at BBCH 96, corresponding to 21 days after the third application. Samples were subjected to conventional and exhaustive extraction.

Conventional extraction and clean-up

Homogenised samples of soya bean forage, hay, straw, and seed were extracted three times with ACN/water (8:2). Individual extracts were filtered and the solids were rinsed with the solvent mixture used for extraction. The solids were dried and aliquots were subjected to combustion.

The extracts were combined and cleaned-up step by SPE which was rinsed with methanol and water and conditioned with ACN/water (8:2). The percolate was collected and the cartridge was rinsed with ACN/water (8:2). The percolate and the rinse were combined. Less polar fractions on the cartridge were eluted by rinsing the cartridge with methanol/THF (1:1). Each combined percolate/rinse solution obtained from SPE purification was evaporated to the aqueous remainder. The purified conventional extracts were analysed by HPLC.

Exhaustive extraction and clean-up

Solids from the conventional extraction of soya bean forage, hay, and straw were exhaustively extracted twice with ACN/water/formic acid (50:50:1:v) with microwave assistance.

For forage and hay, the individual extracts were combined and cleaned-up step by SPE which was rinsed with methanol and water and conditioned with ACN/water (8:2). The percolate was collected and the cartridge was rinsed with ACN/water (8:2). The percolate and the rinse were combined. Less polar fractions on the cartridge were eluted by rinsing the cartridge with methanol/THF (1:1). Each combined percolate/rinse solution obtained from SPE purification was mixed with emulsifier and evaporated to the aqueous remainder. The exhaustive extracts of straw were directly concentrated by evaporation to the aqueous remainder. The final exhaustive extracts of forage and straw were analysed by HPLC.

Characterisation of residues by partitioning

Radioactivity released from soya bean hay by exhaustive extraction with microwave assistance was characterised by partitioning with water and ethyl acetate. The radioactivity in extracts and the PES was determined by combustion/LSC. The total radioactive residue TRR was calculated by summing the radioactivity of the combined extracts and the PES. Pyrazole labelled soya bean forage and seed were stored for a maximum of five months (-18 °C). Soya bean hay and straw were stored for a maximum of 10 and 16 months, respectively (-18 °C).

Parent compound and major metabolites were identified by spectroscopic methods and co-chromatography with reference compounds. Phenyl labelled soya bean forage and seed were stored for a maximum of four months (-18 °C). Soya bean hay and straw were stored for a maximum of 18 and seven months, respectively (-18 °C). A comparison of the metabolic profile via HPLC after 18 and 27 months for soya bean hay and straw, respectively, shows stability of the metabolic profile over the period of frozen storage. The results are shown in Tables 11 and 12.

Table 11 Distribution of radioactivity in the extracts of soya bean commodities following foliar application of [Pyrazole-4-^{14}C] Isoflucypram (S14-01089)

Component/Sample	Forage (TRR = 4.371 mg eq/kg)		Hay (TRR = 4.679 mg eq/kg)		Straw (TRR = 17.715 mg eq/kg)		Seed (TRR = 0.035 mg eq/kg)	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	92.1	4.026	87.4	4.091	94.1	16.669	87.7	0.031
Analysed extracts (HPLC)	91.1	3.981	86.5	4.048	93.2	16.515	76.6	0.027
Losses (not analysed)[1]	1.0	0.045	0.9	0.044	0.9	0.154	11.0	0.004
Exhaustive extract	5.1	0.222	6.9	0.321	2.5	0.441	-	-
Analysed extracts (HPLC)	4.4	0.193	-	-	2.4	0.432	-	-
Partitioning of purified exhaustive extract	-	-	6.4	0.299	-	-	-	-
Ethyl acetate phase	-	-	6.4	0.299	-	-	-	-
Water phase	-	-	NQ	NQ	-	-	-	-
Losses (not analysed)[1]	0.7	0.029	0.5	0.022	< 0.1	0.009	-	-
Total extracted	97.2	4.248	94.3	4.413	96.6	17.110	87.7	0.031
PES	2.8	0.123	5.7	0.266	3.4	0.605	12.3	0.004
Accountability	100.0	4.371	100.0	4.679	100.0	17.715	100.0	0.035

Notes:

[1] Losses during clean up, concentration, degreasing, centrifugation, etc.

NQ: Not quantified.

Table 12 Distribution of radioactivity in the extracts of soya bean commodities following foliar application of [Phenyl-UL-^{14}C] Isoflucypram (S14-01090)

Component/Sample	Forage (TRR = 3.936 mg eq/kg)		Hay (TRR = 1.397 mg eq/kg)		Straw (TRR = 8.527 mg eq/kg)		Seed (TRR = 0.015 mg eq/kg)	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	91.4	3.597	88.6	1.238	92.8	7.914	69.8	0.011
Analysed extracts (HPLC)	90.4	3.557	87.5	1.222	91.8	7.830	69.8	0.011
Losses (not analysed)[1]	1.0	0.040	1.1	0.016	1.0	0.084	NQ	NQ
Exhaustive extract	5.2	0.205	6.3	0.088	3.1	0.264	-	-
Analysed extracts (HPLC)	4.5	0.176	NQ	NQ	3.1	0.264	-	-
Partitioning of purified exhaustive extract	-	-	5.9	0.082	-	-	-	-
Ethyl acetate phase	-	-	5.9	0.082	-	-	-	-
Water phase	-	-	NQ	NQ	-	-	-	-
Losses (not analysed)[1]	0.7	0.029	0.4	0.006	NQ	NQ	-	-
Total extracted	96.6	3.802	94.9	1.326	95.9	8.178	69.8	0.011
PES	3.4	0.134	5.1	0.071	4.1	0.349	30.2	0.005
Accountability	100.0	3.936	100.0	1.397	100.0	8.527	100.0	0.015

Notes:

[1] Losses during clean up, concentration, degreasing, centrifugation, etc.

NQ: Not quantified.

In the conventional extract from soya bean forage, 64.5 percent TRR (2.817 mg eq/kg) [pyrazole] and 69.4 percent TRR (2.729 mg eq/kg) [phenyl] was identified. Parent isoflucypram accounted for 17.6 percent (0.770 mg/kg) [pyrazole] and 17.9 percent TRR (0.703 mg/kg) [phenyl]. Remaining major metabolites included isoflucypram-desfluoro-homoGSH accounting for 21.9 percent TRR (0.955 mg eq/kg) [pyrazole] and 19.6 percent TRR (0.770 mg eq/kg) [phenyl], isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc accounting for 3.4 percent TRR (0.147 mg eq/kg) [pyrazole] and 4.8 percent TRR (0.187 mg eq/kg) [phenyl], isoflucypram-desfluoro-mercapto-lactic acid-OH accounting for

9.5 percent TRR (0.415 mg eq/kg) [pyrazole] and 16.7 percent TRR (0.658 mg eq/kg) [phenyl], isoflucypram-desfluoro-mercapto-lactic acid-Glyc accounting for 3.0 percent TRR (0.129 mg eq/kg) [pyrazole] and 2.7 percent TRR (0.107 mg eq/kg) [phenyl], and isoflucypram-desfluoro-Cys-MA accounting for 9.2 percent TRR (0.400 mg eq/kg) [pyrazole] and 7.7 percent TRR (0.304 mg eq/kg) [phenyl].

In the exhaustive extract from soya bean forage, 2.1 percent TRR (0.091 mg eq/kg) [pyrazole] and 2.8 percent TRR (0.108 mg eq/kg) [phenyl] was further identified. Parent isoflucypram accounted for 1.1 percent TRR (0.049 mg/kg) [pyrazole] and 1.3 percent TRR (0.053 mg/kg) [phenyl]. Remaining major metabolites included isoflucypram-desfluoro-homoGSH accounting for 1.0 percent TRR (0.042 mg eq/kg) [pyrazole] and 0.6 percent TRR (0.023 mg eq/kg) [phenyl], isoflucypram-desfluoro-mercapto-lactic acid-OH accounting for 0.3 percent TRR (0.014 mg eq/kg) [phenyl, only], and isoflucypram-desfluoro-Cys-MA accounting for 0.5 percent TRR (0.019 mg eq/kg) [phenyl, only]. In soya bean forage, up to 17 unknown metabolites were characterised in the extract by chromatographic behaviour, individually accounting for ≤3.9 percent TRR (0.171 mg eq/kg).

In the conventional extract from soya bean hay, 63.2 percent TRR (2.958 mg eq/kg) [pyrazole] and 69.8 percent TRR (0.974 mg eq/kg) [phenyl] was identified. Parent isoflucypram accounted for 10.4 percent TRR (0.487 mg/kg) [pyrazole] and 10.3 percent TRR (0.144 mg/kg) [phenyl]. Remaining metabolites included isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc accounting for 15.2 percent TRR (0.711 mg eq/kg) [pyrazole] and 17.6 percent TRR (0.246 mg eq/kg) [phenyl], isoflucypram-desfluoro-mercapto-lactic acid-Glyc accounting for 11.1 percent TRR (0.520 mg eq/kg) and 10.7 percent TRR (0.150 mg eq/kg), isoflucypram-desfluoro-Cys-MA accounting for 15.4 percent TRR (0.723 mg eq/kg) [pyrazole] and 20.5 percent TRR (0.286 mg eq/kg) [phenyl], isoflucypram-desfluoro-homoGSH accounting for 7.8 percent TRR (0.366 mg eq/kg) [pyrazole] and 7.8 percent TRR (0.109 mg eq/kg) [phenyl], and isoflucypram-desfluoro-mercapto-lactic acid-OH accounting for 3.2 percent TRR (0.151 mg eq/kg) and 2.8 percent TRR (0.040 mg eq/kg).

Residues in the exhaustive extract of soya bean hay were characterised by partitioning using ethyl acetate. Complete radioactivity of the exhaustive extract was found in the ethyl acetate phase after partitioning. In the phenyl study, radioactivity in the concentrated ethyl acetate phase was analysed by HPLC but no peak was detected above the limit of detection due to low radioactivity concentration and high matrix content of the sample. In soya bean hay, up to 13 unknown metabolites were characterised in the extracts by their chromatographic behaviour, individually accounting for ≤ 6.1 percent TRR (≤ 0.208 mg eq/kg).

In the conventional extract from soya bean straw, 81.2 percent TRR (14.380 mg eq/kg) [pyrazole] and 83.3 percent TRR (7.107 mg eq/kg) [phenyl] was identified. Parent isoflucypram was the major component representing 63.6 percent TRR (11.262 mg/kg) [pyrazole] and 69.6 percent TRR (5.934 mg/kg) [phenyl]. Remaining major metabolites included isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc accounting for 3.8 percent TRR (0.667 mg eq/kg) [pyrazole] and 2.1 percent TRR (0.177 mg eq/kg) [phenyl], isoflucypram-desfluoro-homoGSH accounting for 4.8 percent TRR (0.857 mg eq/kg) [pyrazole] and 2.5 percent TRR (0.211 mg eq/kg) [phenyl], isoflucypram-desfluoro-mercapto-lactic acid-OH accounting for 1.5 percent TRR (0.272 mg eq/kg) [pyrazole] and 2.4 percent TRR (0.206 mg eq/kg) [phenyl], isoflucypram-desfluoro-mercapto-lactic acid-Glyc accounting for 3.0 percent TRR (0.533 mg eq/kg) [pyrazole] and 2.7 percent TRR (0.228 mg eq/kg) [phenyl], and isoflucypram-desfluoro-Cys-MA accounting for 4.4 percent TRR (0.788 mg eq/kg) [pyrazole] and 4.1 percent TRR (0.350 mg eq/kg) [phenyl].

In the exhaustive extract from soya bean straw, a further 1.6 percent TRR (0.288 mg eq/kg) [pyrazole] and 3.1 percent TRR (0.264 mg eq/kg) [phenyl] was identified. Parent isoflucypram accounted for 0.9 percent TRR (0.162 mg/kg) [pyrazole] and 0.6 percent TRR (0.049 mg/kg) [phenyl]. Remaining

major metabolites included isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc accounting for 0.1 percent TRR (0.023 mg eq/kg) [pyrazole only], isoflucypram-desfluoro-mercapto-lactic acid-OH accounting for 0.4 percent TRR (0.065 mg eq/kg) [pyrazole] and 0.1 percent TRR (0.009 mg eq/kg) [phenyl], isoflucypram-desfluoro-Cys-MA accounting for 0.2 percent TRR (0.040 mg eq/kg) [pyrazole] and 0.2 percent TRR (0.020 mg eq/kg) [phenyl], and isoflucypram-homoGSH accounting for 0.3 percent TRR (0.024 mg eq/kg) [phenyl only]. In soya bean straw, 20 unknown metabolites were characterised in the extracts by their chromatographic behaviour, individually accounting for ≤2.8 percent TRR (≤ 0.329 mg eq/kg).

Conventional extract from soya bean seed contained only parent isoflucypram, representing 76.6 percent TRR (0.027 mg/kg) [pyrazole] and 69.8 percent TRR (0.011 mg/kg) [phenyl]. The results are shown in Tables 13 to 16.

Table 13 Distribution of parent compound and metabolites in the extracts of soya bean commodities following foliar application of [Pyrazole-4-^{14}C] Isoflucypram (S14-01089)

Sample	Forage (TRR = 4.371 mg eq/kg)		Hay (TRR = 4.679 mg eq/kg)		Straw (TRR = 17.715 mg eq/kg)		Seed (TRR = 0.035 mg eq/kg)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	92.1	4.026	87.4	4.091	94.1	16.669	87.7	0.031
Isoflucypram	17.6	0.770	10.4	0.487	63.6	11.262	76.6	0.027
Isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc	3.4	0.147	15.2	0.711	3.8	0.667	ND	ND
Isoflucypram-desfluoro-homoGSH	21.9	0.955	7.8	0.366	4.8	0.857	ND	ND
Isoflucypram-desfluoro-mercapto-lactic acid-OH	9.5	0.415	3.2	0.151	1.5	0.272	ND	ND
Isoflucypram-desfluoro-mercapto-lactic acid-Glyc	3.0	0.129	11.1	0.520	3.0	0.533	ND	ND
Isoflucypram-desfluoro-Cys-MA	9.2	0.400	15.4	0.723	4.4	0.788	ND	ND
Subtotal identified	64.5	2.817	63.2	2.958	81.2	14.380	76.6	0.027
Subtotal characterised	26.6	1.164	23.3	1.090	12.0	2.135	ND	ND
Exhaustive extract	5.1	0.222	6.9	0.321	2.5	0.441	-	-
Isoflucypram	1.1	0.049	-	-	0.9	0.162	-	-
Isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc	ND	ND	-	-	0.1	0.023	-	-
Isoflucypram-desfluoro-homoGSH	1.0	0.042	-	-	ND	ND	-	-
Isoflucypram-desfluoro-mercapto-lactic acid-OH	ND	ND	-	-	0.4	0.065	-	-
Isoflucypram-desfluoro-mercapto-lactic acid-Glyc	ND	ND	-	-	ND	ND	-	-
Isoflucypram-desfluoro-Cys-MA	ND	ND	-	-	0.2	0.040	-	-
Subtotal identified	2.1	0.091	-	-	1.6	0.288	-	-
Subtotal characterised	2.3	0.102	-	-	0.8	0.144	-	-
Partitioning of purified								

Sample	Forage (TRR = 4.371 mg eq/kg)		Hay (TRR = 4.679 mg eq/kg)		Straw (TRR = 17.715 mg eq/kg)		Seed (TRR = 0.035 mg eq/kg)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
exhaustive extract								
Ethyl acetate phase	-	-	6.4	0.299	-	-	-	-
Water phase	-	-	NQ	NQ	-	-	-	-
Subtotal characterised by partitioning	-	-	6.4	0.299	-	-	-	-
Total not analysed / losses	1.7	0.074	1.4	0.065	0.9	0.163	11.0	0.004
Total identified	66.6	2.908	63.2	2.985	82.8	14.668	76.6	0.027
Total characterised	28.9	1.266	29.7	1.389	12.8	2.279	ND	ND
Total extracted	97.2	4.248	94.3	4.413	96.6	17.110	87.7	0.031
PES	2.8	0.123	5.7	0.266	3.4	0.605	12.3	0.004
Accountability	100.0	4.371	100.0	4.679	100.0	17.715	100.0	0.035

Notes:
ND: Not detected.
NQ: Not quantified.

Table 14 Summary of characterisation and identification of TRR in soya bean commodities following foliar application of [Pyrazole-4-^{14}C] Isoflucypram (S14-01089)

Component/Sample	Forage (TRR = 4.371 mg eq/kg)		Hay (TRR = 4.679 mg eq/kg)		Straw (TRR = 17.715 mg eq/kg)		Seed (TRR = 0.035 mg eq/kg)	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total extracted	97.2	4.248	94.3	4.413	96.6	17.110	87.7	0.031
Isoflucypram	18.7	0.819	10.4	0.487	64.5	11.424	76.6	0.027
Isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc	3.4	0.147	15.2	0.711	3.9	0.690	ND	ND
Isoflucypram-desfluoro - homoGSH	22.9	0.997	7.8	0.366	4.8	0.857	ND	ND
Isoflucypram-desfluoro-mercapto- lactic acid-OH	9.5	0.415	3.2	0.151	1.9	0.337	ND	ND
Isoflucypram-desfluoro-mercapto-lactic acid-Glyc	3.0	0.129	11.1	0.520	3.0	0.533	ND	ND
Isoflucypram-desfluoro-Cys-MA	9.2	0.400	15.4	0.723	4.6	0.828	ND	ND
Total identified	66.6	2.908	63.2	2.958	82.8	14.668	76.6	0.027
Number of unknown peaks	17		13		20		0	
Largest unknown peak	3.9	0.171	4.4	0.208	1.9	0.329	ND	ND
Subtotal characterised by HPLC	28.9	1.266	23.3	1.090	12.8	2.279	-	-
Subtotal characterised by partitioning of exhaustive extract	-	-	6.4	0.299	-	-	-	-
Total characterised	28.9	1.266	29.7	1.389	12.8	2.279	ND	ND
Not analysed / Losses	1.7	0.074	1.4	0.065	0.9	0.163	11.0	0.004
PES	2.8	0.123	5.7	0.266	3.4	0.605	12.3	0.004
Accountability	100.0	4.371	100.0	4.679	100.0	17.715	100.0	0.035

Notes:
ND: Not detected.

Table 15 Distribution of Isoflucypram and Metabolites in the Extracts of Soya bean Commodities Following Foliar Application of [Phenyl-UL-^{14}C] Isoflucypram (S14-01090)

Component/Sample	Forage (TRR = 3.936 mg eq/kg)		Hay (TRR = 1.397 mg eq/kg)		Straw (TRR = 8.527 mg eq/kg)		Seed (TRR = 0.015 mg eq/kg)	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	91.4	3.597	88.6	1.238	92.8	7.914	69.8	0.011
Isoflucypram	17.9	0.703	10.3	0.144	69.6	5.934	69.8	0.011
Isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc	4.8	0.187	17.6	0.246	2.1	0.177	ND	ND
Isoflucypram-desfluoro-homoGSH	19.6	0.770	7.8	0.109	2.5	0.211	ND	ND
Isoflucypram-desfluoro-mercapto-lactic acid-OH	16.7	0.658	2.8	0.040	2.4	0.206	ND	ND
Isoflucypram-desfluoro-mercapto-lactic acid-Glyc	2.7	0.107	10.7	0.150	2.7	0.228	ND	ND
Isoflucypram-desfluoro-Cys-MA	7.7	0.304	20.5	0.286	4.1	0.350	ND	ND
Subtotal identified	69.4	2.729	69.8	0.974	83.3	7.107	69.8	0.011
Subtotal characterised	21.0	0.828	17.7	0.248	8.5	0.723	ND	ND
Exhaustive Extraction[1]	5.2	0.205	6.3	0.088	3.1	0.264	-	-
Isoflucypram	1.3	0.053	ND	ND	0.6	0.049	-	-
Isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc	ND	ND	ND	ND	ND	ND	-	-
Isoflucypram-desfluoro-homoGSH	0.6	0.023	ND	ND	0.3	0.024	-	-
Isoflucypram-desfluoro-mercapto-lactic acid-OH	0.3	0.014	ND	ND	0.1	0.009	-	-
Isoflucypram-desfluoro-mercapto-lactic acid-Glyc	ND	ND	ND	ND	0.1	0.007	-	-
Isoflucypram-desfluoro-Cys-MA	0.5	0.019	ND	ND	0.2	0.020	-	-
Subtotal identified	2.8	0.108	ND	ND	1.3	0.109	-	-
Subtotal characterised by HPLC	1.7	0.068	-	-	1.8	0.155	-	-
Partitioning of purified exhaustive extract								
Ethyl acetate phase	-	-	5.9	0.082	-	-	-	-
Water phase	-	-	NQ	NQ	-	-	-	-
Subtotal characterised	-	-	5.9	0.082	-	-	-	-
Total not analysed/losses	1.7	0.069	1.5	0.022	1.0	0.084	NQ	NQ
Total identified	72.2	2.837	69.8	0.974	84.6	7.216	69.8	0.011
Total characterised	22.8	0.896	23.6	0.330	10.3	0.878	ND	ND
Total extracted	96.6	3.802	94.9	1.326	95.9	8.178	69.8	0.011
PES	3.2	0.134	5.1	0.071	4.1	0.349	30.2	0.005
Accountability	100.0	3.936	100.0	1.397	100.0	8.527	100.0	0.015

Notes:

[1] For hay and straw, microwave extraction was only performed in the course of a second extraction and values given for exhaustive extraction after first conventional extraction were obtained by conversion. Microwave extract of hay was analysed by HPLC but no peak above the detection limit was detected due to low radioactivity and high matrix content.

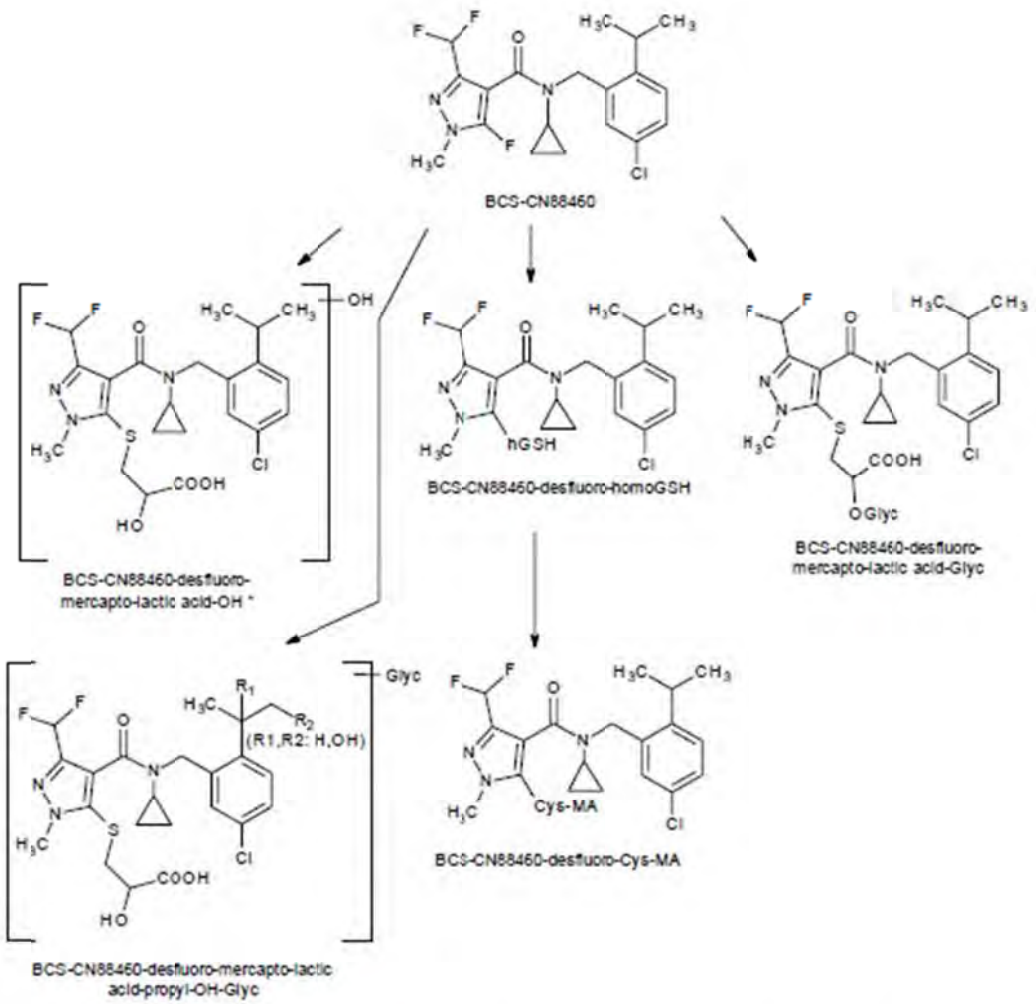
ND: Not detected.

NQ: Not quantified.

Table 16 Summary of characterisation and identification of trr in soya bean commodities following foliar application of [Phenyl-UL-^{14}C] Isoflucypram (S14-01090)

Sample	Forage (TRR = 3.936 mg eq/kg)		Hay (TRR = 1.397 mg eq/kg)		Straw (TRR = 8.527 mg eq/kg)		Seed (TRR = 0.015 mg eq/kg)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total extracted	96.6	3.802	94.9	1.326	95.9	8.178	69.8	0.011
Isoflucypram	19.2	0.756	10.3	0.144	70.2	5.983	69.8	0.011
Isoflucypram-desfluoro-mercapto-lactic acid-propyl- OH-Glyc	4.8	0.187	17.6	0.246	2.1	0.177	ND	ND
Isoflucypram-desfluoro-homoGSH	20.2	0.793	7.8	0.109	2.8	0.235	ND	ND
Isoflucypram-desfluoro-mercapto-lactic acid-OH	17.0	0.672	2.8	0.040	2.5	0.215	ND	ND
Isoflucypram-desfluoro-mercapto-lactic acid-Glyc	2.7	0.107	10.7	0.150	2.8	0.235	ND	ND
Isoflucypram-desfluoro-Cys-MA	8.2	0.323	20.5	0.286	4.3	0.370	ND	ND
Total identified	72.2	2.837	69.8	0.974	84.6	7.216	69.8	0.011
Number of unknown peaks	14		5		20		0	
Largest unknown peak	3.0	0.116	6.1	0.086	2.8	0.230	ND	ND
Subtotal characterised by HPLC	22.8	0.896	17.7	0.248	10.3	0.878	ND	ND
Subtotal characterised by partitioning of exhaustive extract	-	-	5.9	0.082	-	-	-	-
Total characterised	22.8	0.896	23.6	0.330	10.3	0.878	ND	ND
Not analysed / Losses	1.7	0.069	1.5	0.022	1.0	0.084	NQ	NQ
PES	3.4	0.134	5.1	0.071	4.1	0.349	30.2	0.005
Accountability	100.0	3.936	100.0	1.397	100.0	8.527	100.0	0.015

The proposed metabolic pathway of isoflucypram in soya bean is shown in Figure 2



* BCS-CN88460-desfluoro-mercapto-lactic acid-OH: OH position based on mass spectrometry: benzyl moiety.

Figure 2 Metabolic pathway of isoflucypram (BCS-CN88460) in soya bean

Oilseed Rape

Report Nos. S16-01038 and S16-01044.

The metabolism of isoflucypram in oilseed rape plants was investigated following foliar application of [pyrazole-4-^{14}C] and [phenyl-UL-^{14}C] isoflucypram (Botterweck, J.; 2017). Isoflucypram was formulated as an EC 50. The first application was made at BBCH growth stage 14 at 63–64 g ai/ha and the second application was made at BBCH growth stage 77 at 62–63 g ai/ha, for a total application rate of 126 g ai/ha. The timing between applications was 84 days.

Oilseed rape was harvested at BBCH 30 for intermediate harvest, corresponding to two days after the first application. Oilseed rape forage was harvested at BBCH 55, corresponding to 40 days after the first application. Oilseed rape plants and seed were harvested at maturity (BBCH 89), corresponding to 21 days after the second application.

Samples were subjected to conventional and exhaustive extraction and enzyme/acid treatment.

Conventional extraction and clean-up

Homogenised samples of oilseed rape intermediate harvest, forage, mature plants, and seed were extracted three times with ACN/water (8:2). Individual extracts were filtered and the solids were rinsed with the solvent mixture used for extraction. The solids were dried and aliquots were subjected to combustion.

The extracts were combined and cleaned-up step by SPE which was rinsed with methanol and water and conditioned with ACN/water (8:2). The percolate was collected and the cartridge was rinsed with ACN/water (8:2). The percolate and the rinse were combined. Less polar fractions on the cartridge were eluted by rinsing the cartridge with methanol/THF (1:1). Each combined percolate/rinse solution obtained from SPE purification was mixed with emulsifier and evaporated to the aqueous remainder. The purified conventional extracts were analysed by HPLC.

Exhaustive extraction and clean-up

Solids from the conventional extraction of oilseed rape seeds were exhaustively extracted twice with ACN/water/formic acid (50:50:1:v) with microwave assistance. The microwave extracts were cooled down at room temperature and combined. Aliquots of the extracts were cleaned-up step by SPE which was rinsed with methanol and water beforehand. The percolate was collected and the cartridge was rinsed with ACN/water (8:2). Less polar fractions on the cartridge were eluted by rinsing the cartridge with methanol/THF (1:1).

The percolate and the rinse obtained from SPE purification were combined and mixed with emulsifier and evaporated to the aqueous remainder. The final exhaustive extract was analysed by HPLC.

Release of residues upon enzymatic digestion

Solids of oilseed rape seed remaining following exhaustive extraction were incubated with cellulase in sodium acetate buffer (0.1 mol/L) to release additional radioactive residues. The solids were autoclaved (121 °C, 2 bar vapour pressure) in buffer (set to pH 5 using acetic acid) for 2 hours. The solution was mixed with cellulose (100 mg), incubated for 24 hours in a water bath at 37 °C, and centrifuged.

Solids of oilseed rape seed remaining after cellulase treatment were further incubated with amylase in sodium acetate buffer (0.1 mol/L) to release radioactive residues assimilated to carbohydrates. The solids were autoclaved (121 °C, 2 bar) in buffer (set to pH 5 using acetic acid) for 2 hours. The solution was mixed with amylase (50 mg), incubated for 24 hours in a water bath at 37 °C, and centrifuged.

Release of residues upon acidic extraction with HCl

Solids of oilseed rape seed remaining following enzymatic digestion with cellulase and amylase were further extracted with 5 mol/L HCl to release radioactive residues. After addition of HCl the mixture was incubated for 60 minutes at 120 °C with microwave assistance.

Residues in the acid-hydrolysed extracts were characterised by partitioning. The complete extract was neutralised by addition of 10 mol/L NaOH and mixed with ethyl acetate (1:1). The ethyl acetate and water phases were separated, the water phase was again mixed with ethyl acetate (1:1), and the procedure was repeated.

Hydrolysis of the conventional extracts from oilseed rape intermediate harvest

The final purified extract for oilseed rape intermediate harvest was mixed with 10 mol/L HCl and incubated at 100 °C for 1 hour. The mixture was subsequently adjusted to pH 7 with 10 mol/L NaOH and analysed by HPLC.

The radioactivity in extracts and the PES was determined by combustion/LSC. TRR was calculated by summing the radioactivity of the combined extracts and the PES.

Parent compound and major metabolites were identified by spectroscopic methods and co-chromatography with reference compounds. In both radiolabel studies, samples were stored frozen for less than three months at -18 °C. The results are in Tables 17 to 20 and Figure 3 shows the proposed metabolic pathway of isoflucypram in oilseed rape.

Table 17 Distribution of radioactivity in the extracts of oilseed rape commodities following foliar application of [Pyrazole-4-^{14}C] Isoflucypram (S16-01038)

Sample	Intermediate Harvest (TRR = 4.751 mg eq/kg)		Forage (TRR = 0.012)		Mature Plants (TRR = 4.076)		Seed (TRR = 0.099)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	99.5	4.730	85.4	0.010	97.4	3.970	71.0	0.070
Analysed extracts	99.1	4.710	85.4	0.010	97.3	3.964	71.0	0.070
Not analysed	0.4	0.020	NQ	NQ	0.2	0.006	NQ	NQ
Exhaustive extract	-	-	-	-	-	-	9.8	0.010
Analysed extracts[1]	-	-	-	-	-	-	7.6	0.008
Not analysed	-	-	-	-	-	-	2.2	0.002
Enzymatic digestion	-	-	-	-	-	-	1.5	0.002
Acidic extraction and partitioning	-	-	-	-	-	-	11.0	0.011
Ethyl acetate phase	-	-	-	-	-	-	5.4	0.005
Water phase	-	-	-	-	-	-	2.7	0.003
Not analysed	-	-	-	-	-	-	2.9	0.003
Total extracted	99.5	4.730	85.4	0.010	97.4	3.970	93.3	0.093
PES	0.5	0.022	14.6	0.002	2.6	0.106	6.7	0.006
Accountability	100.0	4.751	100.0	0.012	100.0	4.076	100.0	0.099

Notes:
NQ: Not quantified.
[1] No individual peak above detection limit was observed in the HPLC chromatogram of the exhaustive extract of seeds.

Table 18 Distribution of radioactivity in the extracts of oilseed rape commodities following foliar application of [Phenyl-UL-^{14}C] Isoflucypram (S16-01044)

Sample	Intermediate Harvest (TRR = 3.295 mg eq/kg)		Forage (TRR = 0.008 mg eq/kg)		Mature Plants (TRR = 3.934 mg eq/kg))		Seed (TRR = 0.126 mg eq/kg)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	99.7	3.285	77.3	0.006	96.2	3.786	73.6	0.093
Analysed extracts	99.6	3.281	77.3	0.006	96.0	3.776	73.6	0.093
Not analysed	0.1	0.004	NQ	NQ	0.2	0.010	NQ	NQ
Exhaustive extract	-	-	-	-	-	-	10.6	0.013
Analysed extracts[1]	-	-	-	-	-	-	9.6	0.012
Not analysed	-	-	-	-	-	-	1.0	0.001
Enzymatic digestion	-	-	-	-	-	-	1.3	0.002
Acidic extraction and partitioning	-	-	-	-	-	-	7.9	0.010

Sample	Intermediate Harvest (TRR = 3.295 mg eq/kg)		Forage (TRR = 0.008 mg eq/kg)		Mature Plants (TRR = 3.934 mg eq/kg))		Seed (TRR = 0.126 mg eq/kg)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Ethyl acetate phase	-	-	-	-	-	-	4.4	0.005
Water phase	-	-	-	-	-	-	2.1	0.003
Not analysed	-	-	-	-	-	-	1.4	0.002
Total extracted	99.7	3.285	77.3	0.006	96.2	3.786	93.5	0.118
PES	0.3	0.010	22.7	0.002	3.8	0.148	6.5	0.008
Accountability	100.0	3.295	100.0	0.008	100.0	3.934	100.0	0.126

Notes:
NQ: Not quantified.
[1] No individual peak above detection limit was observed in the HPLC chromatogram of the exhaustive extract of seeds.

In the conventional extract from intermediate harvest, 92.3 percent TRR (4.384 mg eq/kg) [pyrazole] and 94.0 percent TRR (3.096 mg eq/kg) [phenyl] was identified. Parent isoflucypram was the major component representing 81.9 percent TRR (3.890 mg/kg) [pyrazole] and 84.1 percent TRR (2.770 mg/kg) [phenyl]. Remaining major metabolites included isoflucypram-hydroxyphenyl-Gluc-MA accounting for 2.3 percent TRR (0.109 mg eq/kg) [pyrazole] and 2.3 percent TRR (0.077 mg eq/kg) [phenyl], isoflucypram-2-propanol-Glyc-MA accounting for 2.2 percent TRR (0.106 mg eq/kg) [pyrazole] and 1.6 percent TRR (0.052 mg eq/kg) [phenyl], isoflucypram-propanol-Glyc-MA accounting for 2.8 percent TRR (0.131 mg eq/kg) [pyrazole] and 2.2 percent TRR (0.071 mg eq/kg) [phenyl], and isoflucypram-hydroxyphenyl-Glyc-MA accounting for 3.1 percent TRR (0.148 mg eq/kg) [pyrazole] and 3.8 percent TRR (0.126 mg eq/kg) [phenyl]. In the conventional extract of intermediate harvest, up to 23 unknown metabolites were characterised, individually accounting for ≤ 1.5 percent TRR (≤ 0.072 mg eq/kg).

Comparison of metabolic profiles before and after acid hydrolysis of intermediate harvest indicated cleavage of isoflucypram-hydroxyphenyl-Gluc-MA, isoflucypram-2-propanol-Glyc-MA, isoflucypram-propanol-Glyc-MA, and isoflucypram-hydroxyphenyl-Glyc-MA to less polar compounds.

In the conventional extract from forage, 85.4 percent TRR (0.010 mg eq/kg) [pyrazole] and 77.3 percent TRR (0.006 mg eq/kg) [phenyl] was analysed. No individual peak was above the background noise due to low radioactivity.

In the conventional extract from mature plants, 91.3 percent TRR (3.719 mg eq/kg) [pyrazole] and 84.4 percent TRR (3.318 mg eq/kg) [phenyl] was identified in total. The parent compound was the major component representing 88.1 percent TRR (3.589 mg/kg) [pyrazole] and 72.0 percent TRR (2.831 mg/kg) [phenyl]. Remaining major metabolites included isoflucypram-hydroxyphenyl-Gluc-MA accounting for 0.7 percent TRR (0.027 mg eq/kg) [pyrazole] and 2.2 percent TRR (0.087 mg eq/kg) [phenyl], isoflucypram-2-propanol-Glyc-MA accounting for 0.9 percent TRR (0.038 mg eq/kg) [pyrazole] and 4.6 percent TRR (0.181 mg eq/kg) [phenyl], isoflucypram-propanol-Glyc-MA accounting for 0.6 percent TRR (0.025 mg eq/kg) [pyrazole] and 2.5 percent TRR (0.097 mg eq/kg) [phenyl], and isoflucypram-hydroxyphenyl-Glyc-MA accounting for 1.0 percent TRR (0.040 mg eq/kg) [pyrazole] and 3.1 percent TRR (0.122 mg eq/kg) [phenyl]. In the conventional extract in mature plants, up to 39 unknown metabolites were characterised, individually accounting for ≤1.3 percent TRR (≤ 0.054 mg eq/kg).

In the conventional extract from seeds, 71.0 percent TRR (0.070 mg eq/kg) [pyrazole] and 73.6 percent TRR (0.093 mg eq/kg) [phenyl] was identified. Parent isoflucypram was the only component, representing 71.0 percent TRR (0.070 mg/kg) [pyrazole] and 73.6 percent TRR (0.093 mg/kg) [phenyl].

Exhaustive extraction of seeds released a further 9.8 percent TRR (0.010 mg eq/kg) [pyrazole] and 10.6 percent TRR (0.013 mg eq/kg) [phenyl], of which and 7.6 percent TRR (0.008 mg eq/kg) [pyrazole] and 9.6 percent TRR (0.012 mg eq/kg) [phenyl] was analysed by HPLC. No individual peak was above the background noise due to low radioactivity.

Table 19 Summary of characterisation and identification of TRR in oilseed rape commodities following foliar application of [Pyrazole-4-^{14}C] Isoflucypram (S16-01038)

Sample	Intermediate Harvest (TRR = 4.751 mg eq/kg)		Forage (TRR = 0.012 mg eq/kg)		Mature Plants (TRR = 4.076 mg eq/kg)		Seed (TRR = 0.099 mg eq/kg)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	99.5	4.730	85.4	0.010	97.4	3.970	71.0	0.070
Isoflucypram	81.9	3.890	ND	ND	88.1	3.589	71.0	0.070
Isoflucypram- hydroxyphenyl-Gluc-MA	2.3	0.109	ND	ND	0.7	0.027	ND	ND
Isoflucypram-2- propanol-Glyc-MA	2.2	0.106	ND	ND	0.9	0.038	ND	ND
Isoflucypram-propanol- Glyc-MA	2.8	0.131	ND	ND	0.6	0.025	ND	ND
Isoflucypram- hydroxyphenyl-Glyc-MA	3.1	0.148	ND	ND	1.0	0.040	ND	ND
Total identified	92.3	4.384	-	-	91.3	3.719	71.0	0.070
Characterised by HPLC	6.8	0.326	-	-	6.0	0.245	-	-
Number of unknown peaks	22		-		16		-	
Largest unknown peak	1.5	0.072	-	-	1.3	0.054	-	-
Total not analysed of conventional extract	0.4	0.020	-	-	0.2	0.006	-	-
Exhaustive extract	-	-	-	-	-	-	9.8	0.010
Analysed by HPLC[1]	-	-	-	-	-	-	7.6	0.008
Not analysed	-	-	-	-	-	-	2.2	0.002
Enzymatic digestion	-	-	-	-	-	-	1.5	0.002
Acidic extraction and partitioning	-	-	-	-	-	-	11.0	0.011
Ethyl acetate phase	-	-	-	-	-	-	5.4	0.005
Water phase	-	-	-	-	-	-	2.7	0.003
Not analysed by partition	-	-	-	-	-	-	2.9	0.003
Total characterised[2]	6.8	0.326	-	-	6.0	0.245	22.3	0.023
Total extracted	99.5	4.730	85.4	0.010	97.4	3.970	93.3	0.093
PES	0.5	0.022	14.6	0.002	2.6	0.106	6.7	0.006
Accountability	100.0	4.751	100.0	0.012	100.0	4.076	100.0	0.099

Notes:

[1] No individual peak above detection limit was observed in the HPLC chromatogram of the exhaustive extract of seeds.

[2] By chromatographic and/or extraction behaviour.

ND: Not detected.

Table 20 Summary of characterisation and identification of TRR in oilseed rape commodities following foliar application of [Phenyl-UL-^{14}C] Isoflucypram (S16-01044)

Sample	Intermediate Harvest (TRR = 3.295 mg eq/kg)		Forage (TRR = 0.008 mg eq/kg)		Mature Plants (TRR = 3.934)		Seed (TRR = 0.126)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	99.7	3.285	77.3	0.006	96.2	3.786	73.6	0.093
Isoflucypram	84.1	2.770	ND	ND	72.0	2.831	73.6	0.093
Isoflucypram- hydroxyphenyl-	2.3	0.077	ND	ND	2.2	0.087	ND	ND

Sample	Intermediate Harvest (TRR = 3.295 mg eq/kg)		Forage (TRR = 0.008 mg eq/kg)		Mature Plants (TRR = 3.934)		Seed (TRR = 0.126)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Gluc- MA								
Isoflucypram-2-propanol-Glyc-MA	1.6	0.052	ND	ND	4.6	0.181	ND	ND
Isoflucypram-propanol- Glyc-MA	2.2	0.071	ND	ND	2.5	0.097	ND	ND
Isoflucypram- hydroxyphenyl-Glyc- MA	3.8	0.126	ND	ND	3.1	0.122	ND	ND
Total identified	94.0	3.096	-	-	84.4	3.318	73.6	0.093
Characterised by HPLC	5.6	0.185	-	-	11.6	0.458	-	-
Number of unknown peaks	23		-		39		-	
Largest unknown peak	1.2	0.038	-	-	1.3	0.050	-	-
Total not analysed of conventional extract	0.1	0.004	-	-	0.2	0.010	-	-
Exhaustive extract	-	-	-	-	-	-	10.6	0.013
Analysed by HPLC[1]	-	-	-	-	-	-	9.6	0.012
Not analysed	-	-	-	-	-	-	1.0	0.001
Enzymatic digestion	-	-	-	-	-	-	1.3	0.002
Acidic extraction and partitioning	-	-	-	-	-	-	7.9	0.010
Ethyl acetate phase	-	-	-	-	-	-	4.4	0.005
Water phase	-	-	-	-	-	-	2.1	0.003
Not analysed by partition	-	-	-	-	-	-	1.4	0.002
Total characterised[2]	5.6	0.185	-	-	11.6	0.458	19.8	0.025
Total extracted	99.7	3.285	77.3	0.006	96.2	3.786	93.5	0.118
PES	0.3	0.010	22.7	0.002	3.8	0.148	6.5	0.008
Accountability	100.0	3.295	100.0	0.008	100.0	3.934	100.0	0.126

Notes:

[1] No individual peak above detection limit was observed in the HPLC chromatogram of the exhaustive extract of seeds.

[2] By chromatographic and/or extraction behaviour.

ND: Not detected.

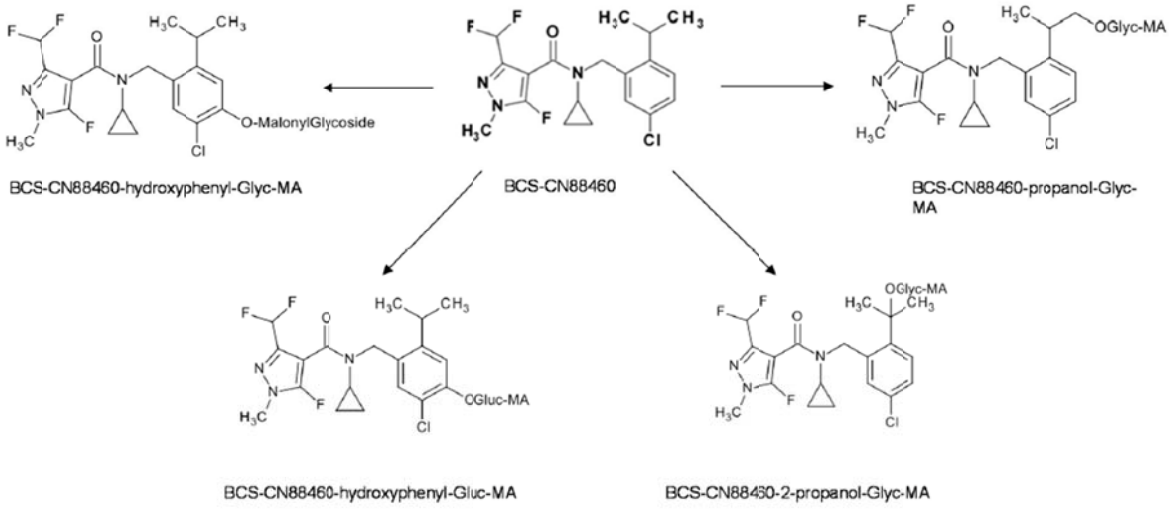


Figure 3 Metabolic pathway isoflucypram (BCS-CN88460) in oilseed rape

Potato Seed Treatment

Report Nos. S17-01394 and S17-01392.

The metabolism of isoflucypram in potatoes was investigated following seed treatment application of [pyrazole-4-^{14}C] and [phenyl-UL-^{14}C] isoflucypram (Botterweck, J.; 2018). Isoflucypram was formulated as an EC 200 and applied to potato tubers prior to planting. Potato seeds were treated at 28 g ai/ha (0.55 mg ai/ tuber and 50,000 plants/ha) for a low dose experiment and 274–280 g ai/ha (5.5 mg ai/tuber and 50,000 plants/ha) for a high dose experiment.

Potato tubers and leaves were harvested at BBCH growth stage 97, corresponding to 119 days after planting. Samples were subjected to conventional and exhaustive extraction.

Conventional extraction and clean-up

Due to the low radioactivity of tubers following the low dose application, no extraction was performed. For conventional extraction of tubers in the high dose experiment and leaves for both dose levels, homogenised samples were extracted three times with ACN/water (8:2). Individual extracts were filtered and the solids were rinsed with the solvent mixture used for extraction. The solids were dried and aliquots were subjected to combustion analysis.

The extracts were combined and cleaned-up by SPE. The percolate was collected and the cartridge was rinsed with ACN/water (8:2). The percolate and the rinse were combined. Less polar fractions on the cartridge were eluted by rinsing the cartridge with methanol/THF (1:1).

Each combined percolate/rinse solution obtained from SPE purification was evaporated to the aqueous remainder and mixed with emulsifier beforehand. The purified conventional extracts were analysed by HPLC.

Exhaustive extraction and clean-up

Solids from the conventional extraction of potato leaves in the low dose pyrazole experiment and potato tubers in the high dose phenyl experiment were exhaustively extracted twice with ACN/water/formic acid (50:50:1:v) with microwave assistance at 120 °C. The microwave extracts were combined and cleaned-up by SPE. The combined fractions from SPE purification were evaporated to the aqueous remainder.

The radioactivity in extracts and the PES was determined by combustion/LSC. The total radioactive residue TRR was calculated by summing the radioactivity of the combined extracts and the PES.

Isoflucypram and the major metabolites were identified by spectroscopic methods and use of co-chromatography with reference compounds.

In the pyrazole study, potato tubers were stored frozen for a maximum of two months at -18 °C. Potato leaves were stored for a maximum of 10 months at -18 °C. Comparison of the metabolite profile in extract stored after 10 months of storage at -18°C demonstrated that the residue profile was stable for at least 10 months.

In the phenyl study, potato tubers and leaves were stored frozen for a maximum of three months at ≤-18 °C.

Table 21 Distribution of radioactivity in the extracts of potato tubers and leaves following seed treatment application of [Pyrazole-4-^{14}C] Isoflucypram (S17-01394)

Sample	Low Dose Experiment		High Dose Experiment			
	Leaves TRR = 0.374 mg eq/kg		Tubers TRR = 0.064 mg eq/kg		Leaves TRR = 1.071 mg eq/kg	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	88.6	0.331	97.7	0.063	93.2	0.998
Analysed extracts	86.9	0.324	97.5	0.063	92.6	0.991
Not analysed	1.7	0.007	0.3	< 0.001	0.6	0.007
Exhaustive extract	7.5	0.028	-	-	-	-
Analysed extracts[1]	6.9	0.026	-	-	-	-
Not analysed	0.6	0.002	-	-	-	-
Total extracted	96.1	0.359	97.7	0.063	93.2	0.998
PES	3.9	0.015	2.3	0.001	6.8	0.073
Accountability	100.0	0.374	100.0	0.064	100.0	1.071

Notes:

[1] No individual peak above detection limit was observed in the HPLC chromatogram of the exhaustive extract of tubers for the overdose experiment.

Table 22 Distribution of radioactivity in the extracts of potato tubers and leaves following seed treatment application of [Phenyl-UL-^{14}C] Isoflucypram (S17-01392)

Component/Sample			High Dose Experiment			
	Leaves TRR = 0.050 mg eq/kg		Tubers TRR = 0.042 mg eq/kg		Leaves TRR = 0.688 mg eq/kg	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	92.6	0.046	82.2	0.034	93.8	0.645
Analysed extracts	92.2	0.046	82.2	0.034	93.3	0.641
Not analysed	0.4	< 0.001	< 0.1	< 0.001	0.5	0.003
Exhaustive extract	-	-	12.9	0.005	-	-
Analysed extracts[1]	-	-	8.7	0.004	-	-
Not analysed	-	-	4.2	0.001	-	-
Total extracted	92.6	0.046	95.1	0.039	93.8	0.645
PES	7.4	0.004	4.9	0.003	6.2	0.043
Accountability	100.0	0.050	100.0	0.042	100.0	0.688

Notes:

[1] No individual peak above detection limit was observed in the HPLC chromatogram of the exhaustive extract of tubers for the overdose experiment.

Low dose experiment

No extraction/profiling was performed in tubers due to low radioactivity (0.002–0.009 mg eq/kg).

In the conventional extract of potato leaves, 88.6 percent TRR (0.331 mg eq/kg) [pyrazole] and 92.6 percent TRR (0.046 mg eq/kg) [phenyl] was released. Parent isoflucypram was a minor component of the residue accounting for 2.0 percent TRR (0.007 mg/kg) [pyrazole] and 7.3 percent TRR (0.004 mg/kg) [phenyl]. Major metabolites identified included isoflucypram-OH-phenyl-Glyc-MA accounting for 6.6 percent TRR (0.025 mg eq/kg) [pyrazole] and 23.4 percent TRR (0.012 mg eq/kg) [phenyl], isoflucypram-2-propanol-Glyc-MA accounting for 14.3 percent TRR (0.053 mg eq/kg) [pyrazole] and 29.0 percent TRR (0.014 mg eq/kg) [phenyl], and isoflucypram-cyclopropyl-pyrazole-carboxamide accounting for 10.7 percent TRR (0.040 mg eq/kg) [pyrazole, only]. Up to 17 unknown metabolites were characterised, individually accounting for ≤19.9 percent TRR (≤ 0.024 mg eq/kg).

Exhaustive extraction released an additional 7.5 percent TRR (0.028 mg eq/kg) [pyrazole]. No detectable peaks were observed in the exhaustive extract. The results are shown in Table 23

Table 23 Summary of characterisation and identification of TRR in potato leaves following low dose seed treatment application of [Pyrazole-4-^{14}C] (Study S17-01394) and [Phenyl-UL-^{14}C] Isoflucypram (Study S17-01392)

Sample	[Phenyl-UL-^{14}C] (TRR = 0.05mg eq/kg)		[Pyrazole-4-^{14}C] (TRR = 0.374 mg eq/kg)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	92.6	0.046	88.6	0.331
Isoflucypram	7.3	0.004	2.0	0.007
Isoflucypram-cyclopropyl-pyrazole-carboxamide	-	-	10.7	0.040
Isoflucypram-OH-phenyl-Glyc-MA	23.4	0.012	6.6	0.025
Isoflucypram-2-propanol-Glyc-MA	29.0	0.014	14.3	0.053
Total identified	59.6	0.030	33.6	0.125
Number of unknown peaks	2		17	
Largest unknown peak	19.9	0.010	6.3	0.024
Subtotal characterised	32.6	0.016	53.3	0.199
Total not analysed of conventional extract	-	-	1.7	0.007
Exhaustive extract	-	-	7.5	0.028
Analysed by HPLC[1]	-	-	6.9	0.026
Not analysed	-	-	0.6	0.002
Subtotal characterised	-	-	6.9	0.026
Total characterised by HPLC	-	-	60.2	0.225
Total extracted	92.6	0.046	96.1	0.359
Total not analysed	0.4	< 0.001	2.3	0.009
PES	7.4	0.004	3.9	0.015
Accountability	100.0	0.050	100.0	0.374

Notes:

[1] No individual peak above detection limit was observed in the HPLC chromatogram of the exhaustive extract of leaves.

High dose experiment

In the conventional extract for potato tubers, 97.7 percent TRR (0.063 mg eq/kg) [pyrazole] and 82.2 percent TRR (0.034 mg eq/kg) [phenyl] was released. Isoflucypram represented the main residue component at 86.4 percent TRR (0.056 mg/kg) [pyrazole] and 69.2 percent TRR (0.029 mg/kg) [phenyl]. The only other identified compound was isoflucypram-cyclopropyl-pyrazole-carboxamide accounting for 11.1 percent TRR (0.007 mg eq/kg) [pyrazole only]. Up to one unidentified metabolite was characterized accounting for 13.0 percent TRR (0.005 mg eq/kg).

In the potato tuber pyrazole study, and additional 12.9 percent TRR (0.005 mg eq/kg) was released in the exhaustive extract. No single peak was identified above the limit of detection.

In the conventional extract for potato leaves, 93.2 percent TRR (0.998 mg eq/kg) [pyrazole] and 93.8 percent TRR (0.645 mg eq/kg) [phenyl] was released. Isoflucypram accounted for 2.5 percent TRR (0.027 mg/kg) [pyrazole] and 4.0 percent TRR (0.027 mg/kg) [phenyl]. Remaining major metabolites included isoflucypram-cyclopropyl-pyrazole-carboxamide accounting for 7.2 percent TRR (0.077 mg eq/kg) [pyrazole only], isoflucypram-OH-phenyl-Glyc-MA accounting for 9.9 percent TRR (0.105 mg eq/kg) [pyrazole] and 15.0 percent TRR (0.103 mg eq/kg) [phenyl], and isoflucypram-2-propanol-Glyc-MA accounting for 13.9 percent TRR (0.148 mg eq/kg) [pyrazole] and 15.0 percent TRR (0.103 mg eq/kg) [phenyl]. In leaves, up to 34 unknown metabolites were characterised, individually

accounting for ≤8.6 percent TRR (≤ 0.064 mg eq/kg). The results are shown in Tables 24 and 25 and the proposed metabolic pathways in Figures 4 and 5.

Table 24 Distribution of isoflucypram and metabolites in the extracts of potato tubers and leaves following high dose seed treatment application of [Pyrazole-4-^{14}C] Isoflucypram (S17-01394)

Sample	Tubers (TRR = 0.064 mg eq/kg)		Leaves (TRR = 1.071 mg eq/kg)	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	97.7	0.063	93.2	0.998
Isoflucypram	86.4	0.056	2.5	0.027
Isoflucypram-cyclopropyl-pyrazole-carboxamide	11.1	0.007	7.2	0.077
Isoflucypram-OH-phenyl-Glyc-MA	ND	ND	9.9	0.105
Isoflucypram-2-propanol-Glyc-MA	ND	ND	13.9	0.148
Total identified	97.5	0.063	33.5	0.357
Number of unknown peaks	0		32	
Largest unknown peak	-	-	6.0	0.064
Total characterised	-	-	59.1	0.634
Total not analysed of conventional extract	0.3	<0.001	0.6	0.007
PES	2.3	0.001	6.8	0.073
Accountability	100.0	0.064	100.0	1.071

Notes:
ND: Not detected.

Table 25 Distribution of isoflucypram and metabolites in the extracts of potato tubers and leaves following high dose seed treatment application of Phenyl-UL-^{14}C] Isoflucypram (S17-01392)

Sample	Tubers TRR = 0.042 mg eq/kg		Leaves TRR = 0.688 mg eq/kg	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	82.2	0.034	93.8	0.645
Isoflucypram	69.2	0.029	4.0	0.027
Isoflucypram-OH-phenyl-Glyc-MA	ND	ND	15.0	0.103
Isoflucypram-2-propanol-Glyc-MA	ND	ND	15.0	0.103
Total identified	69.2	0.029	34.0	0.233
Number of peaks	1		21	
Largest unknown peak	13.0	0.005	8.6	0.059
Subtotal characterised	13.0	0.005	59.3	0.408
Total not analysed of conventional extract	< 0.1	< 0.001	0.5	0.003
Exhaustive extract	12.9	0.005	-	-
Analysed by HPLC[1]	8.7	0.004	-	-
Not analysed	4.2	0.001	-	-
Total characterised by HPLC	21.7	0.009	59.3	0.408
Total extracted	95.1	0.039	93.8	0.645
Total not analysed	4.2	0.001	0.5	0.003
PES	4.9	0.002	6.2	0.043
Accountability	100.0	0.042	100.0	0.688

Notes:
ND: Not detected.
[1] No individual peak above detection limit was observed in the HPLC chromatogram of the exhaustive extract of leaves.

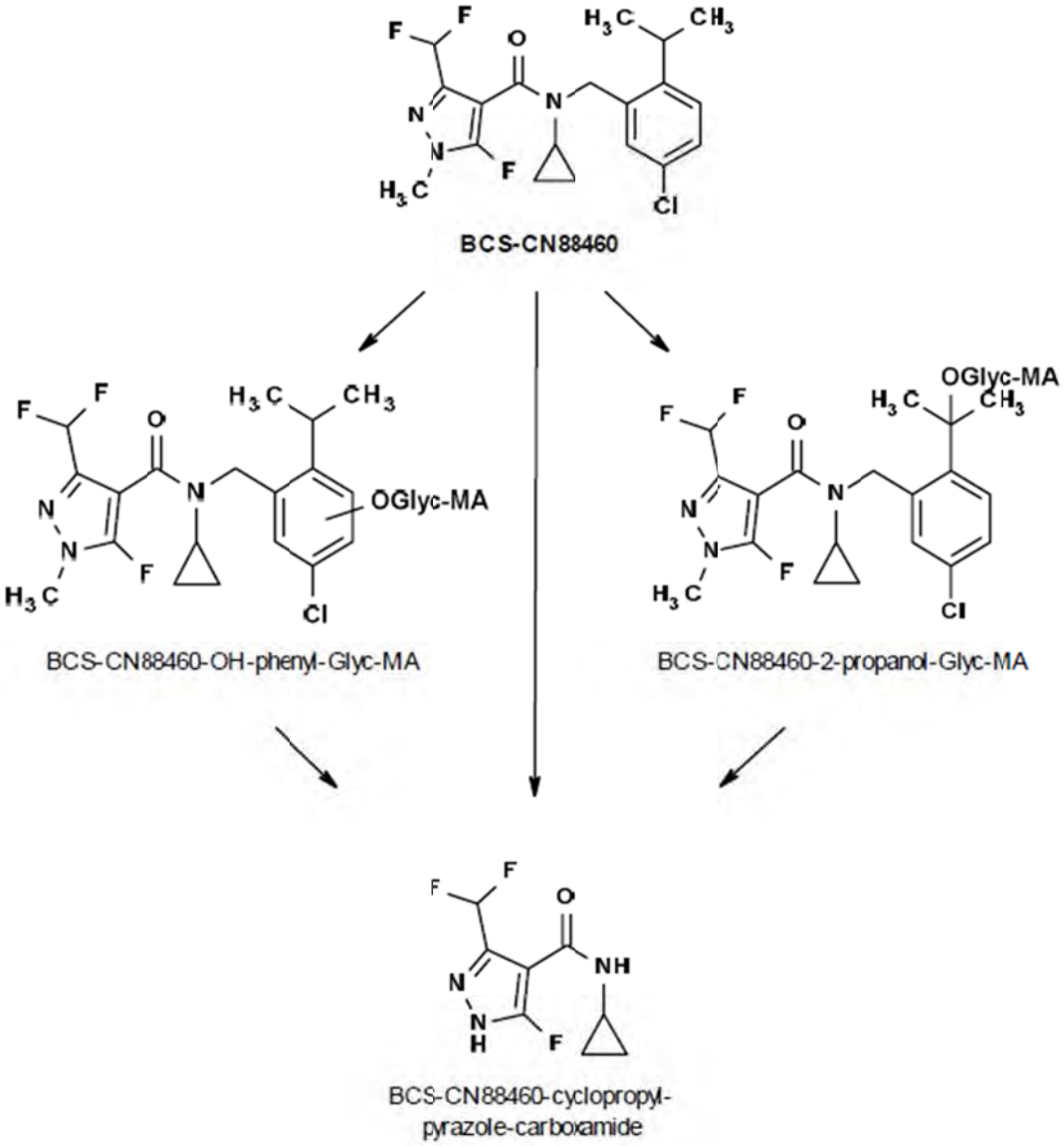


Figure 4 Metabolic Pathway of Pyrazole label isoflucypram (BCS-CN88460) in potato following seed treatment

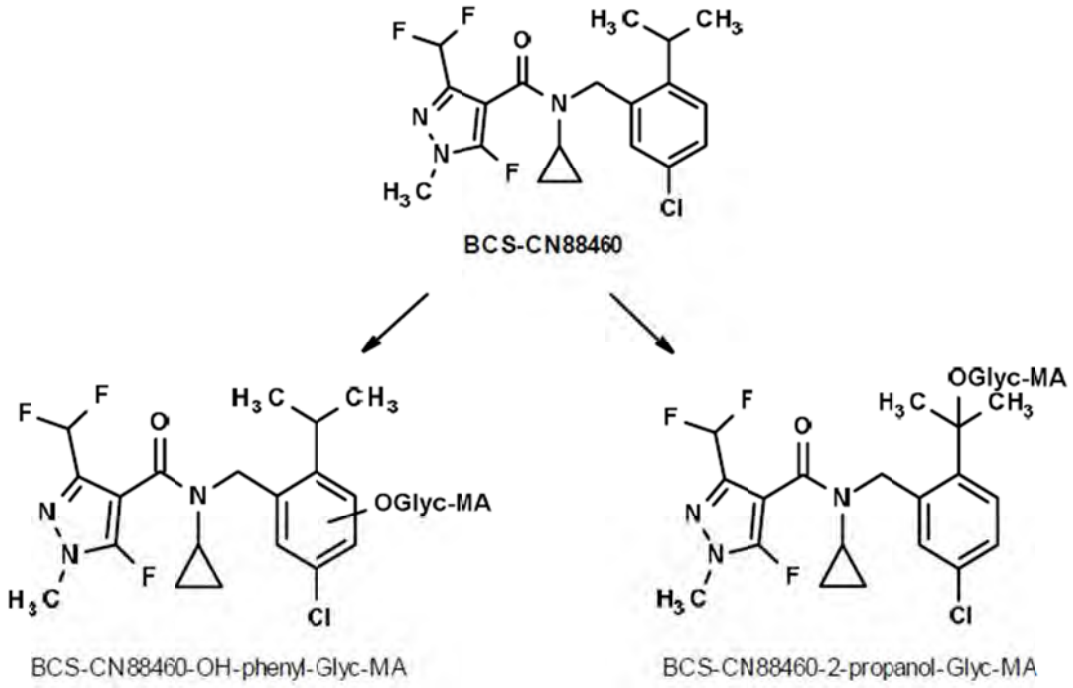


Figure 5 Metabolic pathway of Phenyl label isoflucypram (BCS-CN88460) in potato following seed treatment

Confined rotational crops

Report Nos. EnSa-16-945 and EnSa-17-0128.

Confined rotational crop studies were submitted with [pyrarazol-4-^{14}C] isoflucypram and [phenyl-UL-^{14}C] isoflucypram (Lamshoeft, M., *et al*., 2017). The experimental design and sample work-up was the same for both studies, except as noted below. The test substance was applied to bare sandy loam soil as a single spray application at a rate of 198–210 g ai/ha. Turnips, Swiss chard, and wheat were sown into the treated soil with 30-, 140-, and 287-day plant-back intervals (PBIs). Immature samples of Swiss chard (BBCH 45), wheat forage (BBCH 29), and wheat hay (BBCH 75–83) and mature samples of turnip roots, turnip tops, Swiss chard, wheat straw, and wheat grain were harvested and analysed for residues. The TRRs are shown in Table 26.

Table 26 TRR (mg eq/kg) in confined rotational crops following treatment with label isoflucypram

	Pyrazole-4-14C (EnSa-16-945)			Phenyl-UL-^{14}C (EnSa-17-0128)		
Matrix	30-Day PBI	140-Day PBI	287-Day PBI	30-Day PBI	140-Day PBI	287-Day PBI
Turnip tops	0.018	0.031	0.026	0.004[1]	0.006[1]	0.006[1]
Turnip roots	0.006[1]	0.006[1]	0.006[1]	0.003[1]	0.003[1]	0.003[1]
Swiss chard (immature)	0.031	0.062	0.056	0.029	0.016	0.020
Swiss chard (mature)	0.026	0.062	0.052	0.020	0.016	0.025
Wheat forage	0.041	0.078	0.072	0.023	0.018	0.015
Wheat hay	0.114	0.220	0.187	0.039	0.062	0.036
Wheat straw	0.131	0.247	0.340	0.051	0.070	0.055
Wheat grain	0.004[1]	0.011	0.016	0.001[1]	0.004[1]	0.003[1]

Notes:

[1] Determined by combustion and LSC. Samples were not extracted due to low levels of radioactivity

TRR was determined by combustion and LSC. For all matrices with adequate radioactivity, samples were extracted using ACN/water (4:1). The following matrices were not extracted for analysis due to low levels of radioactivity: turnip roots (all PBIs for the pyrazole label), turnip roots and tops (all PBIs for the phenyl label), and wheat grain (30-day PBI for the pyrazole label and all PBIs for the phenyl label).

PES for wheat hay (all PBIs), wheat straw (all PBIs), and wheat grain (140- and 287-day PBIs) were further extracted using ACN/water/formic acid (50:50:1) with microwave assistance and SPE. Following microwave extraction, solids from pyrazole-label wheat hay and wheat straw were subjected to a second microwave extraction using dioxane and 5 mol/L HCl. Extracts were analysed by HPLC using radiometric and ultraviolet (UV) detection.

For the phenyl label only, extracts of wheat straw and Swiss chard from the 30-day PBI were hydrolysed for 1 hour in 1 mol/L HCl at 100 °C to investigate conjugated residues of parent compound and metabolites.

Compound identification was accomplished by comparison of HPLC profile for the parent compound and by spectroscopic methods with comparison of chromatographic profiles for metabolites.

All samples were stored frozen for a maximum of 41 days. Within a maximum of one month after extraction, the earliest metabolite profiles (used for quantitation of metabolites) were obtained by HPLC-analysis. Therefore, it was concluded that the results of this study were not negatively influenced by storage effects.

In pyrazole-labelled rotational turnip tops, 92.3–93.1 percent TRR (0.017–0.029 mg eq/kg) was extracted. Parent isoflucypram was a minor residue representing 4.8 percent TRR (0.001 mg/kg) at the first PBI and was not detected at longer PBIs. All other identified components retained only the pyrazole moiety and were minor with concentrations ≤ 0.006 mg eq/kg. Up to nine unknown peaks were characterised, each accounting for ≤ 9.5 percent TRR (≤ 0.003 mg eq/kg). The results are in Table 27

Table 27 Residue profile of [Pyrazole-4-^{14}C] isoflucypram in confined rotational turnip tops (EnSa-16-945)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR: 0.018 mg eq/kg		TRR: 0.031 mg eq/kg		TRR: 0.026 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	92.3	0.017	93.1	0.029	92.3	0.024
Isoflucypram	4.8	0.001	-	-	-	-
BCS-CR60082	18.4	0.003	12.7	0.004	9.4	0.002
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala	-	-	8.1	0.002	4.7	0.001
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 1/2)	-/13	-/0.002	6.1/4.6	0.002/ 0.001	5.5/2.9	0.001/ 0.001
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys	7.7	0.001	4.4	0.001	2.9	0.001
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH	25.7	0.005	19.9	0.006	13.9	0.004
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto- Glyc	-	-	2.9	0.001	5.0	0.001
Isoflucypram-desfluoro-N-	12.3	0.002	7.4	0.002	3.8	0.001

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR: 0.018 mg eq/kg		TRR: 0.031 mg eq/kg		TRR: 0.026 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc-MA						
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-desamino-Cys	4.6	0.001	-	-	1.8	<0.001
Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala	5.9	0.001	7.2	0.002	5.8	0.001
Total identified	92.3	0.017	73.3	0.022	55.4	0.014
Number of unknown peaks	0		4		9	
Largest unknown peak	-	-	9.5	0.003	7.3	0.002
Total characterized (by HPLC)	NA	NA	19.7	0.006	36.9	0.01
PES	7.7	0.001	6.9	0.002	7.7	0.002
Accountability	100	0.018	100	0.031	100	0.026

In rotational immature Swiss chard, a total of 94.4–97.2 percent TRR (0.030-0.060 mg eq/kg) [pyrazole] and 96.2–97.7 percent TRR (0.015–0.028 mg eq/kg) [phenyl] was extracted. Parent isoflucypram was a minor component in the pyrazole and phenyl studies representing ≤ 6 percent TRR (≤ 0.002 mg/kg). All remaining identified metabolites retained only the pyrazole moiety. Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH was a major metabolite accounting for 23.0–26.9 percent TRR (0.008–0.016 mg eq/kg). All other metabolites were minor components accounting for ≤ 0.007 mg eq/kg. Up to 19 unknown peaks were characterised based on chromatographic behaviour, each accounting for ≤ 25.4 percent TRR (≤ 0.009 mg eq/kg).

In the hydrolysed extract of phenyl-labelled immature Swiss chard at the 30-day PBI, isoflucypram was identified at 7.4 percent TRR (0.002 mg/kg), isoflucypram-propanol was identified at 10.9 percent TRR (0.003 mg eq/kg), and isoflucypram-carboxylic acid was identified at 37.2 percent TRR (0.011 mg eq/kg). The results are shown in Tables 28 and 29.

Table 28 Residue profile of [Pyrazole-4-^{14}C] isoflucypram in confined rotational immature Swiss chard (EnSa-16-945)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.031 mg eq/kg		TRR = 0.062 mg eq/kg		TRR = 0.056 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	97.2	0.03	95.3	0.06	94.4	0.053
Isoflucypram	6	0.002	0.5	<0.001	0.8	<0.001
BCS-CR60082	18.9	0.006	9	0.006	7.3	0.004
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala	-	-	1.8	0.001	2.3	0.001
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 1/2)	-	-	6.8/-	0.004/-	5.4/1.6	0.003/ 0.001
Isoflucypram-cyclopropyl-pyrazole-carboxamide-OH-lactic acid (isomer 1/2)	-	-	5.2/-	0.003/-	-/0.5	-/<0.001
Isoflucypram-desfluoro- cyclopropyl-pyrazole- carboxamide-Ala	-	-	1.2	0.001	1.3	0.001
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys	26.9	0.008	25.7	0.016	23	0.013

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.031 mg eq/kg		TRR = 0.062 mg eq/kg		TRR = 0.056 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH	21.2	0.007	6.3	0.004	8.4	0.005
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-desamino-Cys	-	-	-	-	0.6	<0.001
Losses	1.1	< 0.001	NA	NA	NA	NA
Total identified	73	0.023	56.4	0.035	51.3	0.029
Number of unknown peaks	3		9		19	
Largest unknown peak	11.1	0.003	15.2	0.009	7.5	0.004
Total characterized (by HPLC)	23.1	0.007	38.9	0.024	43.1	0.024
PES	2.8	0.001	4.7	0.003	5.6	0.003
Accountability	100	0.031	100	0.062	100	0.056

Table 29 Residue profile of [Phenyl-UL-^{14}C] isoflucypram in confined rotational immature Swiss chard (EnSa-17-0128)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.029 mg eq/kg		TRR = 0.016 mg eq/kg		TRR = 0.020 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	97.7	0.028	96.4	0.015	96.2	0.019
Isoflucypram	2.8	0.001	-	-	-	-
Total identified	2.8	0.001	NA	NA	NA	NA
Number of unknown peaks	13		8		17	
Largest unknown peak	25.4	0.007	21.2	0.003	16.2	0.003
Total characterized (by HPLC)	94.9	0.027	96.4	0.015	96.2	0.019
PES	2.3	0.001	3.6	0.001	3.8	0.001
Accountability	100	0.029	100	0.016	100	0.02

In rotational mature Swiss chard, a total of 92.4–96.0 percent TRR (0.025–0.058 mg eq/kg) [pyrazole] and 95.6–97.7 percent TRR (0.015–0.024 mg eq/kg) [phenyl] was extracted. Parent isoflucypram was a minor component in the pyrazole and phenyl studies representing ≤ 6.1 percent TRR (0.001 mg/kg). All remaining identified metabolites retained only the pyrazole moiety. Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys was a major metabolite accounting for 16.4–34.2 percent TRR (0.009–0.011 mg eq/kg). All other metabolites were minor components accounting for ≤ 0.006 mg eq/kg. Up to 22 unknown peaks were characterised based on chromatographic behaviour, each accounting for ≤ 28.3 percent TRR (≤ 0.007 mg eq/kg).

In the hydrolysed extract of phenyl-labelled mature Swiss chard at the 30-day PBI, isoflucypram was identified at 16.8 TRR (0.003 mg/kg), isoflucypram-propanol was identified at 17.4 percent TRR (0.004 mg eq/kg), and isoflucypram-carboxylic acid was identified at 40.5 percent TRR (0.008 mg eq/kg). The results are shown in Tables 30 and 31.

Table 30 Residue profile of [Pyrazole-4-^{14}C] isoflucypram in confined rotational mature Swiss chard (EnSa-16-945)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.026 mg eq/kg		TRR = 0.062 mg eq/kg		TRR = 0.052 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	96	0.025	94.2	0.058	92.4	0.048

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.026 mg eq/kg		TRR = 0.062 mg eq/kg		TRR = 0.052 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Isoflucypram	4.6	0.001	-	-	1.1	0.001
BCS-CR60082	19.4	0.005	3.8	0.002	4.0	0.002
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala	-	-	1.8	0.001	2.8	0.001
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 1/2)	-	-	12.2/2.5	0.008/0.002	9.5/-	0.005/-
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys	34.2	0.009	16.4	0.01	21.1	0.011
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH	22.9	0.006	6.2	0.004	3.7	0.002
Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala	-	-	1.9	0.001	1.6	0.001
Total identified	81.1	0.021	44.7	0.028	43.9	0.023
Number of unknown peaks	2		22		19	
Largest unknown peak	8.8	0.002	11.3	0.007	5.8	0.003
Total characterized	14.9	0.004	49.5	0.031	48.5	0.025
PES	4.0	0.001	5.8	0.004	7.6	0.004
Accountability	100	0.026	100	0.062	100	0.052

Table 31 Residue Profile of [Phenyl-UL-^{14}C] Isoflucypram in Confined Rotational Mature Swiss Chard (EnSa-17-0128)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.020 mg eq/kg		TRR = 0.016 mg eq/kg		TRR = 0.025 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	97.7	0.02	96.6	0.015	95.6	0.024
Isoflucypram	6.1	0.001	-	-	-	-
Total identified	6.1	0.001	NA	NA	NA	NA
Number of unknown peaks	9		9		14	
Largest unknown peak	21.8	0.004	28.3	0.004	15.3	0.004
Total characterized (by HPLC)	91.6	0.019	96.6	0.015	95.6	0.024
PES	2.3	<0.001	3.4	0.001	4.4	0.001
Accountability	100	0.02	100	0.016	100	0.025

In rotational wheat forage, a total of 91.0–92.9 percent TRR (0.038–0.071 mg eq/kg) [pyrazole] and 90.1–94.8 percent TRR (0.014–0.021 mg eq/kg) [phenyl] was extracted. Parent isoflucypram was a minor component in the pyrazole and phenyl studies representing ≤17 percent TRR (≤ 0.004 mg/kg). All remaining identified metabolites retained only the pyrazole moiety. Isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala was a major metabolite accounting for 15.4–25.6 percent TRR (0.009–0.020 mg eq/kg). All other metabolites were minor components accounting for ≤ 0.008 mg eq/kg. Up to 12 unknown peaks were characterised based on chromatographic behaviour, each accounting for ≤30.6 percent TRR (≤ 0.007 mg eq/kg). The results are shown in Tables 32 and 33.

Table 32 Residue profile of [Pyrazole-4-^{14}C] isoflucypram in confined rotational wheat forage (EnSa-16-945)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.041 mg eq/kg		TRR = 0.087 mg eq/kg		0.072 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	92.9	0.038	91.0	0.071	91.9	0.066
Isoflucypram	7.0	0.003	1.4	0.001	-	-
BCS-CR60082	9.2	0.004	7.3	0.006	4.3	0.003
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala	22.4	0.009	25.6	0.02	15.4	0.011
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 2)	-	-	4.2	0.003	3.2	0.002
Isoflucypram-cyclopropyl-pyrazole-carboxamide-OH-lactic acid (isomer 2)	5.8	0.002	10.2	0.008	6.7	0.005
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys	-	-	3.9	0.003	2.0	0.001
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH	9.3	0.004	8.7	0.007	6.3	0.005
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc	4.0	0.002	8.4	0.007	1.8	0.001
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc-MA	-	-	-	-	4.5	0.003
Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala	2.8	0.001	-	-	-	-
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-desamino-Cys	-	-	6.9	0.005	2.1	0.002
Total identified	60.6	0.025	76.6	0.059	46.2	0.033
Number of unknown peaks	3		4		12	
Largest unknown peak	9.2	0.004	4.9	0.004	6.1	0.004
Total characterized by HPLC	32.4	0.013	14.4	0.011	45.8	0.033
PES	7.1	0.003	9.0	0.007	8.1	0.006
Accountability	100	0.041	100	0.078	100	0.072

Table 33 Residue Profile of [Phenyl-UL-^{14}C] Isoflucypram in Confined Rotational Wheat Forage (EnSa-17-0128)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.023 mg eq/kg		TRR = 0.018 mg eq/kg		TRR = 0.015 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	94.8	0.021	93.5	0.017	90.1	0.014
Isoflucypram	17	0.004	12.2	0.002	5.2	0.001
Total identified	17	0.004	12.2	0.002	5.2	0.001
Number of unknown peaks	6		9		8	
Largest unknown peak	30.6	0.007	12.4	0.002	18.7	0.003
Total characterized by HPLC	77.9	0.018	81.3	0.015	84.9	0.013
PES	5.2	0.001	6.5	0.001	9.9	0.002
Accountability	100	0.023	100	0.018	100	0.015

In rotational wheat hay, a total of 83.6–85.7 percent TRR (0.098–0.184 mg eq/kg) [pyrazole] and 82.0–86.3 percent TRR (0.030–0.053 mg eq/kg) [phenyl] was released in the conventional extract. Parent isoflucypram was not recovered in the pyrazole study and a minor component of the residue (≤1.1 percent TRR; ≤ 0.001 mg/kg) in the phenyl study. All remaining identified metabolites retained only the pyrazole moiety. Major metabolites included isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala accounting for 7.9 percent–13.9 percent TRR (0.014–0.031 mg eq/kg), isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 2) accounting for 3.8–5.8 percent TRR (0.007–0.013 mg eq/kg), isoflucypram-cyclopropyl-pyrazole-carboxamide-OH-lactic acid (isomer 2) accounting for 3.2–9.0 percent TRR (0.007–0.016 mg eq/kg), isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys accounting for 5.7–7.0 percent TRR (0.012–0.013 mg eq/kg), isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH accounting for 1.8–6.5 percent TRR (0.003–0.014 mg eq/kg), and isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala accounting for 2.2–4.8 percent TRR (0.004–0.011 mg eq/kg). All other metabolites were minor components accounting for ≤ 0.009 mg eq/kg. Up to 17 unknown peaks were characterised based on chromatographic behaviour, each accounting for ≤17.3 percent TRR (≤ 0.013 mg eq/kg).

An additional 10.5–12.5 percent TRR (0.012–0.027 mg eq/kg) [pyrazole] and 5.5–6.7 percent TRR (0.002–0.003 mg eq/kg) [phenyl] was released in the exhaustive extract. The resuls are shown in Tables 34 and 35.

Table 34 Residue profile of [Pyrazole-4-^{14}C] isoflucypram in confined rotational wheat hay (EnSa-16-945)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.114 mg eq/kg		TRR = 0.220 mg eq/kg		TRR = 0.187 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	85.7	0.098	83.6	0.184	85.4	0.16
Isoflucypram	-	-	-	-	-	-
BCS-CR60082	2.2	0.002	1.1	0.002	2.3	0.004
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala	12.2	0.014	13.9	0.031	7.9	0.015
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 1/2)	7.7/-	0.009/-	3.0/5.8	0.007/0.013	2.9/3.8	0.006/0.007
Isoflucypram-cyclopropyl-pyrazole-carboxamide-OH-lactic acid (isomer 1/2)	-/9.9	-/0.01	-/3.2	-/0.007	3.3/8.4	0.006/0.016
Isoflucypram-cyclopropyl-pyrazole-carboxamide- acetic acid	-	-	2.0	0.004	1.6	0.003
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys	-	-	5.7	0.012	7.0	0.013
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH	5.2	0.006	6.5	0.014	1.8	0.003
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc	2.9	0.003	2.3	0.005	1.9	0.003
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc-MA	4.2	0.005	3.2	0.007	3.6	0.007
Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala	-	-	4.8	0.011	2.2	0.004
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-	1.6	0.002	2.5	0.005	1.1	0.002

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.114 mg eq/kg		TRR = 0.220 mg eq/kg		TRR = 0.187 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
carboxamide-desamino-Cys						
Total identified	44.9	0.051	54.0	0.119	47.7	0.089
Number of unknown peaks	11		13		17	
Largest unknown peak	7.0	0.008	5.8	0.013	3.7	0.007
Exhaustive extract	10.5	0.012	12.0	0.027	12.5	0.023
Microwave extract	7.0	0.008	6.7	0.015	7.9	0.015
Microwave extract – SPE	7.0	0.008	6.7	0.015	7.7	0.014
Microwave extract – SPE losses	<0.10	<0.001	<0.010	<0.001	0.20	<0.001
Dioxan microwave extract	3.4	0.004	5.4	0.012	4.6	0.009
Total extracted	96.1	0.11	95.7	0.211	97.9	0.183
Total characterised (by HPLC)	40.7	0.046	29.6	0.065	37.7	0.071
PES	3.9	0.004	4.3	0.010	2.1	0.004
Accountability	100	0.114	100	0.22	100	0.187

Table 35 Residue profile of [Phenyl-UL-^{14}C] isoflucypram in confined rotational wheat hay (EnSa-17-0128)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.039 mg eq/kg		TRR = 0.062 mg eq/kg		TRR = 0.036 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	86.1	0.033	86.3	0.053	82	0.03
Isoflucypram	1.1	<0.001	-	-	-	-
Total identified	1.1	<0.001	NA	NA	NA	NA
Number of unknown peaks	14		12		12	
Largest unknown peak	14.6	0.006	13.4	0.008	17.3	0.006
Exhaustive extract	6.7	0.003	5.5	0.003	6.4	0.002
Microwave extract	6.7	0.003	5.5	0.003	6.4	0.002
Microwave extract - SPE	6.7	0.003	5.5	0.003	5.5	0.002
Extract - SPE osses	NQ	NQ	<0.1	<0.001	0.8	<0.001
Total extracted	92.8	0.036	91.8	0.057	88.3	0.032
Total characterized (by HPLC)	85	0.033	86.3	0.053	82	0.03
PES	7.2	0.003	8.2	0.005	11.7	0.004
Accountability	100	0.039	100	0.062	100	0.036

In rotational wheat straw, a total of 78.5–83.7 percent TRR (0.103–0.284 mg eq/kg) [pyrazole] and 78.4–82.9 percent TRR (0.040–0.058 mg eq/kg) [phenyl] was released in the conventional extract. Parent isoflucypram was not recovered in either study. All remaining identified metabolites retained only the pyrazole moiety. Major metabolites included BCS-CR60082 (Isoflucypram-N-methyl-cyclopropyl-pyrazole-carboxamide) accounting for 2.8–7.0 percent TRR (0.007–0.020 mg eq/kg), isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala accounting for 2.6 percent–9.1 percent TRR (0.003–0.022 mg eq/kg), isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 1) accounting for 2.3–3.6 percent TRR (0.003–0.011 mg eq/kg), isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 2) accounting for 3.3–3.5 percent TRR (0.005–0.011 mg eq/kg), isoflucypram-cyclopropyl-pyrazole-carboxamide-OH-lactic acid (isomer 2) accounting for 5.9–11.9 percent TRR (0.015–0.029 mg eq/kg), isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys accounting for 2.6–7.6 percent TRR (0.003–0.026 mg eq/kg), isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH accounting for 2.6–6.6 percent TRR (0.009–0.014 mg eq/kg), and isoflucypram-desfuloro-N-methyl-cyclopropyl-pyrazole-carboamide-mercapto-Glyc-MA accounting for 2.9–5.5 percent TRR (0.007–0.010 mg eq/kg). All other metabolites were minor components accounting for

≤ 0.009 mg eq/kg. Up to 18 unknown peaks were characterised based on chromatographic behaviour, each accounting for ≤16 percent TRR (≤ 0.021 mg eq/kg).

An additional 12.1–15.1 percent TRR (0.020–0.041 mg eq/kg) [pyrazole] and 7.6–10.8 percent TRR (0.005–0.006 mg eq/kg) [phenyl] was released in the exhaustive extract.

In the hydrolysed extract of phenyl-labelled wheat straw at the 30-day PBI, isoflucypram-propanol was identified at 5.5 percent TRR (0.003 mg eq/kg) and isoflucypram-carboxylic acid was identified at 10.8 percent TRR (0.006 mg eq/kg). The results are shown in Tables 36 and 37.

Table 36 Residue profile of [Pyrazole-4-^{14}C] isoflucypram in confined rotational wheat straw (EnSa-16-945)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.131 mg eq/kg		TRR = 0.247 mg eq/kg		TRR=0.340 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	78.5	0.103	81.2	0.201	83.7	0.284
Isoflucypram	-	-	-	-	-	-
BCS-CR60082	7.0	0.009	2.8	0.007	5.8	0.020
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala	2.6	0.003	9.1	0.022	2.8	0.010
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 1/2)	2.3/3.5	0.003/ 0.005	3.6/ 3.4	0.009/ 0.008	3.2/ 3.3	0.011/ 0.011
Isoflucypram-cyclopropyl-pyrazole-carboxamide-OH-lactic acid (isomer 1/2)	-/11.9	-/0.016	-/5.9	-/0.015	2.2/8.6	0.008/ 0.029
Isoflucypram-cyclopropyl-pyrazole-carboxamide-acetic acid	-	-	-	-	1.4	0.005
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys	2.6	0.003	5.0	0.012	7.6	0.026
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH	6.6	0.009	5.5	0.014	2.6	0.009
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc	3.7	0.005	3.7	0.009	2.0	0.007
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc-MA	5.5	0.007	3.8	0.009	2.9	0.010
Isoflucypram-desfluoro- cyclopropyl-pyrazole-carboxamide-Ala	-	-	-	-	1.1	0.004
Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-desamino-Cys	-	-	1.1	0.003	1.6	0.005
Total identified	45.9	0.060	44.0	0.109	45.1	0.153
Number of unknown peaks	9		14		18	
Largest unknown peak	6.7	0.009	4.7	0.012	6.2	0.021
Conventional extract – losses	0.60	0.001	0.60	0.001	0.30	0.001
Exhaustive extract	15.1	0.020	14.3	0.035	12.1	0.041
Microwave extract	12.2	0.016	8.1	0.020	6.7	0.023
Microwave extract – SPE	12.2	0.016	8.1	0.020	6.2	0.021
Microwave extract – SPE losses	0.20	<0.001	0.10	<0.001	0.40	0.002
Dioxan microwave extract	2.9	0.004	6.2	0.015	5.5	0.019
Total extracted	93.6	0.123	95.5	0.236	95.8	0.326
Total characterised (by HPLC)	32.1	0.042	36.7	0.091	38.3	0.13
Total not analysed	15.7	0.021	14.9	0.037	12.4	0.042
PES	6.4	0.008	4.5	0.011	4.2	0.014
Accountability	100	0.131	100	0.247	100	0.34

Table 37 Residue profile of [Phenyl-UL-^{14}C] isoflucypram in confined rotational wheat straw (EnSa-17-0128)

	30-Day PBI		140-Day PBI		287-Day PBI	
	TRR = 0.051 mg eq/kg		TRR = 0.070 mg eq/kg		TRR = 0.055 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	78.4	0.04	82.9	0.058	79.9	0.044
Isoflucypram	-	-	-	-	-	-
Total identified	-	-	-	-	-	-
Number of unknown peaks	12		13		11	
Largest unknown peak	13.6	0.007	12.7	0.009	16	0.009
Conventional extract - losses	1.3	0.001	NA	NA	NA	NA
Exhaustive extract	10.1	0.005	7.6	0.005	10.8	0.006
Microwave extract	10.1	0.005	7.6	0.005	10.8	0.006
Microwave extract - SPE	10.1	0.005	7.6	0.005	10.1	0.006
Microwave extract - SPE losses	1.3	0.001	<0.1	<0.001	0.7	<0.001
Total extracted	88.5	0.046	90.4	0.063	90.7	0.05
Total characterized (by HPLC)	77.2	0.04	82.9	0.058	79.9	0.044
Total not analysed	11.4	0.006	7.6	0.005	10.8	0.006
PES	11.5	0.006	9.6	0.007	9.3	0.005
Accountability	100	0.051	100	0.07	100	0.055

In pyrazole labelled rotational wheat grain, a total of 52.9–53.7 percent TRR (0.006–0.009 mg eq/kg) was released in the conventional extract. Parent isoflucypram was not recovered. All identified metabolites retained only the pyrazole moiety. All metabolites were minor components accounting for ≤ 0.002 mg eq/kg. Three unknown peaks were characterised based on chromatographic behaviour, each accounting for ≤ 22.4 percent TRR (≤ 0.004 mg eq/kg). An additional 27.6–31.5 percent TRR (0.003–0.005 mg eq/kg) was released in the exhaustive extract (Table 38).

Table 38 Residue profile of [Pyrazole-4-^{14}C] Isoflucypram in confined rotational wheat grain (EnSa-16-945)

	140-Day PBI		287-Day PBI	
	TRR = 0.011 mg eq/kg		TRR = 0.016 mg eq/kg	
Fraction/Compound	% TRR	mg eq/kg	% TRR	mg eq/kg
Conventional extract	52.9	0.006	53.7	0.009
Isoflucypram	-	-	-	-
Isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala	7.7	0.001	-	-
Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala	13.4	0.002	-	-
Total identified	21.2	0.002	NA	NA
Number of unknown peaks	3		3	
Largest unknown peak	16.2	0.002	22.4	0.004
Exhaustive extract	27.6	0.003	31.5	0.005
Microwave extract	27.6	0.003	31.5	0.005
Microwave extract - SPE	27.6	0.003	24.4	0.004
Microwave extract - SPE Losses	<0.1	<0.001	7.1	0.001
Total extracted	80.4	0.009	85.2	0.014
Total characterized (by HPLC)	31.7	0.004	53.7	0.009
PES	19.6	0.002	14.8	0.002
Accountability	100	0.011	100	0.016

A proposed metabolic pathway of isoflucypram in confined rotational crops is shown in Figure 6

Magnitude of the residue in rotational crops and soil

Report No. 15-2502.

The Meeting received four independent rotational crop trials, one each from Germany, the Netherlands, France, and Italy in 2015 following one broadcast application of an EC formulation containing 50 g ai/L (Freitag, T., *et al.,* 2017). The application rate was 0.18 kg ai/ha. Rotational crops of barley, carrot or turnips, and lettuce were planted in treated plots at PBIs of 20–34 days, 100–201 days, and 299–370 days and analysed for isoflucypram and BCS-CR60082. Additionally, soil samples (from 0–30 cm) were taken at from each plot at each PBI and analysed for isoflucypram and isoflucypram-carboxylic acid. No adjuvants were added to the solution at any trial. The soil characteristics in each trial are shown in Table 39.

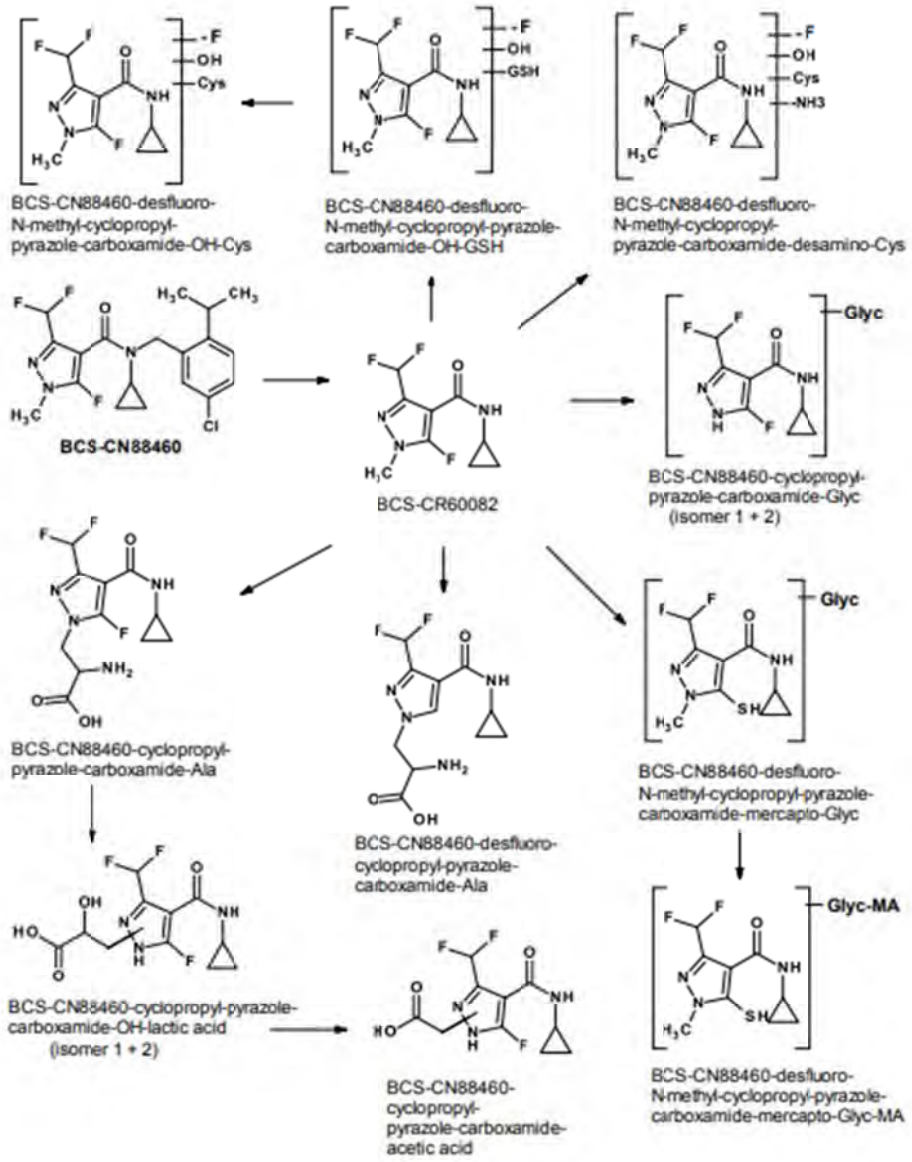


Figure 6 Metabolic pathway in pyrazole-labelled isoflucypram (BCS-CN88460) in confined rotational crops

Table 39 Soil Characteristics (0–30 cm) in the field rotational crops conducted in Europe in 2015

Trial Number (Country)	Classification (USDA)	Clay (%)	Silt (%)	Sand (%)	CEC (meq/100 g)	$CaCO_3$ Content (%)	pH in KCl	Organic Content (%)	WHCmax (g/100g)
15-2502-01 (Germany)	Silt Loam	16.4	76.4	7.2	15.3	<0.1	6.47	1.74	48.3
15-2502-02 (Netherlands)	Loam	15.4	47.1	37.5	31.3	2.1	7.4	2.5	58.1
15-2502-03 (France)	Silt Loam	13.3	65.2	21.5	20.0	41.0	7.97	0.77	39.7
15-2502-04 (Italy)	Silt Loam	16.0	53.1	30.9	24.3	13.4	7.60	1.14	46.9

Irrigation was used at Trials 15-2502-02, 15-2502-03, 15-2502-04. A minimum of 2.02 and 2.08 kg of carrot/turnip leaf and carrot/turnip root were collected from treated plots, respectively; 1.29 kg of lettuce was collected from treated plots; 1.58, 1.07, and 0.600 kg of barley green material, grain, and straw were collected from treated plots, respectively; and 2.70 kg of soil sample was collected from treated plots (however, soil weights were not reported in Trial 15-2502-04). Treated and control samples were frozen within 24 hours after sampling and during shipment to the Laboratory for Sampling (Bayer AG, Monheim am Rhein, Germany). Samples were shredded and homogenized with dry ice and shipped frozen to the analytical laboratory (Bayer S.A.S., Lyon, France).

The maximum storage duration between harvest and extraction for analysis was 299 days for carrot/turnip tops, 300 days for carrot/turnip roots, 341 days for lettuce, 259 days for barley green material, 96 days for barley grain, 103 days for barley straw, and 320 days for soil.

Residues levels of isoflucypram and BCS-CR60082 (isoflucypram-N-methyl-cyclopropyl-pyrazole-carboxamide) were determined in plants by Method 01475. Residue levels of isoflucypram and isoflucypram-carboxylic acid were determined in soil according to Method 01432. There were no residues in control samples. Therefore, residues in treated samples were not corrected for residues in controls.

Average residues of BCS-CR60082 were below the limit of quantitation (LOQ; <0.01 mg eq/kg) in all samples. Average residues of isoflucypram were below the LOQ (<0.01 mg/kg) in all samples except carrot tops at one trial at the 106-day PBI (average residue of 0.066 mg/kg). According to the study report, this plot was close to the barley plot which received an application five days prior to harvest of carrot tops. There was no buffer zone or structure preventing spray drift and a slight wind in the direction of the carrot field. Therefore, the Meeting considers this residue point an outlier and potentially resulting from drift rather than uptake in the field. The results of the residues in the rotational cropos are shown in Table 40 and the residues in the soil in Table 41

Table 40 Residues in rotational crops following application of isoflucypram to bare soil at 0.18 kg ai/ha

Trial No. (Country)	PBI	Crop	BBCH	DAA	Isoflucypram (mg/kg)	BCS-CR60082 (mg eq/kg) [1]
15-2502-01 (Germany)	28	Carrot tops	47, 49	125, 139	<0.01, <0.01	<0.01, <0.01
	28	Carrot roots	47, 49	125, 139	<0.01, <0.01	<0.01, <0.01
	28	Lettuce	46, 49	76, 90	<0.01, <0.01	<0.01, <0.01
	34	Barley green material	30, 75	222, 279	<0.01, <0.01	<0.01, <0.01
	34	Barley grain	89	316	<0.01	<0.01
	34	Barley straw	89	316	<0.01	<0.01
	106	Carrot tops	47, 49	172, 186	0.075[2], 0.057[2]	<0.01[2], <0.01[2]
	106	Carrot roots	47, 49	172, 186	<0.01, <0.01	<0.01, <0.01

Trial No. (Country)	PBI	Crop	BBCH	DAA	Isoflucypram (mg/kg)	BCS-CR60082 (mg eq/kg) [1]
	144	Lettuce	47, 49	172, 186	<0.01, <0.01	<0.01, <0.01
	201	Barley green material	30, 75	389, 446	<0.01, <0.01	<0.01, <0.01
	201	Barley grain	89	483	<0.01	<0.01
	201	Barley straw	89	483	<0.01	<0.01
	368	Carrot tops	47, 49	491, 505	<0.01, <0.01	<0.01, <0.01
	368	Carrot roots	47, 49	491, 505	<0.01, <0.01	<0.01, <0.01
	370	Lettuce	45, 49	424, 438	<0.01, <0.01	<0.01, <0.01
	369	Barley green material	29, 75	425, 466	<0.01, <0.01	<0.01, <0.01
	369	Barley grain	89	515	<0.01	<0.01
	369	Barley straw	89	515	<0.01	<0.01
15-2502-02 (Netherlands)	22	Carrot tops	48, 49	106, 120	<0.01, <0.01	<0.01, <0.01
	22	Carrot roots	48, 49	106, 120	<0.01, <0.01	<0.01, <0.01
	21	Lettuce	45, 49	65, 79	<0.01, <0.01	<0.01, <0.01
	22	Barely green material	30, 75	198, 280	<0.01, <0.01	<0.01, <0.01
	22	Barley grain	89	295	<0.01	<0.01
	22	Barley straw	89	295	<0.01	<0.01
	100	Carrot tops	47, 49	196, 210	<0.01, <0.01	<0.01, <0.01
	100	Carrot roots	47, 49	196, 210	<0.01, <0.01	<0.01, <0.01
	131	Lettuce	46, 49	170, 184	<0.01, <0.01	<0.01, <0.01
	181	Barely green material	30, 75	357, 439	<0.01, <0.01	<0.01, <0.01
	181	Barley grain	89	454	<0.01	<0.01
	181	Barley straw	89	454	<0.01	<0.01
	365	Carrot tops	48, 49	471, 485	<0.01, <0.01	<0.01, <0.01
	365	Carrot roots	48, 49	471, 485	<0.01, <0.01	<0.01, <0.01
	365	Lettuce	45, 49	404, 418	<0.01, <0.01	<0.01, <0.01
	365	Barley green material	30, 75	426, 464	<0.01, <0.01	<0.01, <0.01
	365	Barley grain	89	485	<0.01	<0.01
	365	Barley straw	89	485	<0.01	<0.01
15-2502-03 (France)	20	Turnip tops	48, 49	83, 97	<0.01, <0.01	<0.01, <0.01
	20	Turnip roots	48, 49	83, 97	<0.01, <0.01	<0.01, <0.01
	26	Lettuce	47, 49	55, 69	<0.01, <0.01	<0.01, <0.01
	27	Barley green material	30, 75	153, 234	<0.01, <0.01	<0.01, <0.01
	27	Barley grain	89	271	<0.01	<0.01
	27	Barley straw	89	271	<0.01	<0.01
	139	Turnip tops	48, 49	202, 216	<0.01, <0.01	<0.01, <0.01
	139	Turnip roots	48, 49	202, 216	<0.01, <0.01	<0.01, <0.01
	139	Lettuce	48, 49	175, 189	<0.01, <0.01	<0.01, <0.01
	188	Barley green material	30, 75	314, 395	<0.01, <0.01	<0.01, <0.01
	188	Barley grain	89	432	<0.01	<0.01
	188	Barley straw	89	432	<0.01	<0.01
	350	Turnip tops	48, 49	417, 430	<0.01, <0.01	<0.01, <0.01
	350	Turnip roots	48, 49	417, 430	<0.01, <0.01	<0.01, <0.01
	348	Lettuce	48, 49	385, 398	<0.01, <0.01	<0.01, <0.01
	299	Barley green material	30, 75	378, 417	<0.01, <0.01	<0.01, <0.01
	299	Barley grain	89	446	<0.01	<0.01
	299	Barley straw	89	446	<0.01	<0.01

Trial No. (Country)	PBI	Crop	BBCH	DAA	Isoflucypram (mg/kg)	BCS-CR60082 (mg eq/kg) [1]
15-2502-04	28	Carrot tops	46, 49	93, 107	<0.01, <0.01	<0.01, <0.01
	28	Carrot roots	46, 49	93, 107	<0.01, <0.01	<0.01, <0.01
	28	Lettuce	46, 49	55, 69	<0.01, <0.01	<0.01, <0.01
	34	Barely green material	29, 75	164, 232	<0.01, <0.01	<0.01, <0.01
	34	Barley grain	89	275	<0.01	<0.01
	34	Barley straw	89	275	<0.01	<0.01
	125	Carrot tops	46, 49	195, 209	<0.01, <0.01	<0.01, <0.01
	125	Carrot roots	46, 49	195, 209	<0.01, <0.01	<0.01, <0.01
	125	Lettuce	45, 49	146, 160	<0.01, <0.01	<0.01, <0.01
	195	Barley green material	30, 75	325, 393	<0.01, <0.01	<0.01, <0.01
	195	Barley grain	89	436	<0.01	<0.01
	195	Barley straw	89	436	<0.01	<0.01
	353	Carrot tops	45, 49	419, 433	<0.01, <0.01	<0.01, <0.01
	353	Carrot roots	45, 49	419, 433	<0.01, <0.01	<0.01, <0.01
	353	Lettuce	45, 49	392, 406	<0.01, <0.01	<0.01, <0.01
	345	Barley green material	29, 75	378, 420	<0.01, <0.01	<0.01, <0.01
	345	Barley grain	89	443	<0.01	<0.01
	345	Barley straw	89	443	<0.01	<0.01

Notes:
[1] Expressed in isoflucypram equivalent concentrations.
[2] Mean of three replicates.

Table 41 Residue summary in soil following isoflucypram application to bare soil

Trial No. (Country)	DAT	Sample Material (Plot Number)[1]	Isoflucypram, mg/kg (mean)	Isoflucypram-carboxylic acid (mg eq/kg)[2]
15-2502-01 (Germany)	28	Soil (1)	0.023, 0.026 (0.025)	<0.001, <0.001
	28	Soil (2)	0.024, 0.020 (0.022)	<0.001, <0.001
	32	Soil (3)	0.009, 0.014 (0.012)	<0.001, <0.001
	106	Soil (1)	0.020, 0.020 (0.020)	<0.001, <0.001
	144	Soil (2)	0.018, 0.024 (0.021)	<0.001, <0.001
	201	Soil (3)	0.021, 0.019 (0.020)	<0.001, <0.001
	369	Soil (1)	0.025, 0.020 (0.023)	<0.001, <0.001
	369	Soil (2)	0.028, 0.017 (0.023)	<0.001, <0.001
	369	Soil (3)	0.002, 0.020 (0.011)	<0.001, <0.001
15-2502-02 (Netherlands)	22	Soil (1)	0.030, 0.029 (0.030)	<0.001, <0.001
	22	Soil (2)	0.034, 0.030 (0.032)	<0.001, <0.001
	23	Soil (3)	0.034, 0.035 (0.035)	<0.001, <0.001
	100	Soil (1)	0.027, 0.031 (0.029)	<0.001, <0.001
	131	Soil (2)	0.030, 0.022 (0.026)	<0.001, <0.001
	182	Soil (3)	0.020, 0.036 (0.028)	<0.001, <0.001
	366	Soil (1)	0.028, 0.031 (0.030)	<0.001, 0.001
	365	Soil (2)	0.017, 0.019 (0.018)	<0.001, <0.001
	366	Soil (3)	0.017, 0.021 (0.019)	<0.001, <0.001
15-2502-03 (France)	20	Soil (1)	0.035, 0.031 (0.033)	<0.001, <0.001
	26	Soil (2)	0.022, 0.028 (0.025)	<0.001, <0.001
	27	Soil (3)	0.022, 0.023 (0.023)	<0.001, <0.001
	139	Soil (1)	0.018, 0.019 (0.019)	<0.001, <0.001
	139	Soil (2)	0.018, 0.017 (0.018)	<0.001, <0.001

Trial No. (Country)	DAT	Sample Material (Plot Number)[1]	Isoflucypram, mg/kg (mean)	Isoflucypram-carboxylic acid (mg eq/kg)[2]
	188	Soil (3)	0.012, 0.014 (0.013)	0.001, 0.001
	350	Soil (1)	0.010, 0.011 (0.011)	<0.001, <0.001
	348	Soil (2)	0.012, 0.014 (0.013)	0.001, 0.001
	299	Soil (3)	0.014, 0.013 (0.014)	<0.001, <0.001
15-2502-04 (Italy)	28	Soil (1)	0.040, 0.057 (0.049)	<0.001, <0.001
	28	Soil (2)	0.029, 0.022 (0.026)	<0.001, <0.001
	34	Soil (3)	0.023, 0.027 (0.025)	<0.001, <0.001
	125	Soil (1)	0.020, 0.021 (0.021)	0.001, 0.001
	125	Soil (2)	0.021[3], 0.019 (0.020)	0.001[3], 0.001
	195	Soil (3)	0.020, 0.017 (0.019)	0.002, 0.001
	353	Soil (1)	0.016, 0.015 (0.016)	<0.001, <0.001
	353	Soil (2)	0.017, 0.022 (0.020)	<0.001, 0.001
	345	Soil (3)	0.015, 0.020 (0.018)	<0.001, <0.001

Notes:
[1] Plot 1 corresponds with rotational carrot/turnip trials. Plot 2 corresponds with rotational lettuce trials. Plot 3 corresponds with rotational barley trials.
[2] Expressed in isoflucypram-carboxylic acid.
[3] Average of four analyses.

Environmental degradation

The Meeting received studies investigating the aerobic metabolism/degradation in soil, hydrolytic degradation, photolytic transformation, and theoretical photodegradation in water and environmental half-life.

Aerobic Metabolism/Degradation in Soil

Report No. EnSa-13-1043.

The route and rate of degradation of [pyrazole-4-^{14}C] isoflucypram was studied in four soils (Hellpointner, E., *et. al.*; 2014). Samples were maintained under aerobic conditions in the dark in the laboratory for 120 days at 20.0 °C. Samples were maintained at a moisture content of 53.1 percent of the maximum water holding capacity. The application rate of was 75 g ai/ha, corresponding to 200 µg ai/kg soil dry weight. Soil was collected from the top 20 cm and sieved to a particle size of ≤ 2 mm. The four soils represent different geographical origin and properties (Table 42).

Table 42 Soil properties for aerobic metabolism study for isoflucypram (EnSa-13-1043)

Designation	Source	Texture (USDA)	pH[1]	% organic content
Hanscheider Hof	Burscheid, Germany	Loam	5.7	2.9
Laacher Hof AXXa	Monheim, Germany	Loamy sand	6.3	2.0
Hoefchen Am Hohenseh	Burscheid, Germany	Silt loam	6.6	1.9
Dollendorf II	Blankenheim, Germany	Loam	7.4	5.2

Notes:
[1] pH values were derived from aqueous 0.01M $CaCl_2$ suspensions.

Tests were performed in incubation vessels with 100 g soil (dry weight equivalents) equipped with oxygen-permeable polyurethane (PU) and soda lime traps for volatile organic compounds (VOCs) and CO_2, respectively.

Water loss from evaporation was determined by weighing test systems at the start of the study and after 37, 76, and 107 days of incubation. No significant losses of moisture were observed throughout the study. Determinations of microbial biomass were performed on day after treatment (DAT)-0, DAT50 and DAT120 and demonstrated that the used soils were microbially viable.

Ten sampling intervals were distributed over the incubation period of 120 days. Duplicate samples were processed and analysed on DAT 0, 2, 6, 15, 28, 50, 62, 84, 104, and 120. Prior to processing of soil, possible volatiles were purged into the trap attachment and the trap attachment was removed. CO_2 was released from the soda lime with HCl and re-absorbed to a scintillation cocktail for LSC. VOCs were released with ethyl acetate. No chromatographic analyses were performed for VOCs due to low radioactivity (≤ 0.1 percent applied radioactivity [AR]).

The entire soil of each test vessel was extracted three times under ambient conditions with ACN/water (1:1) followed by one extraction with ACN/water (1:1) with microwave assistance at 70 °C, followed by one extraction with MeOH/water (1:1) with microwave assistance at 50 °C. The radioactivity content was determined by LSC. Non-extracted residues (NER) were determined by combustion/LSC.

The trap attachments containing soda lime and PU foam were stored for a maximum of six days. The first analysis of soil extracts with the primary chromatographic method was usually done within one day after sampling. After analysis, soil extracts were stored at <-18 °C in the dark. Soil extracts of DAT120 samples were re-analysed with the confirmatory method with a maximum sample storage period was four weeks. The exhaustive extracted soils were stored at ambient temperature in the laboratory for a maximum period of two weeks.

Mean material balances were ranged from 97.7–100.3 percent AR. The complete material balances found at all sampling intervals for all soils demonstrated that there was no significant loss of radioactivity from the test systems or during sample processing. The resuts of the degradation study in different soils are shown in Table 43.

Table 43 Degradation of isoflucypram in soil under aerobic conditions, in percent of applied radioactivity (mean ± SD)

Compound	DAT0	DAT2	DAT6	DAT15	DAT28	DAT50	DAT62	DAT84	DAT104	DAT120
Hanscheider Hof										
Isoflucypram	98.2 ± 0.4	97.3 ± 0.9	97.3 ± 1.7	95.8 ± 0.4	93.7 ± 0.8	92.3 ± 0.7	90.0 ± 0.2	86.3 ± 0.1	82.2 ± 0.6	82.6 ± 1.3
ROI 1[1]	ND	ND	ND	0.8 ± 0.2	1.4 ± 0.2	1.6 ± 0.0	1.7 ± 0.3	2.2 ± 0.1	2.6 ± 0.2	2.4 ± 0.1
ROI 2[1]	ND	ND	ND	0.7 ± 0.1	0.9 ± 0.1	1.3 ± 0.1	1.6 ± 0.1	1.4 ± 0.2	1.1 ± 0.2	1.8 ± 0.1
ROI 3[1]	ND	ND	ND	ND	ND	ND	0.7 ± 0.0	0.7 ± 0.0	0.8 ± 0.0	0.8 ± 0.1
ROI 4[1]	ND	ND	ND	ND	ND	ND	0.7 ± 0.0	ND	0.6 ± 0.1	ND
ROI 7[1]	ND	ND	ND	ND	ND	ND	ND	0.7 ± 0.0	0.8 ± 0.0	0.6 ± 0.1
ROI 8[1]	ND	ND	ND	ND	ND	ND	ND	ND	0.6 ± 0.1	ND
Sum of unid./diff. residues	0.9 ± 0.1	ND	ND	0.9 ± 0.1	2.2 ± 0.5	2.2 ± 0.4	3.2 ± 0.4	4.0 ± 0.3	6.4 ± 0.6	5.8 ± 0.4
Total extracted residues	99.0 ± 0.4	97.6 ± 1.2	97.6 ± 1.4	97.5 ± 0.1	97.3 ± 0.2	96.3 ± 0.1	95.6 ± 0.5	93.2 ± 0.3	92.0 ± 0.2	91.8 ± 0.5
CO_2	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	0.2 ± 0.0	0.6 ± 0.0	0.8 ± 0.0	1.2 ± 0.0	1.6 ± 0.1	1.8 ± 0.0
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	1.5 ± 0.1	2.4 ± 0.1	2.0 ± 0.2	2.7 ± 0.0	2.9 ± 0.1	4.2 ± 0.0	4.9 ± 0.2	5.4 ± 0.2	6.3 ± 0.2	5.8 ± 0.1
Total Recovery	100.6 ± 0.4	100.0 ± 1.1	99.6 ± 1.2	100.3 ± 0.1	100.5 ± 0.1	101.1 ± 0.1	101.3 ± 0.4	99.7 ± 0.1	99.9 ± 0.6	99.3 ± 0.6
Compound	DAT0	DAT2	DAT6	DAT15	DAT28	DAT50	DAT62	DAT84	DAT104	DAT120
Laacher Hof AXXa										
Isoflucypram	99.8 ± 0.0.	96.9 ± 0.0	95.6 ± 1.1	93.9 ± 0.2	90.4 ± 1.4	84.8 ± 0.5	81.0 ± 0.4	76.7 ± 1.4	72.5 ± 1.8	70.1 ± 0.3
ROI 1[1]	ND	ND	ND	1.3 ± 0.0	1.9 ± 0.2	3.2 ± 0.1	3.3 ± 0.0	4.0 ± 0.3	4.9 ± 0.1	5.4 ± 0.5

Compound	DAT0	DAT2	DAT6	DAT15	DAT28	DAT50	DAT62	DAT84	DAT104	DAT120
ROI 2[1]	ND	ND	ND	0.9 ± 0.0	1.5 ± 0.1	2.3 ± 0.1	2.6 ± 0.3	2.5 ± 0.4	2.5 ± 0.4	3.4 ± 0.1
ROI 3[1]	ND	ND	ND	ND	0.7 ± 0.1	1.5 ± 0.2	1.9 ± 0.4	2.1 ± 0.2	2.5 ± 0.3	2.8 ± 0.3
ROI 4[1]	ND	ND	ND	ND	ND	0.4 ± 0.0	ND	ND	ND	0.6 ± 0.0
ROI 5[1]	ND	ND	ND	ND	ND	ND	ND	ND	1.1 ± 0.1	1.0 ± 0.1
ROI 6[1]	ND	ND	ND	ND	ND	ND	ND	0.5 ± 0.0	0.6 ± 0.0	0.6 ± 0.0
ROI 7[1]	ND	ND	ND	ND	ND	ND	0.8 ± 0.0	1.1 ± 0.1	1.1 ± 0.1	1.3 ± 0.1
ROI 9[1]	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.7 ± 0.0
Sum of unid./diff. residues	0.6 ± 0.1	0.9 ± 0.1	0.4 ± 0.0	0.9 ± 0.0	2.4 ± 1.0	4.3 ± 0.7	4.3 ± 0.1	6.1 ± 1.1	6.7 ± 1.2	8.9 ± 0.3
Total extracted residues	100.4 ± 0.1	97.8 ± 0.1	96.1 ± 0.7	96.1 ± 0.1	95.4 ± 0.7	93.8 ± 0.5	90.9 ± 0.4	89.3 ± 0.5	87.7 ± 0.2	88.2 ± 0.9
CO_2	NA	<0.1 ± 0.0	<0.1 ± 0.0	0.1 ± 0.0	0.4 ± 0.0	0.9 ± 0.0	1.2 ± 0.1	1.7 ± 0.0	2.2 ± 0.0	2.5 ± 0.1
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	0.8 ± 0.1	1.2 ± 0.0	1.3 ± 0.0	1.8 ± 0.0	2.3 ± 0.0	3.4 ± 0.1	4.1 ± 0.4	5.2 ± 0.3	5.4 ± 0.1	5.8 ± 0.1
Total Recovery	101.2 ± 0.2	99.0 ± 0.1	97.4 ± 0.7	98.0 ± 0.1	98.1 ± 0.7	98.1 ± 0.4	96.2 ± 0.1	96.2 ± 0.8	95.2 ± 0.2	96.5 ± 0.7
Compound	DAT0	DAT2	DAT6	DAT15	DAT28	DAT50	DAT62	DAT84	DAT104	DAT120
Hoefchen Am Hohenseh										
Isoflucypram	98.2 ± 0.1	94.8 ± 0.5	96.4 ± 0.9	93.9 ± 0.2	91.1 ± 1.3	89.5 ± 0.4	85.1 ± 0.8	82.4 ± 2.5	78.6 ± 2.1	77.2 ± 0.1
ROI 1[1]	ND	ND	0.6 ± 0.2	0.9 ± 0.0	1.3 ± 0.2	1.3 ± 0.0	1.5 ± 0.3	1.8 ± 0.4	1.4 ± 0.0	1.6 ± 0.1
ROI 2[1]	ND	ND	ND	0.7 ± 0.1	1.3 ± 0.1	2.0 ± 0.2	1.9 ± 0.1	0.8 ± 0.0	2.5 ± 0.1	2.6 ± 0.2
ROI 3[1]	ND	ND	ND	ND	0.6 ± 0.1	0.9 ± 0.1	1.4 ± 0.3	1.5 ± 0.0	1.7 ± 0.3	1.6 ± 0.1
ROI 5[1]	ND	ND	ND	ND	ND	ND	0.4 ± 0.0	0.6 ± 0.0	0.7 ± 0.2	0.6 ± 0.0
ROI 6[1]	ND	ND	ND	ND	ND	ND	ND	0.5 ± 0.1	ND	ND
ROI 7[1]	ND	ND	ND	ND	ND	ND	0.8 ± 0.1	1.1 ± 0.0	0.7 ± 0.2	1.0 ± 0.0
Sum of unid./diff. residues	ND	0.5 ± 0.1	ND	0.7 ± 0.1	2.9 ± 0.2	3.5 ± 0.4	3.5 ± 0.9	4.2 ± 1.0	5.5 ± 1.0	7.3 ± 0.0
Total extracted residues	98.6 ± 0.5	95.3 ± 0.4	97.3 ± 1.3	95.6 ± 0.2	95.8 ± 0.8	95.3 ± 0.1	92.0 ± 0.2	90.6 ± 1.9	87.9 ± 1.0	88.3 ± 0.2
CO_2	NA	<0.1 ± 0.0	<0.1 ± 0.0	0.1 ± 0.0	0.3 ± 0.0	0.9 ± 0.0	1.0 ± 0.3	1.7 ± 0.0	2.3 ± 0.1	2.8 ± 0.2
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	1.3 ± 0.0	1.8 ± 0.2	1.8 ± 0.0	2.2 ± 0.1	2.5 ± 0.2	4.0 ± 0.0	4.5 ± 0.1	5.9 ± 0.3	8.0 ± 0.0	7.7 ± 0.2
Total Recovery	100.0 ± 0.4	97.1 ± 0.2	99.1 ± 1.4	97.8 ± 0.2	98.7 ± 1.0	100.2 ± 0.0	97.5 ± 0.1	98.2 ± 1.5	98.2 ± 1.0	98.8 ± 0.1
Compound	DAT0	DAT2	DAT6	DAT15	DAT28	DAT50	DAT62	DAT84	DAT104	DAT120
Dollendorf II										
Isoflucypram	95.3 ± 1.4	95.0 ± 0.8	94.6 ± 0.1	93.1 ± 0.1	87.1 ± 2.5	85.1 ± 1.9	82.4 ± 0.1	77.9 ± 2.8	69.3 ± 2.8	72.2 ± 0.3
ROI 1[1]	ND	ND	1.1 ± 0.0	1.3 ± 0.2	1.9 ± 0.0	2.5 ± 0.3	2.4 ± 0.2	2.9 ± 0.2	5.8 ± 1.1	2.5 ± 0.1
ROI 2[1]	ND	ND	ND	0.9 ± 0.2	1.4 ± 0.2	2.1 ± 0.5	2.0 ± 0.3	2.1 ± 0.4	2.6 ± 1.0	2.6 ± 0.1
ROI 3[1]	ND	ND	ND	ND	0.6 ± 0.1	1.3 ± 0.3	1.5 ± 0.1	1.8 ± 0.1	3.8 ± 1.1	2.9 ± 0.3
ROI 4[1]	ND	ND	ND	ND	ND	ND	ND	ND	0.4 ± 0.0	ND
ROI 5[1]	ND	ND	ND	ND	ND	ND	0.7 ± 0.0	ND	0.8 ± 0.1	0.7 ± 0.0
ROI 7[1]	ND	ND	ND	ND	ND	ND	0.8 ± 0.0	1.1 ± 0.3	1.9 ± 0.1	1.1 ± 0.1
Sum of unid./diff. residues	ND	ND	0.4 ± 0.0	0.9 ± 0.2	2.2 ± 0.7	3.2 ± 1.1	3.7 ± 0.3	4.1 ± 0.8	6.4 ± 0.4	7.4 ± 0.2
Total extracted residues	95.7 ± 1.8	95.0 ± 0.8	96.1 ± 0.5	95.3 ± 0.1	91.8 ± 1.8	92.1 ± 0.8	90.7 ± 0.5	86.9 ± 1.6	86.0 ± 0.0	85.6 ± 0.2
CO_2	NA	<0.1 ± 0.0	<0.1 ± 0.0	0.1 ± 0.0	0.3 ± 0.0	0.7 ± 0.0	1.1 ± 0.0	1.8 ± 0.0	2.5 ± 0.1	3.0 ± 0.1
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	3.2 ± 0.6	5.0 ± 0.7	2.8 ± 0.1	3.7 ± 0.2	3.6 ± 0.4	4.9 ± 0.2	5.8 ± 0.3	7.4 ± 0.0	11.6 ± 0.2	10.7 ± 0.4

Notes:

[1] ROI: Region of interest.

Isoflucypram was evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. Three different kinetic models were tested in order to determine the best-fit

kinetic model: single first order (SFO), first order multi-component (FOMC), and double first-order in parallel (DFOP). The best-fit kinetic model was selected on the basis of the chi^2 scaled-error criterion and visual assessment of the goodness of fit. DT_{50} and DT_{90} values were calculated from the resulting kinetic parameters.

The degradation of isoflucypram was best fit with SFO kinetics in all soils according to the lowest chi^2 error values and visual assessments. The calculated DT_{50} and DT_{90} are shown in Table 44.

Table 44 Best fit results of the DT_{50} and DT_{90} calculations for isoflucypram

Soil (Texture)	Best Fit Kinetic Model[1,2]	DT_{50} (Days)	DT_{90} (Days)	Chi^2 Error (%)	Visual Assessment
Hanscheider Hof (loam)	SFO	458	>1,000	0.7	Good
	FOMC[3]	448	>1,000	0.7	Good
	DFOP	459	-[4]	0.7	Good
Laacher Hof AXXa (loamy sand)	SFO	239	795	0.7	Good
	FOMC[3]	285	>1,000	0.6	Good
	DFOP[3]	238	808	0.4	Good
Hoefchen Am Hohenseh (silt loam)	SFO	358	>1,000	0.9	Good
	FOMC	353	>1,000	1.0	Good
	DFOP	359	-[4]	0.9	Good
Dollendorf II (loam)	SFO	267	887	1.5	Good
	FOMC	259	873	1.6	Good
	DFOP	267	888	1.7	Good

Notes:
[1] SFO: Single first order, FOMC: First order multi compartment, DFOP: Double first order in parallel.
[2] Best fit highlighted in bold.
[3] Statistically non-reliable kinetic evaluations.
[4] Could not be calculated/determined.

The metabolic/degradation pathway of isoflucypram (BCS-CN88460) in soil in shown in Figure 7.

Report No. MELNN013.

The route and rate of degradation of [pyrazole-4-1^4C] isoflucypram was studied in two United States soils (Gabbert, D., *et. al.*; 2017). Samples were maintained under aerobic conditions in the dark in the laboratory for 123 days at 20.4 °C. Samples were maintained at moisture content of between pF 2.0 and 2.5. The application rate was 75 g ai/ha, corresponding to a concentration of 200 µg ai/kg soil dry weight. In order to bridge to a higher rate, additional test systems were treated at 430 µg ai/kg soil dry weight (equivalent to ca. 150 g ai/ha) which were additionally used for metabolite identification. Soil was collected from the top 20 cm and sieved to a particle size of ≤ 2 mm. The two soils represent different geographical origin and properties.

Table 45 Soil Properties for aerobic metabolism study for isoflucypram

Designation	Source	Texture (USDA)	pH	% organic content
CA Soil	Sanger, CA	Sandy loam	6.3	0.77
NE Soil	Louisville, NE	Silty clay loam	6.3	2.0

Notes:
[1] pH values were derived from aqueous 0.01M $CaCl_2$ suspensions.

The test was performed with a flow-through system consisting of cylindrical bottles each containing 75 g soil (dry weight equivalents). The bottles were attached to a series of traps for collection of VOCs and CO_2.

Water loss from evaporation was determined by weighing all test systems at study start and the respective each sampling interval. No significant losses of moisture were observed throughout the study. Determinations of microbial biomass were performed on DAT0 and DAT123 and demonstrated that the soils were microbially viable. The CA soil showed a significant decline in activity at the end of the study. Under the conditions of a laboratory experiment a decrease of microbial biological activity is inevitable due to the absence of any further amendment of nutrients.

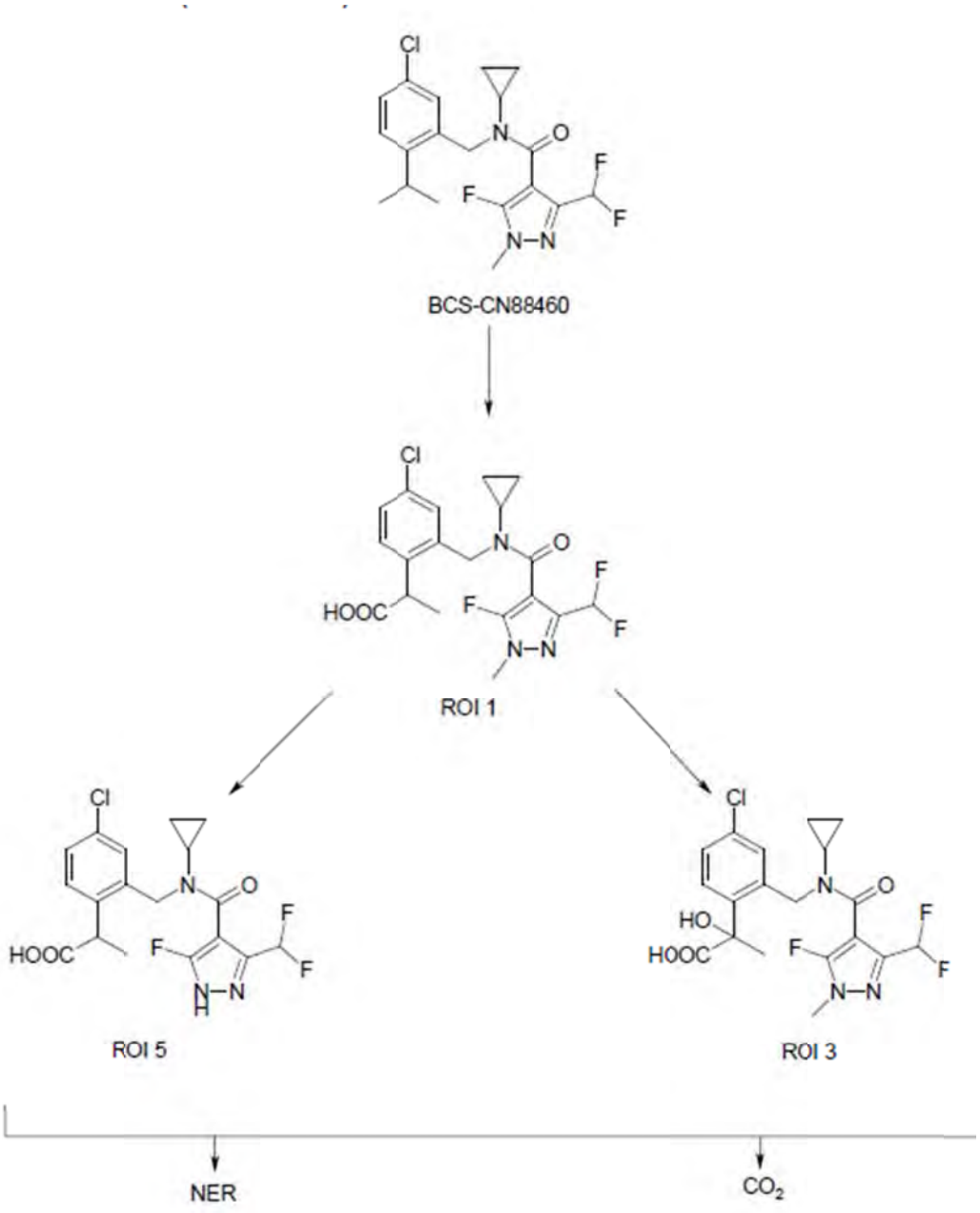


Figure 7 Metabolic/degradation pathway of isoflucypram (BCS-CN88460) in soil

Replicate samples were collected for analysis on DAT0, 6, 14, 21, 28, 60, 88, and 123. The exaggerated application soil samples were collected for analysis on DAT76, 88, and 123. Prior to processing of soil, possible volatiles were purged into the trap attachment and the trap attachment was removed. VOCs and CO_2 were extracted with ethylene glycol and potassium hydroxide, respectively, and

radioactivity was determined by LSC. No chromatographic analyses were performed for VOCs due to low radioactivity (≤ 3.3 percent AR).

At each sampling interval, soil was extracted three times at ambient temperature: once using ACN and additional two times using ACN/water (4:1). Furthermore, two microwave-accelerated extraction steps were performed using ACN/water (4:1) at 70°C and MeOH/water (9:1; v;v) at 50 °C, respectively. The amounts of test substance and degradation products in soil extracts were determined by LSC and by HPLC/radiodetection analysis. The amounts of volatiles and non-extracted residues were determined by LSC and combustion/LSC, respectively.

The test substance and degradation products were identified by LC/ESI-MS and comparison with reference standards. Further, on DAT123, samples were extracted as described above with an additional two ambient extraction steps, first with ethyl acetate and second with hexane. Radioactivity of the combined extracts was determined by LSC and found to be ≤ 0.9 percent AR for the CA soil and ≤ 2.1 percent for NE soil, demonstrating that the principal extraction method was effective at determining extracted residues. Parent and metabolite peaks from these samples were analysed for identification by LC/ESI-MS.

Soil extracts were stored for a maximum of two days at -10°C. Soil NER were stored for a maximum of four days under refrigerated conditions.

Mean material balances were 97.0 percent AR for CA soil and 96.8 percent AR for NE soil. Extracted residues decreased from 94.7 percent AR at DAT0 to 92.3 percent AR at DAT123 in CA soil and from 94.8 percent at DAT0 to 83.0 percent AR DAT123 in NE soil. NER increased from 0.2 percent at DAT0 to 3.4 percent AR at DAT123 in CA soil and from 0.3 percent at DAT0 to 10.7 percent AR at DAT123 in NE soil. Formation of carbon dioxide was ≤ 3.2 percent AR and other VOCs was ≤ 0.1 percent AR in both soil samples.

Trials showed similar trends at both application rates. The results are shown in Tables 46 and 47.

Table 46 Composition of radioactive residues soils under aerobic conditions, in percent of applied radioactivity (mean ± SD) (MELNN013)

Compound	DAT0	DAT6	DAT14	DAT21	DAT28	DAT60	DAT88	DAT123
CA soil								
Isoflucypram	94.7 ± 0.5	97.1 ± 0.8	97.4 ± 1.8	97.0 ± 1.1	95.8 ± 0.6	90.4 ± 1.9	89.8 ± 0.1	86.2 ± 2.8
Isoflucypram-carboxylic acid	ND	ND	ND	ND	ND	1.1 ± 1.5	ND	1.3 ± 1.8
Unknown 2	ND	ND	ND	ND	ND	2.6 ± 0.0	1.9 ± 2.7	4.0 ± 0.5
Unidentified radioactivity	ND	ND	ND	ND	ND	2.6 ± 0.0	1.9 ± 2.7	4.0 ± 0.5
Total extracted radioactivity	94.7 ± 0.5	97.1 ± 0.8	97.4 ± 1.8	97.0 ± 1.1	95.8 ± 0.6	94.1 ± 0.4	91.7 ± 2.7	92.3 ± 1.6
CO_2	NA[1]	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.5 ± 0.0	0.5 ± 0.0	0.9 ± 0.1	1.9 ± 0.9
VOC	NA[1]	ND	0.1 ± 0.0	ND	0.1 ± 0.0	ND	ND	0.1 ± 0.0
Bound residues	0.2 ± 0.0	0.5 ± 0.0	0.7 ± 0.1	0.9 ± 0.1	1.0 ± 0.1	1.9 ± 0.2	3.0 ± 0.2	3.4 ± 0.0
Total percent recovery	94.9 ± 0.4	97.7 ± 0.7	98.2 ± 1.7	98.1 ± 1.2	97.4 ± 0.6	96.5 ± 0.7	95.6 ± 2.9	97.6 ± 2.5
NE soil								
Isoflucypram	94.8 ± 3.0	95.7 ± 1.8	94.6 ± 0.7	93.5 ± 1.7	92.9 ± 2.1	86.4	75.0 ± 1.8	64.4 ± 1.2
Isoflucypram-carboxylic acid	ND	ND	ND	1.4 ± 2.0	ND	4.1	6.7 ± 0.4	9.6 ± 0.7
Unknown 2	ND	ND	ND	ND	ND	ND	3.6 ± 0.7	4.0 ± 0.1
Unknown 3	ND	ND	ND	ND	ND	ND	ND	3.2 ± 0.3
Unidentified radioactivity	ND	ND	ND	ND	ND	ND	3.6 ± 0.7	7.2 ± 0.3

Compound	DAT0	DAT6	DAT14	DAT21	DAT28	DAT60	DAT88	DAT123
CA soil								
Extracted radioactivity	94.8 ± 3.0	95.7 ± 1.8	94.6 ± 0.7	94.9 ± 0.3	92.9 ± 2.1	90.5	85.2 ± 0.7	83.0 ± 2.1
CO_2	NA[1]	0.1 ± 0.0	0.3 ± 0.0	0.4 ± 0.0	0.2 ± 0.0	1.2	2.0 ± 0.1	3.2 ± 0.1
VOCs	NA[1]	ND	0.1 ± 0.0	ND	0.1 ± 0.0	ND	ND	0.1 ± 0.0
Bound residues	0.3 ± 0.0	1.9 ± 1.3	2.0 ± 0.4	2.5 ± 0.0	3.2 ± 0.2	6.0	8.4 ± 0.8	10.7 ± 1.3
Total percent recovery	95.1 ± 3.0	97.7 ± 0.4	96.9 ± 0.4	97.8 ± 0.3	96.4 ± 2.3	97.8	95.6 ± 1.6	97.0 ± 0.6

Notes:
[1] Not trapped or measured on DAT0.
[2] Only one replicate on DAT60.

Table 47 Degradation of isoflucypram at exaggerated application rate in CA and NE soil, in percent of applied radioactivity (mean ± SD)

Compound	DAT76	DAT88	DAT123
CA soil			
Isoflucypram	97.9	99.4	88.9
Unknown 1	<LOQ	<LOQ	1.6
Unknown 2	<LOQ	<LOQ	4.1
Isoflucypram-carboxylic acid	3.0	ND	2.8
Unknown 3	<LOQ	<LOQ	<LOQ
Unknown 4	<LOQ	<LOQ	<LOQ
Total extracted radioactivity	100.9	99.4	97.9
CO_2	0.6	0.7	1.4
Bound residues	2.4	3.2	3.6
Total percent recovery	103.9	103.4	102.9
NE soil			
Isoflucypram	84.9	79.3	64.7
Unknown 1	<LOQ	<LOQ	1.8
Unknown 2	3.4	4.0	5.0
Isoflucypram-carboxylic acid	5.4	7.1	10.0
Unknown 3	<LOQ	<LOQ	4.0
Unknown 4	<LOQ	<LOQ	1.9
Total extracted radioactivity	93.7	90.4	88.0
CO_2	1.6	1.8	3.0
Bound residues	7.2	7.8	11.2
Total percent recovery	102.6	100.0	102.2

Isoflucypram was evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. Three different kinetic models were tested in order to determine the best-fit kinetic model (SFO, FOMC, and DFOP). The best-fit kinetic model was selected on the basis of the chi^2 scaled-error criterion and visual assessment of the goodness of fit. DT_{50}, DT_{75}, and DT_{90} values were calculated from the resulting kinetic parameters.

The degradation of isoflucypram was best fit with SFO kinetics in all soils according to the lowest chi^2 error values and visual assessments (Table 48). Another proposal of degradation of isoflucypuram in soil is shown in Figure 8.

Table 48 Summary of the kinetic evaluation (for trigger values according to FOCUS) of the degradation of isoflucypram in soils under aerobic conditions

Soil Texture (USDA)	Kinetic Model[1]	DT_{50} (Days)	DT_{75} (Days)	DT_{90} (Days)	Chi^2 Error[2] (percent)	Visual Assessment
CA sandy loam	SFO	714	>1,000	>1,000	1.15	Good

Soil Texture (USDA)	Kinetic Model[1]	DT_{50} (Days)	DT_{75} (Days)	DT_{90} (Days)	Chi^2 Error[2] (percent)	Visual Assessment
	FOMC	714	>1,000	>1,000	1.23	Good
	DFOP	714	>1,000	>1,000	1.33	Good
NE silty clay loam	SFO	223	446	741	2.35	Good
	FOMC	223	446	741	2.51	Good
	DFOP	223	446	740	2.71	Good

Notes:
[1] SFO = Single first order. FOMC = First order multi compartment. DFOP = Double first order in parallel.
[2] Best fit highlighted in bold font.

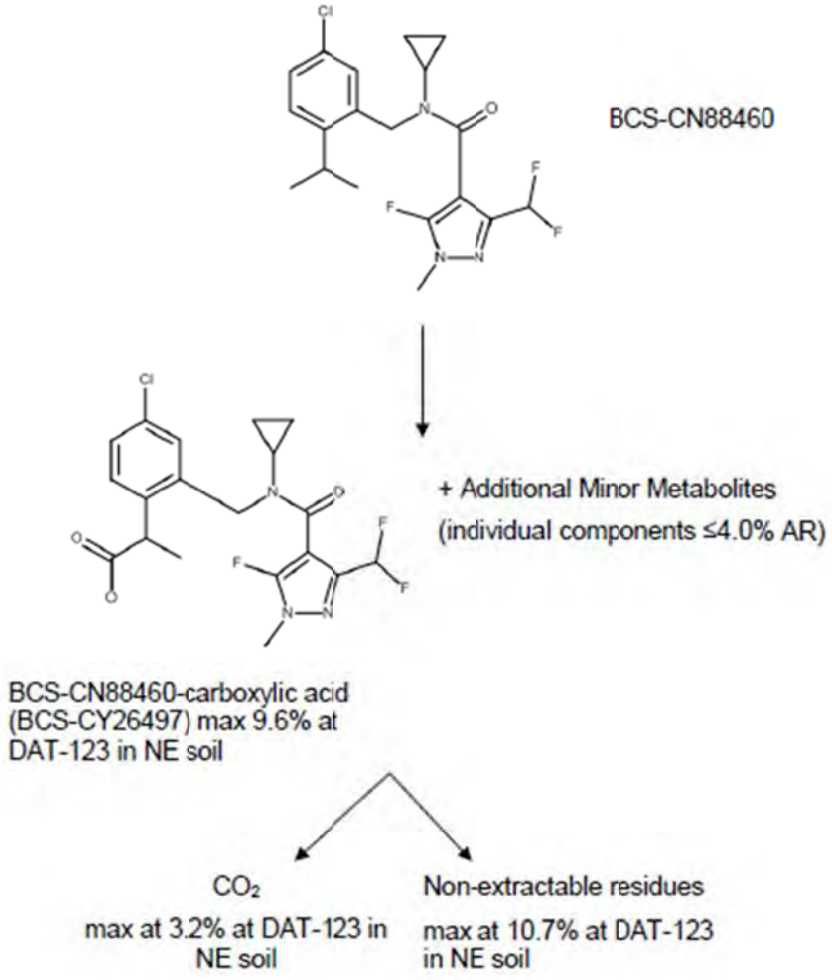


Figure 8 Metabolic/degradation pathway of isoflucypram (BCS-CN88460) in soil

Report No. EnSa-16-0986.

The route and rate of degradation including the radiolabelled testing of the simplified extraction method of [phenyl-UL-^{14}C] isoflucypram were studied (Heinemann, O., *et. al.*, 2017). Samples were maintained under aerobic conditions in the dark in the laboratory for 125 days at 20 °C. Samples were maintained at 55 percent of the maximum water holding capacity. The application rate was 75 g ai/ha, corresponding to

a concentration of 18.8 µg ai/kg soil dry weight. Soil was collected from the top 20 cm and sieved to a particle size of ≤ 2 mm (Table 49.

Table 49 Soil properties for aerobic metabolism study for isoflucypram

Soil	Source	Texture	pH[1]	OC (percent)
Laacher Hof AXXa	Monheim, Germany	Loamy sand	5.8	1.6

Notes:

[1] pH value was derived from aqueous 0.01M $CaCl_2$ suspension.

Tests were performed in incubation vessels with 100 g soil (dry weight equivalents) equipped with oxygen-permeable PU and soda lime traps for VOCs and CO_2, respectively. Water loss from evaporation was determined by weighing all test systems at study start and each sampling interval from DAT14 onwards. The evaporated portion was replaced accordingly by addition of de-ionized water after 28, 50, and 85 days of incubation. No significant losses of moisture were observed throughout the study. Determinations of microbial biomass were performed on DAT0, 65, and 125 and demonstrated that the used soil was microbially viable. Duplicate samples were processed and analysed on DAT0, 2, 6, 14, 28, 50, 65, 85, 100, and 125.

Prior to processing of soil, possible volatiles were purged into the trap attachment and the trap attachment was removed. CO_2 was released from the soda lime with HCl and re-absorbed to a scintillation cocktail for LSC. VOCs were released with ethyl acetate. No chromatographic analyses were performed for VOCs due to low radioactivity (≤ 0.1 percent AR).

At each sampling interval, the soil was extracted three times at ambient temperature using ACN/water (1:1). Furthermore, two microwave-assisted extraction steps were performed using ACN/water (1:1) at 70 °C and MeOH/water (1:1) at 50 °C. The amounts of test item and degradation products in soil extracts were determined by LSC and by HPLC/radiodetection analysis. The amounts of volatiles and non-extracted residues were determined by LSC and combustion/LSC, respectively.

Test item identity was confirmed by HPLC-MS(/MS) including accurate mass determination and degradation products were identified by co-chromatography with reference items. Additionally, samples were processed and analysed on DAT0, 65, and 125 using a simplified extraction method and compared to the exhaustive extraction method (Table 50). The simplified extraction method involved ACN:water:acetic acid (400:100:3) for LSC. The simplified extraction process was not used for sample analysis.

Table 50 Extraction efficiencies of the simplified and exhaustive extraction methods in soils Laacher Hof AXXa and Dollendorf II, in percent of applied radioactivity (mean ± SD)

DAT0		DAT65		DAT125	
Laacher Hof AXXa					
Exhaustive	Simplified	Exhaustive	Simplified	Exhaustive	Simplified
100.3 ± 1.0	80.7 ± 0.7	97.5 ± 0.8	75.2 ± 0.9	92.0 ± 0.2	72.1 ± 0.0
Difference: 19.6		Difference: 22.3		Difference: 19.9	
Dollendorf II					
Exhaustive	Simplified	Exhaustive	Simplified	Exhaustive	Simplified
97.5 ± 0.8	78.7 ± 0.4	92.8 ± 0.5	67.2 ± 0.4	85.3 ± 1.4	66.7 ± 0.0
Difference: 18.9		Difference: 25.7		Difference: 18.5	

Soil samples were stored for a maximum of three days in the dark. Soda lime and PU were stored for a maximum of 22 days. Soil NER were stored for a maximum of three days in the dark.

The material balances ranged from 101 to 105 percent AR. The maximum amount of CO_2 was 5.2 percent AR at study end (DAT125). Formation of VOCs was insignificant as demonstrated by values of ≤ 0.1 percent AR at all sampling intervals. Extracted residues decreased from DAT0 to DAT125 from 100 to 92.0 percent AR. NER increased from DAT0 to DAT125 from 0.7 to 6.4 percent AR. The amount of isoflucypram in the soil extracts decreased from DAT0 to DAT125 from 100 to 75.5 percent AR (Table 51).

Table 51 Composition of radioactive residues under aerobic conditions, in percent of applied radioactivity (mean ± SD) (EnSa-16-0986)

Compound	DAT0	DAT2	DAT6	DAT14	DAT28	DAT50	DAT65	DAT85	DAT100	DAT125
Isoflucypram	100.3 ± 1.0	102.8 ± 0.2	101.0 ± 0.1	97.6 ± 0.3	94.2 ± 1.2	88.2 ± 0.1	88.0 ± 0.4	73.0 ± 0.4	81.5 ± 1.9	75.5 0.2
Isoflucypram-carboxylic acid	ND	ND	ND	1.3 ± 0.2	2.2 ± 0.0	2.7 ± 0.2	3.0 ± 0.0	5.8 ± 0.5	4.2 ± 0.2	6.2 ± 0.4
Sum of unid./diff. residues	ND	ND	ND	ND	1.9 ± 0.5	4.7 ± 0.0	6.5 ± 0.5	13.1 ± 0.0	8.4 ± 0.9	10.3 ± 0.1
Total extracted residues	100.3 ± 1.0	102.8 ± 0.2	101.0 ± 0.1	99.2 ± 0.5	98.3 ± 0.7	95.6 ± 0.2	97.5 ± 0.8	91.9 ± 0.1	94.1 ± 0.9	92.0 ± 0.2
CO_2	NA	<0.1 ± 0.0	0.2 ± 0.0	0.3 ± 0.2	1.1 ± 0.0	2.3 ± 0.0	2.9 ± 0.2	3.5 ± 0.8	4.3 ± 0.2	5.2 ± 0.2
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	0.7 ± 0.0	0.9 ± 0.0	1.2 ± 0.0	1.5 ± 0.0	2.2 ± 0.0	3.2 ± 0.0	3.8 ± 0.0	5.6 ± 0.0	5.3 ± 0.2	6.4 ± 0.2
Total recovery	101.0 ± 1.0	103.7 ± 0.2	102.4 ± 0.1	101.1 ± 0.3	101.6 ± 0.6	101.1 ± 0.2	104.2 ± 1.1	101.0 ± 0.6	103.8 ± 0.6	103.7 ± 0.2

Isoflucypram was evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. Three different kinetic models were tested in order to determine the best-fit kinetic model (SFO, FOMC, and DFOP). The best-fit kinetic model was selected on the basis of the chi^2 scaled-error criterion and visual assessment of the goodness of fit. DT_{50} and DT_{90} values were calculated from the resulting kinetic parameters (Table 52). The degradation of isoflucypram was best fit with SFO kinetics in all soils according to the lowest chi^2 error values and visual assessments.

Table 52 Summary of kinetic evaluation (for trigger values according to FOCUS) of the degradation of isoflucypram in soil under aerobic conditions

Soil	Kinetic Model[1,2]	DT_{50}	DT_{90}	Chi^2 Error	Visual Assessment
Laacher Hof AXXa (Loamy sand)	SFO	263	873	2.7	Moderate
	FOMC	425	>1,000	2.8	Moderate
	DFOP	>1,000	>1,000	2.9	Moderate

Notes:

[1] SFO: single first order. FOMC: first order multi compartment. DFOP: double first order in parallel.

[2] Best fit written in bold.

Aerobic degradation and time-dependent sorption

Report No. EnSa-19-0236

The Meeting received a study investigating the degradation and time-dependence of sorption of [phenyl-UL-^{14}C] isoflucypram (Hellpointner, E., *et. al.*; 2019). Isoflucypram was studied in six soils under aerobic conditions in the laboratory in the dark at 20 °C for 120 days. Samples were maintained at 55 percent of the maximum water holding capacity. The application rate was 75 g ai/ha, corresponding to a

concentration of 204.8 µg ai/kg soil dry weight. Soil was collected from the top 20 cm and sieved to a particle size of ≤ 2 mm (Table 53).

Table 53 Soil properties

Soil	Source	Texture (USDA)	pH[1]	% organic content
Laacher Hof AXXa	Monheim, Germany	Sandy loam	6.9	1.7
Hoefchen am Hohenseh 4a	Burscheid, Germany	Silt loam	5.8	2.3
Hanscheider Hof	Burscheid, Germany	Silt loam	5.3	3.5
Dollendorf II	Blankenheim, Germany	Loam	7.1	5.7
Wurmwiese	Monheim, Germany	Sandy loam	5.1	2.0
Frankenforst	Vinxel, Germany	Silt loam	6.9	3.3

Notes:

[1] pH values were derived from aqueous 0.01 mol/L $CaCl_2$ suspensions.

Tests were performed in incubation vessels with 100 g soil (dry weight equivalents) equipped with oxygen-permeable PU and soda lime traps for VOCs and CO_2, respectively.

Water loss from evaporation was determined by weighing test systems DAT64 and DAT98. The evaporated portion was replaced accordingly by addition of de-ionized water. No significant losses of moisture were observed throughout the study.

Determinations of microbial biomass were performed on DAT0 demonstrated that the soil was microbially viable. Duplicate samples were processed and analysed on DAT0, 3, 7, 14, 28, 49, 70, 84, 98, and 120.

Prior to processing of soil, possible volatiles were purged into the trap attachment and the trap attachment was removed. CO_2 was released from the soda lime with HCl and re-absorbed to a scintillation cocktail for LSC. VOCs were released with ethyl acetate. No chromatographic analyses were performed for VOCs due to low radioactivity (≤ 0.1 percent AR).

At each sampling interval, the soil was extracted three times at ambient temperature; first with 0.01 mol/L $CaCl_2$ followed by two extractions with ACN/water (1:1). Furthermore, two microwave-assisted extraction steps were performed using ACN/water (1:1) at 70°C and MeOH at 50 °C. The amounts of test item and degradation products in soil extracts were determined by LSC and by HPLC/radiodetection analysis. The amounts of volatiles and non-extracted residues were determined by LSC and combustion/LSC, respectively.

Test item identity and degradation products were confirmed by HPLC-MS(/MS) including accurate mass determination.

Mean material balances were 97.9 percent AR for soil Laacher Hof AXXa, 98.8 percent AR for soil Hoefchen am Hohenseh 4a, 98.8 percent AR for soil Hanscheider Hof, 98.1 AR for soil Dollendorf II, 98.1 percent AR for soil Wurmwiese, and 98.1 percent for soil Frankenforst.

Desorbable residues in aqueous 0.01 mol/L $CaCl_2$ solution slightly increased from DAT0 to DAT120 from 15.6 percent to 16.0 percent AR in soil Laacher Hof AXXa, from 4.7 percent to 7.0 percent in soil Dollendorf, from 7.5 percent to 9.8 percent AR in soil Frankenforst, and decreased from 10.5 percent to 9.5 percent AR in soil Hoefchen am Hohenseh 4a, from 5.7 percent to 3.8 percent AR in soil Hanscheider Hof, and from 11.8 percent to 9.5 percent AR in soil Wurmwiese. The results for all soils are shown in Table 54.

Table 54 Degradation of isoflucypram in different soils under aerobic conditions, in percent of applied radioactivity (mean ± SD) (EnSa-19-0236)

Compound/Fraction	DAT0	DAT3	DAT7	DAT14	DAT28	DAT49	DAT70	DAT84	DAT98	DAT120
Laacher Hof AXXa										
Isoflucypram	93.6 ± 0.3	94.0 ± 0.6	94.0 ± 1.5	93.5 ± 0.8	94.4 ± 0.5	87.5 ± 1.4	84.8 ± 0.6	81.0 ± 1.0	81.3 ± 0.3	79.4 ± 0.5
Isoflucypram-carboxylic acid	ND	ND	ND	ND	1.9 ± 0.0	6.3 ± 0.7	8.5 ± 0.3	7.4 ± 0.1	8.6 ± 1.7	10.9 ± 0.3
Isoflucypram-olefine	ND	ND	ND	ND	ND	ND	ND	3.7 ± 0.4	2.8 ± 1.9	2.4 ± 0.6
Sum of unid./diff. residues	ND	ND	0.7 ± 0.0	0.7 ± 0.0	ND	ND	ND	ND	0.7 ± 0.0	ND
Total extracted residues	94.0 ± 0.6	94.0 ± 0.6	94.7 ± 0.8	94.2 ± 0.0	96.8 ± 0.1	94.1 ± 0.3	93.3 ± 0.3	92.6 ± 0.2	93.4 ± 0.8	93.3 ± 0.4
CO_2	NA	0.3 ± 0.0	0.4 ± 0.0	0.2 ± 0.2	0.7 ± 0.1	1.2 ± 0.0	1.4 ± 0.0	1.4 ± 0.0	1.5 ± 0.0	1.6 ± 0.1
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	1.3 ± 0.0	2.0 ± 0.3	1.7 ± 0.1	2.0 ± 0.2	0.9 ± 0.2	2.9 ± 0.1	3.4 ± 0.1	3.6 ± 0.1	3.9 ± 0.1	4.7 ± 0.4
Total recovery	95.3 ± 0.7	96.3 ± 0.9	96.7 ± 0.8	96.4 ± 0.1	98.4 ± 0.3	98.1 ± 0.3	98.2 ± 0.2	97.6 ± 0.0	98.8 ± 0.9	99.5 ± 0.9
Compound/Fraction	DAT0	DAT3	DAT7	DAT14	DAT28	DAT49	DAT70	DAT84	DAT98	DAT120
Hoefchen am Hohenseh 4a										
Isoflucypram	95.5 ± 0.1	92.6 ± 0.6	95.6 ± 0.2	92.9 ± 0.0	95.5 ± 1.2	94.1 ± 0.1	91.5 ± 0.4	90.0 ± 0.4	87.4 ± 0.3	82.0 ± 0.4
Isoflucypram-carboxylic acid	ND	ND	ND	ND	ND	0.9 ± 0.2	2.1 ±0.3	2.6 ± 0.5	4.0 ± 0.1	8.4 ± 0.5
Isoflucypram-olefine	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.4 ± 0.0
Sum of unid./diff. residues	ND	ND	ND	0.9 ± 0.3	1.3 ± 0.0	ND	ND	ND	1.0 ± 0.1	0.8 ± 0.0
Total extracted residues	95.9 ± 0.3	92.6 ± 0.6	95.6 ± 0.2	93.9 ± 0.3	96.7 ± 0.1	95.0 ± 0.3	94.0 ± 0.3	93.1 ± 0.4	92.3 ± 0.3	92.6 ± 0.0
CO_2	NA	0.2 ± 0.0	0.4 ± 0.0	0.3 ± 0.2	0.7 ± 0.1	1.0 ± 0.0	1.1 ± 0.0	1.2 ± 0.0	1.2 ± 0.0	1.2 ± 0.0
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	1.8 ± 0.1	5.3 ± 0.1	2.3 ± 0.0	3.5 ± 0.2	1.6 ± 0.3	3.4 ± 0.1	4.0 ± 0.0	4.1 ± 0.1	4.4 ± 0.1	5.0 ± 0.2
Total recovery	97.7 ± 0.2	98.2 ± 0.5	98.3 ± 0.2	97.7 ± 0.3	99.0 ± 0.4	99.5 ± 0.2	99.1 ± 0.3	98.3 ± 0.3	97.9 ± 0.3	98.8 ± 0.3
Compound/Fraction	DAT0	DAT3	DAT7	DAT14	DAT28	DAT49	DAT70	DAT84	DAT98	DAT120
Hanscheider Hof										
Isoflucypram	94.9 ± 0.1	88.4 ± 0.6	92.5 ± 0.3	91.8 ± 0.3	92.2	90.9 ± 0.3	87.2 ± 0.2	87.0 ± 0.3	86.3 ± 0.3	85.5 ± 0.3
Isoflucypram-carboxylic acid	ND	ND	ND	ND	ND	1.8 ± 0.3	2.3 ± 0.2	1.9 ± 0.5	2.7 ± 0.2	3.0 ± 0.0
Isoflucypram-olefine	ND	ND	ND	ND	ND	0.9 ± 0.1	ND	0.6 ± 0.0	0.7 ± 0.0	1.3 ± 0.3
Sum of unid./diff. residues	ND	ND	ND	0.6 ± 0.0	1.2	0.8 ± 0.0	0.8 ± 0.0	0.9 ± 0.2	ND	0.6 ± 0.0
Total extracted residues	95.3 ± 0.3	88.4 ± 0.6	92.9 ± 0.8	92.4 ± 0.9	93.4	94.4 ± 0.0	90.3 ± 0.8	90.4 ± 0.0	90.2 ± 0.3	90.4 ± 0.0
CO_2	NA	0.3 ± 0.0	0.5 ± 0.0	0.7 ± 0.1	1.1	1.2 ± 0.0	1.2 ± 0.0	1.3 ±0.0	1.3 ± 0.0	1.4 ± 0.0
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	2.9 ± 0.0	8.9 ± 0.5	4.6 ± 0.1	6.8 ± 0.2	4.4	4.9 ± 0.0	6.5 ± 0.2	6.1 ± 0.1	6.9 ± 0.1	6.9 ± 0.0
Total recovery	98.2 ± 0.3	97.5 ± 0.1	98.0 ± 0.9	99.8 ± 1.0	98.8	100.5 ± 0.0	98.0 ± 0.9	97.8 ± 0.1	98.3 ± 0.3	98.7 ± 0.3
Compound/Fraction	DAT0	DAT3	DAT7	DAT14	DAT28	DAT49	DAT70	DAT84	DAT98	DAT120
Dollendorf II										

Compound/Fraction	DAT0	DAT3	DAT7	DAT14	DAT28	DAT49	DAT70	DAT84	DAT98	DAT120
Isoflucypram	93.7 ± 0.0	86.1 ± 1.4	90.4 ± 0.3	88.2 ± 3.5	86.0 ± 0.8	86.5 ± 0.0	81.5 ± 0.0	80.6 ± 1.0	79.5 ± 2.8	80.8 ± 1.3
Isoflucypram-carboxylic acid	ND	ND	ND	ND	2.5 ± 0.8	2.7 ± 0.1	3.7 ± 0.1	2.2 ± 1.3	1.5 ± 0.5	1.8 ± 0.6
Isoflucypram-olefine	ND	ND	ND	ND	ND	1.3 ± 0.0	1.9 ± 0.0	1.4 ± 0.3	2.4 ± 0.4	1.6 ± 0.2
Sum of unid./diff. residues	0.8 ± 0.1	ND	1.0 ± 0.0	ND	ND	ND	0.9 ± 0.2	1.0 ± 0.1	ND	1.0 ± 0.1
Total extracted residues	94.4 ± 0.1	86.5 ± 1.0	91.4 ± 0.3	88.5 ± 3.1	89.1 ± 0.5	90.9 ± 0.3	88.0 ± 0.1	85.1 ± 0.0	83.7 ± 2.3	85.3 ± 0.5
CO_2	NA	0.3 ± 0.0	0.4 ± 0.0	0.7 ± 0.0	0.9 ± 0.0	1.7 ± 0.1	2.7 ± 0.0	3.2 ± 0.1	4.8 ± 0.6	4.7 ± 0.4
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	3.1 ± 0.3	10.5 ± 0.4	4.8 ± 0.2	10.3 ± 2.4	7.0 ± 0.1	6.7 ± 0.1	8.1 ± 0.2	8.5 ± 0.1	9.1 ± 0.7	8.4 ± 0.0
Total recovery	97.6 ± 0.4	97.3 ± 0.6	96.6 ± 0.4	99.5 ± 0.7	97.1 ± 0.7	99.2 ± 0.4	98.8 ± 0.1	96.9 ± 0.0	97.5 ± 1.1	98.3 ± 1.1
Compound/Fraction	DAT0	DAT3	DAT7	DAT14	DAT28	DAT49	DAT70	DAT84	DAT98	DAT120
Wurmwiese										
Isoflucypram	96.0 ± 1.4	93.2 ± 0.5	92.7 ± 0.7	91.7 ± 0.5	93.5 ± 0.5	91.6 ± 0.9	88.9 ± 0.8	88.7 ± 1.1	87.9 ± 0.3	85.8 ± 0.8
Isoflucypram-carboxylic acid	ND	ND	ND	ND	ND	2.4 ± 0.1	2.9 ± 0.3	3.3 ± 0.4	2.8 ± 0.5	4.1 ± 1.0
Isoflucypram-olefine	ND	ND	ND	ND	ND	1.0 ± 0.4	1.3 ± 0.2	1.3 ± 0.0	1.0 ± 0.0	1.3 ± 0.1
Sum of unid./diff. residues	ND	ND	1.1 ± 0.3	1.6 ± 0.0	0.6 ± 0.0	ND	1.1 ± 0.5	0.8 ± 0.1	ND	1.0 ± 0.2
Total extracted residues	96.0 ± 1.4	93.6 ± 0.1	93.8 ± 0.5	93.2 ± 0.5	94.1 ± 0.1	95.3 ± 0.8	94.2 ± 0.2	94.1 ± 0.5	91.8 ± 0.1	92.1 ± 0.1
CO_2	NA	0.3 ± 0.0	0.5 ± 0.0	0.8 ± 0.0	1.0 ± 0.0	1.1 ± 0.0	1.2 ± 0.0	1.2 ± 0.0	1.2 ± 0.0	1.2 ± 0.0
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	1.6 ± 0.1	3.8 ± 0.1	2.3 ± 0.0	4.2 ± 0.6	2.1 ± 0.3	3.2 ± 0.2	3.7 ± 0.0	3.7 ± 0.0	4.0 ± 0.1	4.3 ± 0.2
Total recovery	97.5 ± 1.3	97.6 ± 0.2	96.5 ± 0.5	98.2 ± 0.1	97.2 ± 0.4	99.7 ± 0.6	99.1 ± 0.2	99.0 ± 0.5	97.0 ± 0.0	97.7 ± 0.0
Compound/Fraction	DAT0	DAT3	DAT7	DAT14	DAT28	DAT49	DAT70	DAT84	DAT98	DAT120
Hoefchen am Hohenseh 4a										
Isoflucypram	95.1 ± 0.4	90.0 ± 0.3	91.5 ± 0.0	91.2 ± 1.0	87.3 ± 0.3	87.8 ± 0.1	83.9 ± 0.0	82.0 ± 0.7	82.8 ± 2.6	80.2 ± 1.2
Isoflucypram-carboxylic acid	ND	ND	ND	ND	3.2 ± 0.4	4.7 ± 0.3	6.2 ± 0.0	5.4 ± 0.1	2.4 ± 1.1	4.7 ± 0.0
Isoflucypram-olefine	ND	ND	ND	ND	ND	0.8 ± 0.1	1.4 ± 0.2	1.6 ± 0.2	1.7 ± 0.1	1.7 ± 0.2
Sum of unid./diff. residues	ND	0.6 ± 0.0	1.0 ± 0.2	1.2 ± 0.4	2.2 ± 0.1	ND	ND	1.1 ± 0.2	0.9 ± 0.2	1.1 ± 0.1
Total extracted residues	95.1 ± 0.4	90.6 ± 0.3	92.5 ± 0.3	92.3 ± 0.6	92.7 ± 0.6	93.8 ± 0.1	92.0 ± 0.7	90.1 ± 0.8	87.8 ± 1.6	87.8 ± 1.3
CO_2	NA	0.3 ± 0.0	0.5 ± 0.0	0.5 ± 0.2	1.0 ± 0.0	1.3 ± 0.0	1.8 ± 0.1	2.5 ± 0.3	3.2 ± 0.5	3.6 ± 0.3
VOCs	NA	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0	<0.1 ± 0.0
NER	2.1 ± 0.1	7.4 ± 0.5	2.9 ± 0.1	5.6 ± 0.9	3.2 ± 0.1	3.9 ± 0.2	4.5 ±0.2	5.1 ± 0.4	6.0 ± 0.5	6.3 ± 0.3
Total recovery	97.2 ± 0.5	98.3 ± 0.1	95.9 ± 0.2	98.4 ± 0.5	96.9 ± 0.5	98.9 ± 0.0	98.4 ± 0.4	97.7 ± 0.1	96.9 ± 0.5	97.7 ± 0.5

Notes:

[1] Replicate 2 of DAT28 is an outlier and was therefore excluded from the evaluation as reanalysis of the soil extracts gave the same results as the first analysis.

The time-dependent sorption ratios (R_{TDS}) were calculated by comparing the concentrations of the test item in the organic soil extracts and the decanted desorption solution (Table 55).

Table 55 Time dependent sorption ratios, , in percent of applied radioactivity (mean ± SD)

	Laacher Hof AXXa	Hoefchen am Hohenseh 4a	Hanscheider Hof[1]	Dollendorf II	Wurmwiese	Frankenforst
0	39.5 ± 0.04	64.1 ± 0.07	126 ± 0.00	149 ± 0.05	58.4 ± 0.18	92.9 ± 0.05
3	40.9 ± 0.04	69.7 ± 0.02	135 ± 0.01	135 ± 0.0	71.2 ± 0.01	92.6 ± 0.01
7	39.7 ± 0.01	63.6 ± 0.07	132 ± 0.07	128 ± 0.02	67.7 ± 0.04	93.2 ± 0.01
14	43.0 ± 0.01	79.4 ± 0.01	144 ± 0.03	126 ± 0.05	70.5 ± 0.00	92.0 ± 0.01
28	48.3 ± 0.08	72.2 ± 0.03	135 ± 0.00	146 ± 0.23	71.0 ± 0.01	112 ± 0.02
49	54.3 ± 0.03	88.5 ± 0.01	147 ± 0.00	155 ± 0.03	69.0 ± 0.01	119 ± 0.01
70	57.0 ± 0.04	92.3 ± 0.02	139 ± 0.00	173 ± 0.03	70.4 ± 0.10	109 ± 0.02
84	60.2 ± 0.02	94.1 ± 0.03	138 ± 0.03	118 ± 0.30	72.0 ± 0.09	104 ± 0.00
98	56.6 ± 0.08	84.5 ± 0.01	132 ± 0.03	81.3 ± 0.07	66.5 ± 0.03	71.0 ± 0.14
120	55.3 ± 0.03	95.6 ± 0.12	191 ± 0.32	110 ± 0.18	72.2 ± 0.11	97.6 ± 0.03
R_{TDS} DAT120/R_{TDS} DAT0	1.40	1.49	1.52	0.74	1.24	1.05
Mean Factor	1.24					

Notes:

[1] Replicate 2 of DAT28 is an outlier and was therefore excluded from the evaluation as reanalysis of the soil extracts gave the same results as the first analysis.

Isoflucypram was evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. Three different kinetic models were tested in order to determine the best-fit kinetic model (SFO, FOMC, and DFOP). The best-fit kinetic model was selected on the basis of the chi^2 scaled-error criterion and visual assessment of the goodness of fit. DT_{50} and DT_{90} values were calculated from the resulting kinetic parameters (Table 56).

The degradation of isoflucypram was best fit with SFO kinetics in all soils according to the lowest chi^2 error values and visual assessments.

Table 56 Summary of kinetic evaluation (for trigger values according to FOCUS) of the degradation of isoflucypram in soil under aerobic conditions

Soil	Kinetic Model[1,2]	DT_{50}	DT_{90}	Chi^2 Error	Visual Assessment
Laacher Hof AXXa	SFO	418	>1,000	1.2	Good
	FOMC	472	>1,000	1.3	Good
	DFOP	546	>1,000	1.3	Good
Hoefchen am Hohenseh 4a	SFO	688	>1,000	1.8	Good
	FOMC	688	>1,000	1.9	Good
	DFOP	688	>1,000	2.0	Good
Hanscheider Hof	SFO	875	>1,000	2.0	Moderate
	FOMC	>1,000	>1,000	1.9	Poor
	DFOP	>1,000	>1,000	1.4	Moderate
Dollendorf II	SFO	546	>1,000	2.7	Moderate
	FOMC	>1,000	>1,000	2.1	Poor
	DFOP	614	>1,000	1.6	Moderate
Wurmwiese	SFO	873	>1,000	1.2	Moderate
	FOMC	>1,000	>1,000	1.3	Poor
	DFOP	977	>1,000	0.7	Moderate
Frankenforst	SFO	531	>1,000	1.8	Moderate
	FOMC	>1,000	>1,000	1.6	Poor
	DFOP	567	>1,000	1.0	Moderate

Notes:

[1] SFO: single first order. FOMC: first order multi compartment. DFOP: double first order in parallel.
[2] Best fit written in bold.

Aerobic metabolism/degradation in soil: isoflucypram-carboxylic acid

Report No. S19-22674.

The Meeting received a study investigating the degradation of the isoflucypram-carboxylic acid in laboratory soil (Schwarzkopf, A.; 2020). The degradation of the test item was investigated in five different soils (Laacher Hof AXXa, Dollendorf II, Hoefchen Am Hohenseh 4a, Laacher Hof Wurmwiese, and Hanscheider Hof) under aerobic conditions at 20 °C in the dark. The five soils were chosen to represent diverse agricultural soils. The nominal application rate was 4.12 µg test item/100 g dry soil (actual application rate = 4.68 µg test item/100 g dry soil). This rate was selected based on 2× the value of expected test item in the field based on the application rate for isoflucypram to cereal grains (0.075 kg ai/ha) and maximum formation rate of the degradate based on radiolabelled aerobic soil metabolism studies (9.6 percent AR). This calculation accounted for metabolite weight conversion (1.07×) and assumed homogenous distribution in soil to a depth of 2.5 cm and a soil density of 1.5 g/cm^3. Samples were analysed for isoflucypram-carboxylic acid at various intervals for a period up to DAT120.

Table 57 Summary of soil characteristics

Soil	Soil ID	Source Location	Texture (USDA)	pH: $CaCl_2$ (0.01 M)	% organic content
Laacher Hof AXXa	AX	Monheim, Germany	Sandy loam	6.8	1.6
Dollendorf II	DD	Blankenheim, Germany	Loam	7.4	3.8
Hofchen am Hohenseh	HH	Burscheid, Germany	Silt loam	5.8	2.0
Wurmweise	WW	Monheim, Germany	Loam	5.1	2.0
Hanscheider Hof	HN	Burscheid, Germany	Silt loam	5.4	2.9

Soil in the field was collected from 0–20 cm, passed through a 2mm sieve, and shipped under ambient conditions. For the test systems, 100 g soil (dry weight basis) were used. The average soil moisture content was maintained at approximately 55 percent of the maximum water holding capacity (MWHC) over the entire period of the study.

The biological activity was checked directly after arrival at the test facility, at the start, after 30 days, after 60 days, and at the end of incubation. Soil microbial activity ranged from 25.1–104.7 mg C/100g DS. Flasks were closed with a PU plug with passive diffusion of oxygen. Duplicate test vessels were sampled on DAT0, 1, 3, 7, 14, 30, 60, and 120.

Samples were stored for a maximum of 108 days at -18 °C. The entire soil per flask was extracted three times with ACN:water (1:1) at ambient temperature, followed by an extraction with ACN:water (1:1) at 70°C, followed by an extraction with MeOH:water (1:1) at 50 °C. Each extraction step was followed with centrifugation and all extracts were combined. Internal standard was added and samples were analysed by LC-MS/MS. The LOQ was 0.060 ng/mL (0.0023 mg/kg). Calibration curves were established with coefficients of correlation >0.99. Method validation was performed successfully within the study. In addition, the extraction efficiency during the study was demonstrated by concurrent recovery samples. Successful recoveries were observed at the LOQ and at 22× LOQ.

Blank samples were taken from Laacher Hof AXXa and analysed alongside concurrent recovery samples. No residues above the limit of detection (LOD) were observed in black samples. Residues of isoflucypram-carboxylic acid during in dark soil is shown in Table 58.

Table 58 Residues of isoflucypram-carboxylic acid at multiple time points in dark soil at 20 °C

Soil Sample	DAT0	DAT1	DAT3	DAT7	DAT14	DAT30	DAT60	DAT120
Isoflucypram-carboxylic acid residue[1], mg eq/kg (% AR)								
Laacher Hof AXXa	0.0429 (91.7)	0.0421 (90.0)	0.0400 (85.5)	0.0358 (76.5)	0.0288 (61.5)	0.0204 (43.6)	0.0094 (20.1)	0.0024 (5.1)
Dollendorf II	0.0434 (92.7)	0.0410 (87.6)	0.0370 (79.1)	0.0318 (67.9)	0.0234 (50.0)	0.0141 (30.1)	0.0044 (9.5)	<LOQ
Hofchen am Hohenseh	0.0436 (93.2)	0.0392 (83.8)	0.0369 (78.8)	0.0331 (70.7)	0.0268 (57.3)	0.0196 (41.9)	0.0124 (26.5)	0.0081 (17.3)
Wurmweise	0.0435 (92.9)	0.0416 (88.9)	0.0361 (77.1)	0.0299 (63.9)	0.0249 (53.2)	0.0198 (42.3)	0.0154 (32.9)	0.0113 (24.1)
Hanscheider Hof	0.0438 (93.6)	0.0439 (93.8)	0.0362 (77.4)	0.0319 (68.2)	0.0275 (58.8)	0.0212 (45.3)	0.0152 (32.5)	0.0106 (22.6)

Notes:
[1] Average of 2-3 analyses.

Dissipation kinetics were modelled with the KinGUII software under the FOCUS Guideline. The degradation pathways evaluated were SFO, DFOP, FOMC, and Hockey Stick. The best representative model was chosen based on chi^2 error and goodness of fit (Table 59.

Table 59 Kinetic modelling for isoflucypram-carboxylic acid in dark soil at 20 °C

Soil Sample	Kinetic Model[1,2]	DT_{50} (Days)	DT_{90} (Days)	Chi^2 Error (%)	Visual Assessment
Laacher Hof AXXa	SFO	27.15	90.19	1.457	Good
	FOMC	26.5	95.07	1.334	Good
	DFOP	26.52	93.9	1.331	Good
	HS	27.15	90.19	1.679	Good
Dollendorf II	SFO	17.67	58.71	2.37	Good
	FOMC	16.78	65	1.797	Good
	DFOP	16.82	63.23	1.358	Good
	HS	17.18	61.5	1.584	Good
Hofchen am Hohenseh	SFO	33.03	109.7	7.411	Satisfactory
	FOMC	24.71	275.47	2.293	Good
	DFOP	24.48	197.3	2.756	Good
	HS	30.3	130.6	5.467	Satisfactory
Wurmweise	SFO	39.75	132	11.97	Satisfactory
	FOMC	21.08	>1,000	1.393	Good
	DFOP	20.17	265.4	1.327	Good
	HS	24.77	227.3	2.724	Good
Hanscheider Hof	SFO	39.42	130.9	10.07	Satisfactory
	FOMC	24.01	806.2	3.085	Good
	DFOP	25.16	218.3	3.478	Good
	HS	32.48	173.2	4.943	Good

Notes:
[1] SFO = single first order. FOMC = first order multicomponent. DFOP = double first order in parallel. HS = hockey stick.
[2] Best fit written with bold.

Terrestrial Field Dissipation

The Meeting received several terrestrial field dissipation studies evaluating the rate of degradation of isoflucypram and the rate of formation of the metabolite isoflucypram-carboxylic acid. Analytical reports included chromatograms showing defined symmetrical peaks.

Report No. 14-2750 (Germany)

Dissipation of isoflucypram was studied under aerobic field conditions (Heinemann, *et al.,* 2017). Field dissipation was examined in four replicate plots following application of an isoflucypram & prothioconazole EC 200 (50 g isoflucypram/ha) formulation to bare soil at one site in Burscheid, Germany. The application was made at a rate of 100 g isoflucypram/ha. Soil samples were collected prior to treatment and at various intervals after treatment up to a final sampling interval of DAT713. Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots did not have a slope and therefore run-off was not assessed. Soil characterisation samples were taken prior to application to a depth of 100 cm and sent under ambient conditions for analysis (Table 60).

Table 60 Summary of soil characteristics

Trial Number (Location)	Soil Depth (cm)	Type of Soil (USDA)	pH ($CaCl_2$)	pH (H_2O)	Organic Carbon (percent)
14-2750-01 (Bursheid, Germany)	0-30	Silt loam	5.3	5.4	1.0
	30-50	Silt loam	5.5	5.9	0.2
	50-75	Loam	5.7	6.0	0.1
	75-100	Sandy loam	5.9	6.2	0.0

At all sampling intervals, 16 soil cores were collected from the plots. Treated soil samples were collected to a depth of 10 cm on DAT0; to a depth of 40 cm on DAT3, 7, 13, 28, 70, and 91; and to a depth of 60 cm on DAT110, 160, 209, 370, 538, and 713. Control samples were taken on DAT0, 370, and 713.

Samples were frozen (-18 °C) within 24 hours of collection. Treated samples were stored for a maximum of 530 days. Each sample was separated into 10 cm horizons and homogenized. Samples were analysed according to Method 01432.

Two soil trays were laid out per plot for verification. Soil tray recoveries were in the range of 85-122 percent of the applied amount. Additionally, spray broth analysis resulted in recoveries of 93-102 percent of the intended amount. No quantifiable residues were observed in control samples. The results are shown in Table 61

Table 61 Rate of soil degradation of isoflucypram and formation of isoflucypram-carboxylic acid

Depth (cm)[1]	DAT												
	0	3	7	13	28	70	91	110	160	209	370	538	713
Average isoflucypram residue, g ai/ha (% applied dose)													
0-10	98.2 (98)	93.6 (94)	92.9 (93)	92.7 (93)	84.2 (84)	65.1 (65)	55.0 (55)	54.1 (54)	45.3 (45)	43.6 (44)	35.0 (35)	29.7 (30)	27.4 (27)
10-20	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ-1.60 (≤2)[2]	<LOQ-2.2 (≤2)[2]	<LOQ	< LOQ
Average isoflucypram-carboxylic acid residue, g ai/ha[3] (% applied dose)[3]													
0-10	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	<LOQ-1.66 (≤2)[2]	<LOQ-1.76 (≤2)[2]	< LOQ	<LOQ-1.86 (≤2)[2]	<LOQ-1.87 (≤2)[2]	< LOQ	< LOQ	< LOQ

Notes:

[1] Samples were also analysed for isoflucypram-carboxylic acid at segment depth 10-20 cm and isoflucypram and isoflucypram-carboxylic acid at segment depths 20-30, 30-40 cm, 40-50 cm, and 50-60 cm at select sampling intervals but were not reported due to residues <LOQ.

[2] Range from four replicate trials.

[3] Expressed in parent equivalents.

NA: Not analysed.

Report No. 14-2750 (United Kingdom).

Dissipation of isoflucypram was studied under aerobic field conditions (Heinemann, O., *et al.,*2017). Field dissipation was examined in four replicate plots following application of an isoflucypram & prothioconazole EC 200 (50 g isoflucypram/ha) formulation to bare soil at one site in Great Chishill, United Kingdom. The application was made at a rate of 100 g isoflucypram/ha. Soil samples were collected prior to treatment and at various intervals after treatment up to a final sampling interval of DAT749. Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots did not have a slope and therefore run-off was not assessed. Soil characterisation samples were taken prior to application to a depth of 100 cm and sent under ambient conditions for analysis (Table 62).

Table 62 Summary of soil characteristics

Trial Number (Location)	Soil Depth (cm)	Type of Soil (USDA)	pH ($CaCl_2$)	pH (H_2O)	Organic Carbon (percent)
14-2750-02 (Great Chishill, UK)	0-30	Clay loam	7.0	7.0	2.0
	30-50	Clay	7.5	7.7	0.2
	50-75	Clay	7.6	7.9	1.5
	75-100	Clay	7.6	7.8	1.9

At all sampling intervals, 16 soil cores were collected from the plots. Treated soil samples were collected to a depth of 10 cm on DAT0; to a depth of 40 cm on DAT4, 7, 14, 27, 67, and 111; and to a depth of 60 cm on DAT140, 168, 278, 402, 560, and 749. Control samples were taken on DAT -0 (prior to application) and DAT 402.

Samples were frozen (-18 °C) within 24 hours of collection. Treated samples were stored for a maximum of 597 days. Each sample was separated into 10 cm horizons and homogenized. Samples were analysed according to Method 01432. Two soil trays per plot were laid out for verification. Soil tray recoveries were in the range of 70–105 percent of the applied amount. Additionally, spray broth analysis resulted in recoveries of 91–102 percent of the intended amount. No quantifiable residues were observed in control samples (Table 63)

Table 63 Rate of soil degradation of isoflucypram and formation of isoflucypram-carboxylic acid

Depth (cm)[1]	DAT												
	0	4	7	14	27	67	111	140	168	278	402	560	749
Average isoflucypram residue, g ai/ha (% applied dose)													
0-10	96.8 (97)	92.0 (92)	96.7 (97)	82.9 (83)	80.7 (81)	70.8 (71)	78.3 (78)	39.2 (39)	42.2 (42)	35.7 (36)	31.2 (31)	28.3 (28)	19.1 (19)
10-20	NA	<LOQ-20.1 (≤20)[2]	<LOQ	<LOQ-3.71 (≤2)[2]	<LOQ	<LOQ	<LOQ-1.76 (≤2)[2]	<LOQ	<LOQ-2.05 (≤2)[2]	<LOQ	<LOQ-2.28 (≤2)[2]	<LOQ-4.09 (≤4)[2]	<LOQ
Average isoflucypram-carboxylic acid residue, g ai/ha (% applied dose)[3]													
0-10	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ-2.32 (≤2)[2]	<LOQ	<LOQ-1.65 (≤2)[2]	<LOQ-1.72 (≤2)[2]	<LOQ	<LOQ	<LOQ	<LOQ

Notes:

[1] Samples were also analysed for isoflucypram-carboxylic acid at segment depth 10-20 cm and isoflucypram and isoflucypram-carboxylic acid at segment depth 30-40 cm but not reported due to residues <LOQ.

[2] Range from four replicate trials.

[3] Expressed in parent equivalents.

NA: Not analysed.

Report No. 14-2750 (Spain).

Dissipation of isoflucypram was studied under aerobic field conditions (Heinemann, O., *et al.,*2017). Field dissipation was examined in four replicate plots following application of an isoflucypram & prothioconazole EC 200 (50 g isoflucypram/ha) formulation to bare soil at one site in Vilobi d'Onyar, Spain. The application was made at a rate of 100 g isoflucypram/ha. Soil samples were collected prior to treatment and at various intervals after treatment up to a final sampling interval of DAT714. Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots did not have a slope and therefore run-off was not assessed. Soil characterisation samples were taken prior to application to a depth of 100 cm and sent under ambient conditions for analysis (Table 64).

Table 64 Summary of Soil Characteristics

Trial Number (Location)	Soil Depth (cm)	Type of Soil (USDA)	pH ($CaCl_2$)	pH (H_2O)	Organic Carbon (percent)
14-2750-06 (17185 Vilobi d'Onyar, Spain)	0-30	Loam	5.8	6.1	0.7
	30-50	Sandy clay loam	6.0	6.3	0.4
	50-75	Sandy clay loam	6.5	6.9	0.1
	75-100	Sandy clay loam	6.5	6.8	0.1

At all sampling intervals, 16 soil cores were collected from the plots. Treated soil samples were collected to a depth of 10 cm on DAT0; to a depth of 40 cm on DAT3, 7, 15, 30, 57, and 99; and to a depth of 60 cm on DAT120, 158, 224, 345, 533, and 714. Control samples were taken on DAT -0 (prior to application), 345, and 714.

Samples were frozen (-18 °C) within 24 hours of collection. Treated samples were stored for a maximum of 595 days. Each sample was separated into 10 cm horizons and homogenized. Samples were analysed according to Method 01432. Two soil trays per plot were laid out for verification. Soil tray recoveries were in the range of 62–101 percent of the applied amount. Additionally, spray broth analysis resulted in recoveries of 87–102 percent of the intended amount. No quantifiable residues were observed in control samples (Table 65).

Table 65 rate of soil degradation of isoflucypram and formation of isoflucypram-carboxylic acid

Depth (cm)[1]	DAT												
	0	3	7	15	30	57	99	120	158	224	345	533	714
Average isoflucypram residue, g ai/ha (% applied dose)													
0-10	88.2 (88)	88.6 (89)	81.6 (82)	45.3 (45)	47.6 (48)	35.7 (36)	35.4 (35)	34.7 (35)	28.2 (28)	24.2 (24)	21.1 (21)	13.7 (14)	11.8 (12)
10-20	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ-13.2 (≤13)[2]	<LOQ
Average isoflucypram-carboxylic acid residue, g ai/ha (% applied dose)[3]													
0-10	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

Notes:

[1] Samples were also analysed for isoflucypram-carboxylic acid at segment depth 10-20 cm and isoflucypram and isoflucypram-carboxylic acid at segment depths 30-40 cm, 40-50 cm, and 50-60 cm.

[2] Range from four replicate trials.

[3] Expressed in parent equivalents.

NA: Not analysed.

Report No. 14-2750 (Italy).

Dissipation of isoflucypram was studied under aerobic field conditions (Heinemann, O., *et al.,* 2017). Field dissipation was examined in four replicate plots following application of an isoflucypram & prothioconazole EC 200 (50 g isoflucypram/ha) formulation to bare soil at one site in Albaro di Ronco all Adige, Italy. The application was made at a rate of 100 g isoflucypram/ha. Soil samples were collected prior to treatment and at various intervals after treatment up to a final sampling interval of DAT728. Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots did not have a slope and therefore run-off was not assessed. Soil characterisation samples were taken prior to application to a depth of 100 cm and sent under ambient conditions for analysis (Table 66).

Table 66 Summary of soil characteristics

Trial Number (Location)	Soil Depth (cm)	Type of Soil (USDA)	pH ($CaCl_2$)	pH (H_2O)	Organic Carbon (%)
14-2750-05 (Albaro di Ronco all Adige, Italy)	0-30	Clay	7.0	7.2	2.1
	30-50	Clay	7.1	7.3	1.6
	50-75	Clay	7.3	7.5	0.7
	75-100	Clay loam	7.2	7.6	0.6

At all sampling intervals, 16 soil cores were collected from the plots. Treated soil samples were collected to a depth of 10 cm on DAT0; to a depth of 40 cm on DAT3, 7, 14, 28, 62, and 89; and to a depth of 60 cm on DAT122, 157, 209, 369, 531, and 728. Control samples were taken on DAT -0 (prior to application), 359, and 728.

Samples were frozen (-18 °C) within 24 hours of collection. Treated samples were stored for a maximum of 272 days. Each sample was separated into 10 cm horizons and homogenized. Samples were analysed according to Method 01432.

Two soil trays per plot were laid out for verification. Soil tray recoveries were in the range of 83–99 percent of the applied amount. Additionally, spray broth analysis resulted in recoveries of 89–103 percent of the intended amount. No quantifiable residues were observed in control samples. The results are shown in Table 67.

Table 67 Rate of soil degradation of isoflucypram and formation of isoflucypram-carboxylic acid

Depth (cm)[1]	DAT												
	0	3	7	14	28	62	89	122	157	209	369	531	728
Average isoflucypram residue, g ai/ha (% applied dose)													
0-10	90.1 (90)	87.0 (87)	85.5 (86)	79.5 (80)	66.0 (66)	47.9 (48)	36.0 (36)	36.9 (37)	36.4 (37)	26.1 (26)	30.9 (31)	26.3 (26)	16.8 (17)
10-20	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ-4.29 (≤4)[2]	<LOQ-2.59 (<2)[2]	<LOQ-8.89 (≤9)[2]	<LOQ-2.47 (≤2)[2]	3.6 (4)[2]	<LOQ-4.05 (≤4)[2]	<LOQ-2.70 (<3)[2]
20-30	-	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ-3.8 (≤3)[2]	<LOQ	<LOQ	<LOQ	<LOQ-2.06 (≤2)[2]	<LOQ-6.05 (≤6)[2]	<LOQ-2.18 (≤2)[2]	<LOQ-3.96 (≤4)[2]
Average isoflucypram-carboxylic acid residue, g ai/ha (% applied dose)[3]													
0-10	ND	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1.8 (2)	2.4 (2)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

Notes:

[1] Samples were also analysed for isoflucypram-carboxylic acid at segment depths 10-20 cm and 20-30 cm and isoflucypram and isoflucypram-carboxylic acid at segment depths 30-40, 40-50, and 50-60 cm but were not reported due to residues <LOQ.

[2] Range from four replicate trials.

[3] Expressed in parent equivalents.

NA: Not analysed.

Report No. 14-2750 (South France).

Dissipation of isoflucypram was studied under aerobic field conditions (Heinemann, O., *et al.,* 2017). Field dissipation was examined in four replicate plots following application of an isoflucypram & prothioconazole EC 200 (50 g isoflucypram/ha) formulation to bare soil at one site in St. Etienne du Gres, France. The application was made at a rate of 100 g isoflucypram/ha. Soil samples were collected prior to treatment and at various intervals after treatment up to a final sampling interval of DAT205. Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots did not have a slope and therefore run-off was not assessed. Soil characterisation samples were taken prior to application to a depth of 100 cm and sent under ambient conditions for analysis (Table 68).

Table 68 Summary of soil characteristics

Trial Number (Location)	Soil Depth (cm)	Type of Soil (USDA)	pH ($CaCl_2$)	pH (H_2O)	Organic Carbon (%)
14-2750-04 (St. Etienne du Gres, France)[1]	0-30	Clay loam	7.5	7.8	2.3
	30-50	Silty clay loam	7.6	7.8	2.3
	50-75	Clay loam	7.6	7.9	1.8
	75-100	Clay	7.7	8.0	2.2

Notes:

[1] The trials from Northern and Southern France are separated by approximately 325 miles and application dates are separated by 35 days and are therefore considered independent.

At all sampling intervals, 16 soil cores were collected from the plots. Treated soil samples were collected to a depth of 10 cm on DAT0; to a depth of 40 cm on DAT3, 7, 14, 30, 58, and 92; and to a depth of 60 cm on DAT116, 151, and 205. Control samples were taken on DAT -0 (prior to application).

Samples were frozen (-18 °C) within 24 hours of collection. Treated samples were stored for a maximum of 291 days. Each sample was separated into 10 cm horizons and homogenized. Samples are analysed according to Method 01432.Two soil trays per plot were laid out for verification. Soil tray recoveries were in the range of 74–111 percent of the applied amount. Additionally, spray broth analysis resulted in recoveries of 87–101 percent of the intended amount. No quantifiable residues were observed in control samples. The results are shown in Table 69.

Table 69 Rate of soil degradation of isoflucypram and formation of isoflucypram-carboxylic acid

Depth (cm)[1]	DAT									
	0	3	7	14	30	58	92	116	151	205
Average isoflucypram residue (g ai/ha) [percent applied dose]										
0-10	90.9 (91)	80.8 (81)	66.8 (67)	54.3 (54)	23.5 (24)	11.0 (11)	6.3 (6)	5.9 (6)	7.0 (7)	3.9 (4)
10-20	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	LOQ-1.99 (≤2[2]	<LOQ	<LOQ	<LOQ
Average isoflucypram-carboxylic acid residue (g ai/ha) [percent applied dose][3]										
0-10	<LOQ	<LOQ	<LOQ-1.34 (≤1)[2]	2.2 (2)	3.6 (4)	3.5 (4)	2.5 (3)	2.5 (3)	<LOQ-1.41 (≤1)[2]	<LOQ

Notes:

[1] Samples were also analysed for isoflucypram-carboxylic acid at segment depth 10-20 cm and isoflucypram and isoflucypram-carboxylic acid at segment depth 20-30 cm but not reported due to residues <LOQ.

[2] Range from four replicate trials.

[3] Expressed in parent equivalents.

NA: Not analysed.

Report No. 14-2750 (North France).

Dissipation of isoflucypram was studied under aerobic field conditions (Heinemann, O., *et al.,*2017). Field dissipation was examined in four replicate plots following application of an isoflucypram & prothioconazole EC 200 (50 g isoflucypram/ha) formulation to bare soil at one site in Parcay Meslay, France. The application was made at a rate of 100 g isoflucypram/ha. Soil samples were collected prior to treatment and at various intervals after treatment up to a final sampling interval of DAT701. Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots did not have a slope and therefore run-off was not assessed. Soil characterisation samples were taken prior to application to a depth of 100 cm and sent under ambient conditions for analysis (Table 70).

Table 70 Summary of Soil Characteristics

Trial Number (Location)	Soil Depth (cm)	Type of Soil (USDA)	pH ($CaCl_2$)	pH (H_2O)	Organic Carbon (%)
14-2750-03 (37210 Parcay Meslay, France)[1]	0-30	Loam	5.9	6.3	1.2
	30-50	Loam	6.1	6.5	0.5
	50-75	Clay loam	6.5	6.9	0.2
	75-100	Clay loam	6.4	6.8	0.2

Notes:

[1] The trials from Northern and Southern France are separated by approximately 325 miles and application dates are separated by 35 days and are therefore considered independent.

At all sampling intervals, 16 soil cores were collected from the plots. Treated soil samples were collected to a depth of 10 cm on DAT0; to a depth of 40 cm on DAT3, 7, 14, 29, 63, and 88; and to a depth of 60 cm on DAT121, 143, 210, 357, 519, and 701. Control samples were taken on DAT -0 (prior to application) and 357.

Samples were frozen (-18 °C) within 24 hours of collection. Treated samples were stored for a maximum of 532 days. Each sample was separated into 10 cm horizons and homogenized. Samples were analysed according to Method 01432.

Two soil trays per plot were laid out for verification. Soil tray recoveries were in the range of 63–119 of the applied amount. Additionally, spray broth analysis resulted in recoveries of 84–95 percent of the intended amount. No quantifiable residues were observed in control samples. The results are shown in Table 71.

Table 71 Rate of soil degradation of isoflucypram and formation of isoflucypram-carboxylic acid

Depth (cm)[1]	DAT												
	0	3	7	14	29	63	88	121	143	210	357	519	701
Average isoflucypram residue (g ai/ha) [percent applied dose]													
0-10	88.1 (88)	85.1 (85)	82.5 (83)	79.2 (79)	64.6 (65)	51.9 (52)	50.6 (51)	44.3 (44)	45.2 (45)	41.7 (42)	34.8 (35)	32.5 (33)	30.1 (30)
10-20	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ-2.23 (≤2)[2]	<LOQ-7.74 (≤8)[2]	<LOQ	<LOQ	<LOQ	<LOQ-2.11 (≤2)[2]	<LOQ
Average isoflucypram-carboxylic acid residue (g ai/ha) [percent applied dose][3]													
0-10	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ-1.38 (≤1)[2]	<LOQ-1.21 (≤1)[2]	<LOQ-1.43 (≤1)[2]	<LOQ-1.06 (≤1)[2]	<LOQ	<LOQ-1.05 (≤1)[2]	<LOQ

Notes:

[1] Samples were also analysed for isoflucypram-carboxylic acid at segment depth 10-20 and isoflucypram and isoflucypram-carboxylic acid at segment depths 20-30 cm, 30-40 cm, and 50-60 cm.
[2] Range from four replicate trials.
[3] Expressed as parent equivalents.
NA: Not analysed.

Report No. 14-2750 (Kinetics).

Samples from trials 14-2750-01/02/03/04/05/06 were analysed for rate of degradation. Degradation kinetics were calculated using the FOCUS kinetics model. The model tested SFO, FOMC, and DFOP. DFOP was selected based on chi^2 and visual inspection. Degradation showed biphasic characteristics (Table 72).

Table 72 Kinetic modelling for isoflucypram in soil

Trial Number	Kinetic Model[1,2]	DT_{50} (Days)	DT_{90} (Days)	Visual Assessment[3]	Chi^2 Error (percent)
14-2750-01 Germany	SFO	241	801	Poor	11.98
	FOMC	155	>1,000	Good	3.502
	DFOP	143	>1,000	Good	2.543
14-2750-02 United Kingdom	SFO	239	795	Poor	12.9
	FOMC	181	>1,000	Good	10.41
	DFOP	177	>1,000	Good	10.68
14-2750-03 Parcay, Meslay France[3]	SFO	309	>1,000	Poor	14.04
	FOMC	153	>1,000	Good	2.814
	DFOP	147	>1,000	Good	2.236
14-2750-04 St. Etienne Du Gres, France[3]	SFO	18.0	59.7	Poor	8.801
	FOMC	16.1	77.8	Good	6.861
	DFOP	16.5	69.6	Good	4.537
14-2750-05 Italy	SFO	198	659	Poor	18.41
	FOMC	91.5	>1,000	Moderate	7.388
	DFOP	77.6	>1,000	Moderate	5.523
14-2750-06 Spain	SFO	107	354	Poor	23.39
	FOMC	35.3	>1,000	Good	11.59
	DFOP	25.7	812	Good	10.29

Notes:
[1] SFO: Single First Order. FOMC: First Order Multicompartment. DFOP: Double First Order in Parallel.
[2] Best fits highlighted in bold.
[3] The trials from Northern and Southern France are separated by approximately 325 miles and application dates are separated by 35 days and are therefore considered independent.

Report No. MELNN203.

Dissipation of isoflucypram and isoflucypram-carboxylic acid were studied under aerobic field conditions (Harbin, A., 2019). Field dissipation was examined following broadcast applications of an isoflucypram EC 50 (50 g ai/L) formulation to bare soil at one site in Rosthern, Saskatchewan, Canada. The application was made at a nominal application rate of 150 g ai/ha (actual rate = 148 g ai/ha). Soil samples were collected prior to treatment and at various intervals after treatment up to a final sampling interval of DAT705. Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots did not have a slope and therefore run-off was not assessed. Soil characterisation samples were taken for control and treated plots to a depth of 105 cm, sectioned into 15 cm segments (Table 73).

Table 73 Soil properties for study MELNN203

Property	0-15	15-30	30-45	45-60	60-75	75-90	90-105
Textural Classification	Loam	Clay Loam	Clay Loam	Clay Loam	Loam	Clay Loam	Clay Loam
% Sand	28	28	22	30	44	42	28
% Silt	48	44	46	42	30	28	34
%Clay	24	28	32	28	26	30	38
Bulk density, disturbed (g/cc)	1.07	1.11	1.11	1.09	1.12	1.13	1.10
pH (1:1 soil:water)	5.6	7.3	7.9	8.1	8.4	8.4	8.5
Moisture (% at 1/3 bar)	28.1	24.9	25.5	23.6	19.9	21.9	25.7
Organic matter (%)	5.6	2.4	1.6	1.3	0.9	0.7	0.6
Cation exchange capacity (meq/100 g)	25.0	23.9	30.3	33.6	32.0	34.4	34.1

Soil cores were collected from the treated plots on 0-DAT to a depth of 15 cm and at all other intervals (DAT -2 [prior to application], 6, 12, 27, 60, 96, 120, 336, 371, 434, 490, and 705) to a depth of 105 cm, which were subsequently split into 15 cm horizons. Samples for control analysis were collected at the control plot on DAT -2 (prior to application). Samples for the control plot were also collected on DAT0, 120, and 371, fortified in the field, shipped frozen to the analytical laboratory, and analysed for isoflucypram at all three intervals and for isoflucypram-carboxylic acid on DAT371.

Soil was frozen after sampling. Treated samples were stored for a maximum of 686 days. Analysis for verification pads was accomplished through Method LN-001-F15-01. Soil pans and treated samples were analysed according to Method 01432. An average of 91.3 percent (n=6) and 90.2 percent (n=3) of the expected active ingredient was recovered from solvent saturation pad monitors and soil pan application monitors, respectively. It is noted that 0.15 percent AR was recovered in the control for pan recovery. For both verification pads and pans, the soil was allowed to dry after application and shipped frozen for analysis. No quantifiable residues were observed in control samples. The results are shown in Table 74.

Table 74 Field dissipation of isoflucypram and isoflucypram-carboxylic acid

Depth (cm)[1]	DAT												
Average isoflucypram residue, g ai/ha (% applied dose)[2]													
	-2	0	6	12	27	60	96	120	336	371	434	490	705
0-15	<LOQ	79.3 (52.9)	59.7 (39.8)	69.8 (46.5)	59.2 (39.5)	55.5 (37.0)	64.0 (42.7)	31.5 (21.0)	24.6 (16.4)	28.9 (19.3)	25.2 (16.8)	31.3 (20.9)	18.8 (12.5)
15-30	<LOQ	NA	<LOQ	<LOQ	<LOQ	15.6 (10.4)	4.63 (3.1)	<LOQ	<LOQ	<LOQ	1.95 (1.3)	<LOQ	<LOQ
Average isoflucypram-carboxylic acid, g ai/ha (% applied dose)[2]													
	-2	0	6	12	27	60	96	120	336	371	434	490	705
0-15	<LOQ	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

Notes:

[1] Samples were also analysed for isoflucypram-carboxylic acid at segment depth 15-30 cm and isoflucypram and isoflucypram-carboxylic acid at segment depths 30-45 and 45-60 cm but results were not reported due to residues <LOQ.

[2] Average of three samples.

The kinetics of isoflucypram degradation was evaluated using kinetic modelling (KinGUII2.1 under FOCUS guidance). Goodness of fit was determined by chi^2, visual assessment, and R^2. The FOMC model best fit the data. The DT_{50} value was 260 days, the DT_{75} was 776 days, and the DT_{90} was >1,000 days. Leaching was not a significant degradation pathway as residues were mainly found in the top 15 cm throughout the course of the study (Table 75).

Table 75 Kinetic modelling for isoflucypram in soil

Model[1,2]	DT_{50}	DT_{75}	DT_{90}	Chi^2	R^2	Visual Assessment
SFO	312	624	>1,000	15.87	0.497	Satisfactory
DFOP	254	586	>1,000	16.31	0.5096	Satisfactory
FOMC	259.5	776	>1,000	15.71[3]	0.5079[3]	Satisfactory

Notes:

[1] SFO: Single First Order. FOMC: First Order Multicompartment. DFOP: Double First Order in Parallel.

[2] Best fit highlighted in bold.

[3] Regarded as unreliable due to high Chi^2 and low R^2.

Report No. AUS-0032.

Dissipation of isoflucypram and isoflucypram-carboxylic acid were studied under aerobic field conditions (White, J., *et al.,*2018). Field dissipation was examined following broadcast application of an isoflucypram EC 50 (50 g ai/L) formulation to bare soil at one site in Richland, Iowa, United States. The application was made at a nominal/actual application rate of 150 g ai/ha. Soil samples were collected prior to treatment and at various intervals after treatment up to a final sampling interval of DAT518. Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots had a 0.5 percent slope and therefore run-off was not assessed. Disturbed soil characterisation samples were taken from treated plots to a depth of 120 cm, sectioned into 15 cm segments, and combined. Two disturbed soil cores were taken, one for bulk density and water holding capacity and one for morphology. Soil characteristics are shown in Table 76.

Table 76 Soil properties for Study AUS-0032

Property	0-15	15-30	30-45	45-60	60-75	75-90	90-105	105-120
Textural Classification	Silty Clay Loam	Silty Clay Loam	Silty Clay Loam	Clay Loam	Clay Loam	Clay Loam	Clay Loam	Silty Clay Loam
% Sand	13	15	17	27	33	27	21	18
% Silt	54	52	48	38	32	36	42	47
% Clay	33	33	35	35	35	37	37	35
Bulk density – disturbed (g/cm^3)	1.02	1.07	1.04	1.09	1.06	1.11	1.10	1.08
pH (1:1 soil:water)	6.7	6.5	6.2	5.9	5.9	6.0	6.2	6.4
pH: (0.01 M $CaCl_2$)	6.4	6.1	5.7	5.4	5.4	5.5	5.7	5.9
Organic matter (%)	3.6	3.0	2.1	1.03	0.67	0.54	0.40	0.36
Cation exchange capacity (meq/100 g)	18.9	18.8	20.2	26.4	29.5	28.5	25.9	26.2
Water holding capacity at 1/3 Bar (%)	30.6	31.7	31.4	35.5	37.8	38.2	37.7	35.7
Water holding capacity at 15 Bar (%)	14.9	16.6	18.7	22.7	27.9	27.0	25.0	22.0

Soil cores were collected from the treated plots on 0-DAT to a depth of 15 cm and at all other intervals (DAT -20 [prior to application], 7, 15, 28, 59, 85, 171, 272, 346, 518) to a depth of 120 cm, which were subsequently split into 15 cm horizons. Samples for control/fortified analysis were collected at the control plot on DAT -20 (prior to application), DAT0, and DAT 171.

Soil was frozen after sampling. Treated samples were stored for a maximum of 639 days. Analysis for verification pads was accomplished through Method LN-001-F15-01. Soil pans and treated samples were analysed according to Method 01432. An average of 107 percent (n=3) and 81.7 percent

(n=3) of the expected active ingredient was recovered from solvent saturation pad monitors and soil pan application monitors, respectively. The soil was allowed to dry after application before analysis. There were no quantifiable residues observed in control samples. The results are shown in Table 77.

Table 77 Field dissipation of isoflucypram and isoflucypram-carboxylic acid

Depth (cm)[1]	DAT										
	-20	0	7	15	28	59	85	171	272	346	518
Average isoflucypram residue, g ai/ha (% applied dose)[2]											
0-15	<LOQ	134.02 (89.35)	84.20 (56.13)	47.72 (31.81)	30.25 (20.17)	29.58 (19.72)	32.36 (21.58)	13.31 (8.87)	18.96 (12.64)	11.88 (7.92)	12.01 (8.00)
Isoflucypram-carboxylic acid, g ai/ha (percent applied dose)[2]											
0-15	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

Notes:

[1] Samples were also analysed for isoflucypram and isoflucypram-carboxylic acid at segment depths 15-30 cm, 30-45 cm, 45-60 cm, 60-75, 75-90, 90-105, and 105-120 cm but results were not reported due to residues <LOQ.

[2] Average of three samples.

The kinetics of isoflucypram degradation was evaluated using kinetic modelling (KinGUII2.1 under FOCUS guidance). Goodness of fit was determined by chi^2, visual assessment, and R^2. The FOMC model best fit the data. The DT_{50} value was 9.2 days, the DT_{75} was 46.2 days, and the DT_{90} was >310.1 days. Leaching was not a significant degradation pathway as residues were mainly found in the top 15 cm throughout the course of the study (Table 78).

Table 78 Kinetic modelling for isoflucypram in soil

Model[1,2]	DT_{50}	DT_{75}	DT_{90}	Chi^2	R^2	Visual Assessment
SFO	13.97	27.94	46.4	31.76	0.6522	Satisfactory
DFOP	9.647	30.38	387.9	9.71	0.697	Good
FOMC	9.158	46.25	310.1	13.41	0.6847	Good

Notes:

[1] SFO: Single First Order. FOMC: First Order Multicompartment. DFOP: Double First Order in Parallel.

[2] Best fit highlighted in bold.

Report No. AUS-0031.

Dissipation of isoflucypram and isoflucypram-carboxylic acid were studied under aerobic field conditions (White, J., *et al.,* 2018). Field dissipation was examined following broadcast application of an isoflucypram EC 50 (50 g ai/L) formulation to bare soil at one site in Fresno, California, United States. The application was made at a nominal application rate of 150 g ai/ha (actual application rate = 151 g ai/ha). Soil samples were collected prior to treatment and at various intervals after treatment up to a final sampling interval of DAT705. Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots had a slope of <1 percent and therefore run-off was not assessed. Disturbed soil characterisation samples were taken from treated plots to a depth of 120 cm, sectioned into 15 cm segments, and combined. Two disturbed soil cores were taken, one for bulk density and water holding capacity and one for morphology. Soil characteristics are shown in Table 79.

Table 79 Soil properties for Study AUS 0031

	Depth							
	0-15	15-30	30-45	45-60	60-75	75-90	90-105	105-120
Textural Classification	Sandy Loam	Sandy Loam	Sandy Loam	Sandy Loam	Sandy Loam	Sandy Loam	Sandy Loam	Sandy Loam
% Sand	60	58	60	60	58	64	68	72
% Silt	25	27	27	27	31	29	27	23
%Clay	15	15	13	13	11	7	5	5
Bulk density – disturbed (g/cm^3)	1.15	1.20	1.26	1.27	1.27	1.29	1.34	1.34
pH: (1:1 soil:water)	7.4	7.5	8.1	8.3	8.3	8.2	8.0	8.0
pH: (0.01M $CaCl_2$)	7.2	7.3	7.7	7.8	7.7	7.6	7.5	7.5
Organic matter (%)	1.12	0.85	0.36	0.36	0.22	0.09	0.22	0.09
Cation exchange capacity (meq/100 g)	13.3	14.1	14.4	15.9	15.8	15.7	15.5	14.9
Water holding capacity at 1/3 Bar (%)	14.8	14.9	15.2	15.4	15.9	16.3	14.7	14.0
Water holding capacity at 15 Bar (%)	7.4	7.1	7.0	7.0	6.9	6.9	6.5	6.3

Soil cores were collected from the treated plots on 0-DAT to a depth of 15 cm and at all other intervals (DAT -1 [prior to application], 0, 7, 16, 28, 62, 90, 185, 275, 365, 547, and 705) to a depth of 120 cm, which were subsequently split into 15 cm horizons. Samples for control/fortified analysis were collected at the control plot on DAT -1 (prior to application), DAT 0, and 185.

Soil was frozen after sampling. Treated samples were stored for a maximum of 533 days. Analysis for verification pads was accomplished through Method LN-001-F15-01. Soil pans and treated samples were analysed according to Method 01432. An average of 99.6 percent (n=3) and 65.3 percent (n=3) of the expected active ingredient was recovered from solvent saturation pad monitors and soil pan application monitors, respectively. Soil was allowed to dry before analysed.There were no quantifiable residues in control samples. The results are shown in Table 80.

Table 80 Field dissipation of isoflucypram and isoflucypram-carboxylic acid

Depth (cm)[1]	DAT										
	-1	0	7	16	28	62	90	185	275	365	547
Average isoflucypram residues, g ai/ha (% applied dose)[2]											
0-15	<LOQ	90.97 (60.65)	52.08 (34.72)	52.82 (35.21)	41.07 (27.38)	22.91 (15.27)	21.95 (14.63)	21.56 (14.37)	14.56 (9.71)	16.02 (10.68)	7.60 (5.07)
15-30	<LOQ	NA	2.99 (1.99)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1.82 (1.21)	<LOQ
Average isoflucypram-carboxylic acid residues, g ai/ha (% applied dose)[2,3]											
0-15	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	2.4 (1.63)	1.94 (1.29)	2.41 (1.61)	<LOQ	<LOQ	<LOQ

Notes:

[1] Samples were also analysed for isoflucypram-carboxylic acid at segment depths 15-30 cm and isoflucypram and isoflucypram-carboxylic acid at segment depths 30-45, 45-60, 60-75, 75-90, and 90-105 cm but results were not reported due to residues <LOQ.

[2] Average of three samples.

[3] Expressed in parent equivalents.

NA = Not analysed.

The kinetics of isoflucypram degradation was evaluated using kinetic modelling (KinGUII2.1 under FOCUS guidance). Goodness of fit was determined by chi^2, visual assessment, and R^2. The FOMC model best fit the data. The DT_{50} value was 19.34 days, the DT_{75} was 124.1 days, and the DT_{90} was >1,000 days. Leaching was not a significant degradation pathway as residues were mainly found in the top 15 cm throughout the course of the study (Table 81).

Table 81 Kinetic Modelling for Isoflucypram in Soil

Model[1,2]	DT_{50}	DT_{75}	DT_{90}	Chi^2	R^2	Visual Assessment
SFO	49.90	99.81	165.8	27.05	0.5749	Satisfactory
DFOP	21.33	162.3	666	11.25	0.6557	Good
FOMC	19.34	124.1	>1,000	9.331	0.6612	Good

Notes:
[1] SFO: Single First Order. FOMC: First Order Multicompartment. DFOP: Double First Order in Parallel.
[2] Best fit highlighted in bold.

Report No. AUS-0030.

Dissipation of isoflucypram and isoflucypram-carboxylic acid were studied under aerobic field conditions (White, J., *et al.,* 2018). Field dissipation was examined following broadcast application of an isoflucypram EC 50 (50 g ai/L) formulation to bare soil at one site in Chula, Georgia, United States. The application was made at a nominal application rate of 150 g ai/ha (actual application rate = 149 g ai/ha). Soil samples were collected prior to treatment and at various intervals after treatment up to a final sampling interval of DAT636. Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots had a slope of ~2 percent and therefore run-off was not assessed. Disturbed soil characterisation samples were taken from treated plots to a depth of 120 cm, sectioned into 15 cm segments, and combined. Two disturbed soil cores were taken, one for bulk density and water holding capacity and one for morphology. Soil characterisation is shown in Table 82.

Table 82 Soil properties for Study AUS 0030

	Depth							
	0-15	15-30	30-45	45-60	60-75	75-90	90-105	105-120
Textural Classification	Loamy Sand	Sandy Loam	Sandy Clay Loam	Sandy Clay Loam	Sandy Clay	Sandy Clay	Sandy Clay	Sandy Clay Loam
% Sand	86	80	64	64	58	60	56	60
% Silt	8	8	4	4	6	4	4	6
% Clay	6	12	32	32	36	36	40	34
Bulk density disturbed (g/cm^3)	1.36	1.33	1.14	1.13	1.11	1.11	1.09	1.09
pH: (1:1 soil:water)	6.5	6.5	6.8	6.6	5.7	4.8	4.5	4.5
pH: (0.01 M $CaCl_2$)	5.9	6.0	6.5	6.4	5.5	4.5	4.2	4.1
Organic matter (%)	0.87	0.73	0.41	0.32	0.32	0.05	0.18	0.14
Cation exchange capacity (meq/100 g)	4.1	4.8	5.8	5.5	6.7	7.0	6.8	6.4
Water holding capacity at 1/3 bar (%)	5.8	8.9	15.7	16.9	19.6	18.5	19.9	17.4
Water holding capacity at 15 bar (%)	3.8	5.7	12.2	13.6	15.1	14.9	15.6	13.8

Soil cores were collected from the treated plots on 0-DAT to a depth of 15 cm and at all other intervals (DAT -1 [prior to application], 0, 7, 14, 28, 58, 90, 178, 269, 365, 402, 491, 539, 588, and 636) to a depth of 120 cm, which were subsequently split into 15 cm horizons. Samples for control/fortified analysis were collected at the control plot on DAT -1 (prior to application), 0, and 178 DAT.

Soil was frozen after sampling. Treated samples were stored for a maximum of 519 days. Analysis for verification pads was accomplished through Method LN-001-F15-01. Soil pans and treated samples were analysed according to Method 01432. An average of 123.8 percent (n=3) and 92.7 percent (n=3) of the expected active ingredient was recovered from solvent saturation pad monitors and soil pan application monitors, respectively. For both verification pads and pans, the soil was allowed to dry before analysed. There were no quantifiable residues in control samples. The results are shown in Table 83

Table 83 Field dissipation of isoflucypram and isoflucypram-carboxylic acid

	DAT							
Isoflucypram average residues, g ai/ha (% applied dose)[1]								
Depth(cm)[2]	-1	0	7	14	28	58	90	178
0-15	<LOQ	126.54 (84.36)	95.00 (63.33)	84.42 (56.28)	71.79 (47.86)	38.17 (25.45)	31.24 (20.83)	30.26 (20.17)
15-30	<LOQ	NA	3.97 (2.65)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	269	365	402	491	539	588	636	-
0-15	18.07 (12.05)	22.59 (15.06)	8.22 (5.48)	9.88 (6.59)	11.74 (7.83)	11.32 (7.55)	9.84 (5.48)	-
15-30	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	-
Average isoflucypram-carboxylic acid residues, g ai/ha (percent applied dose[1]								
	-1	0	7	14	28	58	90	178
0-15	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	269	365	402	491	539	588	636	-
0-15	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	-

Notes:

[1] Samples were also analysed for isoflucypram-carboxylic acid at segment depths 15-30 cm and isoflucypram and isoflucypram-carboxylic acid at segment depths 30-45 cm and 45-60 cm but results were not reported due to residues <LOQ.

[2] Average of three samples.

NA = Not analysed.

The kinetics of isoflucypram degradation was evaluated using kinetic modelling (KinGUII2.1 under FOCUS guidance). Goodness of fit was determined by chi^2, visual assessment, and R^2. The FOMC model best fit the data. The DT_{50} value was 31 days, the DT_{75} was 114 days, and the DT_{90} was 471 days. Leaching was not a significant degradation pathway as residues were mainly found in the top 15 cm throughout the course of the study (Table 84).

Table 84 Kinetic modelling for isoflucypram in soil

Model[1,2]	DT_{50}	DT_{75}	DT_{90}	Chi^2	R^2	Visual Assessment
SFO	50.12	100.2	166.5	24.40	0.7964	Satisfactory
DFOP	30.96	108.3	495.6	8.793	0.8289	Good
FOMC	30.78	113.8	470.8	9.311	0.8267	Good

Notes:

[1] SFO: Single First Order. FOMC: First Order Multicompartment. DFOP: Double First Order in Parallel.

[2] Best fit highlighted in bold.

Report No. AUS-0034.

Dissipation of isoflucypram and isoflucypram-carboxylic acid were studied under aerobic field conditions (White, J., *et al.,* 2018). Field dissipation was examined following two broadcast applications of an isoflucypram EC 50 (50 g ai/L) formulation to bare soil and turf grass at one site in Chula, Georgia, United States. The first application was made at a nominal application rate of 350 g ai/ha (actual application rate = 347 g ai/ha). The second application was made 120 days after the first application at a nominal application rate of 150 g ai/ha (actual application rate = 154–155 g ai/ha). Soil samples were collected prior to the first treatment and at various intervals following the first and second applications. The final sampling interval was DAT1-635 (DAT2-515). Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots had a slope of ~2 percent and therefore run-off was not assessed. Disturbed soil characterisation samples were taken from both treated plots to a depth of 120 cm, sectioned into 15 cm segments, and combined. Two disturbed soil cores were taken from each treated plot, one for bulk density and water holding capacity and one for morphology. Soil characterisation is shown in Table 85

Table 85 Soil properties for Study AUS 0034

	Depth							
Property	0-15	15-30	30-45	45-60	60-75	75-90	90-105	105-120
Bare soil								
Textural classification	Loamy sand	Sandy loam	Sandy clay loam	Sandy clay loam	Sandy clay	Sandy clay loam	Sandy clay loam	Sandy clay
% Sand	84	72	60	62	58	62	62	58
% Silt	8	8	8	4	6	4	4	6
% Clay	8	20	32	34	36	34	34	35
Bulk density – disturbed (g/cm^3)	1.37	1.25	1.15	1.14	1.12	1.14	1.14	1.09
pH: (0.01 M $CaCl_2$)	6.2	6.2	6.3	5.5	4.7	4.3	4.2	4.0
Organic matter (%)	0.78	0.55	0.37	0.18	0.18	0.05	0.09	0.00
Cation exchange capacity (meq/100 g)	4.7	5.0	5.7	6.2	6.7	6.1	5.9	5.4
Water holding capacity at 1/3 bar (%)	7.7	13.1	20.4	21.6	23.2	21.1	19.7	20.7
Water holding capacity at 15 bar (%)	4.4	8.2	13.2	14.6	15.7	14.6	13.7	13.8
Turf plot								
Textural classification	Loamy sand	Loamy sand	Sandy clay loam	Sandy clay loam	Sandy clay loam	Sandy clay loam	Sandy clay loam	Sandy clay loam
% Sand	88	84	68	70	66	62	66	68
% Silt	4	6	6	6	6	6	8	4
% Clay	8	10	26	24	28	32	26	28
Bulk density – disturbed (g/cm^3)	1.42	1.38	1.21	1.20	1.19	1.16	1.17	1.13
pH (1:1 soil:water)	6.4	6.4	6.6	6.5	5.9	5.4	5.0	4.7
pH (0.01M $CaCl_2$)	6.0	5.9	6.4	6.3	5.8	5.2	4.7	4.4
Organic matter (%)	0.83	0.73	0.32	0.41	0.23	0.28	0.09	0.00
Cation exchange capacity (meq/100g)	4.4	4.5	5.0	4.9	5.3	5.5	5.7	6.3
Water holding capacity at 1/3 bar (%)	6.4	7.6	14.5	16.1	15.8	16.7	15.3	16.0
Water holding capacity at 15 bar (%)	3.5	4.8	10.2	11.1	12.1	12.6	11.6	12.1

Soil cores were collected from the treated plots on DAT1-0 and 120 (i.e., DAT2-0) to a depth of 15 cm. Samples at all other intervals (DAT1 -5 [prior to application], 0, 7, 14, 29, 62, 90, 119, 120, 127, 134, 148, 181, 211, 244, 301, 391, 401, 490, 566, 587, and 635) were collected to a depth of 120 cm. Samples from the bare soil plot were split into 15 cm horizons. Samples from turf plot were split into a 0-7.5 cm horizon, a 7.5-15 cm horizon, and 15 cm horizons for the remainder of the sample. Samples for control analysis were collected at the control plot on DAT1 -5 (prior to application), 0, and 120.

Soil was frozen after sampling. Treated samples were stored for a maximum of 514 days. Analysis for verification pads was accomplished through Method LN-001-F15-01. Soil pans and treated samples were analysed according to Method 01432.

The theoretical application rate for the spray solution calibration was 99.3 percent for the first application and 103.3 percent for the second application. For application pads, an average of 101.3 percent (n=6) and 93.5 percent (n=6) of the expected active ingredient was recovered from both treated plots following application one and two, respectively. For application pans, an average of 95.9 percent (n=6) and 95.0 percent (n=6) of the expected active ingredient was recovered from both treated plots following application one and two, respectively. Soil was allowed to dry after application before analysed. There were no quantifiable residues in control samples. The results are shown in Tables 86 and 87.

Table 86 Field dissipation of isoflucypram in bare soil

Depth (cm)[1]	DAT1 (DAT2)										
	-5 (-125)	0 (-120)	7 (-113)	14 (-106)	29 (-91)	62 (-58)	90 (-30)	119 (-1)	120 (0)	127 (7)	134 (14)
	Average isoflucypram residues, g ai/ha) (% applied dose)[2]										
0-15	<LOQ	218.1 (62.3)	165.0 (47.1)	172.1 (49.2)	155.7 (44.5)	82.9 (23.7)	30.3 (8.6)	69.6 (19.9)	182.8 (36.6)	186.5 (37.3)	164.1 (32.8)
15-30	<LOQ	NA	3.5 (1.0)	<LOQ	<LOQ	5.1 (1.4)	<LOQ	<LOQ	NA	3.7 (0.7)	<LOQ
	148 (28)	181 (61)	211 (91)	244 (124)	301 (181)	391 (271)	401 (281)	490 (370)	566 (446)	587 (467)	635 (515)
0-15	125.0 (25.0)	97.6 (19.5)	60.5 (12.1)	60.7 (12.1)	76.3 (15.3)	45.0 (9.0)	42.1 (8.4)	47.2 (9.4)	37.0 (7.4)	49.2 (9.8)	34.7 (6.9)
15-30	<LOQ	ND	<LOQ	<LOQ	<LOQ	<LOQ	4.2 (0.8)	5.2 (1.0)	<LOQ	<LOQ	<LOQ
	Average isoflucypram-carboxylic Acid residues, g ai/ha (% applied dose)[2]										
0-15	ND	ND	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	5.9 (1.2)	7.0 (1.4)
	148 (28)	181 (61)	211 (91)	244 (124)	301 (181)	391 (271)	401 (281)	490 (370)	566 (446)	587 (467)	635 (515)
0-15	7.4 (1.5)	1.0 (0.2)	4.0 (0.8)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

Notes:

[1] Average of three samples.

[2] Samples were also analysed for isoflucypram-carboxylic acid at segment depths 15-30 cm, 30-45 cm, 45-60 cm, 60-75 cm, 75-90 cm, and 105-120 cm but results are not reported due to residues below the LOQ.

[3] Expressed in parent equivalents.

NA = Not analysed. ND = Not detected.

Table 87 Field dissipation of isoflucypram and isoflucypram-carboxylic acid in turf plots

Depth (cm)[1]	DAT1 (DAT2)										
	-5 (-125)	0 (-120)	7 (-113)	14 (-106)	29 (-91)	62 (-58)	90 (-30)	119 (-1)	120 (0)	127 (7)	134 (14)
	Average isoflucypram, g ai/ha (% applied dose)[2]										
0-7.5	<LOQ	185.4 (53.0)	139.2 (39.8)	101.8 (29.1)	79.3 (22.6)	33.5 (9.6)	11.8 (3.4)	10.9 (3.1)	107.6 (21.5)	76.2 (15.2)	64.3 (12.9)
7.5-15	<LOQ	11.8 (3.4)	2.5 (0.7)	4.7 (1.3)	1.7 (0.5)	<LOQ	<LOQ	1.4 (0.4)	5.0 (1.0)	3.2 (0.6)	3.3 (0.7)
	148 (28)	181 (61)	211 (91)	244 (124)	301 (181)	391 (271)	401 (281)	490 (370)	566 (446)	587 (467)	635 (515)
0-7.5	41.0 (8.2)	37.1 (7.4)	30.1 (6.0)	23.3 (4.7)	24.9 (5.0)	11.1 (2.2)	7.6 (1.5)	4.1 (0.8)	3.2 (0.6)	3.8 (0.8)	3.6 (0.7)
7.5-15	3.0 (0.6)	1.7 (0.3)	<LOQ	<LOQ	1.4 (0.3)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	Average isoflucypram carboxylic acid (g ai/ha, % applied dose)[2]										
	-5 (-125)	0 (-120)	7 (-113)	14 (-106)	29 (-91)	62 (-58)	90 (-30)	119 (-1)	120 (0)	127 (7)	134 (14)
0-7.5	ND	ND	2.3 (0.7)	3.3 (0.9)	8.0 (2.3)	6.8 (1.9)	1.6 (0.5)	1.7 (0.5)	1.9 (0.4)	5.1 (1.0)	5.8 (1.2)
7.5-15	ND	ND	ND	ND	1.5 (0.4)	1.6 (0.5)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	148 (28)	181 (61)	211 (91)	244 (124)	301 (181)	391 (271)	401 (281)	490 (370)	566 (446)	587 (467)	635 (515)
0-7.5	5.6 (1.1)	3.3 (0.7)	1.6 (0.3)	1.4 (0.3)	1.6 (0.3)	1.0 (0.2)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
7.5-15	<LOQ	1.2 (0.2)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	ND	<LOQ	ND	<LOQ

Notes:

[1] Average of three samples.

[2] Samples were also analysed for isoflucypram-carboxylic acid at segment depths 15-30 cm, 30-45 cm, and 45-60 cm, but results are not reported due to residues below the LOQ.

[3] Expressed in parent equivalents.

ND= Not detected

The kinetics of isoflucypram degradation was evaluated in both soil and turf grass using kinetic modelling (KinGUII2.1 under FOCUS guidance). Goodness of fit was determined by chi^2, visual assessment, and R^2. For the bare soil plot, SFO most closely matched the first application and FOMC most closely matched the second application. For the turf plot, SFO most closely matched following the first application and DFOP most closely matched following the second application. Leaching was not a significant degradation pathway as residues were mainly found in the top 15 cm throughout the course of the study (Table 88).

Table 88 Kinetic modelling for isoflucypram in soil and turf

Model[1,2]	DT_{50}	DT_{75}	DT_{90}	Chi^2	R^2	Visual Assessment
Isoflucypram – bare ground soil, first application						
SFO	52.12	104.2	173.1	11.79	0.79	Good
DFOP	48.39	104.1	177.7	13.49	0.80	Good
FOMC	49.35	108.1	203.1	12.61	0.79	Good
Isoflucypram – bare ground soil, second application						
SFO	153.1	306.0	508.7	22.04	0.74	Satisfactory
DFOP	55.17	339.2	>1,000	9.471	0.88	Good
FOMC	62.58	290.2	>1,000	11.10	0.86	Good

Model[1,2]	DT_{50}	DT_{75}	DT_{90}	Chi^2	R^2	Visual Assessment
Isoflucypram – turf soil, first application						
SFO	22.41	44.82	74.44	7.945	0.97	Good
DFOP	18.85	47.50	85.74	3.724	0.98	Good
FOMC	18.59	43.80	91.39	5.579	0.98	Good
Isoflucypram – turf soil, second application						
SFO	58.33	116.7	193.8	25.16	0.88	Satisfactory
DFOP	19.28	112.8	288.0	7.213	0.96	Good
FOMC	22.49	82.14	334.1	12.13	0.94	Good

Notes:

[1] SFO: Single First Order. FOMC: First Order Multicompartment. DFOP: Double First Order in Parallel.

[2] Best fit highlighted in bold.

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Dissipation of isoflucypram and isoflucypram-carboxylic acid were studied under aerobic field conditions (White, J., *et al.,* 2018). Field dissipation was examined following two broadcast applications of an isoflucypram EC 50 (50 g ai/L) formulation to bare soil and turf grass at one site in North Rose, New York, United States. The first application was made at a nominal application rate of 350 g ai/ha (actual application rate = 360 [bare soil] and 345 [turf] g ai/ha). The second application was made 121 days after the first application at a nominal application rate of 150 g ai/ha (actual application rate = 148–149 g ai/ha). Soil samples were collected prior to the first treatment and at various intervals after the first and second treatments. The final collection interval was DAT1-832 (DAT2-707). Rainfall was supplemented with irrigation as needed. The monthly mean air temperatures during the study were representative of average monthly mean temperatures. The plots had a slope of ≤1 percent and therefore run-off was not assessed. Three disturbed soil characterisation samples were taken from each treated plot to a depth of 120 cm, sectioned into 15 cm segments, and combined. Two disturbed soil cores were taken from each treated plot, one for bulk density and water holding capacity and one for morphology. Soil characterisation is shown in Table 89.

Table 89 Soil properties for Study AUS 0033

	Depth							
Property	0-15	15-30	30-45	45-60	60-75	75-90	90-105	105-120
Bare Soil								
Textural classification	Loam	Loam	Silt loam	Silt loam	Silt loam	Silt loam	Silt loam	Silt loam
% Sand	50	44	42	38	38	36	36	44
% Silt	41	47	53	57	51	53	59	51
% Clay	9	9	5	5	11	11	5	5
Bulk density disturbed (g/cm^3)	1.10	1.11	1.17	1.18	1.18	1.20	1.22	1.23
pH: (1:1 soil:water)	5.7	5.7	5.7	5.7	5.5	5.3	5.3	5.4
pH: (0.01 mol/L $CaCl_2$	5.4	5.3	5.3	5.2	4.9	4.7	4.8	4.8
Organic matter (%)	3.10	2.80	0.81	0.51	0.25	0.13	0.13	0.08
Cation exchange capacity (meq/100 g)	8.4	7.9	5.1	5.0	5.6	5.4	4.6	4.0
Water holding capacity at 1/3 bar (%)	20.4	22.6	20.7	20.5	21.5	20.1	17.9	16.0
Water holding capacity at 15 bar (%)	7.4	7.0	4.1	4.8	6.6	6.2	4.2	3.7
Turf grass								
Textural classification	Loamy sand	Sandy loam	Sandy loam	Silt loam	Silt loam	Silt loam	Silt loam	Silt loam

	Depth							
Property	0-15	15-30	30-45	45-60	60-75	75-90	90-105	105-120
% Sand	76	66	56	38	40	36	40	34
% Silt	23	29	37	57	55	57	51	55
% Clay	1	5	7	5	5	7	9	11
Bulk density disturbed (g/cm^3)	1.20	1.25	1.24	1.22	1.23	1.20	1.21	1.19
pH (1:1 soil:water)	6.0	6.1	5.7	5.3	5.3	5.3	5.1	5.1
pH (0.01M $CaCl_2$)	5.5	5.6	5.2	4.7	4.6	4.5	4.4	4.3
Organic matter (%)	2.20	1.40	0.47	0.25	0.13	0.13	0.13	0.17
Cation exchange capacity (meq/100 g)	6.6	5.8	4.3	4.6	4.7	5.2	5.3	6.0
Water holding capacity at 1/3 bar (%)	12.3	12.9	16.5	18.4	20.5	12.8	20.7	20.8
Water holding capacity at 15 bar (%)	4.8	4.0	3.2	3.7	4.1	5.2	5.3	5.5

Soil cores were collected from the treated plots on DAT1-0 and 120 (i.e., DAT2-0) to a depth of 15 cm. Samples at all other intervals (DAT1 -4 [prior to application], 0, 7, 14, 28, 59, 90, 120, 121, 128, 135, 149, 181, 211, 241, 301, 392, 487, 570, 662, and 828 DAT1) were collected to a depth of 120 cm. Samples from the bare soil plot were split into 15 cm horizons and samples from turf plot were split into a 0–7.5 cm horizon, a 7.5–15 cm horizon, and 15 cm horizons for the remainder of the samples. Samples for control analysis were collected at the control plot on DAT1 -4 (prior to application), 0, and 121.

Soil was frozen after sampling. Treated samples were stored for a maximum of 509 days. Analysis for verification pads was accomplished through Method LN-001-F15-01. Soil pans and treated samples were analysed according to Method 01432.

The theoretical application rate for the spray solution calibration was 100 percent for the first application and 99 percent for the second application. For application pads, an average of 101.3 percent (n=6) and 72.2 percent (n=6) of the expected active ingredient was recovered from both treated plots following application one and two, respectively. For application pans, an average of 37.5 percent (n=6) and 34.7 percent (n=6) of the expected active ingredient was recovered from both treated plots following application one and two, respectively. The soil was allowed to dry after application before analysed analysis. There were no quantifiable residues in control samples. The results are shown in Tables 90 and 91.

Table 90 Field dissipation of isoflucypram and isoflucypram carboxylic acid in bare soil

Depth (cm)[1]	DAT1 (DAT2)										
	-4 (-125)	0 (-121)	7 (-114)	14 (-107)	28 (-93)	59 (-62)	90 (-31)	120 (-1)	121 (0)	128 (7)	135 (14)
	Average Isoflucypram Residues, g ai/ha (% dose)[2]										
0-15	<LOQ	310.2 (88.6)	338.0 (96.6)	263.2 (75.2)	238.8 (68.2)	282.5 (80.7)	191.0 (54.6)	190.0 (54.6)	344.5 (68.9)	280.4 (56.1)	280.4 (56.1)
15-30	<LOQ	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	NA	3.1 (0.6)	3.6 (0.7)
30-45	<LOQ	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	NA	<LOQ	13.6 (2.7)
45-60	<LOQ	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	NA	<LOQ	<LOQ
60-75	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	2.9 (0.6)
75-90	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	<LOQ
	149	181	211	241	301	392	487	570	662	828	

Depth (cm)[1]	DAT1 (DAT2)										
	-4 (-125)	0 (-121)	7 (-114)	14 (-107)	28 (-93)	59 (-62)	90 (-31)	120 (-1)	121 (0)	128 (7)	135 (14)
	Average Isoflucypram Residues, g ai/ha (% dose)[2]										
	(28)	(60)	(90)	(120)	(180)	(271)	(366)	(449)	(541)	(707)	
0-15	265.7 (53.1)	226.8 (45.4)	248.6 (49.7)	247.0 (49.4)	227.7 (45.5)	220.1 (44.0)	232.4 (46.5)	208.4 (41.7)	236.5 (47.3)	183.4 (36.7)	
15-30	13.1 (2.6)	9.6 (1.9)	29.5 (5.9)	<LOQ	23.7 (4.7)	29.6 (5.9)	4.3 (0.9)	46.4 (9,3)	12.3 (2.5)	10.6 (2.1)	
30-45	6.3 (1.3)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	6.3 (1.3)	<LOQ	
45-60	4.8 (1.0)	<LOQ	<LOQ	<LOQ	2.2 (0.4)	<LOQ	1.8 (0.4)	<LOQ	4.3 (0.9)	<LOQ	
60-75	<LOQ	<LOQ	NA	NA	<LOQ	NA	2.8 (0.6)	<LOQ	<LOQ	<LOQ	
75-90	3.9 (0.8)	<LOQ	NA	NA	17.4 (3.5)	NA	5.3 (1.1)	<LOQ	<LOQ	<LOQ	
90-105	<LOQ	<LOQ	NA	NA	18.3 (3.7)	NA	9.6 (1.9)	<LOQ	<LOQ	<LOQ	
105-120	<LOQ	<LOQ	NA	NA	4.4 (0.9)	NA	5.5 (1.1)	<LOQ	<LOQ	<LOQ	
	Average isoflucypram carboxylic-acid residues, g ai/ha (% dose)[2,3]										
	-4 (-125)	0 (-121)	7 (-114)	14 (-107)	28 (-93)	59 (-62)	90 (-31)	120 (-1)	121 (0)	128 (7)	135 (14)
0-15	<LOQ	ND	<LOQ	0.3 (0.1)	0.6 (0.2)	0.9 (0.3)	1.1 (0.3)	1.0 (0.3)	8.4 (1.7)	8.8 (1.8)	10.3 (2.1)
15-30	<LOQ	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	NA	<LOQ	<LOQ
	149 (28)	181 (60)	211 (90)	241 (120)	301 (180)	392 (271)	487 (366)	570 (449)	662 (541)	828 (707)	
0-15	9.0 (1.8)	10.1 (2.0)	10.8 (2.2)	13.0 (2.6)	6.6 (1.3)	10.2 (2.0)	7.3 (1.5)	8.5 (1.7)	6.0 (1.2)	6.7 (1.3)	
15-30	3.7 (0.7)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	

Notes:

[1] Average of three samples.

[2] Samples were also analysed for isoflucypram-carboxylic acid at segment depths 30-45 cm, 45-60 cm, 60-75 cm, 75-90 cm, 90-105 cm, and 105-120 cm but results are not reported due to residues below the LOQ.

[3] Expressed in parent equivalents.

NA = Not analysed.

ND = not detected

Table 91 Field dissipation of isoflucypram in turf plots

Depth (cm)[1]	DAT1 (DAT2)										
	-4 (-125)	0 (-121)	7 (-114)	14 (-107)	28 (-93)	59 (-62)	90 (-31)	120 (-1)	121 (0)	128 (7)	135 (14)
	Average isoflucypram residues, g ai/ha (% applied dose)[2]										
0-7.5	<LOQ	276.4 (79.0)	279.9 (80.0)	218.2 (62.3)	159.8 (45.7)	93.3 (26.7)	64.5 (18.4)	55.1 (15.7)	166.3 (33.3)	171.4 (34.3)	188.7 (37.7)
7.5-15	<LOQ	12.6 (3.6)	6.3 (1.8)	24.3 (6.9)	5.0 (1.4)	6.9 (2.0)	2.5 (0.7)	3.2 (0.9)	19.6 (3.9)	5.4 (1.1)	8.2 (1.6)
15-30	<LOQ	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	NA	3.3 (0.7)	13.6 (2.7)
30-45	<LOQ	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	3.0 (0.6)	3.3 (0.7)
	149	181	211	241	301	392	487	570	662	828	

Depth (cm)[1]	DAT1 (DAT2)										
	-4 (-125)	0 (-121)	7 (-114)	14 (-107)	28 (-93)	59 (-62)	90 (-31)	120 (-1)	121 (0)	128 (7)	135 (14)
	Average isoflucypram residues, g ai/ha (% applied dose)[2]										
	(28)	(60)	(90)	(120)	(180)	(271)	(366)	(449)	(541)	(707)	
0-7.5	155.8 (31.2)	119.9 (24.0)	134.3 (26.9)	109.5 (21.9)	110.9 (22.2)	80.0 (16.0)	55.7 (11.1)	54.7 (10.9)	45.8 (9.2)	23.7 (4.7)	
7.5-15	2.6 (0.5)	3.1 (0.6)	4.1 (0.8)	<LOQ	3.8 (0.8)	4.6 (0.9)	1.9 (0.4)	2.4 (0.5)	2.3 (0.5)	2.2 (0.4)	
15-30	2.8 (0.6)	3.4 (0.7)	<LOQ	<LOQ	2.2 (0.4)	2.6 (0.5)	<LOQ	7.7 (1.5)	10.2 (2.0)	<LOQ	
30-45	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	
	Average isoflucypram carboxylic acid residues, g ai/ha (% applied dose)[2,3]										
	-4 (-125)	0 (-121)	7 (-114)	14 (-107)	28 (-93)	59 (-62)	90 (-31)	120 (-1)	121 (0)	128 (7)	135 (14)
0-7.5	<LOQ	<LOQ	1.9 (0.5)	7.8 (2.2)	16.5 (4.7)	12.5 (3.6)	6.8 (1.9)	6.3 (1.8)	5.5 (1.1)	9.4 (1.9)	10.0 (2.0)
7.5-15	<LOQ	<LOQ	<LOQ	1.4 (0.4)	5.2 (1.5)	4.5 (1.3)	3.9 (1.1)	2.4 (0.7)	2.9 (0.6)	2.4 (0.5)	5.6 (1.1)
15-30	<LOQ	NA	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	2.7 (0.5)	4.3 (0.9)
	149 (28)	181 (60)	211 (90)	241 (120)	301 (180)	392 (271)	487 (366)	570 (449)	662 (541)	828 (707)	
0-7.5	9.2 (1.8)	9.0 (1.8)	9.6 (1.9)	8.4 (1.7)	4.3 (0.9)	6.6 (1.3)	6.0 (1.2)	3.6 (0.7)	2.1 (0.4)	1.9 (0.4)	
7.5-15	5.2 (1.0)	8.5 (1.7)	7.9 (1.6)	11.0 (2.2)	5.5 (1.1)	5.4 (1.1)	2.6 (0.5)	3.1 (0.6)	2.1 (0.4)	1.5 (0.3)	
15-30	6.9 (1.4)	4.3 (0.9)	8.6 (1.7)	3.0 (0.6)	10.4 (2.1)	5.7 (1.1)	3.1 (0.6)	2.4 (0.5)	2.5 (0.5)	<LOQ	
30-45	<LOQ	<LOQ	<LOQ	<LOQ	3.9 (0.8)	2.7 (0.5)	<LOQ	<LOQ	<LOQ	<LOQ	
45-60	<LOQ	<LOQ	<LOQ	<LOQ	1.6 (0.3)	1.6 (0.3)	<LOQ	1.2 (0.2)	<LOQ	<LOQ	

Notes:

[1] Average of three samples.

[2] Samples were also analysed for isoflucypram at segment depths 45-60 cm, 60-75 cm, 75-90 cm, 90-105 cm, and 105-120 cm but results are not reported due to residues below the LOQ.

NA = Not analysed.

The kinetics of isoflucypram degradation was evaluated in both soils using kinetic modelling (KinGUII2.1 under FOCUS guidance). Goodness of fit was determined by chi^2, visual assessment, and R^2. For both plots and applications, the kinetic modelling followed SFO most closely. Leaching was not a significant degradation pathway as residues were mainly found in the top soil segments throughout the course of the study. The results are shown in Table 92.

Table 92 Kinetic modelling for isoflucypram in soil and turf

Model[1,2]	DT_{50}	DT_{75}	DT_{90}	Chi^2	R^2	Visual Assessment
Isoflucypram – bare ground soil, first application						
SFO	164.0[3]	327.7[3]	544.8[3]	8.70	0.33	Satisfactory
DFOP	170.8[3]	371.2[3]	636.3[3]	10.1	0.34	Satisfactory
FOMC	214.3	>1,000[3]	>1,000[3]	9.34	0.34	Satisfactory
Isoflucypram – bare ground soil, second application						
SFO	1,690[3]	3,381[3]	5,616[3]	7.54	0.13	Satisfactory
DFOP	>1,000	>1,000[3]	>1,000[3]	6.22	0.19	Satisfactory

Model[1,2]	DT_{50}	DT_{75}	DT_{90}	Chi^2	R^2	Visual Assessment
FOMC	>1,000[3]	>1,000[3]	>1,000[3]	6.19	0.19	Satisfactory
Isoflucypram – turf soil, first application						
SFO	40.64	81.26	135.0	6.77	0.90	Good
DFOP	35.92	81.70	>1,000	6.76	0.90	Good
FOMC	37.19	82.94	159.9	6.62	0.90	Good
Isoflucypram – turf soil, second application						
SFO	249.8	499.6	829.7	11.2	0.85	Good
DFOP	200.7	537.8	988.5	10.2	0.87	Good
FOMC	194.0	566.8	>1,000	10.2	0.86	Good

Notes:

[1] SFO: Single First Order. FOMC: First Order Multicompartment. DFOP: Double First Order in Parallel.

[2] Best fit highlighted in bold.

[3] Regarded as unreliable due to low R^2 value.

ANIMAL METABOLISM

The Meeting received studies describing the metabolism of isoflucypram in lactating goats and laying hens. Additionally, a brief summary of the WHO's review of metabolism of isoflucypram in laboratory rats is given.

Laboratory Rats

The WHO received studies investigating the absorption, distribution, metabolism, and excretion (ADME) of isoflucypram in rats. One study evaluated a single dose of [pyrazole-4-^{14}C] isoflucypram at 2 or 200 mg/kg bw and a single 2 mg/kg bw dose after bile duct cannulation by oral gavage. A second study evaluated a single dose of [phenyl-UL-^{14}C] isoflucypram at 2 mg/kg bw. Additionally, whole-body autoradiography studies were conducted with both radiolabels.

The absorption of [pyrazole-4-^{14}C] and [phenyl-UL-^{14}C] isoflucypram started immediately after administration, demonstrated by the observed maximum plasma concentration (Cmax) within 1 hour (tmax) our administration for the low dose experiments and within 2/4 hours (male/female) for the pyrazole high dose experiment. Radioactivity could be detected in all plasma samples until 72 hours after dosing, the latest time of plasma sampling, with values ≤ 3.1 percent of the maximum plasma concentration measured, indicating quick elimination. Low dose pyrazole studies with bile-duct cannulated rats showed that 74 percent/82 percent (male/female) of the dose was detected in the bile.

Isoflucypram-cyclopropyl-pyrazole-carboxamide (M58) and isoflucypram-desmethyl-carboxylic acid (M11) were observed at much higher concentrations than isoflucypram. Other metabolites measured in rats were observed at similar or higher concentrations than the parent compound in plasma.

In the pyrazole study, the low dose female rats showed lower organ concentrations compared to male rats, whereas In the phenyl study, radioactivity in organs and tissues of male and female rats was in the same order of magnitude. Radioactivity in bodies (excluding the gastrointestinal tract) amounted to 0.152–0.365 percent of the administered dose in the pyrazole studies and 0.234–0.290 percent of the administered dose in the phenyl studies. The highest concentration of radioactivity was detected in the liver, followed by blood cells, followed by other tissues.

The whole-body autoradiography studies demonstrated that radioactivity was widely distributed among tissues immediately after dosing. Radioactivity was observed predominantly in the liver and kidney. Absorbed radioactivity was quickly and efficiently eliminated within 72 hours after administration.

Excretion was almost completed 72 hours after administration. The main excretion route was faeces, with less radioactivity eliminated through urine. In bile-duct cannulated rats, radioactivity was observed primarily in bile, followed by faeces, followed by urine, indicating that the major elimination route is faeces through bile. An observed lower urinary excretion rate in the high dose pyrazole studies indicates lower absorption of isoflucypram at higher dose rates.

A high number of metabolites were identified in urine, faeces, and bile, suggesting thorough metabolism in rats. Metabolites and conjugates identified at levels >10 percent of the absorbed dose were:

- Isoflucypram-carboxylic acid;
- Isoflucypram-desmethyl;
- Isoflucypram-desmethyl-propanol;
- Isoflucypram-desmethyl-carboxylic acid;
- Isoflucypram-desmethyl-hydroxyphenyl-1,2-propandiol;
- Isoflucypram-desmethyl-hydroxymethyl-carboxylic acid;
- Isoflucypram-propanol-GlucA (isomer 1);
- Isoflucypram-desmethyl-GlucA (isomer 1 and 2);
- Isoflucypram-desmethyl-OH-GlucA (isomer 2);
- Isoflucypram-desmethyl-diOH-GlucA (group of isomers).

Lactating Goats

Report Nos. EnSa-17-0309 and EnSa-17-0308.

The metabolism and excretion of [pyrazole-4-1^{4}C] Isoflucypram and [phenyl-UL-^{14}C] Isoflucypram were investigated in lactating goats (Bongartz *et al*., 2017). The test compounds were orally administered in gelatine capsules at a dose level of 1 mg/kg bw/day to two goats, corresponding to 45.19 ppm and 20.57 ppm in dry feed per day for the pyrazole and phenyl labels, respectively. The goats were dosed once daily (morning) for five consecutive days and were sacrificed approximately six hours after the final dosing. The goats were milked in the morning immediately prior to each dose administration, about eight hours later in the afternoon, and approximately one hour before sacrifice. TRR was determined in each milk sample, as well as liver, kidney, muscle (pooled round and loin), and fat (pooled perirenal and omental; pyrazole only). Additionally, the total radioactivity (percent of total administered dose) was determined in each urine and faeces sample. The phenyl label goat did not have fat for analysis. The goat was healthy according to veterinarian investigation during acclimation and testing period and did not show any abnormality with regard to feed consumption, weight, and common behaviour.

All fractions were minced (if necessary) and measured for radioactivity by LSC. The overall recovery accounted for 64 percent and 51 percent of the administered dose for pyrazole and phenyl labels, respectively. The remaining amount of radioactivity was postulated to still be present in the non-edible part of the animal body and especially in the gastrointestinal tract at sacrifice due to the short time period between last administration and sacrifice. At the time of sacrifice, a cumulative total of 6.94/9.8 percent (pyrazole/phenyl) and 56.09/40.08 percent (pyrazole/phenyl) of the administered dose was excreted in urine and faeces, respectively, with an observed linear increase following one (pyrazole) or

three (phenyl) days after the first dose. Approximately 0.03 percent (pyrazole) and 0.06 percent (phenyl) of the administered dose was secreted with milk, correlating to TRRs ranging from 0.009–0.021 mg eq/kg (pyrazole) and 0.008–0.016 mg eq/kg (phenyl) per individual sample. Per-day pooled milk samples (evening sample plus following day pre-dose sample) reached a plateau after three days. At sacrifice, the sum of radioactive residues in edible fractions was 0.72 percent (pyrazole) and 0.27 percent (phenyl) of the administered dose (AD), correlating to TRRs of 0.717 mg eq/kg (pyrazole) and 0.348 mg eq/kg (phenyl) in liver, 0.189 mg eq/kg (pyrazole) and 0.183 mg eq/kg (phenyl) in kidney, 0.038 mg eq/kg (pyrazole) and 0.011 (phenyl) mg eq/kg for muscle, and 0.102 mg eq/kg in fat (pyrazole only; no fat analysed in phenyl study). The results are shown in Table 93.

Table 93 TRR for lactating goats dosed with isoflucypram

	[Pyrazole-4-^{14}C][1]		[Phenyl-UL-^{14}C]	
Sample	% AD	TRR (mg eq/kg)	% AD	TRR (mg eq/kg)
Urine (0-102 h plus funnel rinsing)	6.94	-	9.81	-
Faeces (0-102 h)	56.09	-	40.78	-
Milk (0-102 h)[2]	0.03	Day 1 -0.009 Day 2 – 0.014 Day 3 – 0.015 Day 4 – 0.014 Day 5 – 0.021	0.06	Day 1 -0.008 Day 2 – 0.013 Day 3 – 0.012 Day 4 – 0.012 Day 5 – 0.015
Muscle	0.23	Round – 0.035 Loin – 0.041	0.06	Round – 0.011 Loin – 0.010
Fat	0.25	Perirenal - 0.095 Omental – 0.110	NA	
Liver	0.23	0.717	0.19	0.348
Kidney	0.01	0.189	0.01	0.183

Notes:

[1] Pyrazole reference: EnSa-17-0309. Phenyl reference: EnSa-17-0308.

[2] Per-day pooled milk samples from Day 1-4 consists of combined milking eight hours and 24-hours post-dose. The day five milk sample consists of one sample collected approximately five hours post-dose, just before slaughter.

NA = not analysed

Samples for all matrices except urine were extracted 2–3× with ACN/water (8/2). In the pyrazole study, milk was additionally extracted once with THF (3/7) and ACN/water (3/7). Conventional extraction released 89.2–98.7 percent (pyrazole) and 88.3–100.0 percent (phenyl) TRR. All samples from the pyrazole study and liver and kidney extracts from the phenyl study were partitioned against n-heptane. The extracts of milk and muscle were purified with SPE cartridges and a subsequent phase separation with NaCl. In edible fractions, very low amounts of radioactivity were detected in the n-heptane organic phase (≤1.4 percent; ≤ 0.002 mg eq/kg). In edible fractions, concentration of the aqueous phases caused minor losses of radioactivity (≤6.3 percent TRR; ≤ 0.009 mg eq/kg).

In muscle from the pyrazole study and liver from both labels, the solids after conventional extraction were exhaustively extracted using ACN/water, microwave assistance, and HCl in the extract of liver from the pyrazole study. Exhaustive extraction in these matrices released an additional 4.3–4.9 percent TRR (pyrazole) and 3.5 percent TRR (phenyl). In all matrices, up to 7.8 percent TRR (0.036 mg eq/kg) [pyrazole] and 8.2 percent TRR (0.029 mg eq/kg) [phenyl] remained in the PES.

The conventional liver and kidney extract were incubated, separately, for 20 hours at 37 °C with β-glucuronidase and arylsulfatase and analysed by HPLC for comparison of the metabolite profile with the pre-hydrolysis extract. Additionally, aliquots of isoflucypram-propenol-GlucA and isoflucypram-propanol-GlucA (isomer 1 and 2) isolated from goat urine [24–48 hours] from the phenyl study were incubated with

ß-glucuronidase and arylsulfatase for four or 20 hours, respectively, at 37 °C. After purification the digested metabolites were analysed by HPLC.

Residue components of the conventional and exhaustive extracts were analysed by HPLC and thin-layer chromatography (TLC), respectively. Compounds were identified and by co-chromatography with reference standards.

Samples were stored for a maximum of three months in the pyrazole study. Comparison of initial extract with extract stored 13 and 14 months in liver and kidney, respectively, showed no degradation. Also, repeat analysis of muscle, fat, and kidney after 25, nine, and nine months, respectively, demonstrated stability of peaks.

Samples were stored for a maximum of four months in the phenyl study. Comparison of initial extract with extract stored 11 months (liver) showed no degradation. Also, repeat analysis of muscle, liver, and kidney after 16, six, and 20 months, respectively, demonstrated stability of peaks.

In milk samples pooled from 32–101 hours post first dose (correlating with the plateau level), parent isoflucypram was the predominant residue at 33.4 percent TRR (0.005 mg/kg) [pyrazole] and 33.9 percent TRR (0.004 mg/kg) [phenyl]. Isoflucypram-2-propanol was observed at 20.3 percent TRR (0.003 mg eq/kg) [pyrazole] and 5.0 percent TRR (0.001 mg eq/kg) [phenyl]. Isoflucypram-desmethyl-propanol and isoflucypram-propanol-GlucA (isomers 1 and 2) were minor metabolites, each accounting for ≤ 6.7 percent TRR (≤ 0.001 mg eq/kg) in the phenyl study and not detected in the pyrazole study. Up to three peaks each accounting for ≤ 38.6 percent TRR (≤ 0.005 mg eq/kg) were characterized based on chromatographic behaviour. When separated between cream and skim fractions, the majority of the radioactivity (approximately 87 percent TRR) remained in the skimmed milk fraction in the pyrazole study, whereas the radioactivity was essentially evenly distributed between the two fractions in the phenyl study, as determined by TLC. Figure 9 shows the TRRs in milk thought the study and Tables 94 and 95 the identification and characterization of the residues

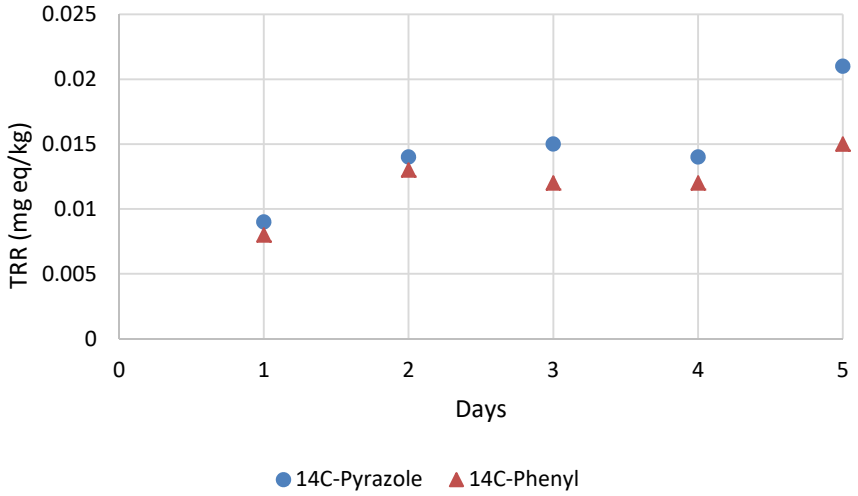


Figure 9 Concentration of TRR in milk following dosing with radiolabelled isoflucypram.

Notes:
* Residues are the average of samples from the evening and following morning, except Day 5, which are only from the evening sample.

Table 94 Identification and characterisation of TRR in milk

Residue Component	[Pyrazole-4-^{14}C][1] (TRR = 0.015 mg eq/kg; 32-101h)		[Phenyl-UL-^{14}C][1] (TRR = 0.013 mg eq/kg; 32-101h)	
	mg eq/kg	%TRR	mg eq/kg	% TRR
Conventional extract	0.015	98.7	0.013	98.5
Isoflucypram	0.005	33.4	0.004	33.9
Isoflucypram-2-propanol	0.003	20.3	0.001	5.0
Isoflucypram-propanol-GlucA (isomer 1/2)	-	-	<0.001/<0.001	2.3/2.5
Isoflucypram-desmethyl-propanol	-	-	0.001	6.7
Total identified	0.008	53.7	0.007	50.4
Number of unidentified peaks	5		3	
Largest unknown peak	0.002	14.3	0.005	38.6
Characterised by HPLC	0.006	42.7	0.006	47.3
Characterised by partitioning (n-heptane phase)	<0.001	1.4	-	-
Total characterised	0.006	44.1	0.006	47.3
Sum of losses	<0.001	1.0	<0.001	0.8
PES	<0.001	1.3	<0.001	1.5
Accountability	0.015	100	0.013	100

Notes:
[1] Pyrazole reference: EnSa-17-0309. Phenyl reference: EnSa-17-0308.

Table 95 Distribution of Residues in Skimmed Milk and Cream

	[Pyrazole-4-^{14}C][1]		[Phenyl-UL-^{14}C][1]	
Sample Description	% TRR	mg eq/kg	% TRR	mg eq/kg
n-heptane phase (cream)	13.3	0.002	49.7	0.006
Aqueous phase (skimmed milk)	86.7	0.013	50.3	0.007
Total	100	0.015	100	0.013

Notes:
[1] Pyrazole reference: EnSa-17-0309. Phenyl reference: EnSa-17-0308.

In pooled muscle, isoflucypram was the predominant residue at 22.3 percent TRR (0.008 mg/kg) [pyrazole] and 21.5 percent TRR (0.002 mg eq/kg) [phenyl]. Other major metabolites included isoflucypram-2-propanol accounting for 17.9 percent TRR (0.006 mg eq/kg) [pyrazole] and 14.2 percent YTT (0.002 mg eq/kg) [phenyl] and isoflucypram-propanol accounting for 10.2 percent TRR (0.004 mg eq/kg) [pyrazole] and 9.0 percent TRR (0.001 mg eq/kg) [phenyl].

Minor metabolites included isoflucypram-carboxylic acid, isoflucypram-propanol-GlucA (isomer 2), and isoflucypram-desmethyl-propanol (≤9.0 percent TRR; ≤ 0.003 percent mg eq/kg). Up to five peaks (≤ 25 percent TRR; ≤ 0.003 mg eq/kg) were characterised based on the chromatographic behaviour in the conventional extract. In the pyrazole exhaustive ACN/water extract, five minor compounds (≤ 1.6 percent TRR; ≤ 0.001 mg eq/kg) were characterised based on the chromatographic behaviour using TLC (Table 95).

Table 96 Identification and characterisation of TRR in pooled muscle

	[Pyrazole-4-^{14}C][1] (TRR = 0.036 mg eq/kg)		[Phenyl-UL-^{14}C][1] (TRR = 0.011 mg eq/kg)	
Residue Component	mg eq/kg	% TRR	mg eq/kg	% TRR
Conventional extract	0.032	89.2	0.011	100.0
Isoflucypram	0.008	22.3	0.002	21.5

	[Pyrazole-4-^{14}C][1] (TRR = 0.036 mg eq/kg)		[Phenyl-UL-^{14}C][1] (TRR = 0.011 mg eq/kg)	
Residue Component	mg eq/kg	% TRR	mg eq/kg	% TRR
Isoflucypram-propanol-GlucA (isomer 2)	0.002	4.8	0.001	6.5
Isoflucypram-desmethyl-propanol	0.002	4.4	<0.001	3.7
Isoflucypram-carboxylic acid	0.003	8.1	0.001	9.0
Isoflucypram-propanol	0.004	10.2	0.001	9.0
Isoflucypram-2-propanol	0.006	17.9	0.002	14.2
Total identified	0.024	67.7	0.007	64.0
Number of unknown peaks	5		2	
Largest unknown peak	0.002	6.8	0.003	25.0
Characterised by HPLC	0.007	20.6	0.003	29.8
Characterised by partitioning (n-heptane phase)	<0.001	0.9	-	-
Exhaustive extraction (ACN/water extract)	0.002	4.3	-	-
Number of unknown peaks	5		-	
Largest unknown peak	0.001	1.6	-	-
Total characterised	0.009	25.8	0.003	29.8
Sum of losses	-	-	0.001	6.3
Total extracted	0.034	93.6	0.011	100
PES	0.002	6.4	-	-
Accountability	0.036	100	0.011	100

Notes:
[1] Pyrazole reference: EnSa-17-0309. Phenyl reference: EnSa-17-0308.

In pooled fat, isoflucypram was the predominant residue at 58.7 percent TRR (0.061 mg/kg). Isoflucypram-2-propanol was a major metabolite accounting for 0.017 mg eq/kg (16.8 percent TRR). Minor metabolites included isoflucypram-carboxylic acid and isoflucypram-propanol (≤ 0.003 mg eq/kg; ≤ 3.3 percent TRR). Four minor peaks (≤ 0.008 mg eq/kg; ≤ 7.5 percent TRR) were characterised based on the chromatographic behaviour using HPLC (Table 97).

Table 97 Identification and characterisation of TRR in pooled fat (EnSa-17-0309)

	[Pyrazole-4-^{14}C][1] (TRR = 0.104 mg eq/kg)	
Residue Component	mg eq/kg	% TRR
Conventional extract	0.102	98.3
Isoflucypram	0.061	58.7
Isoflucypram-carboxylic acid	0.003	3.3
Isoflucypram-propanol	0.003	2.7
Isoflucypram-2-propanol	0.017	16.8
Total identified	0.085	81.5
Number of unknown peaks	4	
Largest unknown peak	0.008	7.5
Characterised by HPLC	0.017	16.1
Characterised by partitioning (n-heptane phase)	0.001	0.8
Total characterised	0.017	16.8
Sum of losses	-	-
PES	0.002	1.7
Accountability	0.104	100

In liver, isoflucypram accounted for 3.5 percent TRR (0.025 mg/kg) [pyrazole] and 5.3 percent TRR (0.018 mg/kg) [phenyl]. In the conventional extract, major metabolites included isoflucypram-2-propanol-GlucA accounting for 13.8 percent TRR (0.099 mg eq/kg) [pyrazole] and 13.0 percent TRR (0.045 mg eq/kg) [phenyl], isoflucypram-propanol-GlucA (isomer 1) accounting for 13.1 percent TRR

(0.094 mg eq/kg) [pyrazole] and 8.8 percent TRR (0.031 mg eq/kg) [phenyl], isoflucypram-propanol-GlucA (isomer 2) accounting for 7.7 percent TRR (0.055 mg eq/kg) [pyrazole] and 5.9 percent TRR (0.021 mg eq/kg) [phenyl], isoflucypram-carboxylic acid accounting for 8.9 percent TRR (0.064 mg eq/kg) [pyrazole] and 4.2 percent TRR (0.015 mg eq/kg) [phenyl], isoflucypram-propanol accounting for 5.8 percent TRR (0.042 mg eq/kg) [pyrazole] and 4.9 percent TRR (0.017 mg eq/kg) [phenyl], isoflucypram-2-propanol accounting for 2.6 percent TRR (0.019 mg eq/kg) [pyrazole] and 2.8 percent TRR (0.010 mg eq/kg) [phenyl], and isoflucypram-lactic acid accounting 1.6 percent TRR (0.011 mg eq/kg) [pyrazole] and 1.3 percent TRR (0.005 mg eq/kg) [phenyl]. The minor metabolites isoflucypram-desmethyl-carboxylic acid and isoflucypram-desmethyl-propanol accounted for ≤ 0.9 percent TRR (≤ 0.007 mg eq/kg).

Up to eighteen peaks, each accounting for ≤ 6.7 percent TRR (≤ 0.029 mg eq/kg), were characterised based on chromatographic behaviour using HPLC. In the exhaustive ACN/water extract, up to 11 minor compounds each accounting for ≤ 1.7 percent TRR (≤ 0.007 mg eq/kg), and in the exhaustive HCl extract, four peaks each accounting for ≤ 2.3 percent TRR (≤ 0.017 mg eq/kg) of the TRR), were characterised based on the chromatographic behaviour using TLC.

The extract enzymatically cleaved with ß-glucuronidase and arylsulfatase resulted in significantly decreased or absent concentrations of isoflucypram-2-propanol-GlucA and isoflucypram-propanol-GlucA (both isomer 1 and 2), whereas their aglycones isoflucypram-2-propanol and isoflucypram-propanol increased. The cleavage of some unknown conjugates resulted in higher amounts of aglycones. Unknown compounds following cleavage accounted for ≤ 9.9 percent TRR (≤ 0.069 mg eq/kg) (Table 98).

Table 98 Identification and characterisation of TRR in liver

	[Pyrazole-4-^{14}C][1] (TRR = 0.717 mg eq/kg)		[Pyrazole-4-^{14}C][1] Enzyme Hydrolysed		[Phenyl-UL-^{14}C][1] (TRR = 0.348 mg eq/kg)		[Phenyl-UL-^{14}C][1] Enzyme Hydrolysed	
Residue Component	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Conventional extract	0.646	90.1	0.585	81.5	0.307	88.3	0.280	80.5
Isoflucypram	0.025	3.5	0.044	6.1	0.018	5.3	0.016	4.7
Isoflucypram-2-propanol-GlucA	0.099	13.8	0.007	0.9	0.045	13.0	0.005	1.3
Isoflucypram-lactic acid	0.011	1.6	0.005	0.7	0.005	1.3	0.002	0.7
Isoflucypram-propanol-GlucA (isomer 1/2)	0.094/0.055	13.1/7.7	-	-	0.031/0.021	8.8 5.9	-	-
Isoflucypram-desmethyl-carboxylic acid	0.007	0.9	0.016	2.1	0.002	0.5	0.005	1.5
Isoflucypram-desmethyl-propanol	0.003	0.5	0.033	4.6	0.002	0.6	0.015	4.3
Isoflucypram- carboxylic acid	0.064	8.9	0.084	11.7	0.015	4.2	0.033	9.6
Isoflucypram-propanol	0.042	5.8	0.149	20.8	0.017	4.9	0.064	18.4
Isoflucypram-2-propanol	0.019	2.6	0.085	11.9	0.010	2.8	0.047	13.4
Total identified	0.418	58.3	0.423	58.9	0.164	47.2	0.187	53.8
Number of unknown peaks	17		-		18		-	-
Largest unknown peak	0.029	4.0	-	-	0.023	6.7	-	-
Characterised by HPLC	0.226	31.5	0.162	22.6	0.132	37.8	0.093	26.7
Characterised by partitioning (n-heptane)	0.002	0.3	-	-	0.002	0.6	-	-
Exhaustive extraction (ACN/water) extract	0.015	2.1	-	-	0.012	3.5	-	-

	[Pyrazole-4-^{14}C][1] (TRR = 0.717 mg eq/kg)		[Pyrazole-4-^{14}C][1] Enzyme Hydrolysed		[Phenyl-UL-^{14}C][1] (TRR = 0.348 mg eq/kg)		[Phenyl-UL-^{14}C][1] Enzyme Hydrolysed	
Residue Component	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Number of unknown peaks	11		-		5		-	-
Largest unknown peak	0.007	0.9	-	-	0.006	1.7	-	-
Exhaustive extraction (HCl) extract	0.020	2.8	-	-	-	-	-	-
Number of unknown peaks	4		-		-		-	-
Largest unknown peak	0.017	2.3	-	-	-	-	-	-
Total characterised	0.262	36.6	0.162	22.6	0.144	41.3	0.093	26.7
Sum of losses	-	-	-	-	0.009	2.6	-	-
Total extracted	0.681	94.9	-	-	0.319	91.8	-	-
PES	0.036	5.1	-	-	0.029	8.2	-	-
Accountability	0.717	100	0.585	81.5	0.348	100	0.280	80.5

Notes:
[1] Pyrazole reference: EnSa-17-0309. Phenyl reference: EnSa-17-0308.

In kidney, isoflucypram was a minor compound accounting for 2.7 percent TRR (0.005 mg/kg) [pyrazole] and 1.6 percent TRR (0.003 mg/kg) [phenyl]. Major metabolites included isoflucypram-carboxylic acid accounting for 18.0 percent TRR (0.034 mg eq/kg) [pyrazole] and 6.8 percent TRR (0.012 mg eq/kg) [phenyl], isoflucypram-lactic acid accounting for 6.1 percent TRR (0.012 mg eq/kg) [pyrazole] and 4.2 percent TRR (0.008 mg eq/kg) [phenyl], isoflucypram-propanol-GlucA (isomer 2) accounting for 7.0 percent TRR (0.013 mg eq/kg) [pyrazole] and 8.6 percent TRR (0.016 mg eq/kg) [phenyl], isoflucypram-propanol accounting for 5.6 percent TRR (0.011 mg eq/kg) [pyrazole] and 2.5 percent TRR (0.004 mg eq/kg) [phenyl], isoflucypram-propenol-GlucA accounting for 3.6 percent TRR (0.007 mg eq/kg) [pyrazole] and 6.2 percent TRR (0.011 mg eq/kg) [phenyl], and isoflucypram-N-methyl-pyrazole-carboxylic acid accounting for 5.8 percent TRR (0.011 mg eq/kg) [pyrazole; not detected in phenyl study]. Minor metabolites included isoflucypram-2-propanol-GlucA, isoflucypram-propanol-GlucA (isomer 1), isoflucypram-desmethyl-carboxylic acid, and isoflucypram-2-propanol in both studies, as well as isoflucypram-desmethyl-propanol in the pyrazole study, only, at concentrations ≤ 0.008 mg eq/kg (≤ 4.2 mg eq/kg).

The extract of kidney enzymatically cleaved with ß-glucuronidase and arylsulfatase resulted in significant decreased concentrations of isoflucypram-2-propanol-GlucA and isoflucypram-propanol-GlucA (isomer 1 and 2), whereas their aglycones isoflucypram-2-propanol and isoflucypram-propanol increased in concentration. The cleavage of some unknown conjugates resulted in higher amounts of aglycones. Unknown compound concentrations following cleavage accounted ≤ 9.3 percent TRR (≤ 0.018 mg eq/kg) (Table 99).

Table 99 Identification and characterisation of TRR in kidney

Residue Component	[Pyrazole-4-^{14}C][1] (TRR = 0.189 mg eq/kg)		[Pyrazole-4-^{14}C][1] Enzyme hydrolysed		[Phenyl-UL-^{14}C][1] (TRR = 0.183 mg eq/kg)		[Phenyl-UL-^{14}C][1] Enzyme hydrolysed	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Conventional extract	0.174	92.2	0.155	82.1	0.179	97.9	0.174	94.2
Isoflucypram	0.005	2.7	0.005	2.6	0.003	1.6	0.002	1.3
Isoflucypram-N-methyl-pyrazole-carboxylic acid	0.011	5.8	0.004	1.9	-	-	-	-
Isoflucypram-2-propanol-GlucA[2]	0.005	2.6	-	-	0.007	4.0	0.003	1.3
Isoflucypram-propenol-GlucA[2]	0.007	3.6	-	-	0.011	6.2	-	-

Residue Component	[Pyrazole-4-^{14}C][1] (TRR = 0.189 mg eq/kg)		[Pyrazole-4-^{14}C][1] Enzyme hydrolysed		[Phenyl-UL-^{14}C][1] (TRR = 0.183 mg eq/kg)		[Phenyl-UL-^{14}C][1] Enzyme hydrolysed	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Isoflucypram- lactic acid	0.012	6.1	0.007	3.4	0.008	4.2	0.004	1.9
Isoflucypram- propanol-GlucA (isomer 1/2)	0.006/ 0.013	3.1/ 7.0	-	-	0.007/ 0.016	3.6/ 8.6	<0.001/ -	0.3/ -
Isoflucypram-desmethyl-carboxylic acid	0.008	4.2	0.008	4.2	0.007	3.6	0.012	6.4
Isoflucypram-desmethyl-propanol	0.003	1.6	0.009	4.6	-	-	0.009	5.0
Isoflucypram-carboxylic acid	0.034	18.0	0.044	23.2	0.012	6.8	0.023	12.7
Isoflucypram- propanol	0.011	5.6	0.020	10.7	0.004	2.5	0.037	20.0
Isoflucypram-2-propanol	0.008	4.2	0.013	6.7	0.003	1.4	0.010	5.5
Number of unknown peaks	16		-		21		-	
Largest unknown peak	0.011	5.7	-	-	0.017	9.1	-	-
Total identified	0.123	65.4	0.108	57.2	0.078	42.4	0.100	54.6
Characterised by HPLC	0.050	26.8	0.047	25.0	0.100	54.4	0.073	39.7
Characterised by partitioning (n-heptane phase)	-	-	-	-	<0.001	0.3	-	-
Total characterised	0.050	26.8	0.047	25.0	0.100	54.4	0.073	39.7
Sum of losses	-	-	-	-	0.002	0.8	-	-
PES	0.015	7.8	-	-	0.004	2.1	-	-
Accountability	0.189	100	0.155	82.1	0.183	100.0	0.173	94.2

Notes:

[1] Pyrazole reference: EnSa-17-0309. Phenyl reference: EnSa-17-0308.

[2] 2-propanol-GlucA and propenol-GlucA were co-eluting on HPLC and were sub-quantified by TLC.

The proposed metabolism pathway of isoflucypram in goats is shown in Figure 10.

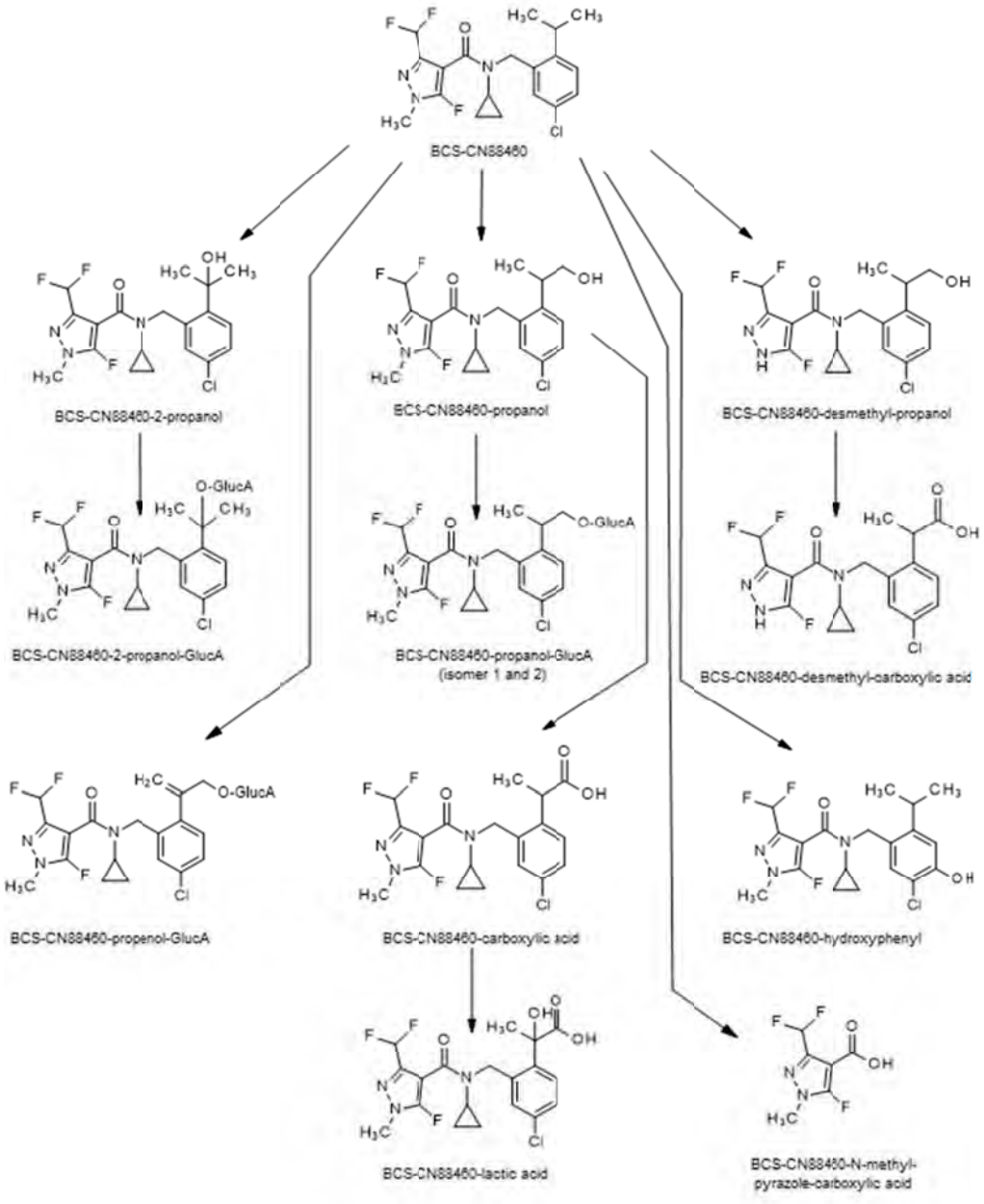


Figure 10 Metabolic pathway in lactating goats

Laying Hen

Report Nos EnSa-17-0307 and EnSa-17-0306.

The metabolism and excretion of [pyrazole-4-^{14}C] Isoflucypram and [phenyl-UL-^{14}C] Isoflucypram was investigated in laying hens as a model for poultry (Bongartz *et al.,* 2017). The test compounds were orally administered to six hens as aqueous 0.5 percent suspensions (via gavage) at **1** mg/kg body weight, corresponding to average doses of 16.57 ppm and 18.12 ppm dry feed per day for the pyrazole and phenyl labels, respectively. The hens received 14 doses at 24-hour intervals in the morning and were sacrificed six hours after the last dosing. Eggs were collected once daily and before sacrifice. Samples were pooled from all hens and TRR was determined in each daily egg sample and in tissues of muscle (leg and thorax),

fat, liver, kidney, skin, and eggs from ovary/oviduct at sacrifice. The percent of the administered dose was determined in each excreta sample.

Solid samples were minced and radioactivity was determined by LSC or combustion/LSC. The overall recovery amounted to approximately 96 percent (pyrazole) and 103.4 percent (phenyl) of the total dose. Approximately 95.8 percent (pyrazole) and 103.0 percent (phenyl) of the total dose was excreted. After the third administration the daily excretion rate was steady at a level ranging from 6.3–7.7 percent (pyrazole) and 7.1–8.8 percent (phenyl). An average of 0.12 percent (pyrazole) and 0.14 percent (phenyl) of the total administered dose was measured in the eggs. At sacrifice, radioactive residues in the tissues were calculated or estimated to be about 0.22 percent (pyrazole) and 0.24 percent (phenyl) of the total administered dose.

TRR in eggs ranged from 0.029 mg eq/kg on day two to 0.057 mg eq/kg at sacrifice in the pyrazole study, and from 0.032 mg eq/kg at day three to 0.066 mg eq/kg at sacrifice in the phenol study. TRR in eggs followed a linear increase until a plateau-level of approximately 0.050 mg eq/kg was reached on the sixth (pyrazole) and fourth day (phenyl). Pooled samples for analysis were collected from Day 6-13 (pyrazole) and 4-13 (phenyl). TRR in tissues amounted to 0.370 mg eq/kg (pyrazole) and 0.373 mg eq/kg (phenyl) in liver, 0.390 mg eq/kg (pyrazole) and 0.360 mg eq/kg (phenyl) in kidney, 0.042 mg eq/kg (pyrazole) and 0.047 mg eq/kg (phenyl) in subcutaneous fat, 0.075 mg eq/kg (pyrazole) and 0.109 mg eq/kg (phenyl) in skin, 0.029 mg eq/kg in leg muscle (both labels), and 0.018 mg eq/kg (pyrazole) and 0.017 mg eq/kg (phenyl) in thorax muscle (Table 100).

Table 100 Summary of TRR in poultry eggs and tissues

	[Pyrazole-4-^{14}C][1]		[Phenyl-UL-^{14}C][1]	
Sample	% AD	% TRR		% TRR
Eggs	0.12	Day 1: NC Day 2: 0.029 Day 3: 0.029 Day 4: 0.038 Day 5: 0.041 Day 6: 0.053 Day 7: 0.054 Day 8: 0.049 Day 9: 0.051 Day 10: 0.049 Day 11: 0.050 Day 12: 0.047 Day 13: 0.049 Day 13.25[2]: 0.057	0.14 % AD	Day 1: NC Day 2: 0.048 Day 3: 0.032 Day 4: 0.043 Day 5: 0.047 Day 6: 0.050 Day 7: 0.062 Day 8: 0.054 Day 9: 0.054 Day 10: 0.051 Day 11: 0.045 Day 12: 0.045 Day 13: 0.047 Day 13.25[2]: 0.066]
Muscle (leg)	0.07	0.029	0.07	0.029
Muscle (thorax)	0.07	0.018	0.07	0.017
Subcutaneous fat	0.04	0.042	0.04	0.047
Skin	0.02	0.075	0.03	0.109
Liver	0.07	0.370	0.07	0.373
Kidney	0.02	0.390	0.01	0.360
Excreta	3.74-7.72. Total = 95.8		4.75-8.84. Total = 103.0	

Notes:

[1] Pyrazole reference: EnSa-17-0307. Phenyl reference: EnSa-17-0306.

[2] TRR of the eggs collected from the ovary and oviduct at sacrifice (0.076 mg eq/kg [pyrazole] and 0.096 mg eq/kg [phenyl]) were a factor of 1.3X (pyrazole) and 1.5X (phenyl) higher than the laid eggs collected at sacrifice.

A total of 83.9–93.4 percent (pyrazole) and 85.4–93.3 percent (phenyl) was extracted using ACN/water (8/2). For sample preparation, the conventional extracts from eggs, muscle, liver, and excreta

were partitioned against n-heptane. Low amounts of radioactivity were detected in the n-heptane phases (≤1.3 percent TRR; 0.001 mg eq/kg). Concentration procedures caused no losses in radioactivity.

In liver, the remaining solids after conventional extraction underwent exhaustive extraction with ACN/water (8/2) using microwave assistance followed by microwave treatment with 0.1 mol/L HCL, which together released an additional 16.1 percent (pyrazole) and 14.5 percent (phenyl) TRR. In the pyrazole study, up to 8.2 percent TRR (thorax muscle) and 0.003 mg eq/kg (egg and fat) remained in the PES. In the phenyl study, up to 8.2 percent TRR (fat) and 0.004 mg eq/kg (egg and fat) remained in the PES.

Aliquots of the conventional liver extracts were incubated with ß-glucuronidase and arylsulfatase. The suspensions were incubated for 96 hours at 37 °C. After incubation, the enzymatic suspensions were analysed by HPLC and compared with the pre-hydrolysis extract. Additionally, aliquots of isoflucypram-desmethyl-1,2-propandiol-N-GlucA and isoflucypram-desmethyl-1,2-propandiol-SA from pyrazole excreta (Day 1) were incubated with ß-glucuronidase and arylsulfatase for 96 and 24 hours, respectively, at 37 °C. An aliquot of isoflucypram-desmethyl-propanol-N-GlucA from the phenyl study was incubated with ß-glucuronidase and arylsulfatase for 96 hours at 37 °C. After purification the digested metabolites were analysed by HPLC.

Extracts were analysed by HPLC in tandem with mass spectrometry (MS) and/or nuclear magnetic resonance (NMR) using MS-electrospray ionisation (ESI)/NMR spectroscopy for quantitation. Additionally, TLC was used for quantitative analyses of the exhaustive extracts of liver as well as for identification of parent compound in the conventional extract of fat.

Identities were assigned by comparison of HPLC profiles among various tissues and across both radiolabels. The identification was performed in isolated fractions from the extract of eggs and excreta by spectroscopic methods and by enzymatic cleavage of selected conjugates. The identified metabolites and their aglycones in the isolated fractions were used as reference compounds. In addition, parent and metabolites were identified in the extract of fat by TLC co-chromatography using metabolites in the isolated fractions from eggs and excreta.

Metabolites isoflucypram-desmethyl-1,2-propandiol-SA, isofliucypram-1,2-propandiol-SA and isoflucypram-propanol-SA showed an additional peak due to necessary sample preparation resulting in a cleavage of the sulphate group. The respective aglycone was identified based on assignment to the radiolabelled reference compound. In case of the isolated metabolites isoflucypram-desmethyl-propanol and isoflucypram-desmethyl-propanol-N-GlucA, an addition of a formyl group was observed during structure elucidation. An exact structure could not be generated and were only present in minor amounts; therefore, they were not reported.

Samples were stored for a maximum of five months in the pyrazole study. Comparison of liver extract after 20 months of storage demonstrated no degradation of parent or metabolites. Re-extraction 3.5–22 months showed no indication of degradation in all profiles, except fat, which showed slight deviations after 23 months due to high matrix content of the extract according to the report.

Samples were stored for a maximum of three months in the phenyl study, a second conventional extraction of liver following nine months of storage demonstrated no significant degradation. Re-analysis of other extracts 4-15 months after extraction showed no indication of degradation.

In eggs, parent compound comprised 3.7 percent TRR (0.002 mg/kg) [pyrazole] and 6.4 percent TRR (0.003 mg/kg) [phenyl]. Isoflucypram-propanol was the most prominent compound at 35.0 percent TRR (0.018 mg eq/kg) [pyrazole] and 33.9 percent TRR (0.017 mg eq/kg) [phenyl] followed by isoflucypram-desmethyl-propanol at 22.3 percent TRR (0.011 mg eq/kg) [pyrazole] and 22.6 percent TRR (0.011 mg eq/kg) [phenyl]. Isoflucypram-desmethyl-1,2-propandiol and isoflucypram-carboxylic acid were

minor components in both studies accounting for ≤ 7.2 percent TRR (≤ 0.004 mg eq/kg); isoflucypram-desmethyl-propanol-N-GlucA was only observed as a minor component in the phenyl study accounting for 3.0 percent TRR (0.002 mg eq/kg). Up to five minor peaks, each accounting for ≤ 7.7 percent TRR (≤ 0.004 mg eq/kg), were characterised based on the chromatographic behaviour. Small portions of the radioactivity (≤ 1.3 percent TRR; 0.001 mg eq/kg) were observed in the n-heptane phase. Levels of TRR in eggs are shown in Figure 11 and identification and characterization of the residues in Table 101.

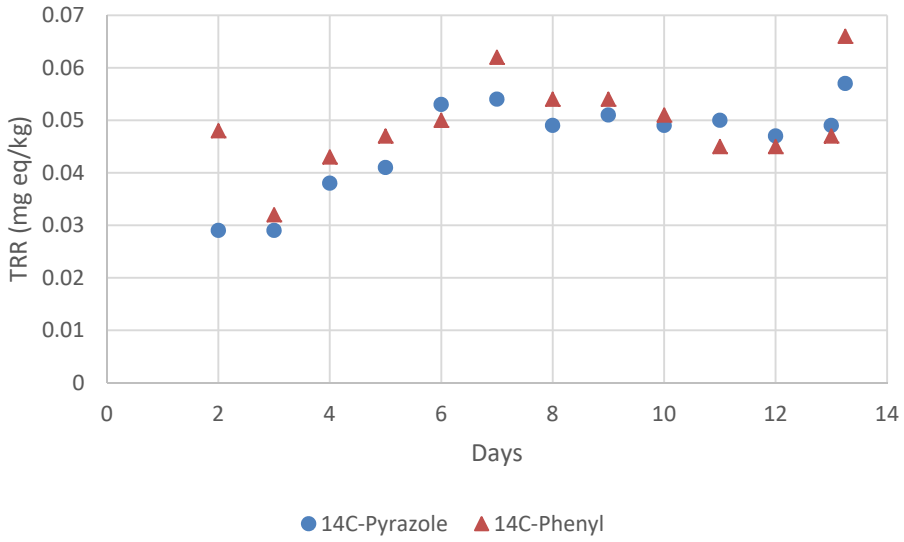


Figure 11 Concentration in Eggs Following Dosing with Radiolabelled Isoflucypram

Table 101 Identification and characterisation of TRR in eggs

	[Pyrazole-4-^{14}C][1] (TRR = 0.050 mg eq/kg)		[Phenyl-UL-^{14}C][1] (TRR = 0.050 mg eq/kg)	
Residue component	mg eq/kg	percent TRR	mg eq/kg	percent TRR
Conventional extract	0.047	93.4	0.046	92.8
Isoflucypram	0.002	3.7	0.003	6.4
Isoflucypram-desmethyl-1,2-propandiol	0.003	5.2	0.003	6.2
Isoflucypram-desmethyl- propanol	0.011	22.3	0.011	22.6
Isoflucypram-carboxylic acid	0.002	3.4	0.004	7.2
Isoflucypram-propanol	0.018	35.0	0.017	33.9
Isoflucypram-desmethyl- propanol-N-GlucA	-	-	0.002	3.0
Number of unknown peaks	5		3	
Largest unknown peak	0.004	7.7	0.002	4.9
Characterised by HPLC	0.011	22.4	0.006	12.4
Total identified	0.035	69.6	0.040	79.2
n-heptane phase	0.001	1.3	0.001	1.2
Total characterised	0.012	23.7	0.007	13.6
PES	0.003	6.6	0.004	7.2
Accountability	0.050	100.0	0.050	100.0

Notes:

[1] Pyrazole reference: EnSa-17-0307. Phenyl reference: EnSa-17-0306.

In muscle, parent compound was a minor residue component and was only identified in leg muscle at ≤ 2.9 percent TRR (0.001 mg eq/kg). In both leg and thorax muscle samples, prominent metabolites included isoflucypram-desmethyl-propanol accounting for 20.9–29.7 percent TRR (0.004–0.009 mg eq/kg) [pyrazole] and 20.9–25.8 percent TRR (0.004–0.007 mg eq/kg) [phenyl], isoflucypram-desmethyl-1,2-propandiol accounting for 15.0–17.9 percent TRR (0.003–0.004 mg eq/kg) [pyrazole] and 14.2–22.3 percent TRR (0.004 mg eq/kg) [phenyl], isoflucypram-desmethyl-carboxylic acid accounting for 12.0–12.1 percent TRR (0.002–0.004 mg eq/kg) [pyrazole] and 19.9–20.2 percent TRR (0.003–0.006 mg eq/kg) [phenyl], and isoflucypram-carboxylic acid accounting for 9.1–11.0 percent TRR (0.002–0.003 mg eq/kg) [pyrazole] and 6.6–8.6 percent TRR (0.001–0.002 mg eq/kg) [phenyl]. Isoflucypram-propanol was a minor component accounting for ≤5.9 percent TRR (≤ 0.002 mg eq/kg). Isoflucypram-desmethyl-1,2-propandiol-N-GlucA and isoflucypram-desmethyl-propanol-N-GlucA were only observed in phenyl-label leg muscle and accounted for ≤ 4.1 percent TRR (0.001 mg eq/kg). Up to four peaks, each accounting for ≤ 19.5 percent TRR (0.004 mg eq/kg) were characterised based on the chromatographic behaviour. Although one peak represented 19.5 percent TRR, it only accounted for 0.004 mg eq/kg was therefore not further analysed (Table 102).

Table 102 Identification and characterisation of TRR in muscle

	[Pyrazole-4-^{14}C][1] Leg (TRR = 0.029 mg eq/kg)		[Pyrazole-4-^{14}C][1] Thorax (TRR = 0.018 mg eq/kg)		[Phenyl-UL-^{14}C][1] Leg (TRR = 0.029 mg eq/kg)		[Phenyl-UL-^{14}C][1] Thorax (TRR = 0.017 mg eq/kg)	
Residue Component	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Conventional extract	0.027	92.8	0.017	91.8	0.027	93.3	0.016	92.4
Isoflucypram	0.001	2.3	-	-	0.001	2.9	-	-
Isoflucypram- desmethyl-1,2- propandiol	0.004	15.0	0.003	17.9	0.004	14.2	0.004	22.3
Isoflucypram- desmethyl-carboxylic acid	0.004	12.1	0.002	12.0	0.006	19.9	0.003	20.2
Isoflucypram- desmethyl-propanol	0.009	29.7	0.004	20.9	0.007	25.8	0.004	20.9
Isoflucypram-carboxylic acid	0.003	9.1	0.002	11.0	0.002	6.6	0.001	8.6
Isoflucypram- propanol	0.002	5.3	0.001	5.9	0.001	2.5	0.001	4.3
Isoflucypram- desmethyl-1,2- propandiol-N-GlucA	-	-	-	-	0.001	4.1	-	-
Isoflucypram- desmethyl-propanol-N-GlucA	-	-	-	-	0.001	3.8	-	-
Total identified	0.021	73.5	0.012	67.6	0.023	79.8	0.013	76.3
Number of unknown peaks	4		2		3		2	
Largest unknown peak	0.003	9.9	0.004	19.5	0.004	13.2	0.003	16.1
Characterised by HPLC	0.005	18.7	0.004	24.2	0.004	13.2	0.003	16.1
n-heptane phase	<0.001	0.6	-	-	<0.001	0.4	-	-
Total characterised	0.005	19.3	0.004	24.2	0.004	13.5	0.003	16.1
PES	0.002	7.2	0.001	8.2	0.002	6.7	0.001	7.6
Accountability	0.029	100.0	0.018	100.0	0.029	100.0	0.017	100.0

Note:
[1] Pyrazole reference: EnSa-17-0307. Phenyl reference: EnSa-17-0306.

In fat, parent was the most prominent compound at 23.6 percent TRR (0.010 mg/kg) [pyrazole] and 20.1 percent TRR (0.009 mg/kg) [phenyl], followed by isoflucypram-propanol at 11.9 percent TRR (0.005 mg eq/kg) [pyrazole] and 6.5 percent TRR (0.003 mg eq/kg) [phenyl] and isoflucypram-desmethyl-propanol at 10.1 percent TRR (0.004 mg eq/kg) [pyrazole] and 8.0 percent TRR (0.004 mg eq/kg) [phenyl].

Minor metabolites included isoflucypram-desmethyl-1,2-propandiol and isoflucypram-carboxylic acid in both labels, and isoflucypram-desmethyl-carboxylic acid in the phenyl label, at concentrations ≤ 7.9 percent TRR (≤ 0.004 mg eq/kg). Up to seven peaks, each accounting for ≤ 11.2 percent TRR (≤ 0.005 mg eq/kg), were characterised based on chromatographic behaviour (Table 103).

Table 103 Identification and characterisation of TRR in fat

	[Pyrazole-4-^{14}C][1] (TRR = 0.042 mg eq/kg)		[Phenyl-UL-^{14}C][1] (TRR = 0.047 mg eq/kg)	
Residue Component	mg eq/kg	percent TRR	mg eq/kg	percent TRR
Conventional extract	0.039	92.7	0.043	91.8
Isoflucypram	0.010	23.6	0.009	20.1
Isoflucypram-desmethyl-1,2-propandiol	0.002	5.1	0.003	5.9
Isoflucypram-desmethyl-propanol	0.004	10.1	0.004	8.0
Isoflucypram-carboxylic acid	0.002	4.8	0.001	3.0
Isoflucypram-propanol	0.005	11.9	0.003	6.5
Isoflucypram-desmethyl-Carboxylic acid	-	-	0.004	7.9
Total identified	0.023	55.5	0.024	51.5
Number of unknown peaks	6		7	
Largest unknown peak	0.005	11.2	0.005	10.0
Total characterised	0.016	37.1	0.019	40.3
PES	0.003	7.3	0.004	8.2
Accountability	0.042	100.0	0.047	100.0

Note:
[1] Pyrazole reference: EnSa-17-0307. Phenyl reference: EnSa-17-0306.

Parent compound was not detected in the liver. In the conventional extract, isoflucypram-desmethyl-carboxylic acid was the most prominent compound at 14.4 percent TRR (0.053 mg/kg) [pyrazole] and 12.9 percent TRR (0.082 mg/kg) [phenyl]. Further prominent components were isoflucypram-carboxylic acid accounting for 11.9 percent TRR (0.044 mg eq/kg) [pyrazole] and 5.8 percent TRR (0.022 mg eq/kg) [phenyl], isoflucypram-desmethyl-1,2-propandiol-N-GlucA accounting for 5.4 percent TRR (0.020 mg eq/kg) [pyrazole] and 9.2 percent TRR (0.034 mg eq/kg) [phenyl], isoflucypram-desmethyl-propanol-N-GlucA accounting for 6.1 percent TRR (0.023 mg eq/kg) [pyrazole] and 10.8 percent TRR (0.040 mg eq/kg) [phenyl], isoflucypram-desmethyl-1,2-propandiol accounting for 6.9 percent TRR (0.025 mg eq/kg) [pyrazole] and 5.6 percent TRR (0.021 mg eq/kg) [phenyl], isoflucypram-desmethyl-propanol accounting for 5.3 percent TRR (0.020 mg eq/kg) [pyrazole] and 2.7 percent TRR (0.010 mg eq/kg) [phenyl], and isoflucypram-desmethyl-2-propanol-N-GlucA accounting for 2.5 percent TRR (0.009 mg eq/kg) [pyrazole] and 3.0 percent TRR (0.011 mg eq/kg) [phenyl]. Isoflucypram-propanol (both labels) and isoflucypram-propanol-SA (phenyl label only) were less abundant and accounted for ≤ 1.7 percent TRR (≤ 0.007 mg eq/kg). Up to 18 minor peaks, each accounting for ≤ 8.4 percent TRR (≤ 0.031 mg eq/kg), were characterised based on the chromatographic behaviour using HPLC.

In the exhaustive ACN/water extract, five compounds each accounting for ≤ 2.8 percent TRR (≤ 0.010 mg eq/kg) were characterised based on the chromatographic behaviour using TLC. In the exhaustive 0.1 mol/L HCl extract, four compounds each accounting for ≤ 8.2 percent TRR (≤ 0.031 mg eq/kg) were characterised based on the chromatographic behaviour using TLC.

The enzymatically digested liver extracts were analysed by HPLC. The amount of isoflucypram-desmethyl-1,2-propandiol-N-GlucA, isoflucypram-desmethyl-propanol-N-GlucA, and isoflucypram-desmethyl-2-propanol-N-GlucA decreased in concentration or were absent following 96 hours incubation, whereas their aglycones isoflucypram-desmethyl-1,2-propandiol and isoflucypram-desmethyl-propanol

increased. The cleavage of some unknown conjugates resulted in higher amounts of the aglycones. Unknown compounds following cleavage accounted for ≤ 5.4 percent TRR (≤ 0.020 mg eq/kg) after enzymatic cleavage (Table 104).

Table 104 Identification and characterisation of TRR in liver

	[Pyrazole-4-^{14}C][1] (TRR = 0.370 mg eq/kg)		[Pyrazole-4-^{14}C][1] Enzyme hydrolysis		[Phenyl-UL-^{14}C][1] (TRR = 0.373 mg eq/kg)		[Phenyl-UL-^{14}C][1] Enzyme hydrolysis[1]	
Residue Component	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Conventional extract	0.310	83.9	0.278	75.2	0.317	85.4	0.292	78.3
Isoflucypram	-	-	-	-	-	-	-	-
Isoflucypram-desmethyl-1,2-propandiol-N-GlucA	0.020	5.4	0.6	0.003	0.034	9.2	0.005	1.4
Isoflucypram- desmethyl-propanol-N-GlucA	0.023	6.1	-	-	0.040	10.8	0.003	0.8
Isoflucypram- desmethyl-2-propanol-N-GlucA	0.009	2.5	0.5	0.002	0.011	3.0	0.002	0.6
Isoflucypram- desmethyl-1,2-propandiol	0.025	6.9	11.5	0.043	0.021	5.6	0.041	11.0
Isoflucypram- desmethyl-carboxylic acid	0.053	14.4	0.064	17.2	0.082	21.9	0.096	25.7
Isoflucypram- desmethyl-propanol	0.020	5.3	0.054	14.6	0.010	2.7	0.053	14.1
Isoflucypram-carboxylic acid	0.044	11.9	0.062	16.6	0.022	5.8	0.033	9.0
Isoflucypram- propanol	0.006	1.7	0.007	1.7	0.006	1.7	0.006	1.7
Isoflucypram- propanol-SA	-	-	-	-	0.005	1.2	-	-
Total identified	0.201	54.3	0.232	62.7	0.231	62.0	0.240	64.4
Number of unknown peaks	18		-		17		-	
Largest unknown peak	0.031	8.4	-	-	0.030	8.0	-	-
Characterised by HPLC	0.107	29.0	-	-	0.086	23.1	-	-
n-heptane phase	0.002	0.6	-	-	0.001	0.3	-	-
Exhaustive extraction ACN/H_2O phase	0.017	4.5	-	-	0.018	4.8	-	-
Number of unknown peaks	5		-		5		-	
Largest unknown peak	0.010	2.8	-	-	0.009	2.4	-	-
Exhaustive extraction 0.1M HCl	0.043	11.5	-	-	0.036	9.6	-	-
Number of unknown peaks	4		-		4		-	
Largest unknown peak	0.031	8.2	-	-	0.025	6.6	-	-
Total characterised	0.169	45.6	0.046	12.6	0.142	37.9	0.052	14.0
Total extracted	0.370	99.9	-	-	0.373	99.9	-	-
PES	<0.001	0.1	-	-	<0.001	0.1	-	-
Accountability	0.370	100.0	0.278	75.2	0.373	100.0	0.292	78.4

Notes:
[1] Pyrazole reference: EnSa-17-0307. Phenyl reference: EnSa-17-0306.

The proposed metabolic pathway of isoflucypram in laying hens is shown in Figure 12.

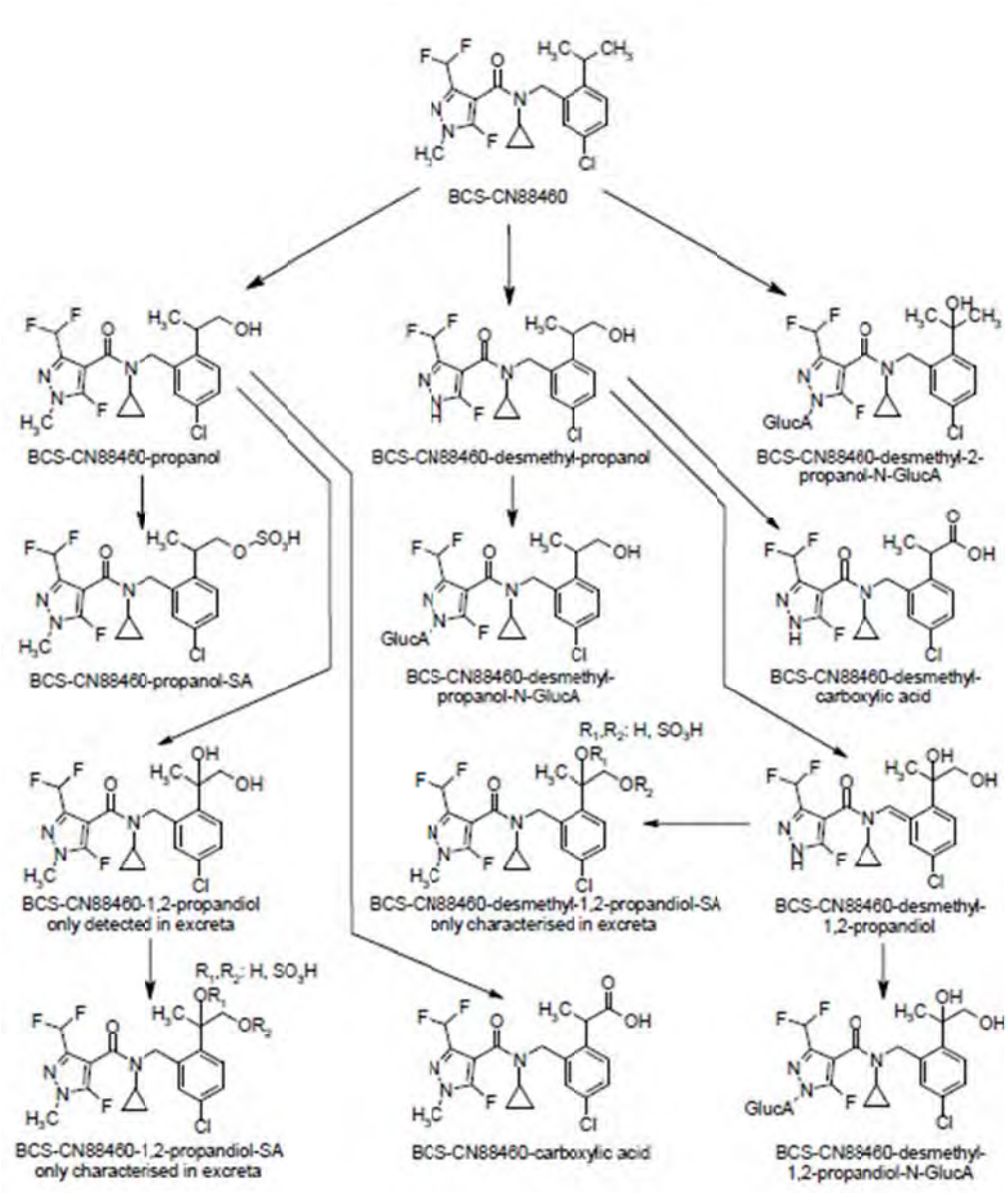


Figure 12 Metabolic pathways in laying hens

METHODS OF RESIDUE ANALYSIS

Plant Matrices

Data Collection Method 01475

Report Nos. MR-16/234, S15-06246, EnSa-16-0204, and EnSa-16-0179.

The Meeting received analytical method descriptions and validation data for the determination of the residues of isoflucypram and its metabolite BCS-CR60082 in/on plant materials by means of HPLC-MS/MS.

Briefly, isoflucypram BCS-CR60082, are extracted from plant material with ACN/water (8:2). The extraction is repeated after blending and centrifugation. The combined extracts are diluted in water and

internal standard is added. The residues are determined by LC-MS/MS using the characteristic m/z 400.2 → 139 (parent) and m/z 234 → 177 (BCS-CR60082) multiple reaction monitoring (MRM) transitions for quantification and m/z 400.2 → 177 (parent) and m/z 234 → 157 (BCS-CR60082) for confirmation using external calibrations with internal standards.

The LOQ for isoflucypram and BCS-CR60082, defined as the lowest validated fortification level, is 0.01 mg/kg in all matrices tested.

The commodities tested for method validation included: tomato fruit (high-water content), orange fruit (high-acid content), rape seed (high-oil content), wheat grain (high-starch content), bean dry seed (high-protein content), and wheat straw (dry). Commodities with low moisture content like wheat grain, rape seed, bean dry seed, and wheat straw are left to soak in water before extraction. Samples were fortified with isoflucypram and BCS-CR60082 at 0.01 and 0.10 mg/kg. Mean recoveries per fortification level were in a range of 70–110 percent, using the quantitation MRM transition with acceptable relative standard deviations (RSDs). Confirmatory procedures for isoflucypram and BCS-CR60082 gave mean recoveries ranging from 82–101 percent for all matrices tested, with acceptable RSD values. Due to a lack of sensitivity and an intercept above 30 percent of the LOQ, it was not possible to determine BCS-CR60082 residues in rape (seed) and bean (dry seed) using a second (confirmatory) MRM transition. The analytical method is fully validated for the accurate and precise determination of residues in crop samples.

For isoflucypram, using the quantification and confirmatory MRM transitions, method linearity was validated over the range of 0.01 to 2.0 µg/mL (internal standard calibration solutions). For isoflucypram, the r values ranged from 0.9997 to 0.9999. For BCS-CR60082, using the quantifier MRM transition, linearity was validated over the range of 0.01 to 1.0 µg/mL (internal standard calibration solutions). For BCS-CR60082, using the confirmatory MRM transition, linearity was validated over the range of 0.01 to 2.0 µg/mL (internal standard calibration solutions). For BCS-CR60082, the r values ranged from 0.9994 to 1.0000. Residues in control samples were <30 percent of the LOQ. The analytical method is fully validated for the accurate and precise determination of residues in crop samples.

The ability of method 01475 to extract incurred residues of isoflucypram and BCS-CR60082 was tested is tomato, wheat, soya bean, oilseed rape, rotational Swiss chard, rotational turnip leaves, and rotational wheat. Extracts of tomato, wheat, soya bean, oilseed rape, Swiss chard, and turnip leaves were stored for approximately 13, 18, 25, 6, 11, and 19 months, respectively, at ≤-18 °C. The stored radioactive residues in samples from primary crop and confined rotational crop studies were analysed according to Method 01475, and the recoveries of the extracted residues were then compared to those in the respective metabolism studies. Method 01475 extracted residues from plant commodities with an efficiency of ≥ 70.8 percent compared to the respective metabolism studies (Table 105). The extraction efficiency of the method is shown in Tables 106 and 107.

Table 105 Recoveries (%) for Method 01475: isoflucypram and BCS-CR60082 in plants (n=5) (MR-16/234)

		Isoflucypram						BCS-CR60082					
	Level (mg/kg)	Primary: Transition 400.2 → 139			Confirmatory: Transition 400.2 → 177			Quantitation: Transition 234 → 177			Confirmatory: Transition 234 → 157		
Matrix		Range	Mean	RSD	Range	Mean	RSD	Range	Mean	RSD	Range	Mean	RSD
Tomato	0.01	87-95	92	3.9	89-95	93	2.7	80-87	84	3.6	81-84	82	1.6
	0.10	89-96	93	2.9	89-95	93	2.7	86-92	90	2.8	85-94	90	3.6
Orange	0.01	96-99	97	1.6	96-101	99	2.2	73	2.3	-	-	-	
	0.10	99-101	100	0.9	101-102	101	0.4	88	2.3	82-92	89	4.3	
Wheat	0.01	90-100	94	3.9	87-97	91	4.0	83-89	85	2.7	79-102	89	9.6

		Isoflucypram						BCS-CR60082					
	Level (mg/kg)	Primary: Transition 400.2 → 139			Confirmatory: Transition 400.2 → 177			Quantitation: Transition 234 → 177			Confirmatory: Transition 234 → 157		
Matrix		Range	Mean	RSD	Range	Mean	RSD	Range	Mean	RSD	Range	Mean	RSD
grain	0.10	91-96	93	1.9	90-95	93	2.1	87-90	88	1.7	85-89	87	1.7
Wheat straw	0.01	86-90	89	1.9	87-92	89	2.1	72-76	75	2.2	-	-	-
	0.10	90-93	92	1.5	89-93	91	2.3	80-85	83	2.3	76-82	80	3.5
Rape seed	0.01	94-97	95	1.2	90-96	94	3.1	102-119	107	6.3	-	-	-
	0.10	97-103	99	2.4	98-105	101	2.8	100-108	103	3.0	-	-	-
Bean dry seed	0.01	93-94	94	0.5	94-98	96	2.1	82-87	86	2.4	-	-	-
	0.10	96-99	97	1.3	97-100	98	1.4	93-96	94	1.2	-	-	-

Table 106 Isoflucypram - recovery results of extraction efficiency testing of Method 01475 from representative matrices taken from plant metabolism studies

	Method 01475		Pyrazole metabolism study		Extraction efficiency[1]
Sample	% TRR	mg/kg	% TRR	mg/kg	%
Primary crops (S15-06246, EnSa-16-0204)					
Tomato fruit	97.8	0.289	96.7	0.165	175
Wheat hay	40.4	1.517	50.0	2.016	75.2
Wheat straw	61.8	9.674	64.0	9.933	97.4
Wheat grain	86.0	0.327	92.0	0.354	92.4
Soya bean forage	20.0	0.731	18.7	0.819	89.3
Soya bean hay	9.0	0.425	10.4	0.487	87.3
Soya bean straw	73.5	12.895	64.5	11.424	112
Soya bean seeds	83.8	0.023	76.6	0.027	85.2
OSR intermediate harvest	79.2	4.292	81.9	3.890	110
OSR seeds	70.5	0.071	71.0	0.070	101
Confined rotational crops[2] (EnSa-16-0179)					
Mature Swiss chard'(1st rotation)	6.7	0.0018	4.6	0.0012	150
Turnip leaves (1st rotation)	3.4	0.0006	4.8	0.0009	66.7

Notes:

[1] Calculated by: extracted radioactivity (mg/kg) from residue analytical method/extracted radioactivity (mg/kg) of method used in the metabolism studies * 100 percent.

[2] Parent compound was not detected in confined rotational crop samples wheat hay, wheat straw, wheat grain, and wheat forage.

Table 107 BCS-CR60082 - recovery results of extraction efficiency testing of Method 01475 from representative matrices taken from plant metabolism studies[2] (EnSa-16-0179)

	Method 01475		Metabolism Studies		Extraction Efficiency[1]
Sample	% TRR	mg/kg	% TRR	mg/kg	%
Wheat hay (3rd rotation)	2.5	0.0050	2.3	0.0043	116
Wheat straw (3rd rotation)	4.2	0.0153	5.8	0.0199	76.9
Wheat forage (3rd rotation)	4.2	0.0031	4.3	0.0031	100
Swiss chard (3rd rotation)	17.0	0.0045	19.4	0.0051	88.2
Turnip leaves (3rd rotation)	17.6	0.0034	18.4	0.0033	103

Notes:

[1] Calculated by: extracted radioactivity (mg eq/kg) from residue analytical method/extracted radioactivity (mg eq/kg) of method used in the metabolism studies * 100 percent.
[2] Metabolite BCS-CR60082 was not detected in confined rotational crop sample wheat grain.

Method 01475 was also utilized for data gathering in the field rotational crop, storage stability, magnitude of the residue, barley processing study, and to screen feed in the lactating cow and laying hen feeding studies.

For all data gathering, calibration curves were established for each analytical set with at least five concentration levels and an r value >0.99. For rotational crops, the extracts were analysed within 24 hours after extraction. The results are shown in Table 108

Table 108 Concurrent recoveries for rotational crops according to Method 01475 (Ref. 15-2502

		Isoflucypram			BCS-CR60082		
Crop	Level (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Recovery (%)	Mean (%)	RSD (%)
Barley grain	0.01	97, 94, 95	95	1.6	92, 86, 91	90	3.6
	0.10	98, 100, 96	98	2.0	95, 95, 95	95	0.0
Barley green material	0.01	104, 108, 101, 101, 107	104	3.1	89, 85, 89, 84, 90	87	3.1
	0.10	103, 97, 117, 106, 103	105	7.0	99, 92, 109, 98, 96	99	6.4
Barley straw	0.01	96, 100, 93	96	3.6	92, 92, 90	91	1.3
	0.10	87, 98, 100	95	7.4	87, 96, 98	94	6.3
Carrot tops	0.01	104, 107, 107	106	1.6	95, 94, 92	94	1.6
	0.10	103, 105, 105	104	1.1	96, 103, 103	101	4.0
Carrot root	0.01	100, 100, 100	100	0.0	94, 94, 92	93	1.2
	0.10	103, 107, 107	106	2.2	99, 99, 101	100	1.2
Lettuce	0.01	98, 102, 96	99	3.1	88, 95, 90	91	4.0
	0.10	101, 104, 98	101	3.0	94, 94, 95	94	0.6
Turnip tops	0.01	99, 104, 103	102	2.6	93, 93, 94	93	0.6
	0.10	99, 109, 105	104	4.8	91, 98, 98	96	4.2
Turnip root	0.01	98, 89, 96	94	5.0	89, 79, 88	85	6.5
	0.10	103, 103, 105	104	1.1	93, 95, 96	95	1.6

For storage stability study MR-17/244, the maximum time between extraction and analysis was 105 hours. For isoflucypram, all concurrent recovery averages were within the acceptable range of 70–110 percent except the storage interval 561 days for bean (dry seed) with an average recovery of 111 percent. The results are shown in Table 109.

Table 109 Summary of concurrent recoveries for isoflucypram and BCS-CR60082 in crops in storage stability study MR-17/244 according to Method 01475

			Isoflucypram			BCS-CR60082		
Crop	Level (mg/kg)	Storage Interval (days)	Recovery (%)	Mean (%)	RSD (%)	Recovery (%)	Mean (%)	RSD (%)
Tomato	0.01	0	100	-	-	88	-	-
		100	96	-	-	101	-	-
		251	103	-	-	100	-	-
		415	106	-	-	97	-	-
		563	97	-	-	90	-	-
		737	99	-	-	100	-	-
		779+ 6[1]	91	-	-	98	-	
	0.20	0	98, 102, 103	101	2.6	94, 95, 93	94	1.1
		100	82, 89	86		84, 86	85	-

			Isoflucypram			BCS-CR60082		
Crop	Level (mg/kg)	Storage Interval (days)	Recovery (%)	Mean (%)	RSD (%)	Recovery (%)	Mean (%)	RSD (%)
		251	106, 103	105		102, 97	100	-
		415	95, 84	90		92, 82	87	-
		563	101, 103	102		95, 102	99	-
		737	96, 99, 94	96	2.6	100, 90, 92	94	5.6
		779+ 6[1]	97, 98, 93	96	2.8	92, 96, 91	93	2.8
Bean dry seed	0.01	0	99	-	-	84	-	-
		100	99	-	-	99	-	-
		246	91	-	-	87	-	-
		415	87	-	-	81	-	-
		561	115	-	-	110	-	-
		743	101	-	-	95	-	-
		783+ 6[1]	97	-	-	92	-	-
	0.20	0	99, 99, 98	99	0.6	93, 94, 94	94	0.6
		100	97, 88	93	-	94, 84	89	-
		246	98, 101	100		91, 95	93	-
		415	76, 82	79	-	74, 77	76	-
		561	113, 109	111	-	97, 94	96	-
		743	97, 95, 91	94	3.2	88, 91, 86	88	2.8
		783+ 6[1]	99, 99, 99[2]	99	0.0	86, 88, 90[2]	88	2.3
Wheat grain	0.01	0	92	-	-	87	-	-
		102	93	-	-	92	-	-
		249	104	-	-	95	-	-
		414	88	-	-	80	-	-
		560	108	-	-	101	-	-
		746	100	-	-	96	-	-
		778+ 6[1]	94	-	-	84	-	-
	0.20	0	95, 94, 97	95	1.6	90, 93, 92	92	1.7
		102	94, 96	95	-	89, 93	91	-
		249	102, 102	102	-	99, 97	98	-
		414	97, 87	92	-	89, 81	85	-
		560	90, 103	97	-	84, 90	87	-
		746	96, 94, 97	96	1.6	88, 85, 89	87	2.4
		778+ 6[1]	96, 95, 93	95	1.6	86, 86, 88	87	1.3
Rape seed	0.01	0	113	-	-	116	-	-
		103	93	-	-	110	-	-
		245	94	-	-	91	-	-
		417	86	-	-	97	-	-
		558	97	-	-	110	-	-
		747	89	-	-	104	-	-
		776+ 6[1]	92	-	-	115	-	-
	0.20	0	100, 99, 96	98	2.1	99, 94, 97	97	2.6
		103	92, 83	88	-	87, 81	84	-
		245	98, 96	97	-	101, 94	98	-
		417	91, 93	92	-	92, 94	93	-
		558	87, 83	85	-	81, 81	81	-
		747	96, 95, 94	95	1.1	93, 79, 95	89	9.8
		776+ 6[1]	97, 97, 100	98	1.8	95, 76, 82[2]	84	11.5
Orange	0.01	0	82	-	-	82	-	-
		6	82	-	-	92	-	-
		34	87	-	-	78	-	-
		106	-[3]	-	-	63	-	-
		254	94	-	-	83	-	-

			Isoflucypram			BCS-CR60082		
Crop	Level (mg/kg)	Storage Interval (days)	Recovery (%)	Mean (%)	RSD (%)	Recovery (%)	Mean (%)	RSD (%)
		415	85	-	-	74	-	-
		559	106	-	-	106	-	-
		742	99	-	-	99	-	-
		785+ 6[1]	96	-	-	83	-	-
	0.20	0	76, 101, 71	83	19.4	75, 87, 67	76	13.2
		6	92, 82, 94	89	7.2	84, 65, 79	76	13.0
		34	104, 77, 88	90	15.1	93, 69, 81	81	14.8
		106	92, 99, 88	93	5.9	84, 87, 81	84	3.6
		254	87, 83, 108	93	14.5	80, 76, 96	84	12.6
		415	89, 80, 96	88	9.1	83, 78, 87	83	5.5
		559	101, 109, 105	105	3.8	92, 97, 101	97	4.7
		742	96, 97, 90	94	4.0	87, 91, 85	88	3.5
		785+ 6[1]	98, 98, 99	98	0.6	83, 85, 82	83	1.8
	0.01	0	108	-	-	93	-	-
		167	93	-	-	77	-	-
		315	103	-	-	91	-	-
	0.20	0	101, 106, 104	104	2.4	94, 99, 94	96	3.0
		167	94, 82, 89	88	6.8	86, 78, 85	83	5.3
		315	101, 102, 97	100	2.6	107, 101, 100	103	3.7

Notes:
[1] Samples stored at -18 °C for ca. 25 months followed by storage at -1 °C for six days.
[2] The recovery was performed with a control sample stored a ca. -18 °C (789 days) because not enough material was available for samples stored at ca. -18 °C followed by storage at -1 °C.
[3] Value not retained.

For barley magnitude of the residue_trials, samples were analysed according to Method 01475. Extracts were analysed within 55 hours of extraction for grain and straw, and within 88 hours of extraction for green material (Table 110).

Table 110 Recovery data for isoflucypram in barley according to Method 01475

Crop	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Trial
Barley grain	0.01	90, 95, 98	94	4.3	15-2114
	0.10	96	-	-	
Barely green material	0.01	97, 98	98	-	
	0.10	95	-	-	
	1.0	100	-	-	
	2.0	92	-	-	
Barley straw	0.01	91	-	-	
	0.10	95	-	-	
	2.0	96	-	-	
Barley grain	0.01	97	-	-	15-2113
	0.10	102, 101	102	-	
Barely green material	0.01	98, 99	99	-	
	0.10	96	-	-	
	2.5	93	-	-	
Barley straw	0.01	104	-	-	
	0.10	97	-	-	
	2.0	99	-	-	
Barley grain	0.01	95, 100	98	-	15-2117
	0.10	105	-	-	

Crop	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Trial
Barley green material	0.01	103	-	-	
	0.10	105	-	-	
	2.0	96	-	-	
	2.5	84	-	-	
Barley straw	0.01	102	-	-	
	0.10	96	-	-	
	2.0	92	-	-	
	5.0	77	-	-	
Barley grain	0.01	95, 111	103	-	15-2118
	0.10	107, 111	109	-	
Barley green material	0.01	98	-	-	
	0.10	106	-	-	
	2.0	98	-	-	
	2.5	94	-	-	
Barley straw	0.01	93	-	-	
	0.10	101	-	-	
	2.0	85	-	-	
Barley grain	0.01	94, 95, 96, 98	96	1.8	15-2066
	0.10	100, 100, 101, 104	101	1.9	
Barley green material	0.01	81, 85, 91, 102	90	10.2	
	0.10	80, 81, 93, 98	88	10.1	
	1.0	96	-	-	
	2.0	97	-	-	
Barley straw	0.01	97, 98, 121	105	12.9	
	0.10	89, 100, 101	97	6.9	
	2.0	101	-	-	
Barley grain	0.01	92, 96	94	-	15-2110
	0.10	94	-	-	
Barley green material	0.01	96	-	-	
	0.10	97	-	-	
	2.5	94	-	-	
Barley straw	0.01	114	-	-	
	0.10	100	-	-	
	2.0	93	-	-	
Barley grain	0.01	91, 93, 94, 105	96	6.6	16-2052
	0.10	90	-	-	
Barley green material	0.01	83, 87, 98, 98, 100	93	8.2	
	0.10	95, 97	96	-	
	1.0	96	-	-	
	2.0	93	-	-	
	4.0	96	-	-	
Barley straw	0.01	99	-	-	
	0.10	97	-	-	
	2.0	101	-	-	
Barley grain	0.01	83, 90, 94	89	6.3	16-2051
	0.10	94, 101	98	-	
Barley green material	0.01	94, 86, 79, 85	86	7.2	
	0.10	93, 95	94	-	
	1.0	94	-	-	
	4.0	95	-	-	
Barley straw	0.01	96	-	-	
	0.10	97	-	-	
	1.0	84	-	-	
Wheat/barley grain	0.01	n=4	96	5.9	PNZ16414
	0.10	n=4	102	5.9	

Crop	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Trial
Wheat/barley forage	0.01	n=4	86	8.1	
	0.10	n=4	92	4.5	
	1.0	n=2	98	-	
Wheat/barley straw	0.01	n=8	81	5.7	
	0.10	n=8	78	10.7	
	1.0	n=2	81	-	
Barley grain	0.01	88, 86, 80	85	4.9	BAYERNZ/GLP/16/04/-a
	0.10	81, 86, 82	83	3.2	
Barley forage	0.01	90, 89	90	-	
	0.10	87, 89	88	-	
Barley straw	0.01	88, 84, 77	83	6.7	
	0.10	79, 80, 74	78	4.1	
Barley grain	0.01	6	89	2.9	S17-07996
	0.10	6	89	3.8	
Barley forage	0.01	5	81	6.6	
	0.10	5	84	4.2	
	5.0	3	75	2.7	
Barley straw	0.01	6	84	6.7	
	0.10	6	81	6.6	
	2.0	3	73	0.8	

For wheat magnitude of the residue trials, samples were analysed according to Method 01475. Extracts were analysed within 58 hours after extraction for grain, 80.4 hours of extraction for green material, and 77 hours after extraction for straw (Table 111).

Table 111 Recovery data for isoflucypram in wheat according to Method 01475

Crop	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Trial
Wheat grain	0.01	91, 92	92	-	15-2115
	0.10	102	-	-	
Wheat green material	0.01	86	-	-	
	0.10	85	-	-	
	2.5	97	-	-	
Wheat straw	0.01	98	-	-	
	0.10	105	-	-	
	2.0	96	-	-	
Wheat grain	0.01	99, 96	98	-	15-2116
	0.10	99	-	-	
Wheat green material	0.01	95	-	-	
	0.10	101	-	-	
	1.0	96	-	-	
	2.0	104	-	-	
Wheat straw	0.01	93	-	-	
	0.10	98	-	-	
	2.0	100	-	-	
Wheat grain	0.01	97, 91	94	-	15-2069
	0.10	107	-	-	
Wheat green material	0.01	89, 97, 104, 96	97	6.1	
	0.10	100, 100, 95, 96, 98	98	2.3	
	2.0	101	-	-	
	2.5	101	-	-	
Wheat straw	0.01	104	-	-	
	0.10	101	-	-	
	2.0	105	-	-	

Crop	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Trial
Wheat grain	0.01	99, 100	100	-	15-2120
	0.10	95, 113, 116	108	10.5	
Wheat green material	0.01	81, 98	90	-	
	0.10	111, 112	112	-	
	2.0	83	-	-	
	3.0	95	-	-	
Wheat straw	0.01	97	-	-	
	0.10	109, 112, 116	112	3.1	
	3.0	94	-	-	
	4.0	99	-	-	
Wheat grain	0.01	98	-	-	15-2119
	0.10	95, 95	95	-	
Wheat green material	0.01	94, 102	98	-	
	0.10	103	-	-	
	3.0	82	-	-	
Wheat straw	0.01	100	-	-	
	0.10	103	-	-	
	2.0	91	-	-	
	2.5	94	-	-	
Wheat grain	0.01	101, 104	103	-	15-2111
	0.10	107	-	-	
Wheat green material	0.01	99, 117, 102	106	9.1	
	0.10	97, 98	98	-	
	2.5	99	-	-	
Wheat straw	0.01	92	-	-	
	0.10	98	-	-	
	2.0	80	-	-	
Wheat grain	0.01	93	-	-	16-2053
	0.10	91, 96	94	-	
Wheat green material	0.01	90, 95	93	-	
	0.10	94	-	-	
	4.0	97	-	-	
Wheat straw	0.01	93	-	-	
	0.10	93	-	-	
	4.0	94	-	-	
Wheat grain	0.01	83	-	-	16-2054
	0.10	92, 92	92	-	
Wheat green material	0.01	82	-	-	
	1.0	91	-	-	
	8.0	96	-	-	
Wheat straw	0.01	98	-	-	
	0.10	92	-	-	
	2.0	93	-	-	
	3.0	92	-	-	
Wheat grain	0.01	92	-	-	BAYERNZ/GLP/16/04/a
	0.10	94	-	-	
Wheat forage	0.01	86, 87, 96	90	6.1	
	0.10	89, 90, 89	89	0.6	
	5.0	96, 90	93	-	
Wheat straw	0.01	92, 92, 90	91	1.3	
	0.10	87, 88, 88	88	0.7	
	1.0	86, 87	87	-	
Wheat grain	0.01	6	87	4.8	S17-07939
	0.10	6	83	3.6	
Wheat forage	0.01	5	91	3.3	

Crop	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Trial
	0.10	5	91	4.4	
	5.0	3	84	0.7	
Wheat straw	0.01	6	74	4.4	
	0.10	6	76	8.4	
	2.0	3	76	4.0	

For the barley processing study and feed, samples were analysed according to Method 01475 (Table 112).

Table 112 Summary of concurrent recoveries for isoflucypram on barley grain and processed commodities according to Method 01475 (Trial 15-3407)

Crop / Sample	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)
Barley grain[1]	0.01	98, 98, 95, 102, 102, 90, 110, 117	102	8.4
	0.10	113, 101	107	-
Barley beer	0.01	81, 92, 93, 98	91	7.9
	0.10	88, 102, 105	98	9.2
Barley brewer's yeast[2]	0.01	112, 104, 106, 106, 97	105	5.1
	0.10	101, 102, 97	100	2.6

Notes:
[1] Representative of brewer's grain, brewer's malt, stored grain, malt sprout, pearled barley, and pearled rub off.
[2] Representative of hops draft.

For the lactating cow feeding study and hen feeding studies, feed was screened for residues of isoflucypram according to Method 01475 (Table 113).

Table 113 Concurrent recoveries for isoflucypram residues in feedstuffs

Sample Material	Fortification Level (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Reference
Cobs mixture and straw	0.01	88, 80, 97, 93	90	8.2	17-8001
	0.10	97, 105, 102, 100	101	3.3	
Dairy cattle concentrate	0.01	97, 99, 96	97	1.6	
	0.10	103, 101, 101	102	1.1	
Cow mineral feed	0.01	106, 98, 100	101	4.1	
	0.10	109, 107, 108	108	0.9	
Hen feed	0.01	106, 104, 105, 95	103	4.9	17-8002
	0.10	105, 104, 107, 106	106	1.2	

Data Collection Method LN-002-P16-01

Report Nos. RALN0017 and M-606616-01-1

The Meeting received analytical method descriptions for the residue analytical method LN-002-P16-01, which is an adaptation of the Method 01475 and was developed as a data collection method for the determination of the residues of isoflucypram in/on plant materials using LC-MS/MS.

Briefly, residues of isoflucypram are extracted by two successive extractions with a mixture of ACN/water (4:1). Before the first extraction, crop samples are left to soak for a minimum of 20 minutes in the ACN/water (4:1) extraction solvent mixture prior to blending for two minutes. After filtration, extracts are combined and an isotopic internal standard is added. An aliquot of this sample is diluted with a mixture of ACN/water (1:4) and then subjected to LC-MS/MS. The quantitation MRM transition is m/z

400.2 → 139.2. The LOQ for isoflucypram, defined as the lowest fortification level of an analyte at which an acceptable recovery can be achieved, was 0.01 mg/kg.

The method validation commodities tested included: tomato fruit (high-water content commodity), orange fruit (high-acid content commodity), wheat grain (high starch commodity), soya bean seed, and canola seed (both high-oil content commodities). The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified with isoflucypram at concentrations of 0.01 and 0.10 mg/kg. Mean recoveries per fortification level were in a range of 70–110 percent. Residues in control samples were <30 percent of the LOQ.

Standard solution concentrations ranged from 0.05 ng/mL to 100 ng/mL with R^2 values ≥ 0.998 using at least five concentration levels. The analytical method is fully validated for accuracy, precision, and linearity for determination of isoflucypram residues in crop samples. The results are shown in Table 114.

Table 114 Recovery results for isoflucypram from the method validation of Method LN-002-P16-01

Matrix	n	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Reference
Orange fruit	7	0.01	84-89	86	2	RALN0017
	5	0.10	91-99	95	4	
Wheat grain	7	0.01	86-93	89	2	
	5	0.10	93-98	96	2	
Soya bean seed	7	0.01	87-90	88	1	
	5	0.10	89-94	92	2	
Canola seed	7	0.01	81-87	84	3	
	5	0.10	89-97	93	4	
Tomato fruit	7	0.01	72-97	82	9	
	5	0.10	65-90	81	13	
Wheat grain	11	0.010	91-109	97	6	RALNN137
	6	0.100	97-102	99	2	
Wheat bran	3	0.010	94-103	98	5	
		0.100	93-95	94	1	
Wheat flour, white	3	0.010	99,-101	100	1	
		0.100	95-100	97	3	
Wheat flour, whole meal	3	0.010	91-99	95	4	
		0.100	89-95	93	3	
Wheat germ	3	0.010	95-99	98	2	
		0.100	88-101	95	7	
Wheat widdlings	3	0.010	99-101	100	1	
		0.100	98-102	100	2	
Wheat shorts	3	0.010	93-118	108	12	
		0.100	99-102	101	2	
Wheat pasta, fresh	3	0.010	80-95	87	9	
		0.100	83-95	89	7	
Wheat pasta, dry	3	0.010	83-94	89	6	
		0.100	92-93	91	4	
Wheat pasta, cooked	3	0.010	91-120	105	14	
		0.100	97-99	98	1	
Wheat pasta, dried and cooked	3	0.010	94-99	96	3	
		0.100	97-101	100	2	
Wheat gluten	3	0.010	95-110	100	9	
		0.100	97-101	99	2	
Wheat starch	3	0.010	98-117	105	10	
		0.100	100-101	101	1	
Wheat AGF	3	0.010	90-100	95	5	
		2.50	100-106	104	3	

Matrix	n	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Reference
Wheat white bread	3	0.010	109, 94, 99	101	8	
		0.100	96, 93, 96	95	2	
Wheat whole meal bread	3	0.010	97, 92, 97	95	3	
		0.100	95, 91, 91	92	3	

Data Collection Method 01564

Report Nos. P602186501 and EnSa-18-1075.

The Meeting received analytical method descriptions and validation data for the determination of isoflucypram, isoflucypram-desmethyl-propanol (free and conjugated), and isoflucypram-propanol (free and conjugated) in wheat grain, green material, straw.

Briefly, samples are spiked with an isotopic internal standard of isoflucypram-desmethyl-propanol (1 mg/L), combined with 5 mol/L HCl (15 mL), and heated at 98 °C for 30 minutes. The solution is cooled, combined with water (100 mL), and centrifuged. The supernatant is decanted over a cartridge with a filter frit. The solid pellet is extracted by two successive extractions with ACN/water (4:1). After filtration, extracts are combined with the first supernatant, and isotopic internal standards of isoflucypram and isoflucypram-propanol are added to the sample. An aliquot of this sample is diluted with ACN/water (1:4) and subjected to LC-MS/MS. The quantitation MRM transition for isoflucypram is m/z 400 → 167, for isoflucypram-desmethyl-propanol is m/z 402 → 220, and for isoflucypram-propanol is m/z 416 → 234.

The LOQ for isoflucypram, isoflucypram-desmethyl-propanol, and isoflucypram-propanol was validated at 0.01 mg/kg in wheat grain, green material, and straw. Method validation mean recoveries for each fortification level were within the 70–110 percent range for recoveries at fortification levels of 0.01 mg/kg and 0.10 mg/kg for wheat grain, green material, and straw, expressed as parent equivalents. RSDs were below 20 percent for each analyte in all investigated sample materials.

For each analyte, method linearity was validated over the range of 0.015 to 1.0 µg/L (internal standard calibration solutions) with r values >0.99. Residues in control samples were < 30 percent of the LOQ. The overall performance (extractability and hydrolysis) of the residue analytical Method 01564 was determined for isoflucypram, isoflucypram-desmethyl-propanol (free and conjugated), and isoflucypram-propanol (free and conjugated). Wheat straw and hay extracts were stored for approximately 3.5 years at ≤-18 °C. The stored extracts were analysed using Method 01564 and compared for radioactive residues in the wheat hay and wheat straw primary crop metabolism studies. The extracts contained similar residue profiles prior to hydrolysis.

The extracts underwent the acid hydrolysis step of the analytical method with 5 mol/L HCl at 98 °C for 30 minutes for comparison with the acidic conditions of the metabolism study (1 mol/L HCl at 100 °C for 60 minutes). The extraction efficiencies of the individual compounds and the cleavage of the conjugates were ≥ 83.8 percent. In the metabolism study approximately 5–7 percent of the conjugates were not cleaved to the respective aglycon, which resulted in slightly higher efficiency for the residue analytical method compared to the metabolism study.

The repeatability of the extraction efficiency and hydrolysis yield of the residue analytical method was assessed by five replicates per sample material. The relative standard deviations of the five replicates were low and in the range of 0.4 percent to 4.1 percent which is an indication of the good reproducibility of the extraction procedure as well as the cleavage of the conjugates. Isoflucypram,

isoflucypram-propanol, and isoflucypram-desmethyl-propanol did not show any degradation during acidic hydrolysis (Table 115). Table 116 shows the extraction efficiency of the method for wheat hay and straw.

Table 115 Recovery results (n=5) from the method validation of Method 01564 for isoflucypram and metabolites (Ref. P602186501)

Matrix	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)
Isoflucypram, Transition 400 → 167				
Wheat, grain	0.01	98-108	105	3.7
	0.10	96-102	100	2.5
Wheat, green material	0.01	87-105	97	7.8
	0.10	95-107	100	4.6
Wheat, straw	0.01	101-107	104	2.1
	0.10	99-108	104	3.3
Isoflucypram-desmethyl-propanol, Transition 402 → 220				
Wheat, grain	0.01	84-92	89	3.7
	0.10	87-96	92	4.0
Wheat, green material	0.01	80-92	87	5.1
	0.10	81-84	82	1.6
Wheat, straw	0.01	70-72	71	1.3
	0.10	71-80	74	5.0
Isoflucypram-propanol, Transition 416 → 238				
Wheat, grain	0.01	79-82	81	1.5
	0.10	88-94	90	3.0
Wheat, green material	0.01	91-96	93	2.5
	0.10	95-105	99	3.9
Wheat, straw	0.01	80-96	89	7.6
	0.10	88-97	92	4.2

Table 116 recovery results of extraction efficiency and efficiency of hydrolysis testing of Method 01564 from wheat hay samples from plant pyrazole metabolism studies (Ref. EnSa-18-1075)

	Wheat hay					Wheat straw				
	Method 01564 (5 mol/L HCl)		Metabolism study (1 mol/L HCl)		Extraction Efficiency[1]	Method 01564 (5 mol/L HCl)		Metabolism study (1 mol/L HCl)		Extraction Efficiency[1]
Compound	% TRR	mg eq/kg	% TRR	mg eq/kg	%	% TRR	mg eq/kg	% TRR	mg eq/kg	%
Isoflucypram	45.8	1.668	44.4	1.791	93.1	69.8	10.756	67.0	10.397	103
Isoflucypram-propanol-Glyc	ND	ND	0.8	0.031	NC	ND	ND	0.2	0.024	NC
Isoflucypram-propanol-Glyc-MA	ND	ND	0.9	0.036	NC	ND	ND	0.3	0.056	NC
Isoflucypram-propanol	28.1	1.021	22.3	0.901	113	12.5	1.932	10.5	1.625	119
Sum Isoflucypram-propanol + conjugates	28.1	1.021	24.0	0.968	105	12.5	1.932	11.0	1.705	113
Isoflucypram-desmethyl-propanol-Glyc-MA	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Isoflucypram-desmethyl-propanol	6.2	0.226	6.9	0.277	81.6	3.0	0.465	3.6	0.564	82.4

Notes:

[1] Calculated as concentration (mg eq/kg) from Method 01564 /concentration (mg eq/kg) from the metabolism studies * 100 percent.

Method 01564 was used for data collection in storage stability and magnitude of the residue studies. Calibration curves were established with at least five calibration standards and an r value >0.99

for each analytical set. For storage stability study P642186502, the maximum time between extraction and analysis was 24 hours. The results are shown in Table 117.

Table 117 Summary of Concurrent Recoveries for isoflucypram-desmethyl-propanol and isoflucypram-propanol in wheat commodities according to Method 01564 (Ref. P642186502)

Sample Material	Fortification Level (mg/kg)	Storage interval (days)	Recovery (%)	Mean (%)	RSD (%)
Isoflucypram-desmethyl-propanol					
Wheat grain (Method validation)	0.01	0	99, 93, 92	95	4.0
	0.10	0	105, 97, 109	104	5.9
Wheat grain (Concurrent recovery)	0.1	33	92, 104	98	-
		97	98, 93	96	-
		183	97, 98	98	-
		358	104, 93	99	-
		544	104, 94	99	-
		728	110, 94	102	-
		904	99, 101	100	-
Wheat green material (Method validation)	0.01	2[1]	98, 102, 88	96	7.5
	0.10	2[1]	108, 99, 108	105	4.9
Wheat green material (Concurrent recovery)	0.1	33	105, 98	102	-
		97	96, 116	106	-
		183	90, 89	90	-
		358	95, 86	91	-
		544	92, 94	93	-
		727	101, 102	102	-
		903	91, 88	90	
Wheat straw (Method validation)	0.01	0	98, 99, 93	97	3.3
	0.10	0	104, 100, 101	102	2.0
Wheat straw (Concurrent recovery)	0.1	30	106, 108	107	-
		97	89, 97	93	-
		183	86, 103	95	-
		358	103, 100	102	-
		544	82, 85	84	-
		728	103, 103	103	-
		909	84, 91	88	
Isoflucypram-propanol					-
Wheat grain (Method validation)	0.01	0	92, 71, 92	85	14.3
	0.10	0	97, 88, 95	93	5.1
Wheat grain (Concurrent recovery)	0.1	33	88, 86	87	-
		97	83, 86	85	-
		183	93, 87	90	-
		358	89, 94	92	-
		544	93, 88	91	-
		728	91, 96	94	-
		904	94, 88	91	-
Wheat green material (Method validation)	0.01	2[1]	86, 90, 82	86	4.7
	0.10	2[1]	91, 92, 98	94	4.0
Wheat green material (Concurrent recovery)	0.1	33	88, 86	87	-
		97	86, 87	87	-
		183	87, 90	89	-
		358	85, 91	88	-
		544	92, 95	94	-
		727	94, 94	94	-
		903	90, 89	90	
Wheat straw	0.01	0	86, 84, 83	84	1.8

Sample Material	Fortification Level (mg/kg)	Storage interval (days)	Recovery (%)	Mean (%)	RSD (%)
(Method validation)	0.10	0	82, 87, 86	85	3.1
Wheat straw	0.1	30	89, 86	88	-
(Concurrent recovery)		97	82, 83	83	-
		190	93, 86	90	-
		358	86, 90	88	-
		544	85, 85	85	-
		728	93, 91	92	-
		909	85, 88	87	-

Notes:
[1] The extraction of the 10×LOQ recoveries had to be repeated two days after extracting the storage samples at Day 0.

For barley magnitude of the residue_Trials 16-2052 and 16-2051 (amendments P672186503 and P672186504; isoflucypram-desmethyl-propanol and isoflucypram-propanol only) and E19RP054, E19RP055, E19RP056, 17-2017, and 17-2018 (all three analytes), samples were analysed according to Method 01564. Extracts were analysed within six days of for grain, green material, and straw (Table 117).

Table 118 Recovery Data for isoflucypram, isoflucypram-desmethyl-propanol, and isoflucypram-propanol in barley grain according to Method 01564

Crop Commodity	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Reference
Isoflucypram-desmethyl-propanol					Amendments P672186503 and P672186504 to studies 16-2052 and 16-2051
Barley grain	0.01	99, 102, 97	99	2.5	
	1.0	101, 101, 95	99	3.5	
Barley straw	0.01	78, 72, 73	74	4.3	
	1.0	76, 74, 76	75	1.5	
Isoflucypram-propanol					
Barley grain	0.01	77, 93, 90	87	9.8	
	1.0	71, 89, 79	80	11.3	
Barley straw	0.01	91, 87, 86	88	3.0	
	1.0	91, 86, 79	85	7.1	
Isoflucypram					E19RP054
Barley grain	0.01	101, 101, 104	102	1.7	
	0.5	98, 98, 97	98	0.6	
Barley green	0.01	93, 100, 91	95	5.0	
	2.0	99, 99, 98	99	0.6	
Barley straw	0.01	89, 93, 94	92	2.9	
	1.0	85, 92, 86	88	4.3	
	2.0	92, 96, 90	93	3.3	
Isoflucypram-desmethyl-propanol					
Barley grain	0.01	95, 99, 104	99	4.5	
	0.5	84, 88, 89	87	3.0	
Barley green	0.01	95, 95, 98	96	1.8	
	2.0	89, 90, 91	90	1.1	
Barley straw	0.01	98, 97, 84	93	8.4	
	1.0	76, 79, 76	77	2.2	
	2.0	70, 74, 73	72	2.9	
Isoflucypram-propanol					
Barley grain	0.01	88, 87, 88	88	0.7	
	0.5	86, 92, 88	89	3.4	
Barley green	0.01	94, 95, 94	94	0.6	
	2.0	95, 94, 93	94	1.1	
Barley straw	0.01	86, 90, 91	89	3.0	

Crop Commodity	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Reference
	1.0	83, 84, 81	83	1.8	
	2.0	77, 84, 80	80	4.4	
Isoflucypram					E19RP055
Barley grain	0.01	92, 93, 98	94	3.4	
	0.10	93, 94, 95	94	1.1	
Barley green	0.01	75, 86, 88	83	8.4	
	0.10	84, 86, 95	88	6.6	
	2.0	82, 87, 94	88	6.9	
Barley straw	0.01	94, 105, 105	101	6.3	
	0.50	94, 97, 99	97	2.6	
	2.0	82, 88, 91	87	5.3	
Isoflucypram-desmethyl-propanol					
Barley grain	0.01	97, 98, 103	99	3.2	
	0.10	99, 101, 104	101	2.5	
Barley green	0.01	95, 95, 97	96	1.2	
	0.10	95, 95, 99	96	2.4	
	2.0	78, 85, 88	84	6.1	
Barley straw	0.01	89, 91, 93	91	2.2	
	0.50	90, 91, 93	91	1.7	
Isoflucypram-propanol					
Barley grain	0.01	78, 83, 86	82	4.9	
	0.10	87, 87, 89	88	1.3	
Barley green	0.01	76, 84, 93	84	10.1	
	0.10	85, 86, 94	88	5.6	
	2.0	79, 89, 94	87	8.7	
Barley straw	0.01	75, 82, 84	80	5.9	
	0.50	85, 86, 87	86	1.2	
	2.0	79, 82, 84	82	3.1	
Isoflucypram					E19RP056
Barley grain	0.01	95, 100, 107	101	6.0	
	1.0	99, 100, 103	101	2.1	
Barley green	0.01	101, 101, 106	103	2.8	
	1.0	97, 98, 99	98	1.0	
	2.0	97, 101, 104	101	3.5	
Barley straw	0.01	99, 103, 106	103	3.4	
	1.0	98, 101, 102	100	2.1	
Isoflucypram-desmethyl-propanol					
Barley grain	0.01	95, 98, 104	99	4.6	
	1.0	77, 82, 83	81	4.0	
Barley green	0.01	102, 104, 108	105	2.9	
	1.0	86, 90, 91	89	3.0	
	2.0	89, 91, 93	91	2.2	
Barley straw	0.01	80, 84, 96	87	9.6	
	1.0	71, 74, 77	74	4.1	
Isoflucypram-propanol					
Barley grain	0.01	84, 89, 93	89	5.1	
	1.0	87, 87, 89	88	1.3	
Barely green	0.01	92, 96, 99	96	3.7	
	1.0	95, 95, 95	95	0.0	
	2.0	95, 96, 97	96	1.0	
Barley straw	0.01	86, 89, 91	89	2.8	
	1.0	90, 91, 91	91	0.6	
Isoflucypram					17-2017
Barley grain	0.01	97, 99, 99	98	1.2	

Crop Commodity	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Reference
	0.50	97, 100, 103	100	3.0	
Barley green	0.01	72, 72, 77, 82, 88, 89, 89	81	9.5	
	4.0	89, 89, 102	93	8.0	
Barley straw	0.01	90, 94, 98	94	4.3	
	2.0	87, 88, 89	88	1.1	
Isoflucypram-desmethyl-propanol					
Barley grain	0.01	83, 85, 97	88	8.6	
	0.50	84, 85, 86	85	1.2	
Barley green	0.01	70, 76, 78, 79, 84, 91, 91	81	9.6	
	4.0	82, 82, 84	83	1.4	
Barley straw	0.01	67, 73, 75	72	5.8	
	2.0	67, 71, 76	71	6.3	
Isoflucypram-propanol					
Barley grain	0.01	83, 86, 87	85	2.4	
	0.50	90, 90, 95	92	3.1	
Barley green	0.01	63, 70, 86, 89, 90, 93, 94	84	14.5	
	4.0	90, 92, 92	91	1.3	
Barley straw	0.01	76, 80, 91	82	9.4	
	2.0	83, 83, 84	83	0.7	
Isoflucypram					17-2018
Barley grain	0.01	81, 86, 88	85	4.2	
	1.0	95, 95, 98	96	1.8	
Barley green	0.01	73, 73, 75	74	1.6	
	2.0	75, 78, 85	79	6.5	
Barley straw	0.01	81, 89, 97	89	9.0	
	1.0	87, 91, 96	91	4.9	
Isoflucypram-desmethyl-propanol					
Barley grain	0.01	89, 91, 94	91	2.8	
	1.0	78, 82, 85	82	4.3	
Barley green	0.01	89, 92, 100	94	6.1	
	2.0	95, 98, 99	97	2.1	
Barley straw	0.01	73, 78, 85	79	7.7	
	1.0	73, 75, 76	75	2.0	
Isoflucypram-propanol					
Barley grain	0.01	80, 80, 80	80	0.0	
	1.0	83, 85, 88	85	2.9	
Barley green	0.01	71, 72, 78	74	5.1	
	2.0	71, 72, 79	74	5.9	
Barley straw	0.01	78, 79, 83	80	3.3	
	1.0	81, 83, 95	86	8.8	

For wheat magnitude of the residue trials, samples were analysed according to Method 01564. Samples were analysed within seven days of extraction for wheat grain, green material, and straw (Table 119).

Table 119 Recovery Data for Isoflucypram, Isoflucypram-Desmethyl-Propanol, and Isoflucypram-Propanol According to Method 01564

Crop Commodity	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Reference
Isoflucypram-desmethyl-propanol					Amendments P672186503 and P672186504 to studies 16-2053
Wheat grain	0.01	106, 98, 101, 97	101	4.0	
	1.0	94, 87, 96	92	5.1	
Wheat straw	0.01	73, 79, 73	75	4.6	

Crop Commodity	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Reference
	0.10	79, 78, 83	80	3.3	and 16-2054
	1.0	75, 79, 77	77	2.6	
Isoflucypram-propanol					
Wheat grain	0.01	96, 89, 86, 84	89	5.9	
	1.0	100, 91, 90	94	5.9	
Wheat straw	0.01	78, 78, 74	77	3.0	
	0.10	71, 74, 77	74	4.1	
	1.0	70, 76, 78	75	5.6	
Isoflucypram					18-2014
Wheat grain	0.01	92, 97, 99	96	3.8	
	2.0	77, 81, 82	80	3.3	
Wheat green material	0.01	99, 100, 102	100	1.5	
	2.0	96, 98, 106	100	5.3	
Wheat straw	0.01	101, 103, 104	103	1.5	
	2.0	79, 85, 87	84	5.0	
Isoflucypram-desmethyl-propanol					
Wheat grain	0.01	96, 104, 107	102	5.6	
	2.0	98, 99, 104	100	3.2	
Wheat green material	0.01	92, 94, 100	95	4.4	
	2.0	97, 99, 111	102	7.4	
Wheat straw	0.01	78, 83, 100	87	13.3	
	2.0	78, 87, 93	86	8.8	
Isoflucypram-propanol					
Wheat grain	0.01	90, 96, 99	95	4.8	
	2.0	75, 76, 78	76	2.0	
Wheat green material	0.01	86, 99, 102	96	8.9	
	2.0	96, 97, 107	100	6.1	
Wheat straw	0.01	98, 101, 103	101	2.5	
	2.0	72, 74, 80	75	5.5	
Isoflucypram					18-2135
Wheat grain	0.01	94, 97, 97	96	1.8	
	2.0	76, 79, 79	78	2.2	
Wheat green material	0.01	94, 98, 105	99	5.6	
	4.0	81, 83, 95	86	8.8	
Wheat straw	0.01	94, 100, 101, 101	99	3.4	
	2.0	99, 99, 101	100	1.2	
Isoflucypram-desmethyl-propanol					
Wheat grain	0.01	85, 88, 97	90	6.9	
	2.0	90, 92, 94	92	2.2	
Wheat green material	0.01	87, 94, 96	92	5.1	
	4.0	83, 101, 104	96	11.8	
Wheat straw	0.01	84, 89, 92, 97	91	6.0	
	2.0	97, 97, 98	97	0.6	
Isoflucypram-propanol					
Wheat grain	0.01	93, 97, 98	96	2.8	
	2.0	70, 75, 76	74	4.4	
Wheat green material	0.01	97, 98, 102	99	2.7	
	4.0	74, 79, 86	80	7.6	
Wheat straw	0.01	85, 93, 97, 101	94	7.3	
	2.0	88, 90, 92	90	2.2	
Isoflucypram					17-2020
Wheat grain	0.01	87, 91, 98	92	6.1	

Crop Commodity	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Reference
	0.50	78, 78, 89	82	7.8	
Wheat green material	0.01	74, 88, 90	84	10.4	
	3.0	95, 95, 98	96	1.8	
Wheat straw	0.01	87, 97, 105	96	9.4	
	4.0	96, 98, 105	100	4.7	
Isoflucypram-desmethyl-propanol					
Wheat grain	0.01	88, 101, 106	98	9.4	
	0.50	82, 84, 84	83	1.4	
Wheat green material	0.01	78, 80, 88	82	6.5	
	3.0	76, 78, 78	77	1.5	
Wheat straw	0.01	86, 95, 98	93	6.7	
	4.0	74, 74, 76	75	1.5	
Isoflucypram-propanol					
Wheat grain	0.01	73, 75, 86	78	9.0	
	5.0	72, 77, 81	77	5.9	
Wheat green material	0.01	70, 83, 84	79	9.9	
	3.0	82, 84, 87	84	3.0	
Wheat straw	0.01	74, 100, 100	91	16.4	
	4.0	92, 96, 101	96	4.7	
Isoflucypram					17-2019
Wheat grain	0.01	97, 106, 108	104	5.7	
	0.10	100, 102, 103	102	1.5	
Wheat green	0.01	107, 97, 98, 98	100	4.7	
	0.10	83	-	-	
	2.5	92, 94, 96	94	2.1	
Wheat straw	0.01	104, 106, 111	107	3.4	
	0.10	90, 97, 101	96	5.8	
	2.0	92, 92, 94	93	1.2	
Isoflucypram-desmethyl-propanol					
Wheat grain	0.01	87, 89, 94	90	4.0	
	0.10	89, 95, 96	93	4.1	
Wheat green	0.01	85, 75, 82, 89	83	7.1	
	0.10	86	-	-	
	2.5	81, 81, 82	81	0.7	
Wheat straw	0.01	76, 72, 85	78	8.6	
	0.10	69, 73, 76	73	4.8	
Isoflucypram-propanol					
Wheat grain	0.01	87, 88, 103	93	9.7	
	0.10	91, 99, 103	98	6.3	
Wheat green	0.01	100, 93, 98, 108	100	6.3	
	0.10	88	-	-	
	2.5	95, 100, 104	100	4.5	
Wheat straw	0.01	81, 83, 89	84	4.9	
	0.10	81, 85, 87	84	3.6	
	2.0	78, 80, 84	81	3.8	

Data collection Method 46437, Version 1

Method 46437, version 1 is based on Method 01564. The LOQ was 0.01 mg/kg. Extracts are heated with HCl at 98 °C, extracted twice with ACN:water, combined, and analysed by LC-MS/MS with internal standards.

For barley magnitude of the residue trials GLP658 and S18-07828, samples were analysed by Method 46437, version 1. For each analytical set, a calibration curve was performed with a coefficient of determination (R^2) above 0.99. Samples were analysed on the same day as extraction (Table 120).

Table 120 Recovery data for isoflucypram, isoflucypram-desmethyl-propanol, and isoflucypram-propanol in barley forage and straw according to Method 46437, Version 1

Crop Commodity	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)
Isoflucypram-desmethyl-propanol, GLP658 (Amendment to study S17-07996)				
Barley grain	0.01	5	104	3.8
	0.10	6	102	1.5
Barley forage	0.01	6	105	4.6
	0.10	6	105	4.2
	1.0	3	95	6.0
Barley straw	0.01	5	98	6.2
	0.10	5	105	2.5
	1.0	3	95	1.1
Isoflucypram-propanol, GLP658 (Amendment to study S17-07996)				
Barley grain	0.01	5	76	6.4
	0.10	5	79	8.0
Barley forage	0.01	6	85	7.0
	0.10	6	89	8.9
	1.0	3	81	1.2
Barley straw	0.01	5	86	5.3
	0.10	5	90	4.0
	1.0	3	83	3.2
Isoflucypram, S18-07828				
Barley grain	0.01	3	95	6.5
	0.10	3	97	7.8
Barley forage	0.01	5	87	6.0
	0.10	5	88	4.0
	5.0	3	96	4.0
Barley straw	0.01	5	86	12.5
	0.10	5	93	5.0
	1.0	3	92	9.3
Isoflucypram-desmethyl-propanol, S18-07828				
Barley grain	0.01	3	107	2.2
	0.10	3	104	1.0
Barley forage	0.01	5	101	4.3
	0.10	5	98	2.9
	5.0	3	75	2.8
Barley straw	0.01	5	97	11.8
	0.10	5	103	3.8
	1.0	3	99	9.2
Isoflucypram-propanol, S18-07828				
Barley grain	0.01	3	92	8.7
	0.10	3	82	5.2
Barley forage	0.01	5	80	5.0
	0.10	5	80	3.2
	5.0	3	85	5.4
Barley straw	0.01	5	79	11.5
	0.10	5	81	4.6
	1.0	3	84	13.0

For magnitude of the residue trials GLP655 and S18-07829, samples were analysed for residues by Method 46437, version 1. For each analytical set, a calibration curve was performed with an R^2 above 0.99. Samples were analysed within six days of extraction (Table 121).

Table 121 Recovery data for isoflucypram, isoflucypram-desmethyl-propanol, and isoflucypram-propanol in wheat green material and straw according to Method 46437, Version 1

Crop Commodity	Concentration (mg/kg)	Recovery (%)	Mean (%)	RSD (%)
Isoflucypram-desmethyl-propanol, GLP655 (Amendment to study S17-07939)				
Wheat grain	0.01	6	93	4.2
	0.10	6	94	4.3
Wheat forage	0.01	6	101	5.8
	0.10	5	100	7.2
	1.0	3	102	3.9
Wheat straw	0.01	5	102	11.0
	0.10	5	99	4.9
	1.0	3	104	2.0
Isoflucypram-propanol, GLP655 (Amendment to study S17-07939)				
Wheat grain	0.01	6	75	10.5
	0.10	6	78	2.7
Wheat forage	0.01	6	80	9.5
	0.10	5	80	8.2
	1.0	3	87	1.8
Wheat straw	0.01	5	80	6.1
	0.10	5	82	6.0
	1.0	3	84	4.9
Isoflucypram, S18-07829				
Wheat grain	0.01	3	95	12.2
	0.10	3	86	5.2
Wheat forage	0.01	3	95	9.8
	0.10	3	94	2.6
	2.5	2	99	-
Wheat straw	0.01	5	82	8.9
	0.10	5	92	11.4
	1.0	3	77	9.0
Isoflucypram-desmethyl-propanol, S18-07829				
Wheat grain	0.01	3	100	0.0
	0.10	3	100	0.4
Wheat forage	0.01	3	107	2.2
	0.10	3	105	1.3
	2.5	2	83	-
Wheat straw	0.01	5	104	3.8
	0.10	5	106	6.3
	1.0	2	104	-
Isoflucypram-propanol, S18-07829				
Wheat grain	0.01	3	87	2.7
	0.10	3	81	1.9
Wheat forage	0.01	3	89	6.8
	0.10	3	88	5.7
	2.5	2	90	-
Wheat straw	0.01	4	80	10.8
	0.10	4	82	9.1
	1.0	3	76	2.7

Monitoring Method 01520

Report Nos. MR-17/239 and P 4386 G.

The Meeting received analytical method descriptions and validation data for the determination of isoflucypram residues in/on plants using LC-MS/MS Method 01520. Method 01520 uses the same extraction conditions as Method 01475 which was shown capable to extract the residues of isoflucypram from plant samples. Matrix-matched standards are used.

For method validation, samples were extracted from tomato (fruit), orange (fruit), wheat (grain), coffee (green bean), oilseed rape (seed), and bean (dry seed) by two successive extractions using a high-speed blender with a mixture of ACN/water (8:2). Commodities with low moisture content (wheat grain, rape seed, bean dry seed, and coffee green bean) were left for soaking in water before extraction. After centrifugation the combined extracts were adjusted volumetrically, filtered, and diluted with a mixture of ACN/water (17:73) for LC-MS/MS determination. Two MRM transitions were monitored for isoflucypram and each matrix tested m/z 400 → 139 as 1st MRM (quantification) and m/z 400 → 177 as 2nd MRM (confirmation).

Method 01520 was also independently validated for the determination of isoflucypram residues in/on plant using LC-MS/MS. The LOQ for isoflucypram is 0.01 mg/kg in the six tested crop matrix types using both the quantification and the confirmatory MRM transitions. The correlation between the injected amount of substance and the detector response was linear for matrix matched standards ranging from 0.005 µg/L to 2.0 µg/L. with r values > 0.99.

Mean recoveries for each fortification level and the overall mean recovery were within the 70–110 percent range for all matrices with acceptable RSD (Table 122).

Table 122 Recovery results (n=5) from the method validation of Method 01520–Isoflucypram

Matrix	Spiking Level (mg/kg)	Quantitation: Transition 400→ 139			Confirmatory: Transition 400 → 177			Reference
		Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)	
Tomato	0.01	100-107	103	2.8	96-105	101	3.2	MR-17/239
	0.1	100-105	102	1.9	99-104	101	1.9	
Orange	0.01	83-97	91	6.2	91-104	96	5.2	
	0.1	91-102	95	4.6	91-102	95	4.4	
Wheat grain	0.01	98-117	108	8.5	98-120	108	7.7	
	0.1	97-101	99	1.7	97-103	99	2.3	
Coffee green bean	0.01	89-94	91	2.1	85-93	89	3.2	
	0.1	91-101	98	4.2	89-100	96	4.7	
Rape seed	0.01	84-92	88	4.3	87-90	88	1.7	
	0.1	89-93	91	1.8	88-90	90	2.0	
Bean dry seed	0.01	91-98	94	3.1	93-103	97	4.0	
	0.1	94-101	97	3.1	92-99	96	3.0	
Tomato fruit	0.01	97-104	101	2.6	97-107	102	3.6	P 4386 G
	0.1	97-100	99	1.4	96-100	98	1.7	
Orange fruit	0.01	80-109	96	13	85-114	99	12	
	0.1	69-106	91	16	69-106	91	16	
Wheat grain	0.01	91-100	94	4.0	84-95	90	4.7	
	0.1	96-100	98	1.4	97-102	98	2.2	
Oilseed rape seed	0.01	79-91	84	6.6	79-99	85	9.9	
	0.1	92-101	96	3.3	90-96	94	2.6	
Bean dry seed	0.01	89-126	104	15	85-118	99	12	
	0.1	85-107	98	9.3	90-103	98	6.3	
Coffee green bean	0.01	64-98	81	18	68-98	82	16	

Matrix	Spiking Level (mg/kg)	Quantitation: Transition 400→ 139			Confirmatory: Transition 400 → 177			
		Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)	Reference
	0.1	84-93	87	4.2	83-91	87	3.7	

Multi-Residue Methods

Report Nos. EnSa-17-0483, EnSa-17-0551, and S16-05413.

The extraction efficiency of the QuEChERS analytical method was tested for the determination of isoflucypram in tomatoes, wheat, soya bean, oilseed rape, rotational turnip leaves, rotational Swiss chard, and rotational wheat grain. Extracts of tomato, wheat commodities, soya bean commodities, oilseed rape commodities, turnip leaves, and Swiss chard were stored for a maximum of 13, 25, 26, 6, 19, and 19 months, respectively, at ≤-18 °C. Residues in the stored extracts were analysed using the QuEChERS method and the recoveries were compared to those in the respective metabolism studies Table 123.

Table 123 Isoflucypram - extraction efficiency of the QuEChERS analytical method from representative matrices from plant pyrazole metabolism studies

	QuEChERS Extraction		Extraction in metabolism studies[1]		Extraction efficiency[2]
Sample	% TRR	mg/kg	% TRR	mg/kg	%
Primary crops, S16-05413, EnSa-17-0483					
Tomato fruit	82.3	0.181	96.7	0.165	110
Wheat hay	24.7	0.986	50.0	2.016	48.9
Wheat straw	35.1	5.955	64.0	9.933	60.0
Wheat grain	78.6	0.210	92.0	0.354	59.3
Soya bean forage	8.4	0.354	18.7	0.819	43.2
Soya bean hay	4.5	0.138	10.4	0.487	28.3
Soya bean straw	47.2	8.859	64.5	11.424	77.5
Soya bean seeds	67.1	0.019	76.6	0.027	70.3
OSR intermediate harvest	60.8	2.795	81.9	3.890	71.9
OSR seeds	54.9	0.051	71.0	0.070	72.9
Confined rotational crops[3], EnSa-17-0551					
Turnip leaves (1st rotation)	3.4[4]	0.0006[4]	4.8	0.0009	66.7
Immature Swiss chard (1st rotation)	5.7[4]	0.0020[4]	6.0	0.0019	105

Notes:

[1] Combined conventional and exhaustive extractions, if applicable.

[2] Calculated by: Concentration (mg/kg) from QuEChERS analytical method/concentration (mg/kg) from metabolism studies * 100 percent.

[3] No HPLC analysis of confined rotational wheat grain extracts was performed as isoflucypram was not detected in this matrix in the confined rotational wheat grain metabolism study.

[4] Mean of three replicates.

Animal Matrices

Data Collection Method 01511

Report Nos. P603166029 and M-605551-01-1

The Meeting received analytical method descriptions and validation data for the determination of free residues of isoflucypram, isoflucypram-2-propanol, isoflucypram-carboxylic acid, isoflucypram-propanol, isoflucypram-desmethyl-propanol, isoflucypram-desmethyl-carboxylic acid in/on animal tissues, milk, and eggs by HPLC-MS/MS. Method 01511 also allows the determination of the sum of isoflucypram-propanol and its conjugated residue and the sum of isoflucypram-2-propanol and its conjugated residues in cow liver and kidney and the sum of isoflucypram-desmethyl-propanol and its conjugated residue in hen liver by HPLC-MS/MS.

Briefly, residues are extracted from milk, muscle, kidney and eggs samples with a mixture of ACN/water (4:1). After addition of internal standards, the combined raw extracts (extract A) are diluted and subjected to HPLC-MS/MS for the analysis of free residues of isoflucypram, isoflucypram-2-propanol, isoflucypram-carboxylic acid, isoflucypram-propanol, isoflucypram-desmethyl-propanol, and isoflucypram-desmethyl-carboxylic acid.

For free and conjugated residues of isoflucypram-2-propanol and isoflucypram-propanol, an aliquot of extract is evaporated to dryness at 50 °C, followed by a redissolution in water. The obtained extracts are enzymatically hydrolysed with ß-glucuronidase/arylsulfatase for 20 hours at 37 °C. The extracts are cleaned-up using an Oasis SPE column. The eluted solution is evaporated to dryness at 50 °C and redissolved in a mixture of ACN/water (4:1). The final extract is then subjected to HPLC-MS/MS analysis.

For free and conjugated residues of isoflucypram-desmethyl-propanol, an aliquot of extract A is evaporated to dryness at 50 °C, followed by a redissolution in water. The obtained extracts are enzymatically hydrolysed with ß-glucuronidase/arylsulfatase for 96 hours at 37 °C. The extracts are cleaned-up using an Oasis SPE column. The eluted solution is evaporated to dryness at 50 °C and redissolved in a mixture of ACN/water (4:1). The final extract is then subjected to HPLC-MS/MS for the analysis. The LOQ for all analytes is 0.01 mg/kg except for cattle (milk) for which the LOQ is 0.005 mg/kg. Table 124 shows the MRM transitions monitored in the method

Table 124 MRM transitions were monitored for each matrix tested and for each analyte

Analyte	1st MRM (quantitation)	2nd MRM (confirmation)
Isoflucypram	m/z 400.1 → 167.1	400.1 → 139.1
Isoflucypram-2-propanol	m/z 416.1 → 177.0	416.1 → 398.0
Isoflucypram-carboxylic acid	m/z 430.1 → 177.0	430.1 → 412.1
Isoflucypram-propanol	m/z 416.2[1] → 234.1	416.2 → 177.0
Isoflucypram-desmethyl-propanol	m/z 402.1 → 220.1	402.1 → 58.1
Isoflucypram-desmethyl-carboxylic acid	m/z 416.1 → 236.2	416.1 → 208.2

Notes:

[1] Due to fluctuation in the instrument during the measurement on this study there was a slight shift observed from 416.1 to 416.2 m/z during the course of the study.

The linearity range of the detector used was determined for each analyte using internal standards. The tested concentrations were 0.025 µg/L to 5.0 µg/L with r values >0.99. For method validation, eggs, milk, cattle muscle, cattle fat, cattle liver, cattle kidney, and hen liver were analysed. For each matrix, at least five recovery-tests were conducted at the LOQ and five further recovery-tests were

conducted at 10× LOQ. Using the quantification MRM the mean recoveries per fortification level, matrix and analyte ranged between 87 percent and 113 percent (Table 125).

A waiver for extraction efficiency data was received (Report M-605551-01-1) as samples are extracted with the same conventional conditions and with the same solvent mixture of ACN/water (8:2) as performed in the animal metabolism studies. Based on the metabolism studies, the extraction steps used in Method 01511 release > 91.8 percent TTR in all matrices investigated.

Table 125 Recovery results from the method validation of Method 01511 for isoflucypram and metabolites (Ref. P60316602)

Matrix	n	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
	Isoflucypram							
	Quantitation: Transition 400.1 → 167				Confirmatory: Transition 400.1 → 139.1			
Hen egg	5	0.01	95-101	98	2.6	95-103	97	3.4
	5	0.10	94-107	103	5.1	100-111	107	4.0
Cow milk	7	0.005	98-105	102	2.5	99-105	101	2.0
	5	0.05	107-110	108	1.1	110-114	112	1.5
Cow muscle	5	0.01	94-101	97	3.1	94-98	96	1.9
	5	0.10	100-104	102	1.6	104-109	106	1.9
Cow fat	5	0.01	98-100	99	1.1	99-102	101	1.3
	5	0.10	102-105	104	1.5	101-107	104	2.4
Cow liver	5	0.01	84-104	97	8.2	84-104	96	8.7
	5	0.10	95-105	101	3.6	96-105	102	3.6
Cow kidney	5	0.01	95-99	97	1.7	97-103	99	2.6
	5	0.10	105-109	107	1.4	107-112	110	1.8
Hen liver	5	0.01	94-100	98	2.6	92-101	97	3.3
	5	0.10	99-104	101	1.9	102-107	105	2.1
	Isoflucypam-2-Propanol							
	Quantitation: Transition 416.1 → 177.0				Confirmatory: Transition 416.1 → 398.0			
Hen egg	5	0.01	91-117	102	9.2	90-131[1]	111	13.7
	5	0.10	102-118	111	5.5	92-133[1]	106	16.3
Cow milk	7	0.005	84-118	98	13.3	87-131[1]	107	15.1
	5	0.05	101-120	111	8.2	85-124[1]	101	16.7
Cow muscle	5	0.01	96-117	104	8.4	112-145	131	9.1
	5	0.10	96-116	104	7.9	78-137	106	21.7
Cow fat	5	0.01	79-102	93	10.1	78-94	87	7.3
	5	0.10	98-113	105	5.3	90-117	106	10.0
Cow liver	5	0.01	74-111	91	14.6	78-110	89	13.5
	5	0.10	84-106	94	8.7	85-108	93	9.4
Cow liver after hydrolysis[2]	5	0.01	100-129[1]	113	10.8	NA	NA	NA
	5	0.10	96-108	100	4.8	87-114	103	9.9
Cow kidney	5	0.01	84-114	94	12.4	74-108	97	14.2
	5	0.10	89-117	98	11.9	98-119	108	8.1
Cow kidney after hydrolysis[2]	5	0.01	88-113	99	10.5	NA	NA	NA
	5	0.10	104-119	109	5.4	91-153	131	18.8
Hen liver	5	0.01	76-109	92	12.9	80-100	93	9.1
	5	0.10	97-112	104	6.2	86-137[1]	107	18.2
	Isoflucypram-carboxylic acid							
	Quantitation: Transition 430.1 → 177.0				Confirmatory: Transition 430.1 → 412.1			
Hen egg	5	0.01	92-106	101	5.3	94-105	98	4.7
	5	0.10	99-110	104	3.8	92-105	98	5.7
Cow milk	7	0.005	94-116	106	7.3	88-118	101	9.7
	5	0.05	102-120	110	6.0	100-118	105	7.2
Cow muscle	5	0.01	86-97	92	4.5	85-108	96	9.9

Matrix	n	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
	5	0.10	100-105	102	1.9	95-98	96	1.2
Cow fat	5	0.01	98-106	103	3.3	86-110	97	9.4
	5	0.10	99-112	105	5.9	93-108	100	5.5
Cow liver	5	0.01	84-111	97	10.0	87-113	102	9.5
	5	0.10	81-100	94	8.1	86-93	89	2.8
Cow kidney	5	0.01	102-116	108	5.2	90-113	100	10.0
	5	0.10	105-112	109	3.1	101-108	104	2.6
Hen liver	5	0.01	96-102	98	2.6	89-110	101	8.3
	5	0.10	93-103	97	3.9	90-110	99	7.4
	Isoflucypram-propanol							
	Quantitation: Transition 416.2[2] → 234.1				Confirmatory: Transition 416.2[2] → 177.0			
Hen egg	5	0.01	101-111	105	4.4	96-105	101	3.9
	5	0.10	97-108	102	4.5	104-112	107	3.1
Cow milk	7	0.005	95-109	104	4.7	102-110	104	2.9
	5	0.05	98-114	104	6.1	102-114	108	4.1
Cow muscle	5	0.01	94-105	97	4.5	96-105	100	3.4
	5	0.10	94-99	97	1.9	96-104	101	3.8
Cow fat	5	0.01	95-108	102	5.5	90-107	97	6.8
	5	0.10	94-103	100	3.4	93-103	97	4.0
Cow liver	5	0.01	85-104	97	7.7	84-99	92	7.2
	5	0.10	89-97	95	3.7	88-101	94	5.1
Cow liver after hydrolysis	5	0.01	75-102	91	11.1	72-97	89	11.0
	5	0.10	88-97	93	3.9	94-96	95	1.8
Cow kidney	5	0.01	94-106	98	4.6	94-105	100	4.5
	5	0.10	103-107	105	1.7	106-112	110	2.3
Cow kidney after hydrolysis	5	0.01	96-106	100	3.9	96-112	105	6.6
	5	0.10	102-110	105	3.0	104-112	109	2.9
Hen liver	5	0.01	95-102	99	3.1	92-100	94	3.6
	5	0.10	95-99	97	1.6	99-108	102	3.6
	Isoflucypram-desmethyl-propanol							
	Quantitation: Transition m/z 402.1 → 220.1				Confirmatory: Transition 402.1 → 58.1			
Hen eggs	5	0.01	89-109	99	9.5	94-110	101	6.5
	5	0.10	88-107	98	7.3	87-102	96	6.5
Cow milk	7	0.005	98-107	102	3.1	74-102	93	10.6
	5	0.05	94-108	102	5.2	90-101	97	4.5
Cow muscle	5	0.01	93-100	95	2.9	91-109	99	7.3
	5	0.10	87-95	92	3.4	83-97	91	6.6
Cow fat	5	0.01	93-100	96	2.8	87-109	98	8.6
	5	0.10	93-103	98	4.5	95-106	101	5.0
Cow liver	5	0.01	75-102	88	12.2	89-102	95	7.1
	5	0.10	86-94	90	3.8	84-102	90	7.6
Cow kidney	5	0.01	98-108	102	4.0	97-106	103	3.7
	5	0.10	97-107	102	3.9	94-108	103	5.4
Hen liver	5	0.01	92-106	98	5.8	98-112	105	6.3
	5	0.10	97-102	100	2.2	97-107	101	3.7
Hen liver after hydrolysis	5	0.01	88-116	99	11.5	92-108	98	6.8
	5	0.10	95-109	101	5.1	101-113	105	4.7
	Isoflucypram-desmethyl-carboxylic acid							
	Quantitation: Transition m/z 416.1 → 236.2				Confirmatory: Transition 416.1 → 208.2			
Hen eggs	5	0.01	85-91	88	2.5	85-99	92	5.5
	5	0.10	89-110	96	8.4	91-114	97	10.1
Cow milk	7	0.005	88-104	96	6.1	86-108	93	8.9
	5	0.05	99-112	105	4.8	105-114	109	3.1
Cow muscle	5	0.01	70-95	87	11.8	89-109	99	7.7

Matrix	n	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
	5	0.10	94-101	97	2.8	93-104	99	4.1
Cow fat	5	0.01	105-111	108	2.2	100-120	111	6.6
	5	0.10	97-105	100	3.6	95-110	103	6.6
Cow liver	5	0.01	83-100	91	8.3	74-112	97	14.3
	5	0.10	89-95	91	2.9	90-100	95	4.4
Cow kidney	5	0.01	104-117	111	5.1	114-120	118	1.9
	5	0.10	101-109	104	3.2	93-111	102	6.5
Hen liver	5	0.01	82-107	96	10.5	98-121[2]	111	7.5
	5	0.10	95-109	101	5.1	114-96	102	7.2

Notes:

[1] These results are considered acceptable because they were not identified as outliers by a Grubbs outlier test with a level of significance of 95 percent.

[2] Due to fluctuation in the instrument during the measurement on this study there was a slight shift observed from 416.1 to 416.2 m/z during the course of the study.

Method 01511 was used for data collection in the lactating cow and laying hen feeding studies. Calibration curves were established with coefficients of correlation >0.99. For the lactating cow study, the maximum time from extraction to analysis was 27 days. The maximum period of demonstrated stability for residues in the extract is 23 days. Due to acceptable concurrent recoveries in cream and whey samples, the additional four days between extraction and analysis beyond the demonstrated period is considered acceptable. The results are shown in Table 126.

Table 126 Concurrent recoveries for isoflucypram and metabolites in cattle matrices according to Method 01511 (Ref. 17-8001)

Sample Material	Fortification Level (mg/kg)	Recovery (%) Range (n)	Mean (%)	RSD (%)
Isoflucypram				
Cattle milk	0.005	91-116 (48)	102	5.4
	0.05	102-116 (34)	110	3.2
Cattle cream	0.005	102-108 (5)	105	2.5
	0.20	105-112 (3)	109	3.3
Cattle whey	0.005	100-112 (6)	105	4.1
	0.05	103-111 (3)	108	4.0
Cattle muscle	0.01	103-120 (4)	109	7.4
	0.25	105-113 (4)	109	3.2
Cattle fat (mesenteric)	0.01	93-118 (4)	103	11.2
	0.25	100-108 (4)	103	3.3
Cattle fat (perirenal)	0.01	96-101 (4)	98	2.4
	0.25	99-104 (4)	101	2.0
Cattle fat (subcutaneous)	0.01	93-104 (4)	99	4.8
	0.25	98-106 (4)	102	3.4
Cattle kidney	0.01	97-105 (4)	102	3.6
	0.25	104-108 (4)	106	1.6
Cattle liver	0.01	96-106 (3)	102	5.0
	0.25	102-106 (4)	104	1.8
Isoflucypram-2-propanol				
Cattle milk	0.005	62-150 (46)	104	21.7
	0.05	70-132 (33)	97	15.6
Cattle cream	0.005	107-116 (6)	111	3.4
	0.20	106-110 (3)	108	1.9
Cattle whey	0.005	96-106 (5)	101	4.0
	0.05	107-116 (3)	111	4.1
Cattle muscle	0.01	80-112 (3)	99	16.9

Sample Material	Fortification Level (mg/kg)	Recovery (%) Range (n)	Mean (%)	RSD (%)
	0.25	81-108 (4)	98	13.4
Cattle fat	0.01	65-101 (4)	84	19.2
(mesenteric)	0.25	91-102 (4)	99	5.1
Cattle fat	0.01	93-119 (4)	106	10.3
(perirenal)	0.25	94-106 (4)	100	4.9
Cattle fat	0.01	79-107 (4)	97	13.0
(subcutaneous)	0.25	96-111 (4)	103	7.4
Cattle kidney	0.01	96-114 (3)	108	9.4
	0.25	93-108 (4)	100	6.3
Cattle liver	0.01	82-109 (4)	101	13.4
	0.25	102-108 (4)	104	2.8
Isoflucypram-carboxylic acid				
Cattle milk	0.005	85-125 (48)	103	7.7
	0.05	96-124 (33)	109	5.7
Cattle cream	0.005	104-119 (6)	111	5.4
	0.20	107-119 (3)	114	5.5
Cattle whey	0.005	94-110 (6)	105	5.5
	0.05	102-115 (3)	109	6.1
Cattle muscle	0.01	108-111 (4)	110	1.4
	0.25	104-116 (4)	110	4.5
Cattle fat	0.01	103-108 (4)	104	2.4
(mesenteric)	0.25	97-102 (4)	100	2.1
Cattle fat	0.01	93-98 (4)	95	2.5
(perirenal)	0.25	98-107 (4)	102	4.0
Cattle fat	0.01	99-111 (4)	106	6.1
(subcutaneous)	0.25	94-112 (4)	102	7.7
Cattle kidney	0.01	96-103 (4)	99	2.9
	0.25	95-117 (4)	107	10.2
Cattle liver	0.01	99-112 (4)	106	5.7
	0.25	88-112 (4)	99	101
Isoflucypram-propanol				
Cattle milk	0.005	87-116 (48)	104	6.4
	0.05	88-119 (34)	108	5.5
Cattle whey	0.005	95-12 (6)	104	6.6
	0.05	103, 104, 110 (3)	106	3.6
Cattle cream	0.005	99-106 (6)	103	2.4
	0.20	100-109 (3)	104	4.6
Cattle muscle	0.01	94-109 (4)	102	6.4
	0.25	98-107 (4)	102	3.7
Cattle fat	0.01	95-113 (4)	101	8.1
(mesenteric)	0.25	96-106 (4)	102	5.2
Cattle fat	0.01	101-107 (4)	104	2.4
(perirenal)	0.25	97-108 (4)	104	4.5
Cattle fat	0.01	92-106 (4)	101	6.0
(subcutaneous)	0.25	91-105 (4)	99	5.9
Cattle kidney	0.01	90-104 (4)	96	6.1
	0.25	94-96 (4)	95	0.9
Cattle liver	0.01	101-111 (4)	106	4.9
	0.25	101-108 (4)	104	3.2
Isoflucypram-desmethyl-propanol				
Cattle milk	0.005	83-125 (48)	101	8.9
	0.05	92-121 (34)	104	6.6
Cattle cream	0.005	96-112 (6)	105	7.1

Sample Material	Fortification Level (mg/kg)	Recovery (%) Range (n)	Mean (%)	RSD (%)
	0.20	93, 99, 104 (3)	99	5.6
Cattle whey	0.005	100-113 (6)	106	4.4
	0.05	95-104 (3)	100	4.7
Cattle muscle	0.01	96-103 (3)	101	3.3
	0.25	97-109 (4)	103	5.5
Cattle fat (mesenteric)	0.01	99-107 (4)	103	4.2
	0.25	93-106 (4)	101	6.0
Cattle fat (perirenal)	0.01	92-108 (4)	99	7.1
	0.25	94-102 (4)	98	3.9
Cattle fat (subcutaneous)	0.01	91-103 (4)	98	5.2
	0.25	93-110 (4)	100	7.7
Cattle kidney	0.01	94-110 (4)	103	6.5
	0.25	100-107 (4)	105	3.0
Cattle liver	0.01	95-113 (4)	102	7.8
	0.25	94-108 (4)	101	5.8
Isoflucypram-desmethyl-carboxylic acid				
Cattle milk	0.005	83-127 (48)	105	9.7
	0.05	89-120 (34)	103	7.2
Cattle cream	0.005	106-118 (5)	111	4.2
	0.20	85-110 (3)	95	13.7
Cattle whey	0.005	103-115 (6)	109	4.1
	0.05	98-104 (3)	101	3.0
Cattle muscle	0.01	88-114 (4)	103	12.3
	0.25	95-111 (4)	102	6.7
Cattle fat (mesenteric)	0.01	88-114 (3)	98	14.3
	0.25	92-93 (4)	93	0.6
Cattle fat (perirenal)	0.01	64-109 (4)	88	27
	0.25	61-106 (4)	85	26
Cattle fat (subcutaneous)	0.01	100-112 (4)	107	4.8
	0.25	86-96 (4)	92	4.6
Cattle kidney	0.01	91-105 (4)	101	6.6
	0.25	97-104 (4)	100	2.9
Cattle liver	0.01	89-104 (4)	97	6.5
	0.25	104-112 (4)	107	3.6
Free and conjugated isoflucypram-2-propanol				
Cattle kidney	0.01	85-117 (4)	105	13.3
	0.25	106-111 (4)	107	2.5
Cattle Liver	0.01	81-114 (4)	95	21.5
	0.25	98-105 (4)	102	3.0
Free and conjugated isoflucypram-propanol				
Cattle kidney	0.01	83-106 (4)	95	10.0
	0.25	90-112 (4)	101	9.1
Cattle liver	0.01	94-110 (4)	99	7.6
	0.25	96-110 (4)	103	7.0

For the laying hen feeding study. All extracts of eggs and tissues were analysed within 13 days (eggs, yolk and egg white) after extraction. The results are shown in Table 127.

Table 127 Concurrent recoveries for isoflucypram and metabolites according to Method 01511 (Ref. 17-8002)

Sample Material	Fortification Level (mg/kg)	Recovery (%) Range (n)	Mean (%)	RSD (%)
Isoflucypram				
Whole egg	0.01	94-118 (27)	104	6.2
	0.10	91-113 (27)	103	4.3
Egg white	0.01	99-114 (4)	105	6.4
	0.10	92-103 (4)	99	4.7
Egg yolk	0.01	105-116 (4)	112	4.2
	0.10	102-109 (4)	105	2.8
Hen fat	0.01	103-116 (5)	107	5.1
	0.10	104	-	-
	1.0	90-93 (4)	92	1.6
Hen liver	0.01	106-114 (3)	110	3.6
	0.10	109	-	-
	1.0	93, 95	94	-
Hen muscle	0.01	106-118 (6)	112	3.6
	0.10	101-108 (4)	105	3.0
Isoflucypram-2-propanol				
Egg whole	0.01	77-136 (27)	105	15.0
	0.10	82-125 (27)	103	10.2
Egg white	0.01	75-99 (4)	92	12.4
	0.10	89-106 (4)	97	7.7
Egg yolk	0.01	94-113 (4)	106	7.7
	0.10	98-114 (4)	107	6.6
Fat	0.01	69-102 (4)	86	18.0
	0.10	109	-	-
	1.0	89-102 (4)	94	6.3
Liver	0.01	83-124 (3)	109	20.9
	0.10	106	-	-
	1.0	89, 102	96	-
Muscle	0.01	79-128 (6)	107	14.9
	0.10	102-104 (4)	103	0.9
Isoflucypram-carboxylic acid				
Egg whole	0.01	93-116 (27)	102	6.1
	0.10	93-112 (26)	102	5.1
Egg white	0.01	108-114 (4)	111	2.3
	0.10	90-110 (4)	101	8.4
Egg yolk	0.01	106-110 (4)	108	1.6
	0.10	98-107 (4)	101	4.0
Fat	0.01	91-113 (5)	103	8.4
	0.10	112	-	-
	1.0	95-98 (4)	97	1.8
Liver	0.01	102-115 (3)	110	6.2
	0.10	112	-	-
	1.0	98, 104	101	-
Muscle	0.01	102-113 (5)	107	4.5
	0.10	93-107 (4)	101	5.8
Isoflucypram-propanol				
Egg whole	0.01	86-127 (27)	104	8.9
	0.10	89-108 (27)	101	4.4
Egg white	0.01	91-118 (4)	106	10.7
	0.10	99-109 (4)	103	4.1

Sample Material	Fortification Level (mg/kg)	Recovery (%) Range (n)	Mean (%)	RSD (%)
Egg yolk	0.01	102-113 (4)	106	4.7
	0.10	105-111 (4)	107	2.7
Fat	0.01	96-108 (5)	103	5.0
	0.10	110	-	-
	1.0	92-101 (4)	96	3.9
Liver	0.01	104-118 (3)	109	7.4
	0.10	105	-	-
	1.0	98, 102	100	-
Muscle	0.01	96-117 (6)	103	7.3
	0.10	99-106 (4)	103	2.9
Isoflucypram-desmethyl-propanol				
Egg whole	0.01	79-123 (27)	103	12.3
	0.10	89-130 (27)	104	11.0
Egg white	0.01	102-113 (4)	106	4.6
	0.10	91-104 (4)	98	5.4
Egg yolk	0.01	96-112 (4)	107	7.1
	0.10	101-110 (4)	104	3.9
Fat	0.01	90-116 (5)	104	9.4
	0.10	90	-	-
	1.0	96-104 (4)	100	3.5
Liver	0.01	97-125	109	13.2
	0.10	103	-	-
	1.0	86, 89	88	-
Muscle	0.01	105-120 (6)	111	4.4
	0.10	97-109 (4)	101	5.4
Isoflucypram-desmethyl-carboxylic acid				
Egg whole	0.01	74-128 (27)	99	12.8
	0.10	75-121 (27)	98	9.9
Egg white	0.01	97-114 (4)	108	7.2
	0.10	87-112 (4)	97	11.1
Egg yolk	0.01	103-123 (4)	112	7.5
	0.10	95-100 (4)	98	2.4
Fat	0.01	89-115 (5)	99	9.9
	0.10	98	-	-
	1.0	87-97 (4)	91	4.7
Liver	0.01	87-97 (3)	92	5.5
	0.10	117	-	-
	1.0	93, 96	95	-
Muscle	0.01	100-119 (6)	107	6.3
	0.10	96-109 (4)	100	6.2
Isoflucypram-desmethyl-propanol free and conjugated				
Liver	0.01	103-122 (4)	112	8.5
	0.10	115	-	-
	1.0	75, 85	80	-

Monitoring Method 01300/M034

Report Nos. RALN0050, P683176031, and EnSa-17-0647.

The Meeting received analytical method descriptions and validation data for the determination of residues of isoflucypram, isoflucypram-carboxylic acid, isoflucypram-propanol, and isoflucypram-desmethyl-carboxylic acid. The validated matrices are cattle (muscle), cattle (fat), cattle (liver), cattle (kidney), cattle

(milk), hen (muscle), and hen (eggs). The extracts were subjected to HPLC-MS/MS. A further independent laboratory validation study was carried on same matrices.

The residues are extracted from animal matrices according to the QuEChERS multi-residue method. For cow liver, muscle, and milk, a 5.0 g sample aliquot is shaken after the addition of water and ACN. For fat samples, a 5.0 g sample aliquot is shaken after the addition of ACN. QuEChERS salts were added, samples are shaken and centrifuged; and an aliquot is diluted with ACN/water (1:4). Identification and quantitation were performed by LC-MS/MS. The LOQ for all analytes is 0.01 mg/kg except for cattle (milk) for which the LOQ is 0.005 mg/kg. Table 128 shown the monitored MRM transitions

Table 128 MRM transitions were monitored for each matrix tested and for each analyte:

Analyte	1st MRM (quantitation)	2nd MRM (confirmation)
Isoflucypram	m/z 400.1 → 167.0	400.1 → 139.0
Isoflucypram-carboxylic acid	m/z 430.1 → 177.0	430.1 → 412.0
Isoflucypram-propanol	m/z 416.1 → 234.1	416.2 → 177.0
Isoflucypram-desmethyl-carboxylic acid	m/z 416.1 → 236.2	416.1 → 208.1

The linearity range of the detector used was determined for each analyte using matrix-matched standards or internal standards. The correlation between the injected amount of substance and the detector response was linear for concentrations ranging from 0.025 µg/L to 5 µg/L with r values >0.99. For each matrix at least five recovery-tests were conducted at the LOQ and five recovery-tests were conducted at 10× LOQ. Mean recoveries for each fortification level were within the 70–120 percent range for all analyte/matrix combinations and both investigated MRM transitions.

The extraction efficiency of the QuEChERS analytical method was tested for the determination of the TRR, isoflucypram, isoflucypram-desmethyl-carboxylic acid, isoflucypram-propanol, isoflucypram-carboxylic acid, isoflucypram-propanol, and isoflucyrpam-2-propanol in animals. Extracts of eggs, hen muscle (leg and thorax), hen muscle, hen fat, hen liver, goat muscle, goat fat, goat liver, and goat kidney were stored for a maximum of 18 months at ≤-18 °C. Stored extracts were analysed using the QuEChERS method and compared to results of the hen and goat metabolism studies (Table 129). The efficiency of extraction of isoflucypram and metabolites is shown in Table 130.

Table 129 Recovery results from the method validation of method 01300/M034 – isoflucypram and metabolites

Matrix	No	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)	Study
			Isoflucypram						
			Quantitation: Transition 400.1 → 167			Confirmatory: Transition 400.1 → 139.0			
Hen egg	10	0.01	100-118	110	6.1	102-119	112	5.0	P683176031
	12	0.1	85-103	93	6.3	85-104	93	7.1	
Cow milk	12	0.005	95-115	104	6.4	93-110	103	5.4	
	12	0.05	88-96	94	3.0	86-98	93	4.1	
Cow muscle	12	0.01	77-95	86	5.4	81-93	88	4.4	
	10	0.1	77-89	84	4.6	81-87	84	2.3	
Cow fat	12	0.01	89-101	95	3.4	93-100	96	2.9	
	12	0.1	83-99	92	4.9	80-96	92	5.1	
Cow liver	12	0.01	88-108	95	6.6	87-109	94	7.9	
	12	0.1	71-89	77	7.7	70-89	78	8.0	
Cow kidney	14	0.01	60-100	83	11.8	62-101	85	13.1	
	14	0.1	65-95	81	13.2	64-95	81	13.5	
Hen Muscle	12	0.01	84-102	93	5.3	89-104	94	5.6	

Matrix	No	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)	Study
	14	0.1	70-114	86	17.2	73-112	86	17.5	
Cow liver	5	0.01	69-74	71	3	70-73	72	2	RALN0050
	5	0.1	70-74	71	2	69-72	71	2	
Cow muscle	5	0.01	69-77	74	4	70-74	72	2	
	5	0.1	67-73	70	3	66-72	69	3	
Cow fat	5	0.01	95-97	96	1	93-98	96	2	
	5	0.1	93-100	96	3	92-98	95	2	
Cow milk	5	0.005	72-81	77	4	67-79	73	6	
	5	0.05	69-70	70	1	70-71	70	1	
			Isoflucypram Carboxylic Acid						
			Quantitation: Transition 430.1 → 177			Confirmatory: Transition 430.1 → 412.0			
Hen egg	10	0.01	87-94	92	3.1	89-97	94	2.6	P683176031
	12	0.1	83-96	91	4.5	84-94	91	3.6	
Cow milk	12	0.005	83-101	93	5.5	75-105	92	9.9	
	12	0.05	88-103	97	5.0	87-102	98	4.4	
Cow muscle	12	0.01	88-99	92	4.3	90-99	95	3.1	
	10	0.1	85-95	89	3.6	87-96	92	4.0	
Cow fat	12	0.01	92-102	97	3.2	94-101	97	2.7	
	12	0.1	90-99	96	3.0	91-101	97	3.2	
Cow liver	12	0.01	88-95	91	2.9	71-100	88	10.2	RALN0050
	12	0.1	82-89	86	2.4	81-90	85	3.4	
Cow kidney	14	0.01	75-89	82	4.6	77-94	84	5.7	
	14	0.1	81-95	86	6.1	75-97	86	7.3	
Hen Muscle	12	0.01	84-95	90	3.4	84-101	91	5.3	
	14	0.1	73-98	86	9.7	75-96	86	8.5	
			Isoflucypram-propanol						
			Quantitation: Transition 416.0 → 234.1			Confirmatory: Transition 416.0 → 177.0			
Hen egg	12	0.01	93-107	99	4.0	97-107	102	3.7	P683176031
	12	0.1	90-102	97	3.9	93-103	97	3.7	
Cow milk	12	0.005	84-106	96	7.1	84-104	94	8.1	
	12	0.05	83-102	95	6.1	82-103	95	6.4	
Cow muscle	12	0.01	72-81	77	4.0	72-80	77	3.8	
	10	0.1	66-79	74	4.9	71-80	76	3.4	
Cow fat	12	0.01	99-107	103	2.5	97-107	102	2.9	
	12	0.1	95-106	100	3.2	96-107	100	4.0	
Cow liver	12	0.01	92-100	95	3.5	92-102	97	3.5	RALN0050
	12	0.1	85-95	91	3.4	86-94	91	2.7	
Cow kidney	14	0.01	80-98	89	7.7	79-97	89	7.6	
	14	0.1	77-97	86	7.3	76-98	86	6.9	
Hen Muscle	12	0.01	73-82	78	3.4	74-83	78	3.6	
	10	0.1	62-100	77	17.8	61-98	77	17.7	
			Isoflucypram- desmethyl-carboxylic acid						
			Quantitation: Transition 416.0 → 236.2			Confirmatory: Transition 416.0 → 208.1			
Hen egg	12	0.01	80-97	88	4.9	84-97	89	4.9	P683176031
	12	0.1	81-89	86	2.8	77-96	90	5.4	
Cow milk	12	0.005	78-102	88	9.6	68-111	92	14.1	
	12	0.05	82-105	98	7.1	79-104	97	7.0	
Cow muscle	12	0.01	82-91	87	3.2	79-96	86	6.2	
	10	0.1	84-96	89	4.0	85-92	88	3.3	
Cow fat	12	0.01	84-98	91	4.0	85-103	94	5.8	
	12	0.1	85-98	92	4.2	89-98	94	4.2	
Cow liver	12	0.01	75-89	84	4.6	72-96	86	8.8	RALN0050

Matrix	No	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)	Study
	12	0.1	80-85	83	1.9	84-89	86	2.2	
Cow kidney	14	0.01	72-88	80	7.6	73-91	82	7.9	
	14	0.1	72-103	84	10.3	71-98	84	8.3	
Hen Muscle	12	0.01	83-98	89	4.5	81-94	88	4.4	
	10	0.1	70-93	83	9.5	67-97	82	13.7	

Table 130 Extraction efficiency of the QuEChERS analytical method from representative matrices from pyrazole animal metabolism studies (Ref. EnSa-17-0647)

	QuEChERS		Metabolism Studies		Extraction Efficiency[1]
Sample	% TRR	mg/kg	% TRR	mg/kg	%
Isoflucypram					
Eggs (hen)	2.1	0.001	3.7	0.002	50.0
Leg muscle (hen)	3.8	0.001	2.3	0.001	100
Fat (hen)	17.7	0.007	23.6	0.010	70.0
Milk (goat)	39.9	0.006	33.4	0.005	120
Muscle (goat)	19.5	0.007	22.3	0.008	87.5
Fat (goat)	39.8	0.041	58.7	0.061	67.2
Liver (goat)	7.2	0.051	3.5	0.025	204
Kidney (goat)	2.3	0.004	2.7	0.005	80.0
Isoflucypram-Propanol					
Eggs (hen)	30.0	0.015	35.0	0.018	83.3
Leg muscle (hen)	3.6	0.001	5.3	0.002	50.0
Thorax muscle (hen)	2.0	<0.001	5.9	0.001	<100
Fat (hen)	8.8	0.004	11.9	0.005	80.0
Liver (hen)	1.4	0.005	1.7	0.006	83.3
Muscle (goat)	10.0	0.004	10.2	0.004	100
Fat (goat)	2.4	0.002	2.7	0.003	66.7
Liver (goat)	8.6	0.062	5.8	0.042	148
Kidney (goat)	3.6	0.007	5.6	0.011	63.6
Isoflucypram-Carboxylic Acid					
Eggs (hen)	4.2	0.002	3.4	0.002	100
Leg muscle (hen)	7.3	0.002	9.1	0.003	66.7
Thorax muscle (hen)	7.4	0.001	11.0	0.002	50.0
Fat (hen)	2.6	0.001	4.8	0.002	50.0
Liver (hen)	10.1	0.038	11.9	0.044	86.4
Muscle (goat)	4.1	0.001	8.1	0.003	33.3
Fat (goat)	2.0	0.002	3.3	0.003	66.7
Liver (goat)	6.2	0.045	8.9	0.064	70.3
Kidney (goat)	15.2	0.029	18.0	0.034	85.3
Isoflucypram-Desmethyl-Propanol					
Eggs (hen)	18.3	0.009	22.3	0.011	81.8
Leg muscle (hen)	25.9	0.008	29.7	0.009	88.9
Thorax muscle (hen)	17.1	0.003	20.9	0.004	75.0
Fat (hen)	6.8	0.003	10.1	0.004	75.0
Liver (hen)	4.6	0.017	5.3	0.020	85.0
Muscle (goat)	3.1	0.001	4.4	0.002	50.0
Liver (goat)	0.9	0.006	0.5	0.003	200
Kidney (goat)	-	-	1.6	0.003	NC
Isoflucypram-Desmethyl-Carboxylic Acid					

	QuEChERS		Metabolism Studies		Extraction Efficiency[1]
Sample	% TRR	mg/kg	% TRR	mg/kg	%
Leg muscle (hen)	7.5	0.002	12.1	0.004	50.0
Thorax muscle (hen)	10.0	0.002	12.0	0.002	100
Fat (hen)	2.7	0.001	-	-	NC
Liver (hen)	11.3	0.042	14.4	0.053	79.2
Liver (goat)	0.9	0.006	0.9	0.007	85.7
Kidney (goat)	2.3	0.004	4.2	0.008	50.0
Isoflucypram-2-Propanol					
Milk (goat)	14.7	0.002	20.3	0.003	66.7
Muscle (goat)	16.7	0.006	17.9	0.006	100
Fat (goat)	12.7	0.013	16.8	0.017	76.5
Liver (goat)	4.2	0.030	2.6	0.019	158
Kidney (goat)	3.3	0.006	4.2	0.008	75.0

Notes:
[1] Calculated by: Extracted radioactivity (mg eq/kg) from the QuEChERS analytical method/extracted radioactivity (mg eq/kg) from the metabolism studies * 100 percent.

Environmental Matrices

Method 01432

Report No. MR-14/077.

The Meeting received analytical method descriptions and validation data for the determination of residues of isoflucypram and isoflucypram-carboxylic acid residues in/on soil and sediment.

Briefly, soil and sediment samples of 20 g were extracted in a microwave extractor with ACN/water/acetic acid (4000:1000:30:v). The extracts were centrifuged to remove fine particles of the soil. Possible matrix effects of isoflucypram and isoflucypram-carboxylic acid are eliminated by using an internal standard solution of isotopically labelled reference items. Identification and quantitation of the active ingredient was done by high performance liquid chromatography using MS/MS detection in the Multiple Reaction Monitoring mode.

The LOQ for each single analyte was 1.0 µg/kg in soil. Two MRM transitions were monitored for each compound and each soil tested m/z 400.1 → 139.0 for quantitation and m/z 400.1 → 167.1 for confirmation of isoflucypram and m/z 430.1 → 177.0 for quantitation and m/z 430.1 → 412.1 for confirmation of isoflucypram-carboxylic acid. Mean recoveries for each fortification level were within the range of 94 -101 percent for isoflucypram and isoflucypram-carboxylic acid.

The correlation between the injected amount of substance and the detector response was linear for standards ranging from 0.1 µg/L to 200 µg/L, equivalent to a concentration ranging from 0.2 µg/kg to 400 µg/kg with r values ranging from 0.9996 to 0.9999. Relative standard deviations were for each fortification level were between 0.5 to 2.8 percent for isoflucypram and isoflucypram-carboxylic acid (Table 131).

Table 131 Recovery results from the method validation (n=5) of Method 01432 in soil (Ref. MR-14/077)

Matrix	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
Isoflucypram							
		Quantitation: Transition 400.1 → 167			Confirmatory: Transition 400.1 → 139.1		
Soil Höfchen	1.0	98-100	99	0.8	98-99	98	0.6
	10.0	100-101	100	0.5	101-104	102	1.2

Matrix	Spiking Level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
Soil Laacher Hof	1.0	96-99	98	1.3	97-100	99	1.4
	10.0	100-102	101	1.1	99-103	101	1.5
Soil Dollendorf	1.0	95-99	97	1.8	99-101	100	0.9
	10.0	97-100	98	1.2	99-101	100	0.8
Sediment	1.0	97-100	99	1.2	93-100	97	3.1
	10.0	97-100	99	1.2	100-101	101	0.5
Isoflucypram-Carboxylic Acid							
		Quantitation: Transition 430.0 → 177.0			Confirmatory: Transition 430.0 → 412.0		
Soil Höfchen	1.0	94-100	97	2.7	86-96	91	4.4
	10.0	98-101	99	1.4	99-104	101	1.9
Soil Laacher Hof	1.0	98-101	99	1.3	89-94	92	2.3
	10.0	99-100	100	0.5	97-103	99	2.4
Soil Dollendorf	1.0	95-101	98	2.2	83-96	90	5.5
	10.0	95-102	99	2.8	95-98	97	1.3
Sediment	1.0	90-96	94	2.7	82-98	91	6.8
	10.0	98-100	98	0.9	99-104	101	2.1

Method 01432 was used in the terrestrial field dissipation studies, the soil analysis portion of the limited field rotational crops study, and the soil storage stability study. Calibration curves were established for each analytical set with coefficients of determination > 0.99. Results are shown in Tables 132 and 133.

Table 132 Concurrent recovery data for terrestrial field dissipation for method Method 01432

Matrix	Fortification Level (µg/kg)	Recoveries (%) Range (n)	Mean Recovery (%)	RSD (%)	Study
Isoflucypram					14-2750
Soil	1	63-112 (203)	99	8.3	
	10	64-112 (183)	98	7.4	
	100	101	101	-	
	400	87-99 (7)	95	5.7	
	500	80, 81	81	-	
	1000	72-91 (7)	84	7.3	
Isoflucypram-carboxylic acid					
Soil	1	66-116 (196)	99	8.9	
	9	66-116 (173)	99	8.5	
	90	105	105	-	
	300	90-107 (5)	99	7.5	
Isoflucypram					MELNN203
Soil (Method validation)	1.0	86, 88	87	-	
	5.0	79, 81	80	-	
	20	80, 85	83	-	
Soil (Concurrent recovery)	10	78-95 (n=14)	88	5	
Soil (Field spike)	83	0[1]-78 (n=9)	50	48	
Isoflucypram-carboxylic acid					
Soil (Method validation)	1.0	78, 82	80	-	
	5.0	85, 81	83	-	
	20	90, 87	88	-	
Soil (Concurrent recovery)	10	83-102 (n=15)	94	6	
Soil (Field spike)	83	57, 70, 37	55	30	
Isoflucypram					AUS 0032
Soil (Method validation)	1	83, 82	83	-	

Matrix	Fortification Level (µg/kg)	Recoveries (%) Range (n)	Mean Recovery (%)	RSD (%)	Study
	10	79, 81	80	-	
Soil (Concurrent recovery)	1	83, 91, 82	85	6	
	10	81-98 (11)	89	6	
	1,500	89, 91, 90	90	1	
	3,000	91, 90, 91	91	1	
Isoflucypram-carboxylic acid					
Soil (Method validation)	1	85, 85	85	-	
	10	90, 90	90	-	
Soil (Concurrent recovery)	1	85, 92, 85	87	5	
	10	85-90 (11)	90	4	
Isoflucypram					AUS 0031
Soil (Method validation)	1	85, 81	83	-	
	10	91, 91	91	-	
Soil (Concurrent recovery)	1	85, 81	83	-	
	10	74-93 (12)	85	7	
	1,500	85, 80, 84	83	3	
	3,000	88, 86, 87	87	1	
Isoflucypram-carboxylic acid					
Soil (Method validation)	1	83, 77	80	-	
	10	97, 98	98	-	
Soil (Concurrent recovery)	1	83, 77	80	-	
	10	79-98 (12)	90	7	
Isoflucypram					AUS 0030
Soil (Method validation)	1	87, 89	88	-	
	10	91, 92	91	-	
Soil (Concurrent recovery)	10	84-90 (13)	86	2	
	1,500	82, 82, 81	82	1	
	3,000	87, 89, 88	88	1	
Isoflucypram-carboxylic acid					
Soil (Method validation)	1	85, 86	86	-	
	10	94, 95	95	-	
Soil (Concurrent recovery)	10	90-98 (13)	94	2	
Isoflucypram					AUS 0034
Soil (Method validation)	1	94, 95	94	-	
	10	85, 84	84	-	
Soil (Concurrent recovery)	1	94, 94	94	-	
	10	76-94 (63)	86	4	
Isoflucypram-carboxylic acid					
Soil (Method validation)	1	93, 92	93	-	
	10	96, 94	95	-	
Soil (Concurrent recovery)	1	93, 92	93	-	
	10	84-102 (63)	94	4	
Isoflucypram					AUS 0033
Soil (Method Validation)	1	89, 85	87	-	
	10	79, 84	82	-	
Soil (Concurrent recovery)	1	70-91 (8)	83	10	
	10	74-104 (80)	86	7	
	1,500	73, 84, 76	78	7	
	3,000	81, 84, 83	83	2	
Isoflucypram-carboxylic acid					
Soil (Method validation)	1	77, 71	74	-	
	10	84, 89	87	-	
Soil (Concurrent recovery)	1	71-94 (8)	85	10	

Matrix	Fortification Level (µg/kg)	Recoveries (%) Range (n)	Mean Recovery (%)	RSD (%)	Study
	10	76-102 (84)	93	7	
Isoflucypram					15-2502
Soil 0-30 cm	0.001	95-104 (16)	99	2.4	
	0.01	97102 (17)	99	1.7	
Isoflucypram-carboxylic acid					
Soil 0-30 cm	0.001	73-105 (17)	94	9.9	
	0.01	90-108 (17)	100	4.7	

Table 133 Summary of concurrent recoveries for according to Method 01432 in the soil storage stability Study P641 14 1803

Analyte/Matrix	Fortification level (µg/kg)	Storage interval (Days)	Recovery (%)	Mean (%)	RSD (%)
Isoflucypram/soil Höfchen	10	0	101, 102, 103, 103	102	0.9
		93	103, 104, 100, 102	102	1.7
		194	100, 99, 99, 98	99	0.8
		308	101, 104, 104, 99	102	2.4
		363	104, 104, 104, 101	103	1.5
		539	101, 100, 101, 101	101	0.5
		720	102, 93, 99, 100	99	3.9
Isoflucypram/soil Dollendorf	10	0	102, 99, 98, 99	100	1.7
		93	101, 104, 105, 104	104	1.7
		194	100, 100, 100, 99	100	0.5
		308	100, 100, 100, 99	100	0.5
		363	99, 104, 100, 100	101	2.2
		539	101, 102, 99, 100	101	1.3
		720	102, 101, 98, 95	99	3.2
Isoflucypram-carboxylic acid/soil Höfchen	10	0	104, 100, 102, 100	102	1.9
		93	105, 99, 106, 107	104	3.4
		194	103, 104, 104, 102	103	0.9
		308	91, 96, 98, 105	98	6.0
		363	102, 100, 100, 94	99	3.5
		539	98, 97, 95, 101	98	2.6
		720	106, 100, 104, 103	103	2.4
Isoflucypram-carboxylic acid/soil Dollendorf	10	0	105, 100, 96, 100	100	3.7
		93	100, 106, 100, 109	104	4.3
		194	105, 108, 99, 104	104	3.6
		308	101, 91, 92, 100	96	5.4
		363	99, 102, 99, 108	102	4.2
		539	101, 100, 102, 104	102	1.7
		720	119, 103, 103, 105	108	7.2

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

The Meeting received three studies investigating various analytes in various commodities under frozen storage conditions. Analytical reports are sufficiently detailed and included chromatograms showing defined symmetrical peaks.

Plants

Report No. MR-17/244.

The Meeting received a study evaluating the stability of isoflucypram and BCS-CR60082 (Isoflucypram-N-methyl-cyclopropyl-pyrazole-carboxamide) during freezer storage (≤-18 °C) for a period of 24–25 months in tomato (fruit), bean (dry seed), wheat (grain), rape (seed), and orange (fruit) (Uceda, L.; 2018). The study also investigated the stability of isoflucypram and BCS-CR60082 following frozen storage for 25–26 months at -18 °C followed by six days of storage at -1 ± 2 °C.

Samples were fortified, separately, with isoflucypram and BCS-CR60082 at 0.20 mg/kg and stored at an average temperature of -18 °C or below. Tomato (fruit), bean (dry seed), wheat (grain), and rape (seed) were analysed at the nominal storage intervals of 0, 3, 8, 13, 18, and 24 months. Samples of orange (fruit) were analysed at the nominal storage intervals of 0 and 6 days, and 1, 3, 8, 13, 18, and 24 months. Additionally, second set of orange (fruit) data were investigated due to high variability of recoveries in orange (fruit) on Day-0. The second set of orange (fruit) was analysed following 0, 5, and 10 months.

After approximately 25 months of storage at -18 °C, some samples of all commodities were transferred to a refrigerator with a temperature of -1 ± 2 °C and stored for six days. After the six additional days of storage, samples were observed to have partially thawed and no fungal formation was observed.

Samples were analysed according to Method 01475. At each storage interval, residues of isoflucypram and BCS-CR60082 in the control samples were below the LOQ. Therefore, treated samples were not corrected for residues in controls.Residues of isoflucypram and BCS-CR60082 were stable (mean recoveries greater than 70 percent) at all sampling intervals. Therefore, isoflucypram and BCS-CR60082 are considered to be stable in diverse crop matrices for a period of 24–25 months at -18 °C, as well as a period of 25–26 months at -18 °C plus six days at approximately 1 °C. The results are shown in Table 134.

Table 134 Storage stability for isoflucypram and BCS-CR60082 ((isoflucypram-N-methyl-cyclopropyl-pyrazole-carboxamide) in crops

Commodity	Fortification level (mg/kg)	Storage period (days)	% remaining	Mean (%)
Isoflucypram				
Tomato fruit	0.2	0	99, 99, 104	101
		100	100, 97	99
		251	99, 102	101
		415	86, 90	88
		563	96, 101	99
		737	94, 91, 91	92
		779 + 6[1]	88, 93, 91	91
Bean dry seed	0.2	0	102, 100, 102	101
		100	103, 103	103
		246	98, 98	98
		415	84, 84	84
		561	104, 103	104
		743	89, 90, 91	90
		783 + 6[1]	92, 95, 94	94
Wheat grain	0.2	0	97, 95, 97	96
		102	94, 94	94
		249	100, 97	99
		414	89, 91	90
		560	94, 85	90

Commodity	Fortification level (mg/kg)	Storage period (days)	% remaining	Mean (%)
		746	84, 85, 84	84
		778 + 6[1]	84, 82, 82	83
Rape seed	0.2	0	101, 94, 95	97
		103	80, 97	89
		245	92, 100	96
		417	97, 93	95
		558	90, 88	89
		747	88, 88, 87	88
		776 + 6[1]	89, 88, 91	89
Orange fruit	0.2	0	126, 88, 97	104
		6	95, 91, 88	91
		34	107, 101, 91	100
		106	61, 87, 93	80
		254	90, 94, 100	95
		415	84, 92, 92	89
		559	103, 97, 100	100
		742	88, 90, 91	90
		785 + 6[1]	89, 88, 92	90
Orange fruit (Second study)	0.2	0	114, 104, 111	110
		167	93, 86, 83	87
		315	102, 95, 102	100
BCS-CR60082				
Tomato fruit	0.2	0	96, 93, 94	94
		100	98, 96	97
		251	100, 102	101
		415	88, 87	88
		563	90, 92	91
		737	87, 95, 88	90
		779 + 6[1]	84, 86, 86	85
Bean dry seed	0.2	0	91, 95, 91	92
		100	94, 102	98
		246	97, 98	98
		415	77, 85	81
		561	86, 87	87
		743	84, 91, 84	86
		783 + 6[1]	80, 82, 80	81
Wheat grain	0.2	0	94, 90, 91	92
		102	96, 94	95
		249	104, 104	104
		414	89, 88	89
		560	85, 79	82
		746	79, 80, 77	79
		778 + 6[1]	82, 78, 80	80
Rape seed	0.2	0	114, 113, 104	110
		103	76, 77	77
		245	100, 95	98
		417	91, 97	94
		558	86, 89	88
		747	77, 74, 75, 82[2], 77[2], 80[2]	78
		776 + 6[1]	76, 80, 81	79
Orange fruit	0.2	0	98, 88, 90	92
		6	80, 60, 72	71
		34	89, 87, 67	81
		106	95, 86, 93	91

Commodity	Fortification level (mg/kg)	Storage period (days)	% remaining	Mean (%)
		254	93, 87, 93	91
		415	83, 71, 74	76
		559	97, 99, 109	102
		742	75, 85, 86	82
		785 + 6[1]	85, 81, 75	80
Orange fruit (Second study)	0.2	0	99, 104, 102	102
		167	90, 84, 77	84
		315	89, 106, 104	100

Notes:
[1] Samples stored at -18 °C for ca. 25 months followed by storage at -1 °C for six days.
[2] Results confirmed after 782 days with new samples.

Report No. P642186502.

The Meeting received a study investigating the stability of isoflucypram-desmethyl-propanol and isoflucypram-propanol in/on wheat grain, green material, and straw under frozen storage conditions (Stuke, S.; 2021). Isoflucypram-desmethyl-propanol and isoflucypram-propanol were fortified, separately, at 0.1 mg/kg. Samples were stored at <-18 °C and analysed at nominal intervals of ca. 0, 1, 3, 6, 12, 18, 24, and 30 months of storage. It is noted that a power outage occurred, requiring samples to be moved to a separate freezer with a temperature of ca. -26 °C for approximately 53 hours.

Samples were analysed according to Method 01564. Residues in all control samples were below the LOQ. Therefore, residues in treated samples were not corrected for control samples.Residues of isoflucypram-desmethyl-propanol and isoflucypram-propanol were stable (mean recoveries greater than 70 percent) at all sampling intervals. The interim study results demonstrate that the residues of isoflucypram-desmethyl-propanol and isoflucypram-propanol are stable in wheat commodities for at least 18 months under deep-freezer storage conditions (≤-18 °C) (Table 135).

Table 135 Summary of storage stability recoveries for isoflucypram-desmethyl-propanol and isoflucypram-propanol in wheat commodities

Commodity	Fortification level (mg/kg)	Storage period (days)	% remaining	Mean (%)
Isoflucypram-desmethyl-propanol				
Wheat grain	0.1	0	109, 99, 103, 101, 95	101
		33	100, 98, 105	101
		97	100, 96, 92	96
		183	96, 97, 98	97
		358	95, 96, 107	99
		544	96, 96, 97	96
		728	100, 103, 103	102
		904	95, 101, 102	99
Wheat green material	0.1	0	106, 110, 105, 100, 101	104
		33	101, 97, 106	101
		97	108, 103, 99	103
		183	97, 101, 94	97
		358	83, 94, 84	87
		544	94, 100, 91	95
		727	103, 101, 104	103
		903	90, 90, 80	87
Wheat straw	0.1	0	110, 99, 106, 99, 94	102
		30	111, 95, 107	104
		97	112, 93, 98	101
		180	95, 98, 99	97

Commodity	Fortification level (mg/kg)	Storage period (days)	% remaining	Mean (%)
		358	90, 89, 79	86
		544	84, 74, 79	79
		728	92, 101, 95	96
		909	81, 92, 93	89
Isoflucypram-propanol				
Wheat grain	0.1	0	90, 94, 87, 93, 93	91
		33	93, 94, 92	93
		97	88, 86, 88	87
		183	72, 88, 77	79
		358	97, 94, 97	96
		544	86, 92, 92	90
		728	94, 91, 93	93
		904	98, 96, 96	97
Wheat green material	0.1	0	96, 96, 95, 96, 92	95
		33	94, 90, 92	92
		97	94, 90, 87	90
		183	89, 75, 79	81
		358	95, 93, 96	95
		544	94, 92, 93	93
		727	93, 96, 98	96
		903	91, 94, 89	91
Wheat straw	0.1	0	93, 85, 74, 89, 75	83
		30	91, 89, 87	89
		97	67, 72, 84	74
		190	86, 82, 83	84
		358	91, 91, 90	91
		544	87, 82, 87	85
		728	89, 96, 92	92
		909	90, 94, 94	93

Soil

Report No. P641 14 1803.

The Meeting received a study evaluating the stability of isoflucypram and isoflucypram-carboxylic acid in soil under freezer storage conditions (Koch, V.; 2016). Untreated soil samples of soil Höfchen (silt loam) and soil Dollendorf (clay loam) were fortified, separately, with isoflucypram and isoflucypram-carboxylic acid at 10 µg/kg for each analyte. The characteristics of the soils are shown in Table 136

Table 136 Soil Characteristics

	Soil Höfchen	Soil Dollendorf
Description	0-30 cm soil layer	0-20 cm soil layer
pH (in $CaCl_2$ solution)	6.7	7.3
pH (in H_2O)	7.4	7.4
Organic carbon (percent)	0.92	5
Organic matter (percent)[1]	1.58	8.6
Cation exchange capacity (meq/100 g dry soil)	12.4	20.6
Maximum water holding capacity (g/100 g dry soil)	39.5	79.1
Clay (<0.002 mm) (percent)	19.4	31
Silt (0.002-0.050 mm) (percent)	76.3	38

	Soil Höfchen	Soil Dollendorf
Sand (0.050-2.000 mm) (percent)	4.3	31
Soil type	Silt loam	Clay loam

Notes:
[1] Organic matter = Organic carbon X 1.72.

Soil samples were analysed following 0, 93, 194, 308, 363, 539, and 720 days of storage. Samples were generally maintained at ≤-18 °C; however, due to an error on day 235, the temperature rose to an average of -12.2 °C for 5 hours and 55 minutes. This deviation is not expected to have an impact on the study.

Soil samples were analysed according to Method 01432. All control samples were below the LOQ. Therefore, treated samples were not corrected for residues in controls. Residues of isoflucypram and isoflucypram-carboxylic acid were stable (mean recoveries greater than 70 percent) at all sampling intervals. The results demonstrate that residues of isoflucypram and isoflucypram-carboxylic acid are stable in soil for at least 720 days under frozen storage conditions (Table 137).

Table 137 Summary of storage stability for isoflucypram and isoflucypram-carboxylic acid in soil

Analyte/Matrix	Fortification level (µg/kg)	Storage interval (Days)	% remaining	Mean
Isoflucypram/soil Höfchen	10	0	101, 101, 100, 100	101
		93	98, 101, 98, 101	100
		194	97, 101, 101, 101	100
		308	102, 104, 103, 103	103
		363	101, 106, 103, 102	103
		539	105, 102, 103, 101	103
		720	102, 105, 106, 104	104
Isoflucypram/soil Dollendorf	10	0	100, 99, 100, 97	99
		93	100, 103, 101, 102	102
		194	100, 100, 101, 100	100
		308	105, 106, 105, 98	104
		363	103, 105, 105, 105	105
		539	103, 103, 103, 101	103
		720	104, 104, 103, 105	104
Isoflucypram-carboxylic acid/soil Höfchen	10	0	96, 103, 98, 96	98
		93	96, 105, 110, 94	101
		194	101, 114, 98, 96	102
		308	99, 103, 102, 100	101
		363	107, 99, 94, 96	99
		539	95, 97, 101, 99	98
		720	98, 108, 108, 101	104
Isoflucypram-carboxylic acid/soil Dollendorf	10	0	98, 98, 100, 96	98
		93	106, 99, 107, 103	104
		194	102, 100, 101, 104	102
		308	94, 108, 101, 100	101
		363	98, 100, 99, 101	100
		539	100, 97, 102, 95	99
		720	105, 105, 102, 107	105

USE PATTERN

The Meeting received two labels from New Zealand, both for ECs containing 50 g ai/L. The labels included use patterns for isoflucypram on wheat, barley, triticale, and ryegrass seed crop (Table 138).

Table 138 Summary of use patterns for isoflucypram in New Zealand [1,2]

Crop	Maximum Application Rate (kg ai/ha)	Number of Applications	Withholding Period (Days)	Additional Instructions
Wheat	0.075	1	Grain and straw/stubble: 42 Feed/silage: 28	1st appearance of disease or before disease appearance Up to BBCH 69
Barley	0.075	1	Grain and straw/stubble: 56 Forage green feed/silage: 42	1st appearance of disease Up to BBCH 61
Triticale	0.075	1	Grain and straw/stubble: 42 Feed/silage: 28	1st appearance of disease Up to BBCH69

Notes:
[1] Use 100-300 L water/ha for ground applications and 50 L water/ha for air applications.
[2] Neither label specifies PBI for planting rotational crops.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received several magnitude of the residue studies for isoflucypram on wheat and barley. In many cases, residues of multiple active ingredients were analysed. The following evaluation only addresses data submitted pertaining to isoflucypram, and in some cases, the metabolites isoflucypram-propanol and isoflucypram-desmethyl-propanol.

The field trial reports included dates from critical events during the study, including application, harvest, storage, and analysis, and detailed information on the field site and treatment parameters. Analytical reports were sufficiently detailed and included example chromatograms and example calculations. Chromatogram peaks were clean and symmetrical.

Field trial residues for metabolites are expressed in parent equivalents. Residues below the LOQ were reported as "<0.01 mg/kg" and were calculated at the LOQ (0.01 mg/kg). Several European trials reported percent moisture content and all New Zealand trials report percent dry matter. New Zealand study reports included calculations of residues corrected for percent dry matter; therefore, residues from New Zealand are shown before correction ("as received") and after correction ("dry matter").

In the summary tables, values used for maximum residue level recommendations and dietary burden/intake are underlined. Table 139 summarizes the trials submitted to the Meeting.

Table 139 Supervised trials for isoflucypram

Crop Group	Commodity	Table No
Cereal Grains	Barley (GC 0640)	140
	Wheat (GC 0654)	141
Straw, fodder, and forage of cereal grains	Barley (AS 0640)	142
	Wheat (AS 0654)	13143

Cereal Grains

Barley

The Meeting received 63 supervised residue trials from multiple European countries, New Zealand and the United Kingdom. Trials were conducted with an EC formulation containing 50 g ai/L or 42 g ai/L. Trials were conducted on a variety of spring, winter, and malting barley. Residues were investigated in

forage/green material, grain, and straw. For all trials, a foliar-directed spray application was made at BBCH 61. Grain was harvested at BBCH 89, except where indicated. No adjuvants were used at any of the trials.

Irrigation was conducted at Trials 15-2115-01, 15-2117-03, 15-2066-01, PNZ16414-01, PNZ16414-02, 17-2018-02, BAYERNZ/GLP/16/04/a-03, S17-07998-01, S17-07998-03, and S18-07828-04. In general, a minimum of 1.0 kg was collected for barley grain; however, the minimum sample weight for treated barley grain from Trial 16-2052 was 0.96 kg.

Treated and control samples were frozen within 24 hours after sampling and during shipment to the Laboratory for Sampling. Samples were shredded and homogenized with dry ice and then shipped frozen to the analytical laboratory. Samples were generally stored below -18 °C. There were several exceptions with storage temperatures briefly reaching temperatures up to 10 °C; howeverr, these deviations are not expected to adversely impact the validity of the study results. Frozen storage conditions were not reported for P672186504.

In general, the maximum storage duration for barley grain was 544 days. However, in samples from study P672186504 (amendment to studies 16-2051 and 16-2052), residues of isoflucypram-desmethyl-propanol and isoflucypram-propanol were extracted for analysis after frozen storage of 819–885 days. Additionally, frozen storage duration from study GLP658 (amendment to study S17-07996) were not reported for isoflucypram-desmethyl-propanol and isoflucypram-propanol. The Meeting calculated the maximum duration of frozen storage of 481 days for barley grain based on harvest data and date of extraction for analysis.

Samples were analysed according to Methods 01475 and 46437, version 1. There were no residues in control samples. For samples analysed according to Method 01564, residues of isoflucypram, isoflucypram-propanol, or isoflucypram-desmethyl-propanol were generally below the LOQ in control samples, except for residues of isoflucypram and isoflucypram-desmethyl-propanol were 0.013 and 0.036 mg eq/kg in untreated barley grain from Trial 17-2018-01. The results are shown in Table 140.

Table 140 Residues of isoflucypram and metabolites (expresses as parent) in barley grain treated once with EC formulation at 50 or 42 g ai/L at BBCH 61

Trial No. (Location	Variety	% dry matter	Application rate g ai/ha	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, dw, mg/kg	Isoflucypram-desmethyl-propanol, mg/kg	Isoflucypram-desmethyl-propanol, dw, mg/kg	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, dw, mg/kg
15-2114-01 Santarem, Portugal[2,3]	Winter barley Pewter	-	75	60	<u><0.01</u>	-	-	-	-	-
15-2066-01 Santarem, Portugal[2,3]	Winter barley Pewter	-	75	60	<0.01	-	-	-	-	-
15-2114-02 Etienne du Gres, France[2,3]	Winter barley Baraka	-	75	69	<u><0.01</u>	-	-	-	-	-
15-2117-01 Nimes Languedoc Roussillon, France[2,4]	Barley Jallon	-	63	48	<0.01	-	-	-	-	-
15-2066-02 Etienne du Gres, France[2,4]	Winter barley Baraka	-	75	69	<0.01	-	-	-	-	-
15-2114-03 Marchena, Spain[2,5]	Traveler malting	-	75	57	<u><0.01</u>	-	-	-	-	-

Trial No. (Location	Variety	% dry matter	Application rate g ai/ha	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, dw, mg/kg	Isoflucypram-desmethyl-propanol, mg/kg	Isoflucypram-desmethyl-propanol, dw, mg/kg	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, dw, mg/kg
15-2066-03 Marchena, Spain[2,5]	Traveler malting	-	75	57	<0.01	-	-	-	-	-
15-2114-04 Bouloc, France[2,6]	Winter barley Cacia	-	75	45	0.012	-	-	-	-	-
15-2066-04 Bouloc, France[2,6]	Winter barley Cacia	-	75	45	0.022	-	-	-	-	-
15-2113-01 Wieringwerf, Netherlands[2,7]	Spring barley Triple summer	-	75	61	<u><0.01</u>	-	-	-	-	-
15-2110-01 Middenmeer, Netherlands[2,7]	Spring barley Odyssey	-	75	55	<0.01	-	-	-	-	-
15-2113-02 Burscheid, Germany[2,8]	Spring barley Streif	-	75	68	<u><0.01</u>	-	-	-	-	-
15-2110-02 Burscheid, Germany[2,8]	Spring barley Streif	-	75	68	<0.01	-	-	-	-	-
15-2113-03 Esvres sur Indre, France[2,9]	Winter barley Etincel	-	75	62	<u><0.01</u>	-	-	-	-	-
15-2110-03 Esvres sur Indre, France[2,9]	Winter barley Etincel	-	75	62	<0.01	-	-	-	-	-
15-2113-04 Little Shelford, Cambridge, United Kingdom[2,10]	Winter barley Glacier	-	75	77	<u><0.01</u>	-	-	-	-	-
15-2110-04 Little Shelford, Cambridge, United Kingdom[2,10]	Winter barley Glacier	-	75	77	<0.01	-	-	-	-	-
15-2117-02 Guarene, Italy[2]	Barley Sfera	-	63	35	0.037	-	-	-	-	-
15-2117-03 Mahora, Spain[2]	Barley Shakira	-	63	46	<0.01	-	-	-	-	-
15-2117-04 Cartaxo, Portugal[2]	Barley Pewter	-	63	41	0.027	-	-	-	-	-
15-2118-01 Linconshire, PE12 9 PQ, United Kingdom[2]	Winter barley Cassia	-	63	63	<u><0.01</u>	-	-	-	-	-
15-2118-02 La Chapelle de Guinchay, Bourgogne, France[2]	Winter barley Esterel	-	63	55	0.020	-	-	-	-	-
15-2118-03 9763 Vasszecseny, Hungary[2]	Spring barley Mandolina	-	63	35	0.013	-	-	-	-	-
15-2118-04 Zlinsky Kraj, Czech Republic[2]	Spring barley Kangoo	-	63	55	<0.01	-	-	-	-	-
16-2052-01 Santarem, Portugal[2]	Barley Pewter	-	75	67	<u><0.01</u>	-	<0.01	-	<0.01	-
16-2052-02 St Etienne du Gres, France[2]	Winter barley Augusta	-	75	54	<u><0.01</u>	-	0.025	-	<0.01	-

Trial No. (Location	Variety	% dry matter	Application rate g ai/ha	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, dw, mg/kg	Isoflucypram-desmethyl-propanol, mg/kg	Isoflucypram-desmethyl-propanol, dw, mg/kg	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, dw, mg/kg
16-2052-03 41600 Arahal, Spain[2]	Malting barley Odissey	-	75	57	<0.01	-	<0.01	-	<0.01	-
16-2052-04 Gargas, France[2]	Winter barley Cacia	-	75	49	<0.01	-	0.024	-	<0.01	-
16-2051-01 CB22 5EU Little Shelford, Cambridge, United Kingdom[2]	Spring barley Odyssey	-	75	57	<0.01	-	0.012	-	<0.01	-
16-2051-02 Burscheid, Germany[2]	Spring barley Vespa	-	75	60	<0.01	-	0.014	-	<0.01	-
16-2051-03 Chambourg sur Indre, Northern France[2]	Winter barley Obit	-	75	53	<0.01	-	<0.01	-	<0.01	-
16-2051-04 1681 ND Zwaagdijk, Netherlands[2]	Winter barley Quadrigo	-	75	37	0.041	-	0.051	-	0.032	-
E19RP054-01 Burscheid, Germany	Spring barley Avalon	-	76	57	<0.01	-	<0.01	-	<0.01	-
E19RP054-02 Kommingen, Germany	Spring barley Avalon	-	75	27	0.049	-	0.016	-	<0.01	-
E19RP054-03 Werl-Niederbergstrabe, Germany	Winter barley Meridian	-	77	47	<0.01	-	<0.01	-	<0.01	-
E19RP054-04 Tilly, Belgium	Winter barley Keeper	-	74	50	<0.01	-	0.034	-	<0.01	-
E19RP055-01 Caprais, South France	Barley Cassia	-	73	56	<0.01	-	0.018	-	<0.01	-
E19RP055-02 Maire, South France	Barley Etincel	-	78	51	<0.01	-	<0.01	-	<0.01	-
E19RP055-03 44123 Boara Ferrara, Italy	Barley Marjorie	-	76	41	0.019	-	0.028	-	0.015	-
E19RP055-04 Sommacampagna via Cessarina, Italy	Barley Calanque	-	74	48	<0.01	-	0.051	-	<0.01	-
E19RP056-01 Borgo d'Ale, Italy	Barley Tunika	-	64	42	<0.01	-	<0.01	-	<0.01	-
E19RP056-02 Zafarraya, Spain	Barley Yuriko	-	63	50	0.025	-	0.030	-	<0.01	-
17-2017-01 Kranzlin, Germany[2]	Spring barley Simba	-	63	55	<0.01	-	0.015	-	<0.01	-
17-2017-02 Tinglev, Denmark[2]	Spring barley Overture	-	63	50	0.017	-	0.014	-	<0.01	-
17-2017-03 Oxfordshire, United Kingdom[2]	Spring barley Octavia	-	63	57	<0.01	-	0.011	-	<0.01	-
17-2017-04 Juvincourt et Dammary, North France[2]	Spring barley Irina	-	63	41	0.010	-	<0.01	-	<0.01	-

Trial No. (Location	Variety	% dry matter	Application rate g ai/ha	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, dw, mg/kg	Isoflucypram-desmethyl-propanol, mg/kg	Isoflucypram-desmethyl-propanol, dw, mg/kg	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, dw, mg/kg
17-2018-01 Upie, Southern France[2,11]	Barley Maltesse winter	-	63	38	0.011	-	0.042	-	0.010	-
17-2018-02 Settala, Italy[2]	Barley Concerto	-	63	44	<0.01	-	<0.01	-	<0.01	-
17-2018-03 Montamaneu, Spain[2]	Barley Meseta winter	-	63	48	0.010	-	0.018	-	<0.01	-
17-2018-04 Drymos, Greece[2]	Barley Hyvito	-	63	43	0.010	-	0.014	-	<0.01	-
PNZ16414-03 Timaru, New Zealand	Barley 776	80	76	61	0.103	0.128	-	-	-	-
PNZ16414-04 St. Andrews, New Zealand[12]	Barley Sanette	86	77	49	0.134, 0.120 (0.127)	0.156, 0.140 (0.148)	-	-	-	-
BAYERNZ/GLP/16/0 Hawkes Bay, New Zealand[2]	Barley Sumit	83	75	56	<0.01	<0.01	-	-	-	-
BAYERNZ/GLP/16/04/a-04, Manawatu, New Zealand[2]	Barley Fairview	86	75	56	<0.01	<0.01	-	-	-	-
S17-07996-01/GLP658-01 Ashburton, New Zealand[13]	Spring barley Bumpa	65	116[14]	87	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
S17-07996-02/GLP658-02 Springston, New Zealand	Spring barley Bumpa	82	106[14]	56	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
S17-07996-03/GLP658-03 Kairanga, New Zealand[2,15]	Spring barley Bumpa	82	75	49	0.028	0.034	0.021	0.025	<0.01	<0.01
S17-07996-04/GLP658-04 Bulls, New Zealand[2,15]	Spring barley Calibre	82	75	61	<0.01, <0.01 (<0.01)	<0.01, <0.01	0.030, 0.027 (0.029)	0.037, 0.033 (0.035)	0.016, 0.012 (0.014)	0.020, 0.014 (0.017)
S18-07828-01 Otane, New Zealand[2]	Spring barley Jimpy	86	75	56	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
S18-07828-02 Cheltenham, New Zealand[1,16]	Spring barley Planet	87	75	45	0.029	0.033	0.035	0.040	0.012	0.014
S18-07828-03 Beconsfield, New Zealand[2,16]	Spring barley Cassia	87	75	53	0.013	0.015	0.018	0.020	<0.01	<0.01
S18-07828-04 Tinwald, New Zealand[2,17]	Spring barley Milford	91	75	54	0.023	0.025	0.012	0.013	<0.01	<0.01
S18-07828-05 Mitcham, New Zealand[2,17]	Spring barley Tavern	89	75	57	0.013, 0.012 (0.013)	0.015, 0.014 (.015)	0.019, 0.018 (0.019)	0.021, 0.021 (.021)	<0.01, <0.01 (0.01)	<0.01, <0.01 (<0.01)

Notes:

Dw = dry weight.

[1] DALA = Days after last application.

[2] Application rates are reported as the nominal values if the measured rates were ±5 percent of the planned rates.

[3] Trials 15-2114-01 and 15-2066-01 are not independent.

[4] Trials 15-2114-02, 15-2117-01, and 15-2066-02 are not independent.

[5] For Trials 15-2114-03 and 15-2066-03, applications were made at BBCH 53. Additionally, Trials 15-2114-03 and 15-2066-03 are not independent.

[6] Trials 15-2114-04 and 15-2066-04 are not independent.
[7] Trials 15-2113-01 and 15-2110-01 are not independent.
[8] For Trials 15-2113-02 and 15-2110-02, applications were made at BBCH 59. Additionally, Trials 15-2113-02 and 15-2110-02 are not independent.
[9] Trials 15-2113-03 and 15-2110-03 are not independent.
[10] Trials 15-2113-04 and 15-2110-04 are not independent.
[11] Quantifiable residues in control sample. Residues presented are not corrected for residues in controls.
[12] Isoflucypram applied at BBCH 63.
[13] Grain harvested at BBCH 92.
[14] Trials S17-07996-01/GLP658 and S17-07996-02/GLP658 were accidentally overdosed.
[15] Trials S17-07996-03/GLP658-03 and S17-07996-04/GLP658-04 are not independent.
[16] Trials S18-07828-02 and S18-07828-03 are not independent.
[17] For Trials S18-07828-04 and S18-07828-05, grain was harvested at BBCH 93. Additionally, Trials S18-07828-04 and S18-07828-05 are not independent.

Wheat

The Meeting received 62 supervised residue trials from multiple European countries, the United Kingdom, and New Zealand. Trials were conducted with an EC formulation containing either 50 g ai/L or 42 g ai/L. Trials were conducted on a variety of spring, winter, and durum wheat. Residues were generally investigated in forage/green material, grain, and straw; however, grain and straw were not analysed at Trial 15-2116-03 and Trial 15-2069-03 due to insufficient material harvested. For all trials, isoflucypram was applied as a single foliar-directed application. Grain was harvested at BBCH 89, except where indicated. No adjuvants were added to the solution at any trial.

Irrigation was only conducted at Trials 15-2116-01, 15-2116-04, 15-2069-01, 15-2069-04, 15-2119-02, and S17-07939-02/GLP655-02. In general, a minimum of 1.00 kg of treated grain was collected. However, in Trial S17-07939-02/GLP655-02, a minimum of 0.50 kg of grain was collected; the low collection weight was not addressed in the study report. Additionally, wheat grain from Trial BAYER NZ/GLP/16/04-01 was specified to be below the required amount of 1.0 kg due to poor yield; however, grain was collected from 12 areas adequately representative of the entire plot.

Treated and control samples were frozen within 24 hours after sampling and during shipment to the Laboratory for Sampling. Samples were shredded and homogenized with dry ice and then shipped frozen to the analytical laboratory.

Samples were generally maintained below -18 °C. In several cases, samples were briefly stored at higher temperature which are not generally expected to adversity impact the residue results. Samples from Trial 18-2014-03 were replaced with reserve samples due to an increase to -0.5 °C during transit. Frozen storage conditions were not reported for study P672186504.

In general, the maximum storage duration for wheat grain was 544 days. However, in samples from study P672186504 (amendment to studies 16-2053 and 16-2054), residues of isoflucypram-desmethyl-propanol and isoflucypram-propanol were extracted for analysis after frozen storage for 817–894 days. Additionally, storage durations were not provided for isoflucypram-desmethyl-propanol and isoflucypram-propanol for study GLP655 (amendment to study S17-07939). The Meeting calculated a maximum frozen storage duration of 483 days based on the harvest date and date of extraction for analysis. No residues were observed in control samples. The results are shown in Table 141.

Table 141 Residues of isoflucypram and metabolites (expresses as parent) in wheat grain treated once with EC formulation at 50 or 42 g ai/L

Trial no. Location	Crop Variety	BBCH at application	Percent Dry Matter	Application rate g ai/ha	DALA[1]	Isoflucypram, mg/kg	Isoflucyprammg/kg, dw	Isoflucypram-Desmethyl-Propanol, mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg, dw	soflucypram-Propanol, mg/kg	soflucypram-Propanol , mg/kg dw
15-2115-01 Parcay Meslay, France[2,3]	Winter wheat Rubisco	69	-	75	45	<0.01	-	-	-	-	-
15-2111-01 Parcay Meslay, France[2,3]	Winter wheat Rubisko	69	-	75	45	<0.01	-	-	-	-	-
15-2115-02 Little Shelford, United Kingdom[2,4]	Winter wheat KWS Chasel Nabim Group 2	69	-	75	43	<0.01	-	-	-	-	-
15-2111-02 Little Shelford, United Kingdom[2,4]	Winter wheat KWS Cashel Nabin Group 2	69	-	75	43	<0.01	-	-	-	-	-
15-2115-03 Wieringerwerf, The Netherlands[2,5]	Spring wheat Quintes	69	-	75	68	<0.01	-	-	-	-	-
15-2111-03 Wieringerwerf, Netherlands[2,5]	Spring wheat Quintes	69	-	75	68	<0.01	-	-	-	-	-
15-2115-04 Bursheid, Germany[2,6]	Spring wheat Chamsin	69	-	75	49	<0.01	-	-	-	-	-
15-2111-04 Bursheid, Germany[2,6]	Spring wheat KWS Chasmin	69	-	75	49	<0.01	-	-	-	-	-
15-2116-01 Santerem, Portugal[2,7]	Winter wheat Jordao	69	-	75	45	<0.01	-	-	-	-	-
15-2069-01 Santarem, Portugal[2,7]	Winter wheat Jordao	69	-	75	45	<0.01	-	-	-	-	-
15-2116-02 Etienne du Gres, France[2,8]	Winter wheat Aubusson	69	-	75	49	<0.01	-	-	-	-	-
15-2119-01 Nimes, Languedoc Roussillon, France[2,8]	Wheat P22R58	69	-	66	65	<0.01	-	-	-	-	-
15-2069-02 Etienne du Gres, France[2,8]	Winter wheat Aubusson	69	-	75	49	<0.01	-	-	-	-	-
15-2116-04 Brenes, Spain[2,9]	Durum wheat Euroduro	65	-	75	51	<0.01	-	-	-	-	-
15-2069-04 Brenes, Spain[2,9]	Durum wheat Vitron	65	-	75	53	<0.01	-	-	-	-	-
15-2120-01 Stowbridge, PE34 3NR, United Kingdom[2]	Winter wheat Skyfall	69	-	63	49	<0.01	-	-	-	-	-
15-2120-02 Vasszecseny, Hungary[2]	Winter wheat GK Szala	69	-	63	34	<0.01	-	-	-	-	-
15-2120-03	Spring wheat	69	-	63	24	<0.01	-	-	-	-	-

Trial no. Location	Crop Variety	BBCH at application	Percent Dry Matter	Application rate g ai/ha	DALA[1]	Isoflucypram, mg/kg	Isoflucyprammg/kg, dw	Isoflucypram-Desmethyl-Propanol, mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg, dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg dw
La Chapelle de Guinchay, France[2]	Togano										
15-2120-04 Piekary, Poland[2]	Spring wheat Tybalt	69	-	63	36	<0.01	-	-	-	-	-
15-2119-02 Mahora, Albacete, Spain[2]	Wheat Sarina	69	-	63	40	<0.01	-	-	-	-	-
15-2119-03 Cartaxo, Ribatejo, Portugal[2]	Wheat Valbona	69	-	63	41	0.042	-	-	-	-	-
15-2119-04 Castallaneta, TA, Italy[2]	Durum wheat Duilio	69	-	63	38	<0.01	-	-	-	-	-
16-2053-01 Chemery, Northern France[2]	Winter wheat Sy moisson	69	-	75	52	<0.01	-	<0.01	-	<0.01	-
16-2053-02 Mellery, Belgium[2]	Winter wheat Rubisco	65	-	75	60	<0.01	-	<0.01	-	<0.01	-
16-2053-03 TR Pesse, Netherlands[2]	Spring wheat Tybalt	69	-	75	57	<0.01	-	<0.01[9]	-	0.019[9]	-
16-2053-04 Leichlingen, Germany[2]	Spring wheat Tybalt	69	-	75	64	<0.01	-	<0.01	-	<0.01	-
16-2054-01 C. da Terrebianche Misterbianco CT, Italy[2]	Durum wheat Anco Marzio	69	-	75	49	<0.01	-	<0.01	-	<0.01	-
16-2054-02 41310 Brenes, Spain[2]	Wheat Artur Nick	69	-	75	49	<0.01	-	<0.01	-	<0.01	-
16-2054-03 Bonnieux, Southern France[2]	Winter wheat Calabro	65	-	75	45	<0.01	-	<0.01	-	<0.01	-
16-2054-04 Ceaux en Loudun, Southern France[2]	Winter wheat Orgrain	69	-	75	52	<0.01	-	<0.01	-	<0.01	-
16-2054-05 Kissa Village, Kozano, Greece[2]	Winter wheat Achilleas	65	-	75	53	<0.01	-	<0.01	-	<0.01	-
18-2014-01 T1 32160 St Aunix Lengros, France	Wheat Oregrain	39	-	76	51	<0.01	-	<0.01	-	<0.01	-
18-2014-01 T2 32160 St Aunix Lengros, France	Wheat Oregrain	49	-	78	43	<0.01	-	<0.01	-	<0.01	-
18-2014-01 T3 Aunix Lengros, France	Wheat Oregrain	59	-	78	35	<0.01	-	<0.01	-	<0.01	-
18-2014-01 T4 Aunix Lengros, France	Wheat Oregrain	69	-	77	29	<0.01	-	<0.01	-	<0.01	-
18-2014-02 T1 Settala, Italy	Wheat Illico	39	-	77	55	<0.01	-	<0.01	-	<0.01	-
18-2014-02 T2	Wheat	49	-	76	49	<0.01	-	<0.01	-	<0.01	-

Trial no. Location	Crop Variety	BBCH at application	Percent Dry Matter	Application rate g ai/ha	DALA[1]	Isoflucypram, mg/kg	Isoflucyprammg/kg, dw	Isoflucypram-Desmethyl-Propanol, mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg, dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg dw
Settala, Italy	Illico										
18-2014-02 T3 Settala, Italy	Wheat Illico	59	-	75	47	<0.01	-	<0.01	-	<0.01	-
18-2014-02 T4 Settala, Italy	Wheat Illico	69	-	76	42	<u><0.01</u>	-	<0.01	-	<0.01	-
18-2014-03 T1 Zafarraya, Spain	Wheat Marius	39	-	77	64	<0.01	-	<0.01	-	<0.01	-
18-2014-03 T2 Zafarraya, Spain	Wheat Marius	49	-	79	55	<0.01	-	<0.01	-	<0.01	-
18-2014-03 T3 Zafarraya, Spain	Wheat Marius	59	-	80	50	<0.01	-	<0.01	-	<0.01	-
18-2014-03 T4 Zafarraya, Spain	Wheat Marius	69	-	73	41	<u>0.015</u>	-	<0.01	-	<0.01	-
18-2014-04 T1 Drymos, Greece	Durum wheat Cannavaro	39	-	74	55	<0.01	-	<0.01	-	<0.01	-
18-2014-04 T2 Drymos, Greece	Durum wheat Cannavaro	49	-	75	53	<0.01	-	<0.01	-	<0.01	-
18-2014-04 T3 Drymos, Greece	Durum wheat Cannavaro	59	-	76	48	<0.01	-	<0.01	-	<0.01	-
18-2014-04 T4 Drymos, Greece	Durum wheat Cannavaro	69	-	76	42	<u><0.01</u>	-	<0.01	-	<0.01	-
18-2135-01 T1 Burscheid, Germany	Wheat Elixer	39	-	75	78	<0.01	-	<0.01	-	<0.01	-
18-2135-01 T2 Burscheid, Germany	Wheat Elixer	45	-	78	69	<0.01	-	<0.01	-	<0.01	-
18-2135-01 T3 Burscheid, Germany	Wheat Elixer	58	-	75	62	<0.01	-	<0.01	-	<0.01	-
18-2135-01 T4 Burscheid, Germany	Wheat Elixer	69	-	76	44	<u><0.01</u>	-	<0.01	-	<0.01	-
18-2135-02 T1 Mellet, Belgium	Wheat Mistral	39	-	84	74	<0.01	-	<0.01	-	<0.01	-
18-2135-02 T2 Mellet, Belgium	Wheat Mistral	49	-	74	71	<0.01	-	<0.01	-	<0.01	-
18-2135-02 T3 Mellet, Belgium	Wheat Mistral	59	-	74	66	<0.01	-	<0.01	-	<0.01	-
18-2135-02 T4 Mellet, Belgium	Wheat Mistral	69	-	77	57	<u><0.01</u>	-	<0.01	-	<0.01	-
18-2135-03 T1 Great Chishill, United Kingdom	Wheat KWS Trinity	41	-	79	76	<0.01	-	<0.01	-	<0.01	-
18-2135-03 T2 SG8 8GS Great Chishill, United Kingdom	Wheat KWS Trinity	49	-	75	72	<0.01	-	<0.01	-	<0.01	-
18-2135-03 T3 SG8 8GS Great Chishill, near Royston, United Kingdom	Wheat KWS Trinity	59	-	76	65	<0.01	-	<0.01	-	<0.01	-

Trial no. Location	Crop Variety	BBCH at application	Percent Dry Matter	Application rate g ai/ha	DALA[1]	Isoflucypram, mg/kg	Isoflucypram mg/kg, dw	Isoflucypram-Desmethyl-Propanol, mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg, dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg dw
18-2135-03 T4 SG8 8GS Great Chishill, United Kingdom	Wheat KWS Trinity	69	-	74	53	<0.01	-	<0.01	-	<0.01	-
18-2135-04 T1 Zwaagdijk, Netherlands	Wheat Nobless	39	-	75	58	<0.01	-	<0.01	-	<0.01	-
18-2135-04 T2 Zwaagdijk, Netherlands	Wheat Nobless	49	-	77	54	<0.01	-	<0.01	-	<0.01	-
18-2135-04 T3 Zwaagdijk, Netherlands	Wheat Nobless	59	-	76	49	<0.01	-	<0.01	-	<0.01	-
18-2135-04 T4 1681 ND Zwaagdijk, Netherlands	Wheat Nobless	69	-	75	35	0.014	-	<0.01	-	<0.01	-
17-2020-01 St Etienne du Gres, Southern France[2]	Wheat Arkeos	69	-	63	44	<0.01	-	<0.01	-	<0.01	-
17-2020-02 Paradas, Spain[2]	Durum Wheat Amilcar	69	-	63	57	<0.01	-	<0.01	-	<0.01	-
17-2020-03 50100 Thymaria Kozanis, Greece[2]	Wheat Zanzibar	69	-	63	41	<0.01	-	<0.01	-	<0.01	-
17-2020-04 Mineo, Italy[2]	Durum Wheat Alemanno	69	-	63	51	<0.01	-	<0.01	-	<0.01	-
17-2019-01 Burscheid, Germany[2]	Winter wheat Potential	69	-	63	66	<0.01	-	<0.01	-	<0.01	-
17-2019-02 Chambourg Sur Indre, Northern France[2]	Winter wheat Venezio	69	-	63	42	<0.01	-	<0.01	-	<0.01	-
17-2019-03 Anrochte-Berge, Germany[2]	Spring wheat Cernetto	69	-	63	43	<0.01	-	<0.01	-	<0.01	-
17-2019-04 ND Zwaagdijk, Netherlands[2]	Spring wheat Tybalt	69	-	63	39	<0.01	-	<0.01	-	<0.01	-
PNZ16414-01 Irwell, New Zealand	Wheat B12	69	87	70	63	<0.01	<0.01	-	-	-	-
PNZ16414-02 Rakaia, New Zealand	Wheat Starfire	69	86	72	63	<0.01	<0.01	-	-	-	-
BAYERNZ/GLP/16/04- Hawkes Bay, New Zealand[2]	Wheat Starfire	69	88	75	59	<0.01	<0.01	-	-	-	-
BAYERNZ/GLP/16/04-02, Manawatu, New Zealand[2]	Wheat Rappels	69	83	75	56	<0.01	<0.01	-	-	-	-
S17-07939-01/GLP655-01[11]	Spring wheat Raffles	69	82	116[12]	54	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Trial no. Location	Crop Variety	BBCH at application	Percent Dry Matter	Application rate g ai/ha	DALA[1]	Isoflucypram, mg/kg	Isoflucyprammg/kg, dw	Isoflucypram-Desmethyl-Propanol, mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg, dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg dw
Timaru, New Zealand											
S17-07939-02/GLP655-02 Laureston, New Zealand	Spring wheat Raffles	69	75	116[12]	54	<0.01	<0.01	<0.01, <0.01	<0.01, <0.0	0.026, <0.01	0.034, <0.01
S17-07939-03/GLP655-03 Opiki, New Zealand[2,13]	Spring wheat Sensas	69	79	75	49	0.019	0.024	<0.01, <0.01	<0.01, <0.0	0.020, <0.01, <0.01	0.025, <0.01, <0.01
S17-07939-04/GLP655-04 Fielding, New Zealand[2,13]	Spring wheat Sensas	69	76	75	47	<0.01	<0.01	<0.01, <0.01	<0.01, <0.0	<0.01, <0.01	<0.01, <0.01
S18-07829-01 Kairanga, New Zealand[14]	Spring wheat Discovery	69	86	75	54	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
S18-07829-02 Cheltenham, New Zealand[14]	Spring wheat Sensas	69	87	75	45	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
S18-07829-03 Opiki, New Zealand[2]	Spring wheat Sensas	69	86	75	58	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
S18-07829-04 Ruapuna, New Zealand[2]	Spring wheat Raffles	69	86	75	54	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
S18-07829-05 Mitcham, New Zealand[2]	Spring wheat Raffles	69	85	75	57	0.01, <0.01 (<0.01)	<0.01, <0.01	<0.01, <0.01	<0.01, <0.0	<0.01, <0.01	<0.01, <0.01

Notes:

dw = dry wight.

[1]DALA = Days after last application.

[2] Application rates are reported as the nominal values if the measured rates were ±5 percent of the planned rates.

[3] Trials 15-2115-01 and 15-2111-01 are not independent.

[4] Trials 15-2115-02, and 15-2111-02 are not independent.

[5] Trials 15-2115-03 and 15-2111-03 are not independent.

[6] Trials 15-2115-04 and 15-2111-04 are not independent.

[7] Trials 15-2116-01 and 15-2069-01 are not independent.

[8] Trials 15-2116-02, 2119-01, and 15-2069-02 are not independent.

[9] Trials 15-2116-04 and 15-2069-04 are not independent.

[10] Average of duplicate analyses.

[11] Samples from Trial 07939-01 were harvested at BBCH 87.

[12] Trials S17-07939-01/GLP655-01 and S17-07939-02/GLP655-02 were accidentally overdosed.

[13] Trials S17-07939-03/GLP655-03 and S17-07939-04/GLP655-04 are not independent.

[14] Trials S18-07829-01 and S18-07829-02 are not independent. Samples were harvested at BBCH 93.

Straw, fodder, and forage or cereal grains

Residue trials for barley and wheat green material and straw matrices are the same as those for barley and wheat grain described above.

Barley

A minimum of 1.0 kg and 0.50 kg of treated sample was collected for barley green material and straw, respectively. In general, the maximum storage duration for barley green material and straw was 544 days which is supported by storage stability data. However, in straw samples from P672186503 (amendment to studies 16-2051 and 16-2052), residues of isoflucypram-desmethyl-propanol and isoflucypram-propanol were extracted for analysis after frozen storage for 786–852 days. Additionally, frozen storage durations from Trial GLP658 (amendment to study S17-07996) were not reported for isoflucypam-desmethyl-propanol and isoflucypram-propanol. The Meeting calculated the maximum duration of frozen storage of 481 days for green material and straw based on harvest date and date of extraction for analysis.

Residues of isoflucypram, isoflucypram-propanol, or isoflucypram-desmethyl-propanol were generally below the LOQ in control samples, with the following of residues of isoflucypram, isoflucypram-desmethyl-propanol, and isoflucypram-propanol were 0.19, 0.037, and 0.13 mg eq/kg in untreated barley straw from Trial 17-2018-01. The results are in Table 142.

Table 142 Residues of isoflucypram and metabolites (expresses as parent) in barley green material and straw treated once with EC formulation at 50 or 42 g ai/L at BBCH 61

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application Rate (g ai/ha)	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, mg/kg dw	Isoflucypram-Desmethyl-Propanol, mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg dw
15-2114-01 Santarem, Portugal[2,3] (Pewter)	Green material	61	36	75	0	1.7	-	-	-	-	-
		65	39		6	1.4	-	-	-	-	-
		71	45		14	0.50	-	-	-	-	-
		73	54		21	0.42	-	-	-	-	-
		75	60		28	0.29	-	-	-	-	-
	Straw	89	-		60	<u>1.0</u>	-	-	-	-	-
15-2066-01 Santarem, Portugal[2,3] (Pewter)	Green material	61/61	36	75	0	1.9	-	-	-	-	-
		65	44		6	1.5	-	-	-	-	-
		71	48		14	0.52	-	-	-	-	-
		73	53		21	0.37	-	-	-	-	-
		75	58		28	0.23	-	-	-	-	-
	Straw	89	-		60	0.85	-	-	-	-	-
15-2114-02 Etienne du Gres, France[2,4] (Baraka)	Green material	61/61	21	75	0	1.0	-	-	-	-	-
		69	25		7	0.24	-	-	-	-	-
		71	29		14	0.15	-	-	-	-	-
		75	37		21	0.10	-	-	-	-	-
		77	33		27	0.074	-	-	-	-	-
	Straw	89	-		69	<u>0.16</u>	-	-	-	-	-
15-2117-01 Nimes Languedoc Roussillon, France[2,4] (Jallon)	Green material	61/61	30	63	0	0.87	-	-	-	-	-
		71	32		7	0.14	-	-	-	-	-
		75	50		13	0.077 0.091 (0.084)	-	-	-	-	-
		77	47		21	0.065	-	-	-	-	-
		83	54		28	0.050	-	-	-	-	-
	Straw	89	-		48	0.13	-	-	-	-	-
15-2066-02	Green material	61/61	29	75	0	1.6	-	-	-	-	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application Rate (g ai/ha)	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, mg/kg dw	Isoflucypram-Desmethyl-Propanol , mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg, dw
St. Etienne du Gres, France[2,5] (Baraka)		69	31		7	0.28	-	-	-	-	-
		71	34		14	0.14	-	-	-	-	-
		75	41		21	0.091	-	-	-	-	-
		77	43		27	0.076	-	-	-	-	-
	Straw	89	-		69	0.14	-	-	-	-	-
15-2114-03 Marchena, Spain[2,5] (Traveler malting)	Green material	53/53	20	75	0	1.6	-	-	-	-	-
		61	30		7	1.1	-	-	-	-	-
		71	35		14	0.65	-	-	-	-	-
		75	39		21	0.27	-	-	-	-	-
		75	46		22	0.32	-	-	-	-	-
		83	48		28	0.20	-	-	-	-	-
	Straw	89	-		57	<u>0.29</u>	-	-	-	-	-
15-2066-03 Marchena, Spain[2,5] (Traveler malting)	Green material	53/53	25	75	0	2.0	-	-	-	-	-
		61	32		7	1.2	-	-	-	-	-
		71	39		14	0.55	-	-	-	-	-
		75	40		21	0.28	-	-	-	-	-
		75	43		22	0.25	-	-	-	-	-
		83	52		28	0.20	-	-	-	-	-
	Straw	89	-		57	0.22	-	-	-	-	-
15-2114-04 Bouloc, France[2,6] (Cacia)	Green material	61/61	30	75	0	1.8	-	-	-	-	-
		69	45		7	1.1	-	-	-	-	-
		73	43		14	0.42	-	-	-	-	-
		75	46		17	0.45	-	-	-	-	-
		77	54		21	0.37	-	-	-	-	-
		83	57		28	0.47	-	-	-	-	-
	Straw	89	-		45	0.96	-	-	-	-	-
15-2066-04 Bouloc, France[2,6] (Cacia)	Green material	61/61	41	75	0	1.8	-	-	-	-	-
		69	50		7	1.0	-	-	-	-	-
		73	55		14	0.28	-	-	-	-	-
		75	56		17	0.39	-	-	-	-	-
		77	54		21	0.37	-	-	-	-	-
		83	58		28	0.41	-	-	-	-	-
	Straw	89	-		45	0.65	-	-	-	-	-
15-2113-01 Wieringwerf, Netherlands[2,7] (Triple summer)	Green material	61/61	29	75	0	1.8	-	-	-	-	-
		65	31		7	0.41	-	-	-	-	-
		73	36		14	0.23	-	-	-	-	-
		75	38		16	0.12	-	-	-	-	-
		75	44		21	0.11	-	-	-	-	-
		77	48		28	0.051	-	-	-	-	-
	Straw	89	-		61	<u>0.049</u>	-	-	-	-	-
15-2110-01 Middenmeer, Netherlands[2,7] (Odyssey)	Green material	61/61	34	75	0	1.5	-	-	-	-	-
		61	39		7	0.41	-	-	-	-	-
		75	43		14	0.20	-	-	-	-	-
		77	49		21	0.12	-	-	-	-	-
		83	51		28	0.12	-	-	-	-	-
	Straw	89	-		55	0.11	-	-	-	-	-
15-2113-02	Green material	59/59	24	75	0	2.0	-	-	-	-	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application Rate (g ai/ha)	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, mg/kg dw	Isoflucypram-Desmethyl-Propanol , mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg, dw
Burscheid, Germany[2,8] (Streif)		61	28		7	1.1	-	-	-	-	-
		65	37		14	0.55	-	-	-	-	-
		75	39		19	0.39	-	-	-	-	-
		77	44		21	0.28	-	-	-	-	-
		83	47		28	0.20	-	-	-	-	-
	Straw	89	-		68	<u>0.24</u>	-	-	-	-	-
15-2110-02 Burscheid, Germany[2,8] (Streif)	Green material	59/59	31	75	0	2.2	-	-	-	-	-
		61	36		7	0.98	-	-	-	-	-
		65	42		14	0.33	-	-	-	-	-
		75	42		19	0.22	-	-	-	-	-
		77	48		21	0.18	-	-	-	-	-
		83	53		28	0.092	-	-	-	-	-
	Straw	89	-		68	0.12	-	-	-	-	-
15-2113-03 Esvres sur Indre, France[2,9] (Etincel)	Green material	61/61	24	75	0	1.1	-	-	-	-	-
		69	25		7	0.18	-	-	-	-	-
		71	39		14	0.11	-	-	-	-	-
		73	35		21	0.080	-	-	-	-	-
		75	46		28	0.051	-	-	-	-	-
	Straw	89	-		62	<u>0.20</u>	-	-	-	-	-
15-2110-03 Esvres sur Indre, France[2,9] (Etincel)	Green material	61/61	30	75	0	1.2	-	-	-	-	-
		69	32		7	0.18	-	-	-	-	-
		71	36		14	0.16	-	-	-	-	-
		73	45		21	0.091	-	-	-	-	-
		75	50		28	0.098	-	-	-	-	-
	Straw	89	-		62	0.18	-	-	-	-	-
15-2113-04 Little Shelford, Cambridge, United Kingdom[2,10] (Glacier)	Green material	61/61	26	75	0	1.7	-	-	-	-	-
		69	33		7	1.2	-	-	-	-	-
		69	33		14	0.69	-	-	-	-	-
		71	41		20	0.46	-	-	-	-	-
		73	46		28	0.26	-	-	-	-	-
		75	55		33	0.28	-	-	-	-	-
	Straw	89	-		77	<u>0.94</u>	-	-	-	-	-
15-2110-04 Little Shelford, Cambridge, United Kingdom[2,10] (Glacier)	Green material	61/61	38	75	0	1.3	-	-	-	-	-
		69	39		7	1.2	-	-	-	-	-
		69	46		14	0.59	-	-	-	-	-
		71	50		20	0.35	-	-	-	-	-
		73	54		28	0.22	-	-	-	-	-
		75	52		33	0.18	-	-	-	-	-
	Straw	89	-		77	0.60	-	-	-	-	-
15-2117-02 Guarene, Italy[2] (Sfera)	Green material	61/61	32	63	0	1.1	-	-	-	-	-
		71	44		7	0.75	-	-	-	-	-
		75	38		14	0.31	-	-	-	-	-
		77	57		21	0.24	-	-	-	-	-
		87	82		28	0.34	-	-	-	-	-
	Straw	89	-		35	0.29	-	-	-	-	-
15-2117-03 Mahora, Spain[2]	Green material	61/61	25	63	0	1.3	-	-	-	-	-
		71	29		7	0.20	-	-	-	-	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application Rate (g ai/ha)	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, mg/kg dw	Isoflucypram-Desmethyl-Propanol , mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg, dw
(Shakira)		75	35		13	0.11	-	-	-	-	-
		77	36		20	0.054	-	-	-	-	-
		77	42		27	0.048	-	-	-	-	-
	Straw	89	-		46	0.021	-	-	-	-	-
15-2117-04 Cartaxo, Portugal[2] (Pewter)	Green material	61/61	43	63	0	1.7	-	-	-	-	-
		75	59		8	0.83	-	-	-	-	-
		77	64		14	0.83	-	-	-	-	-
		83	87		20	1.4	-	-	-	-	-
		87	92		27	1.4	-	-	-	-	-
	Straw	89	-		41	3.1	-	-	-	-	-
15-2118-01 Linconshire, PE12 9 PQ, United Kingdom[2] (Cassia)	Green material	61/61	29	63	0	1.2	-	-	-	-	-
		65	34		8	0.74	-	-	-	-	-
		71	40		13	0.25	-	-	-	-	-
		75	47		21	0.16	-	-	-	-	-
		83	58		28	0.075	-	-	-	-	-
	Straw	89	-		63	<u>0.16</u>	-	-	-	-	-
15-2118-02 La Chapelle de Guinchay, Bourgogne, France[2] (Esterel)	Green material	61/61	32	63	0	1.2	-	-	-	-	-
		69	38		7	0.12	-	-	-	-	-
		75	43		14	0.14	-	-	-	-	-
		85	56		21	0.095	-	-	-	-	-
		87	71		28	0.094	-	-	-	-	-
	Straw	89	-		55	0.51	-	-	-	-	-
15-2118-03 Vasszecseny, Hungary[2] (Mandolina)	Green material	61/61	37	63	0	0.87	-	-	-	-	-
		73	45		7	0.53	-	-	-	-	-
		75	50		14	0.31	-	-	-	-	-
		83	62		21	0.35	-	-	-	-	-
		85	63		28	0.55	-	-	-	-	-
	Straw	89	-		35	0.96	-	-	-	-	-
15-2118-04 Zlinsky Kraj, Czech Republic[2] (Kangoo)	Green material	61/61	32	63	0	2.4	-	-	-	-	-
		69	35		7	1.5	-	-	-	-	-
		75	42		14	0.91	-	-	-	-	-
		83	52		20	0.63	-	-	-	-	-
		85	61		28	0.56	-	-	-	-	-
	Straw	89	-		55	1.2	-	-	-	-	-
PNZ16414-03 Timaru, New Zealand (776)	Green material	61/61	38	76	0	0.93	2.4	-	-	-	-
		83	76		35	0.59	0.78	-	-	-	-
		85	69		42	<u>0.35</u>	<u>0.51</u>	-	-	-	-
		87	78		49	0.24	0.31	-	-	-	-
	Straw	89	77		56	0.31, 0.24 (0.28)	0.40, 0.31 (0.36)	-	-	-	-
		89	72		65	0.39, 0.40 (0.40)	0.54, 0.55 (0.55)	-	-	-	-
PNZ16414-04 St. Andrews, New Zealand (Sanette)	Barley green material	63/63	31	77	0	0.27 0.30, 0.31 (0.29)	0.88, 0.97, 1.01 (0.95)	-	-	-	-
		85	67		42	0.36, 0.28 (<u>0.32</u>)	0.54, 0.41 (<u>0.48</u>)	-	-	-	-
	Straw	89	65		49	0.51, 0.59,	0.79, 0.92,	-	-	-	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application Rate (g ai/ha)	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, mg/kg dw	Isoflucypram-Desmethyl-Propanol , mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg, dw
						0.60 (0.57)	0.93 (0.88)				
BAYERNZ/GLP/16/04	Green material	61/61	30	75	0	2.7	9.0	-	-	-	-
/a-03 Hawkes Bay, New Zealand[2] (Sumit)		85	68		34	.139, 0.15 0.118 (0.14)	0.21, 0.23 0.174 (0.20)	-	-	-	-
		87	83		42	0.199	0.24	-	-	-	-
		87	86		49	0.179	0.21	-	-	-	-
	Straw	89	81		56	0.33 0.29, 0.31 (0.31)	0.41 0.35, 0.38 (0.38)	-	-	-	-
BAYERNZ/GLP/16/04	Barley green material	61/61	18	75	0	1.36, 1.35 (1.36)	7.6, 7.6 (7.6)	-	-	-	-
/a-04 Manawatu, New Zealand[2] (Fairview)		87	46		42	.045 0.040 (0.043)	0.099 0.08 (0.094)	-	-	-	-
	Straw	89	60		56	.100 0.093 (0.097)	0.167 0.15 (0.16)	-	-	-	-
E19RP054-01 Burscheid, Germany (Avalon)	Green material	61/61	-	76	0	2.22	-	<0.01	-	<0.01	-
	Straw	89	-		57	0.406	-	0.019	-	0.338	-
E19RP054-02 Kommingen, Germany (Avalon)	Green material	61/61	-	75	0	1.51	-	<0.01	-	<0.01	-
	Straw	89	-		27	2.11	-	0.020	-	0.114	-
E19RP054-03 Werl- Niederbergstrabe, Germany (Meridian)	Green material	61/61	-	77	0	1.29	-	<0.01	-	<0.01	-
	Straw	89	-		47	0.258	-	0.040	-	0.072	-
E19RP054-04 Tilly, Belgium (Keeper)	Green material	61/61	-	74	0	0.852	-	<0.01	-	<0.01	-
	Straw	89	-		50	0.084	-	0.049	-	0.064	-
E19RP055-01 St Caprais, France (Cassia)	Green material	61/61	-	73	0	1.05	-	<0.01	-	<0.01	-
	Straw	89	-		56	0.164	-	0.043	-	0.229	-
E19RP055-02 Maire, France (Etincel)	Green material	61/61	-	78	0	1.58	-	<0.01	-	<0.01	-
	Straw	89	-		51	0.264	-	0.064	-	0.169	-
E19RP055-03 Boara Ferrara, Italy (Marjorie)	Green material	61/61	-	76	0	1.24	-	<0.01	-	<0.01	-
	Straw	89	-		41	0.036	-	0.029	-	0.118	-
E19RP055-04 Sommacampagna via Cessarina, Italy (Calanque)	Green material	61/61	-	74	0	1.48	-	<0.01	-	<0.01	-
	Straw	89	-		48	0.034	-	0.050	-	0.112	-
E19RP056-01 Borgo d'Ale, Italy (Tunika)	Green material	61/61	-	64	0	1.8	-	<0.01	-	<0.01	-
	Straw	89	-		42	0.073	-	0.031	-	0.13	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application Rate (g ai/ha)	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, mg/kg dw	Isoflucypram-Desmethyl-Propanol , mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg, dw
E19RP056-02 Zafarraya, Spain (Yuriko)	Green material	61/61	-	63	0	0.99	-	<0.01	-	<0.01	-
	Straw	89	-		50	0.48	-	0.041	-	0.13	-
17-2017-01 Kranzlin, Germany[2] (Simba)	Green material	61/61	-	63	0	3.6	-	<0.01	-	<0.01	-
		73	-		7	1.0	-	0.068	-	0.14	-
		75	-		14	0.72	-	0.087	-	0.17	-
		77	-		22	0.25	-	0.028	-	0.069	-
		83	-		30	<0.01	-	<0.01	-	<0.01	-
	Straw[12]	89	-		55	0.23	-	0.037	-	0.13	-
17-2017-02 Tinglev, Denmark[2] (Overture)	Green material	61/61	-	63	0	1.4	-	<0.01	-	<0.01	-
		73	-		7	0.32	-	0.016	-	0.030	-
		75	-		9	0.30	-	0.024	-	0.044	-
		77	-		15	0.068	-	0.012	-	0.012	-
		83	-		21	0.15	-	0.012	-	0.024	-
		85	-		28	0.15	-	0.010	-	0.026	-
	Straw	89	-		50	0.25	-	0.010	-	0.048	-
17-2017-03 (Oxfordshire, United Kingdom[2] (Octavia)	Green material	61/61	-	63	0	1.8	-	<0.01	-	<0.01	-
		65	-		7	0.69	-	0.023	-	0.086	-
		73	-		13	0.47	-	0.028	-	0.11	-
		73	-		21	0.32	-	0.011	-	0.056	-
		75	-		27	0.21	-	0.031	-	0.13	-
	Straw	89	-		57	0.24	-	0.041	-	0.14	-
17-2017-04 (Juvincourt et Dammary, France[2] (Irina)	Green material	61/61	-	63	0	1.4	-	<0.01	-	<0.01	-
		71	-		7	0.78	-	0.011	-	0.043	-
		75	-		14	0.35	-	0.015	-	0.064	-
		83	-		20	0.39	-	0.012	-	0.075	-
		87	-		28	0.40	-	0.010	-	0.082	-
	Straw	89	-		41	0.37	-	0.016	-	0.13	-
17-2018-01 Upie, France[2] (Maltesse winter)	Green material	61/61	-	63	0	0.54	-	<0.01	-	<0.01	-
		65	-		7	0.10	-	0.025	-	0.013	-
		69	-		14	0.12	-	0.055	-	0.046	-
		73	-		22	0.046	-	0.040	-	0.033	-
		75	-		28	0.025	-	0.037	-	0.026	-
	Straw	89	-		38	0.21	-	0.041	-	0.16	-
17-2018-02 Settala, Italy[2] (Concerto)	Green material	61/61	-	63	0	1.1	-	<0.01	-	<0.01	-
		65	-		7	0.15	-	0.010	-	0.023	-
		73	-		14	0.10	-	0.010	-	0.029	-
		75	-		22	0.078	-	<0.01	-	0.028	-
		83	-		28	0.097	-	0.010	-	0.039	-
	Straw	89	-		44	<0.01	-	<0.01	-	<0.01	-
17-2018-03 Montamaneu, Spain[2] (Meseta winter)	Green material	61/61	-	63	0	0.50	-	<0.01	-	<0.01	-
		69	-		8	0.30	-	0.033	-	0.044	-
		73	-		14	0.34	-	0.040	-	0.095	-
		75	-		18	0.23	-	0.019	-	0.045	-
		75	-		20	0.15	-	0.057	-	0.089	-
		83	-		27	0.20	-	0.023	-	0.067	-
	Straw	89	-		48	0.40	-	0.072	-	0.38	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application Rate (g ai/ha)	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, mg/kg dw	Isoflucypram-Desmethyl-Propanol , mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg, dw
17-2018-04 Drymos, Greece[2] (Hyvito)	Green material	61/61	-	63	0	1.9	-	<0.01	-	<0.01	-
		71	-		7	0.51	-	0.031	-	0.029	-
		73	-		14	0.31	-	0.15	-	0.11	-
		73	-		21	0.36	-	0.17	-	0.20	-
		75	-		23	0.22	-	0.032	-	0.031	-
		77	-		27	0.31	-	0.089	-	0.10	-
	Straw	89	-		43	0.26	-	0.057	-	0.095	-
16-2052-01 Santarem, Portugal[2] (Pewter)	Green material	61/61	30	75	0	1.4	-	-	-	-	-
		65	26		7	0.37	-	-	-	-	-
		71	31		14	0.28	-	-	-	-	-
		73	31		21	0.16	-	-	-	-	-
		75	38		28	0.079	-	-	-	-	-
	Straw	89	-		67	0.24	-	0.044	-	0.53	-
16-2052-02 St Etienne du Gres, France[2] (Augusta)	Green material	61/61	25	75	0	1.8	-	-	-	-	-
		69	30		7	1.3	-	-	-	-	-
		71	33		12	0.69	-	-	-	-	-
		73	42		20	0.30	-	-	-	-	-
		75	44		27	0.23	-	-	-	-	-
	Straw	89	-		54	0.31	-	0.094	-	0.34	-
16-2052-03 Arahal, Spain[2] (Odissey)	Green material	61/61	27	75	0	3.1	-	-	-	-	-
		71	21		7	0.90	-	-	-	-	-
		73	34		14	0.89	-	-	-	-	-
		75	53		21	0.95	-	-	-	-	-
		77	34		28	0.32	-	-	-	-	-
	Straw	89	-		57	0.85	-	0.047	-	0.33	-
16-2052-04 Gargas, France[2] (Cacia)	Green material	61/61	28	75	0	0.94	-	-	-	-	-
		71	37		8	0.55	-	-	-	-	-
		75	40		14	0.30	-	-	-	-	-
		77	39		21	0.23	-	-	-	-	-
		83	39		28	0.091	-	-	-	-	-
	Straw	89	-		49	0.18	-	0.058	-	0.23	-
16-2051-01 Cambridge, United Kingdom[2] (Odyssey)	Green material	61/61	28	75	0	2.4	-	-	-	-	-
		69	32		8	0.25	-	-	-	-	-
		73	35		15	0.16	-	-	-	-	-
		75	44		22	0.10	-	-	-	-	-
		75	42		22	0.13	-	-	-	-	-
		83	50		29	0.090	-	-	-	-	-
	Straw	89	-		57	0.40	-	0.039	-	0.28	-
16-2051-02 Burscheid, Germany[2] (Vespa)	Green material	61/61	27	75	0	2.4	-	-	-	-	-
		71	36		7	0.53	-	-	-	-	-
		75	36		14	0.17	-	-	-	-	-
		75	44		14	0.17	-	-	-	-	-
		77	45		21	0.13	-	-	-	-	-
		83	59		28	0.10	-	-	-	-	-
	Straw	89	-		60	0.32	-	0.034	-	0.16	-
16-2051-03 Chambourg sur Indre,	Green material	61/61	25	75	0	1.2	-	-	-	-	-
		69	22		7	0.45	-	-	-	-	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application Rate (g ai/ha)	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, mg/kg dw	Isoflucypram-Desmethyl-Propanol , mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg, dw
France[2] (Obit)		71	31		14	0.30	-	-	-	-	-
		73	32		21	0.18	-	-	-	-	-
		75	30		27	0.11	-	-	-	-	-
	Straw	89	-		53	0.13	-	0.028	-	0.15	-
16-2051-04 Zwaagdijk, Netherlands[2] (Quadrigo)	Green material	61/61	39	75	0	1.6	-	-	-	-	-
		65	43		7	0.97	-	-	-	-	-
		65	45		14	0.83	-	-	-	-	-
		71	56		21	0.54	-	-	-	-	-
		83	60		28	0.39	-	-	-	-	-
		83	66		34	0.41	-	-	-	-	-
	Straw	89	-		37	0.44	-	0.013	-	0.070	-
S17-07996- 01/GLP658-01 Ashburton, New Zealand (Bumpa)	Green material	61/61	26	116[13]	0	3.1	12.1	<0.01	<0.01	<0.01	<0.01
		73	36		35	.113, 0.12 (0.117)	0.32, 0.34 (0.33)	0.059	0.165	0.26	0.74
		75	49		42	<u>0.113</u>	<u>0.23</u>	0.093	0.191	0.39	0.80
		77	37		49	0.060	0.164	0.063	0.171	0.190	0.52
		83	57		55	0.072	0.127	0.054	0.094	0.32	0.57
	Straw	92	49		87	0.033	0.068	0.020	0.042	0.058	0.119
S17-07996- 02/GLP658 -02, Springston, New Zealand (Bumpa)	Green material	61/61	27	106[13]	0	3.5	12.9	<0.01	<0.01	<0.01	<0.01
		87	43		42	<u>0.059</u>	<u>0.137</u>	0.061	0.141	0.21	0.50
	Straw	89	63		56	0.126	0.20	0.044	0.071	0.33	0.52
S17-07996- 03/GLP658-03 Kairanga, New Zealand)[14] (Bumpa)	Green material	61/61	28	75	0	1.16	4.1	<0.01	<0.01	<0.01	<0.01
		87	68		35	0.23, 0.31 (0.27)	0.34, 0.45 (0.40)	0.025	0.037	0.087	0.128
		87	61		42	<u>0.195</u>	<u>0.32</u>	0.019	0.032	0.062	0.103
		89	78		49	0.197	0.25	0.019	0.025	0.050	0.065
	Straw	89	73		49	<u>0.24</u>	<u>0.33</u>	0.020	0.027	0.056	0.077
S17-07996- 04/GLP658-04 Bulls, New Zealand[2,14] (Calibre)	Green material	61/61	26	75	0	1.62, 1.64 (1.63)	6.2, 6.3 (6.3)	<0.01	<0.01	<0.01	<0.01
		85-87	60		43	0.035	0.058	0.056	0.093	0.24	0.40
		89	71		56	0.018	0.025	0.059	0.083	0.179	0.25
	Straw	89	62		61	0.055	0.088	0.040, 0.042 (0.041)	.064, 0.06 (0.066)	0.21, 0.23 (0.22)	0.33, 0.37 (0.35)
S18-07828-01 Otane, New Zealand[2] (Jimpy)	Green material	61/61	21	75	0	3.1	14.9	<0.01	<0.01	<0.01	<0.01
		87	57		35	.22, 0.177 (0.199)	0.39, 0.31 (0.35)	0.062, 0.051 (0.057)	.109, 0.09 (0.100)	0.25, 0.19 (0.222)	0.44, 0.34 (0.39)
		87	66		43	<u>0.21</u>	<u>0.32</u>	0.077	0.117	0.190	0.29
		87	82		49	0.22	0.27	0.077	0.094	0.21	0.25
	Straw	89	65		56	<u>0.45</u>	<u>0.69</u>	0.070	0.109	0.37	0.57
S18-07828-02 Cheltenham, New Zealand[2,15] (Planet)	Green material	61/56-61	24	75	0	1.9	7.7	<0.01	<0.01	<0.01	<0.01
		89	85		42	<u>0.31</u>	<u>0.37</u>	0.171	0.20	0.43	0.51
	Straw	89	88		45	0.81	0.91	0.127	0.144	0.59	0.67
S18-07828-03 Beconsfield, New	Green material	61/59-61	20	75	0	1.6	8.2	<0.01	<0.01	<0.01	<0.01
		85-87	72		42	0.22	0.31	0.134	0.188	0.33	0.47

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application Rate (g ai/ha)	DALA[1]	Isoflucypram, mg/kg	Isoflucypram, mg/kg dw	Isoflucypram-Desmethyl-Propanol , mg/kg	Isoflucypram-Desmethyl-Propanol, mg/kg dw	Isoflucypram-Propanol, mg/kg	Isoflucypram-Propanol , mg/kg, dw
Zealand[2,15] (Cassia)	Straw	87-89	62		53	0.55, 0.49 (0.52)	0.88, 0.79 (0.84)	0.076, 0.072 (0.074)	.122, 0.11 (0.119)	0.39, 0.35 (0.37)	0.62, 0.56 (0.59)
S18-07828-04 Tinwald, New Zealand[2,16] (Milford)	Green material	61/61	22	75	0	1.4	6.5	<0.01	<0.01	<0.01	<0.01
		87	73		40	0.076	0.104	0.013	0.018	0.025	0.035
	Straw	93	90		54	0.22	0.25	0.050	0.056	0.121	0.135
S18-07828-05 Mitcham, New Zealand[2,16] (Tavern)	Green material	61/61	18	75	0	1.83, 2.0 (1.92)	10.1, 11.2 (10.7)	<0.01, <0.01	<0.01, <0.0	<0.01, <0.0	<0.01, <0.01
		77-80	46		36	.076, 0.06 (0.071)	.167, 0.14 (0.16)	0.029, 0.026 (0.028)	.063, 0.05 (0.060)	.063, 0.06 (0.065)	0.138, 0.144 (0.14)
		80-85	43		43	.030, 0.03 (0.034)	.071, 0.08 (0.079)	0.016, <0.01 (0.013)	.036, <0.0 (0.023)	.020, 0.02 (0.020)	0.047, 0.047 (0.047)
		87	66		51	.063, 0.07 (0.071)	.096, 0.12 (0.11)	0.019, 0.034 (0.027)	.029, 0.05 (0.041)	.047, 0.06 (0.058)	0.072, 0.104 (0.088)
	Straw	93	90		57	0.22, 0.23, 0.24 (0.23)	0.25, 0.26 (0.26)	0.101, 0.100, 0.075 (0.09)	.113, 0.11 0.083 (0.10)	0.32, 0.38 0.32 (0.34)	.36, 0.42, 0.35 (0.38)

Notes:
dw = dry weight.
[1] DALA = Days after last application.
[2] Application rates are reported as the nominal values if the measured rates were ±5 percent of the planned rates.
[3] Trials 15-2114-01 and 15-2066-01 are not independent.
[4] Trials 15-2114-02, 15-2117-01, and 15-2066-02 are not independent.
[5] Trials 15-2114-03 and 15-2066-03 are not independent.
[6] Trials 15-2114-04 and 15-2066-04 are not independent.
[7] Trials 15-2113-01 and 15-2110-01 are not independent.
[8] Trials 15-2113-02 and 15-2110-02 are not independent.
[9] Trials 15-2113-03 and 15-2110-03 are not independent.
[10] Trials 15-2113-04 and 15-2110-04 are not independent.
[12] Quantifiable residues in control samples. Residues presented are not corrected for residues in controls.
[13] Trials S17-07996-01/GLP658 and S17-07996-02/GLP658 were accidentally overdosed.
[14] Trials S17-07996-03/GLP658-03 and S17-07996-04/GLP658-04 are not independent.
[15] Trials S18-07828-02 and S18-07828-03 are not independent.
[16] Trials S18-07828-04 and S18-07828-05 are not independent.

Wheat

In general, a minimum of 1.00 kg and 0.500 kg of treated sample were collected for green material and straw, respectively.

In general, the maximum storage duration for wheat green material and straw was 544 days which is supported by storage stability data. However, in straw samples from study P672186503 (amendment to studies 16-2053 and 16-2054), residues of isoflucypram-desmethyl-propanol and

isoflucypram-propanol were extracted for analysis after frozen storage for 782–859 days. Additionally, storage durations were not provided for isoflucypram-desmethyl-propanol and isoflucypram-propanol for study GLP655 (amendment to study S17-07939). The Meeting calculated a maximum duration of 483 days based on the harvest date and date of extraction for analysis.

For samples analysed according to Methods 01475 and 46437, version 1, there were no residues in control samples. Therefore, residues in treated samples were not corrected for residues in controls. For samples analysed according to Method 01564, residues of isoflucypram, isoflucypram-propanol, or isoflucypram-desmethyl-propanol were generally below the LOQ in control samples, with the following exceptions: Isoflucypram and isoflucypram-propanol were 0.053 mg/kg and 0.013 mg eq/kg in control wheat straw from Trial 18-2135-03 of 0.053 mg eq/kg (with corresponding field trial residues of 0.35–0.99 mg/kg and 0.12–0.23 mg eq/kg, respectively). Residues in treated samples were not corrected for residues in controls. The results are shown in Table 143.

Table 143 Residue for isoflucypram and metabolites (expressed as parent) in wheat green material and straw treated once with EC formulation at 50 or 42 g ai/L

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application rate (g ai/ha)	DALA[1]	Isoflucypram (mg/kg)	Isoflucypram, mg/kg dw	Isoflucypram-desmethyl-propanol, mg eq/kg	Isoflucypram-desmethyl-propanol, mg /kg dw	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, mg /kg dw
15-2115-01 Parcay Meslay, France[2,3] (Rubisco)	Green material	69	38	75	0	1.0	-	-	-	-	-
		71	41		7	0.61	-	-	-	-	-
		75	50		14	0.79	-	-	-	-	-
		77	47		21	0.46	-	-	-	-	-
		83	64		28	0.49	-	-	-	-	-
	Straw	89	-		45	0.84	-	-	-	-	-
15-2111-01 Parcay Meslay, France[2,3] (Rubisko)	Green material	69	51	75	0	1.6	-	-	-	-	-
		71	55		7	1.3	-	-	-	-	-
		75	59		14	0.92	-	-	-	-	-
		77	60		21	0.53	-	-	-	-	-
		83	66		28	0.57	-	-	-	-	-
	Straw	89	-		45	0.94	-	-	-	-	-
15-2115-02 Little Shelford, United Kingdom[2,4] (KWS Chasel Nabim Group 2)	Green material	69	55	75	0	0.97	-	-	-	-	-
		73	49		7	0.72	-	-	-	-	-
		75	54		14	0.48	-	-	-	-	-
		77	62		22	0.54	-	-	-	-	-
		85	70		29	0.49	-	-	-	-	-
	Straw	89	-		43	1.2	-	-	-	-	-
15-2111-02 Little Shelford, United Kingdom[2,4] (KWS Cashel Nabin Group 2)	Green material	69	55	75	0	1.8	-	-	-	-	-
		73	58		7	1.4	-	-	-	-	-
		75	55		14	0.62	-	-	-	-	-
		77	69		22	0.87	-	-	-	-	-
		85	77		29	0.73	-	-	-	-	-
	Straw	89	-		43	1.7	-	-	-	-	-
15-2115-03 Wieringerwerf, The Netherlands[2,5] (Quintes)	Green material	69	29	75	0	1.5	-	-	-	-	-
		71	36		7	0.74	-	-	-	-	-
		75	43		14	0.40	-	-	-	-	-
		75	43		21	0.30	-	-	-	-	-
		77	49		28	0.15	-	-	-	-	-
	Straw	89	-		68	0.12	-	-	-	-	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application rate (g ai/ha)	DALA[1]	Isoflucypram (mg/kg)	Isoflucypram, mg/kg dw	oflucypram-desmethyl-propano mg eq/kg	Isoflucypram-desmethyl-propanol, mg /kg dw	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, mg /kg dw
15-2111-03 Wieringerwerf, Netherlands[2,5] (Quintes)	Green material	69	37	75	0	2.0	-	-	-	-	-
		71	47		7	0.87	-	-	-	-	-
		75	54		14	0.37	-	-	-	-	-
		75	55		21	0.24	-	-	-	-	-
		77	57		28	0.12	-	-	-	-	-
	Straw	89	-		68	0.074	-	-	-	-	-
15-2115-04 Bursheid, Germany[2,6] (Chamsin)	Green material	69	35	75	0	2.1	-	-	-	-	-
		71	38		7	1.5	-	-	-	-	-
		75	43		14	1.1	-	-	-	-	-
		83	49		21	0.78	-	-	-	-	-
		85	56		28	0.61	-	-	-	-	-
	Straw	89	-		49	0.82	-	-	-	-	-
15-2111-04 Bursheid, Germany[2,6] (KWS Chasmin)	Green material	69	44	75	0	2.1	-	-	-	-	-
		71	50		7	1.4	-	-	-	-	-
		75	54		14	1.2	-	-	-	-	-
		83	49		21	0.52	-	-	-	-	-
		85	60		28	0.44	-	-	-	-	-
	Straw	89	-		49	0.43	-	-	-	-	-
15-2116-01 Santerem, Portugal[2,7] (Jordao)	Green material	69	37	75	0	1.1	-	-	-	-	-
		71	49		7	0.80	-	-	-	-	-
		73	46		14	0.66	-	-	-	-	-
		75	60		21	0.57	-	-	-	-	-
		83	64		28	0.83	-	-	-	-	-
	Straw	89	-		45	1.9	-	-	-	-	-
15-2069-01 Santarem, Portugal[2,7] (Jordao)	Green material	69	41	75	0	1.6	-	-	-	-	-
		71	49		7	0.90	-	-	-	-	-
		73	47		14	0.66	-	-	-	-	-
		75	51		21	0.63	-	-	-	-	-
		83	63		28	0.57	-	-	-	-	-
	Straw	89	-		45	1.6	-	-	-	-	-
15-2116-02 St. Etienne du Gres, France[2,8] (Aubusson)	Green material	69	35	75	0	1.6	-	-	-	-	-
		71	45		6	1.1	-	-	-	-	-
		75	41		14	0.68	-	-	-	-	-
		77	47		21	0.88	-	-	-	-	-
		83	55		28	0.84	-	-	-	-	-
	Straw	89	-		49	0.87	-	-	-	-	-
15-2119-01 Nimes, Languedoc Roussillon, France[2,8] (P22R58)	Green material	69	33	66	0	1.5	-	-	-	-	-
		71	42		8	0.97	-	-	-	-	-
		75	48		14	0.70	-	-	-	-	-
		83	53		21	0.56	-	-	-	-	-
		85	59		29	0.51	-	-	-	-	-
	Straw	89	-		65	1.4	-	-	-	-	-
15-2069-02 St. Etienne du Gres, France[2,8] (Aubusson)	Green material	69	39	75	0	1.7	-	-	-	-	-
		71	39		6	0.98	-	-	-	-	-
		75	43		14	0.92	-	-	-	-	-
		77	48		21	0.83	-	-	-	-	-
		83	60		28	0.85	-	-	-	-	-
	Straw	89	-		49	0.71	-	-	-	-	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application rate (g ai/ha)	DALA[1]	Isoflucypram (mg/kg)	Isoflucypram, mg/kg dw	Isoflucypram-desmethyl-propanol mg eq/kg	Isoflucypram-desmethyl-propanol, mg /kg dw	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, mg /kg dw
15-2116-03 Le Burguad, France[2,9] (Arezzo)	Green material	69	39	75	0	1.3	-	-	-	-	-
		73	45		7	0.48	-	-	-	-	-
		73	48		14	0.39	-	-	-	-	-
		77	57		21	0.38	-	-	-	-	-
		83	68		28	<u>0.20</u>	-	-	-	-	-
15-2069-03 Le Burguad, France[2,9] (Arezzo)	Green material	69	38	75	0	1.3	-	-	-	-	-
		71	45		7	0.50	-	-	-	-	-
		73	51		14	0.42	-	-	-	-	-
		77	61		21	0.30	-	-	-	-	-
		83	65		28	0.15	-	-	-	-	-
15-2116-04 Brenes, Spain[2,10] (Euroduro)	Green material	65	44	75	0	2.2	-	-	-	-	-
		73	44		7	1.6	-	-	-	-	-
		83	46		14	0.97	-	-	-	-	-
		85	54		21	0.74	-	-	-	-	-
		87	64		28	<u>0.78</u>	-	-	-	-	-
	Straw	89	-		51	<u>1.9</u>	-	-	-	-	-
15-2069-04 Brenes, Spain[2,10] (Vitron)	Green material	65	38	75	0	2.3	-	-	-	-	-
		77	41		7	1.9	-	-	-	-	-
		83	48		14	0.89	-	-	-	-	-
		85	50		21	0.66	-	-	-	-	-
		85	59		28	0.67	-	-	-	-	-
	Straw	89	-		53	1.2	-	-	-	-	-
15-2120-01 Stowbridge, PE34 3NR, United Kingdom[2] (Skyfall)	Green material	69	45	63	0	1.0	-	-	-	-	-
		71	45		6	0.88	-	-	-	-	-
		75	52		14	0.42	-	-	-	-	-
		83	50		20	0.34	-	-	-	-	-
		83	52		28	<u>0.20</u>	-	-	-	-	-
	Straw	89	-		49	<u>0.38</u>	-	-	-	-	-
15-2120-02 Vasszecseny, Hungary[2] (GK Szala)	Green material	69	38	63	0	1.3	-	-	-	-	-
		75	47		7	0.95	-	-	-	-	-
		83	59		14	1.2	-	-	-	-	-
		85	62		21	1.3	-	-	-	-	-
		87	78		28	<u>0.70</u>	-	-	-	-	-
	Straw	89	-		34	3.6	-	-	-	-	-
15-2120-03 La Chapelle de Guinchay, France[2] (Togano)	Green material	69	50	63	0	2.0	-	-	-	-	-
		75	58		7	1.4	-	-	-	-	-
		81	67		14	1.5	-	-	-	-	-
		87	89		21	2.5	-	-	-	-	-
	Straw	89	-		24	3.3	-	-	-	-	-
15-2120-04 Piekary, Poland[2] (Tybalt)	Green material	69	41	63	0	1.4	-	-	-	-	-
		73	47		7	0.91	-	-	-	-	-
		77	43		14	0.58	-	-	-	-	-
		83	58		21	0.66	-	-	-	-	-
		85	72		28	<u>0.84</u>	-	-	-	-	-
	Straw	89	-		36	1.5	-	-	-	-	-
15-2119-02 Mahora, Albacete, Spain[2] (Sarina)	Green material	69	33	63	0	1.1	-	-	-	-	-
		71	35		7	0.28	-	-	-	-	-
		75	44		14	0.17	-	-	-	-	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application rate (g ai/ha)	DALA[1]	Isoflucypram (mg/kg)	Isoflucypram, mg/kg dw	oflucypram-desmethyl-propanol mg eq/kg	Isoflucypram-desmethyl-propanol, mg /kg dw	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, mg /kg dw
		77	50		21	0.16	-	-	-	-	-
		83	58		28	0.19	-	-	-	-	-
	Straw	89	-		40	0.33	-	-	-	-	-
15-2119-03 Cartaxo, Ribatejo, Portugal[2] (Valbona)	Green material	69	51	63	0	0.76	-	-	-	-	-
		71	57		7	0.59	-	-	-	-	-
		75	72		13	0.74	-	-	-	-	-
		77	91		21	1.2	-	-	-	-	-
		85	91		27	1.5	-	-	-	-	-
	Straw	89	-		41	2.3	-	-	-	-	-
15-2119-04 Castallaneta, TA, Italy[2] (Duilio)	Green material	69	49	63	0	2.5	-	-	-	-	-
		73	48		7	0.79	-	-	-	-	-
		75	59		14	1.1	-	-	-	-	-
		77	64		20	0.84	-	-	-	-	-
		83	73		28	1.1	-	-	-	-	-
	Straw	89	-		38	1.8	-	-	-	-	-
PNZ16414-01 Lake Road South, Irwell, New Zealand (B12)	Green material	69	2	70	0	0.92	3.2	-	-	-	-
		77	40		35	0.01, <0.01 (<0.01)	0.01, <0.01 (<0.01)	-	-	-	-
		83-85	48		42	0.01, <0.01 (<0.01)	0.01, <0.01 (<0.01)	-	-	-	-
		87-89	69		49	0.01, <0.01 (<0.01)	0.01, <0.01 (<0.01)	-	-	-	-
	Straw	89	84		56	0.01, <0.01 (<0.01)	0.01, <0.01 (<0.01)	-	-	-	-
		89	77		63	0.32, 0.47 (0.40)	0.42, 0.61 (0.52)	-	-	-	-
PNZ16414-02 (Rakaia, New Zealand) (Starfire)	Green material	69	26	72	0	0.69	2.7	-	-	-	-
		83	40		42	.062, 0.06, 0.061 (0.062)	158, 0.163, 0.154 (0.158)	-	-	-	-
	Straw	89	59		63	.117, 0.133 (0.125)	0.20, 0.22 (0.21)	-	-	-	-
BAYERNZ/GLP/16/04-01 Hawkes Bay, New Zealand[2] (Starfire)	Green material	69/69	21	75	0	0.60	2.9	-	-	-	-
		75	29		35	058, 0.052 0.058 (0.056)	.20, 0.181, 0.20 (0.19)	-	-	-	-
		75	43		44	0.140	0.33	-	-	-	-
		85	43		49	.052, 0.079 (0.067)	120, 0.182 (0.15)	-	-	-	-
	Straw	87	39		56	0.096	0.25	-	-	-	-
		89	45		59	0.107	0.24	-	-	-	-
BAYERNZ/GLP/16/04-02 Manawatu, New Zealand[2] (Rappels)	Green material	69	22	75	0	1.34	6.0	-	-	-	-
		77	40		42	106, 0.096 0.061 (0.088)	0.26, 0.24, 0.151 (0.22)	-	-	-	-
	Straw	89	42		56	.160, 0.131 (0.15)	0.38, 0.31 (0.35)	-	-	-	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application rate (g ai/ha)	DALA[1]	Isoflucypram (mg/kg)	Isoflucypram, mg/kg dw	Isoflucypram-desmethyl-propanol mg eq/kg	Isoflucypram-desmethyl-propanol, mg /kg dw	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, mg /kg dw
18-2014-01 T1 St Aunix Lengros, France (Oregrain)	Green material	39	-	76	-0	<0.01	-	<0.01	-	<0.01	-
		39	-		0	1.0	-	<0.01	-	<0.01	-
	Straw	89	-		51	0.11	-	0.075	-	0.089	-
18-2014-01 T2 St Aunix Lengros, France (Oregrain)	Green material	49	-	78	-0	<0.01	-	<0.01	-	<0.01	-
		49	-		0	0.53	-	<0.01	-	<0.01	-
	Straw	89	-		43	0.10	-	0.20	-	0.10	-
18-2014-01 T3 St Aunix Lengros, France (Oregrain)	Green material	59	-	78	-0	<0.01	-	<0.01	-	<0.01	-
		59	-		0	0.51	-	<0.01	-	<0.01	-
	Straw	89	-		35	0.064	-	0.13	-	0.072	-
18-2014-01 T4 St Aunix Lengros, France (Oregrain)	Green material	69/69	-	77	-0	<0.01	-	<0.01	-	<0.01	-
		69	-		0	0.57	-	<0.01	-	<0.01	-
	Straw	89	-		29	0.092	-	0.091	-	0.096	-
18-2014-02 T1 Settala, Italy (Illico)	Green material	39	-	77	-0	<0.01	-	<0.01	-	<0.01	-
		39	-		0	1.8	-	<0.01	-	<0.01	-
	Straw	89	-		55	<0.01	-	<0.01	-	<0.01	-
18-2014-02 T2 Settala, Italy (Illico)	Green material	49/49	-	76	-0	<0.01	-	<0.01	-	<0.01	-
		49	-		0	2.2	-	<0.01	-	<0.01	-
	Straw	89	-		49	<0.01	-	<0.01	-	<0.01	-
18-2014-02 T3 Settala, Italy (Illico)	Green material	59	-	75	-0	<0.01	-	<0.01	-	<0.01	-
		59	-		0	1.8	-	<0.01	-	<0.01	-
	Straw	89	-		47	<0.01	-	<0.01	-	<0.01	-
18-2014-02 T4 Settala, Italy (Illico)	Green material	69	-	76	-0	<0.01	-	<0.01	-	<0.01	-
		69	-		0	1.7	-	<0.01	-	<0.01	-
	Straw	89	-		42	<0.01	-	<0.01	-	<0.01	-
18-2014-03 T1 Zafarraya, Spain (Marius)	Green material	39	-	77	-0	<0.01	-	<0.01	-	<0.01	-
		39	-		0	0.72	-	<0.01	-	<0.01	-
	Straw	89	-		64	0.10	-	0.023	-	0.020	-
18-2014-03 T2 Zafarraya, Spain (Marius)	Green material	49	-	79	-0	<0.01	-	<0.01	-	<0.01	-
		49	-		0	0.83	-	<0.01	-	<0.01	-
	Straw	89	-		55	0.39	-	0.054	-	0.048	-
18-2014-03 T3 Zafarraya, Spain (Marius)	Green material	59	-	80	-0	<0.01	-	<0.01	-	<0.01	-
		59	-		0	0.50	-	<0.01	-	<0.01	-
	Straw	89	-		50	0.38	-	0.043	-	0.056	-
18-2014-03 T4 Zafarraya, Spain (Marius)	Green material	69	-	73	-0	<0.01	-	<0.01	-	<0.01	-
		69	-		0	0.62	-	<0.01	-	<0.01	-
	Straw	89	-		41	0.14	-	0.012	-	0.016	-
18-2014-04 T1 Drymos, Greece (Cannavaro)	Green material	39	-	74	-0	<0.01	-	<0.01	-	<0.01	-
		39	-		0	1.9	-	<0.01	-	<0.01	-
	Straw	89	-		55	0.97	-	0.082	-	0.26	-
18-2014-04 T2 Drymos, Greece (Cannavaro)	Green material	49	-	75	-0	<0.01	-	<0.01	-	<0.01	-
		49	-		0	2.0	-	<0.01	-	<0.01	-
	Straw	89	-		53	1.3	-	0.08	-	0.38	-
18-2014-04 T3 Drymos, Greece (Cannavaro)	Green material	59	-	76	-0	<0.01	-	<0.01	-	<0.01	-
		59	-		0	1.7	-	<0.01	-	<0.01	-
	Straw	89	-		48	1.4	-	0.15	-	0.41	-
18-2014-04 T4	Green	69	-	76	-0	<0.01	-	<0.01	-	<0.01	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application rate (g ai/ha)	DALA[1]	Isoflucypram (mg/kg)	Isoflucypram, mg/kg dw	oflucypram-desmethyl-propanol mg eq/kg	Isoflucypram-desmethyl-propanol, mg /kg dw	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, mg /kg dw
Drymos, Greece	material	69	-		0	0.96	-	<0.01	-	<0.01	-
(Cannavaro)	Straw	89	-		42	1.6	-	0.10	-	0.31	-
18-2135-01 T1	Green	39	-	75	-0	<0.01	-	<0.01	-	<0.01	-
Burscheid, Germany	material	39	-		0	0.85	-	<0.01	-	<0.01	-
(Elixer)	Straw	89	-		78	0.090	-	0.062	-	0.11	-
18-2135-01 T2	Green	45	-	78	-0	<0.01	-	<0.01	-	<0.01	-
Burscheid, Germany	material	45	-		0	0.85	-	<0.01	-	<0.01	-
(Elixer)	Straw	89	-		69	0.087	-	0.10	-	0.10	-
18-2135-01 T3	Green	58	-	75	-0	<0.01	-	<0.01	-	<0.01	-
Burscheid, Germany	material	58	-		0	1.0	-	<0.01	-	<0.01	-
(Elixer)	Straw	89	-		62	0.20	-	0.15	-	0.20	-
18-2135-01 T4	Green	69	-	76	-0	<0.01	-	<0.01	-	<0.01	-
Burscheid, Germany	material	69	-		0	0.47	-	<0.01	-	<0.01	-
(Elixer)	Straw	89	-		44	0.28	-	0.095	-	0.12	-
18-2135-02 T1	Green	39	-	84	-0	<0.01	-	<0.01	-	<0.01	-
Mellet, Belgium	material	39	-		0	0.93	-	<0.01	-	<0.01	-
(Mistral)	Straw	89	-		74	0.044	-	0.038	-	0.072	-
18-2135-02 T2	Green	49	-	74	-0	<0.01	-	<0.01	-	<0.01	-
Mellet, Belgium	material	49	-		0	1.2	-	<0.01	-	<0.01	-
(Mistral)	Straw	89	-		71	0.047	-	0.047	-	0.091	-
18-2135-02 T3	Green	59	-	74	-0	<0.01	-	<0.01	-	<0.01	-
Mellet, Belgium	material	59	-		0	0.52	-	<0.01	-	<0.01	-
(Mistral)	Straw	89	-		66	0.044	-	0.050	-	0.047	-
18-2135-02 T4	Green	69	-	77	-0	<0.01	-	<0.01	-	<0.01	-
Mellet, Belgium	material	69	-		0	0.50	-	<0.01	-	<0.01	-
(Mistral)	Straw	89	-		57	0.15	-	0.068	-	0.085	-
18-2135-03 T1	Green	41	-	79	-0	<0.01	-	<0.01	-	<0.01	-
Great Chishill, United	material	41	-		0	0.45	-	<0.01	-	<0.01	-
Kingdom[12]	Straw	89	-		76	0.35	-	0.12	-	0.21	-
(KWS Trinity)											
18-2135-03 T2	Green	49	-	75	-0	<0.01	-	<0.01	-	<0.01	-
Great Chishill, United	material	49	-		0	0.35	-	<0.01	-	<0.01	-
Kingdom[12]	Straw	89	-		72	0.35	-	0.14	-	0.12	-
(KWS Trinity)											
18-2135-03 T3	Green	59	-	76	-0	<0.01	-	<0.01	-	<0.01	-
Great Chishill, United	material	59	-		0	0.45	-	<0.01	-	<0.01	-
Kingdom[12]	Straw	89	-		65	0.55	-	0.13	-	0.17	-
(KWS Trinity)											
18-2135-03 T4	Green	69	-	74	-0	<0.01	-	<0.01	-	<0.01	-
Great Chishill, United	material	69	-		0	0.71	-	<0.01	-	<0.01	-
Kingdom[12]	Straw	89	-		53	0.99	-	0.11	-	0.23	-
(KWS Trinity)											
18-2135-04 T1	Green	39	-	75	-0	1.7[2]	-	<0.01	-	<0.01	-
Zwaagdijk, Netherlands	material	39	-		0	<0.01	-	<0.01	-	<0.01	-
(Nobless)	Straw	89	-		58	0.15	-	0.12	-	0.14	-
18-2135-04 T2	Green	49	-	77	-0	0.011	-	<0.01	-	<0.01	-
Zwaagdijk, Netherlands	material	49	-		0	1.9	-	<0.01	-	<0.01	-
(Nobless)	Straw	89	-		54	0.17	-	0.11	-	0.16	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application rate (g ai/ha)	DALA[1]	Isoflucypram (mg/kg)	Isoflucypram, mg/kg dw	Isoflucypram-desmethyl-propanol mg eq/kg	Isoflucypram-desmethyl-propanol, mg /kg dw	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, mg /kg dw
18-2135-04 T3 Zwaagdijk, Netherlands (Nobless)	Green material	59	-	76	-0	<0.01	-	<0.01	-	<0.01	-
		59	-		0	0.81	-	<0.01	-	<0.01	-
	Straw	89	-		49	0.64	-	0.20	-	0.37	-
18-2135-04 T4 Zwaagdijk, Netherlands (Nobless)	Green material	69	-	75	-0	<0.01	-	<0.01	-	<0.01	-
		69	-		0	1.4	-	<0.01	-	<0.01	-
	Straw	89	-		35	1.6	-	0.087	-	0.16	-
17-2020-01 St Etienne du Gres, Southern France[2] (Arkeos)	Green material	69	-	63	0	0.99	-	<0.01	-	<0.01	-
		73	-		7	0.74	-	0.040	-	0.071	-
		75	-		14	0.53	-	0.060	-	0.097	-
		83	-		21	0.42	-	0.090	-	0.12	-
		87	-		28	0.49	-	0.090	-	0.15	
	Straw	89	-		44	1.1	-	0.060	-	0.23	-
17-2020-02 Paradas, Spain[2] (Amilcar)	Green material	69	-	63	0	2.1	-	<0.01	-	<0.01	-
		73	-		7	1.8	-	0.031	-	0.077	-
		77	-		14	2.0	-	0.072	-	0.15	-
		83	-		21	1.3	-	0.061	-	0.11	-
		87	-		28	1.8	-	0.057	-	0.13	-
	Straw	89	-		57	3.2	-	0.11	-	0.27	-
17-2020-03 Thymaria Kozanis, Greece[2] (Zanzibar)	Green material	69	-	63	0	1.9	-	<0.01	-	<0.01	-
		75	-		7	0.29	-	0.019	-	0.022	-
		83	-		14	0.16	-	0.035	-	0.029	-
		85	-		21	0.12	-	0.048	-	0.039	-
		87	-		28	0.11	-	0.055	-	0.057	-
	Straw	89	-		41	0.34	-	0.023	-	0.070	-
17-2020-04 Mineo, Italy[2] (Alemanno)	Green material	69	-	63	0	1.3	-	<0.01	-	<0.01	-
		69	-		7	0.95	-	<0.01	-	0.024	-
		71	-		14	0.98	-	0.022	-	0.059	-
		72	-		21	0.71	-	0.036	-	0.084	-
		75	-		28	0.62	-	0.040	-	0.092	-
	Straw	89	-		51	1.4	-	0.044	-	0.13	-
17-2019-01 Burscheid, Germany[2] (Potential)	Green material	69	-	63	0	1.2	-	<0.01	-	<0.01	-
		71	-		7	0.63	-	0.027	-	0.11	-
		75	-		14	0.33	-	0.051	-	0.15	-
		77	-		21	0.28	-	0.060	-	0.16	-
		83	-		28	0.24	-	0.066	-	0.15	-
	Straw	89	-		66	0.48	-	0.038	-	0.28	-
17-2019-02 Chambourg Sur Indre, Northern France[2] (Venezio)	Green material	69	-	63	0	1.2	-	<0.01	-	<0.01	-
		71	-		7	0.96	-	0.040	-	0.15	-
		75	-		14	0.67	-	0.053	-	0.18	-
		77	-		21	0.57	-	0.068	-	0.19	-
		83	-		28	0.63	-	0.080	-	0.31	-
	Straw	89	-		42	1.4	-	0.078	-	0.56	-
17-2019-03 Anrochte-Berge, Germany[2] (Cernetto)	Green material	69	-	63	0	1.8	-	<0.01	-	<0.01	-
		71	-		6	0.62	-	0.020	-	0.052	-
		73	-		14	0.33	-	0.037	-	0.085	-
		75	-		21	0.26	-	0.034	-	0.080	-
		83	-		27	0.17	-	0.038	-	0.10	-
	Straw	89	-		43	0.53	-	0.049	-	0.23	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application rate (g ai/ha)	DALA[1]	Isoflucypram (mg/kg)	Isoflucypram, mg/kg dw	Isoflucypram-desmethyl-propanol, mg eq/kg	Isoflucypram-desmethyl-propanol, mg /kg dw	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, mg /kg dw
17-2019-04 Zwaagdijk, Netherlands[2] (Tybalt)	Green material	69	-	63	0	1.3	-	<0.01	-	<0.01	-
		71	-		7	0.21	-	0.013	-	0.037	-
		75	-		14	0.14	-	0.018	-	0.051	-
		85	-		21	0.10	-	0.017	-	0.049	-
		87	-		28	0.073	-	0.019	-	0.056	-
	Straw	89	-		39	0.069	-	0.013	-	0.048	-
16-2053-01 Chemery, Northern France[2] (Sy moisson)	Green material	69	38	75	0	1.7	-	-	-	-	-
		71	40		7	0.55	-	-	-	-	-
		71	35		13	0.23	-	-	-	-	-
		75	50		20	0.21	-	-	-	-	-
		77	53		28	0.14	-	-	-	-	-
	Straw	89	-		52	0.19	-	0.060	-	0.38	-
16-2053-02 Mellery, Belgium[2] (Rubisco)	Green material	65	35	75	0	1.2	-	-	-	-	-
		71	35		7	0.38	-	-	-	-	-
		75	39		14	0.16	-	-	-	-	-
		77	45		21	0.12	-	-	-	-	-
		83	46		28	0.087	-	-	-	-	-
	Straw	89	-		60	0.054	-	0.015	-	0.051	-
16-2053-03 TR Pesse, Netherlands[2] (Tybalt)	Green material	69	30	75	0	1.7	-	-	-	-	-
		69	38		7	0.73	-	-	-	-	-
		73	39		14	0.48	-	-	-	-	-
		83	44		21	0.32	-	-	-	-	-
		85	54		28	0.27	-	-	-	-	-
	Straw	89	-		57	0.40	-	0.013	-	0.11	-
16-2053-04 Leichlingen, Germany[2] (Tybalt)	Green material	69	31	75	0	1.8	-	-	-	-	-
		73	36		7	0.31	-	-	-	-	-
		75	38		14	0.13	-	-	-	-	-
		77	44		21	0.081	-	-	-	-	-
		83	47		28	0.065	-	-	-	-	-
	Straw	89	-		64	0.071	-	0.025	-	0.11	-
16-2054-01 C. da Terrebianche Misterbianco CT, Italy[2] (Anco Marzio)	Green material	69	35	75	0	1.2	-	-	-	-	-
		71	41		7	1.1	-	-	-	-	-
		83	49		14	0.88	-	-	-	-	-
		85	53		21	0.46	-	-	-	-	-
		87	63		28	0.70	-	-	-	-	-
	Straw	89	-		49	1.6	-	0.052	-	0.21	-
16-2054-02 Brenes, Spain[2] (Artur Nick)	Green material	69	32	75	0	1.7	-	-	-	-	-
		71	33		7	1.2	-	-	-	-	-
		75	47		14	0.89	-	-	-	-	-
		75	37		21	0.22	-	-	-	-	-
		85	51		28	0.21	-	-	-	-	-
	Straw	89	-		49	1.3	-	0.24	-	0.58	-
16-2054-03 Bonnieux, Southern France[2] (Calabro)	Green material	65	40	75	0	1.7	-	-	-	-	-
		71	45		6	1.3	-	-	-	-	-
		75	51		14	1.3	-	-	-	-	-
		77	55		21	0.78	-	-	-	-	-
		83	68		28	0.91	-	-	-	-	-
	Straw	89	-		45	2.4	-	0.084	-	0.29	-

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application rate (g ai/ha)	DALA[1]	Isoflucypram (mg/kg)	Isoflucypram, mg/kg dw	oflucypram-desmethyl-propanol mg eq/kg	Isoflucypram-desmethyl-propanol, mg /kg dw	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, mg /kg dw
16-2054-04 Ceaux en Loudun, Southern France[2] (Orgrain)	Green material	69	30	75	0	1.1	-	-	-	-	-
		71	35		6	0.51	-	-	-	-	-
		71	32		14	0.27	-	-	-	-	-
		75	44		21	0.23	-	-	-	-	-
		77	42		28	0.14	-	-	-	-	-
	Straw	89	-		52	0.22	-	0.040	-	0.17	-
16-2054-05 Kissa Village, Kozano, Greece[2] (Achilleas)	Green material	65	37	75	0	1.6	-	-	-	-	-
		71	38		7	0.45	-	-	-	-	-
		73	45		14	0.46	-	-	-	-	-
		77	46		21	0.18	-	-	-	-	-
		83	55		28	0.20	-	-	-	-	-
	Straw	89	-		53	0.41	-	0.034	-	0.12	-
S17-07939-01/GLP655-01 Timaru, New Zealand (Raffles)	Green material	69/69	28	116[13]	0	3.2	11.5	<0.01	<0.01	<0.01	<0.01
		77	51		34	0.90, 0.72, 0.73 (0.78)	1.74, 1.41, 1.43 (1.53)	0.19	0.37	0.53	1.04
		77	80		42	1.18, 1.10 (1.14)	1.48, 1.38 (1.43)	0.20	0.26	0.71	0.89
		77	84		48	1.14, 1.20 (1.17)	1.36, 1.43 (1.40)	0.127	0.152	0.51	0.61
	Straw	87	80		54	1.69, 2.1 (1.90)	2.1, 2.6 (2.35)	0.078	0.098	0.40	0.50
S17-07939-02/GLP655-02 Laureston, New Zealand (Raffles)	Green material	69	26	116[13]	0	3.1	11.8	<0.01	<0.01	<0.01	<0.01
		77	54		42	0.105	0.193	0.163	0.30	0.49	0.91
	Straw	89	41		54	0.067	0.165	0.128	0.31	0.42	1.03
S17-07939-03/GLP655-03 Opiki, New Zealand[2,14] (Sensas)	Green material	69	33	75	0	2.9	9.0	<0.01	<0.01	<0.01	<0.01
		85	67		35	0.45, 0.37 (0.41)	0.67, 0.56 (0.62)	0.25	0.38	0.61	0.92
		87-89	79		42	0.42	0.53	0.199	0.25	0.54	0.69
		89-92	77		49	0.25	0.32	0.122	0.159	0.35	0.46
	Straw	89-92	69		49	0.94	1.37	0.161	0.23	0.53	0.77
S17-07939-04/GLP655-04 Fielding, New Zealand[2,14] (Sensas)	Green material	69	37	75	0	1.70, 1.56 (1.63)	4.6, 4.2 (4.4)	<0.01 <0.01 (<0.01)	<0.01, <0.01 (<0.01	<0.01, <0.0 (0.01)	<0.01, <0.01 (<0.01)
		87--89	80		42	0.27	0.34	0.111	0.140	0.22	0.27
	Straw	89-92	75		47	0.67, 0.70 (0.69)	0.90, 0.94 (0.92)	0.102, 0.10 (0.102)	0.137, 0.135 (0.136)	0.25, 0.23 (0.24)	0.34, 0.30 (0.320
S18-07829-01 (Kairanga, New Zealand)[15] (Discovery)	Green material	69	24	75	0	2.0	8.4	<0.01	<0.01	<0.01	<0.01
		85-87	52		35	0.56	1.08	0.086	0.164	0.175	0.33
		85-87	65		41	0.4	0.7	0.106	0.164	0.159	0.24
		87-89	71		49	0.39	0.56	0.063	0.089	0.169	0.24
	Straw	89	73		54	0.25	0.35	0.026	0.035	0.110	0.151
S18-07829-02	Green	69	27	75	0	2.1	7.7	<0.01	<0.01	<0.01	<0.01

Trial No. Location (variety)	Sample	BBCH at harvest	Dry Matter (%)	Application rate (g ai/ha)	DALA[1]	Isoflucypram (mg/kg)	Isoflucypram, mg/kg dw	oflucypram-desmethyl-propanc mg eq/kg	Isoflucypram-desmethyl-propanol, mg /kg dw	Isoflucypram-propanol, mg/kg	Isoflucypram-propanol, mg /kg dw
Cheltenham, New Zealand[15] (Sensas)	material	89	84		42	0.77	0.92	0.138	0.165	0.25	0.30
	Straw	89	79		45	1.05	1.33	0.088	0.112	0.20	0.26
S18-07829-03 Opiki, New Zealand[2] (Sensas)	Green material	69	24	75	0	1.64	7.0	<0.01	<0.01	0.015	0.065
		77	42		44	0.121	0.29	0.064	0.154	0.108	0.26
		87	55		55	0.093	0.168	0.030	0.054	0.065	0.118
	Straw	89	53		58	0.142	0.27	0.058	0.109	0.137	0.26
S18-07829-04 Ruapuna, New Zealand[2] (Raffles)	Green material	69	21	75	0	2.1	10.3	<0.01	<0.01	<0.01	<0.01
		77	51		40	0.085	0.167	0.026	0.052	0.043	0.085
	Straw	93	86		54	0.123	0.143	0.076	0.089	0.088	0.103
S18-07829-05 Mitcham, New Zealand[2] (Raffles)	Green material	69	24	75	0	2.0, 2.0 (2.00)	8.3, 8.5 (8.4)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01	<0.01, <0.01 (<0.01
		77-80	47		36	.071, 0.085 (0.078)	151, 0.181 (0.166)	0.024, 0.026 (0.025)	.051, 0.056 (0.054)	0.032, 0.03 (0.034)	0.068, 0.075 (0.072)
		77-80	43		43	0.077	0.2	0.027	0.063	0.039	0.093
		87	76		51	.080, 0.070 (0.075)	106, 0.093 (0.10)	0.028, 0.029 (0.029)	.037, 0.038 (0.038)	0.047, 0.04 (0.048)	0.063, 0.065 (0.064)
	Straw	93	85		57	0.117	0.137	0.082	0.096	0.095	0.112

Notes:
dw=dry weight.
[1] DALA = Days after last application.
[2] Application rates are reported as the nominal values if the measured rates were ±5 percent of the planned rates.
[3] Trials 15-2115-01 and 15-2111-01 are not independent.
[4] Trials 15-2115-02 and 15-2111-02 are not independent.
[5] Trials 15-2115-03 and 15-111-03 are not independent.
[6] Trials 15-2115-04 and 15-2111-04 are not independent.
[7] Trials 15-2116-01 and 15-2069-01 are not independent.
[8] Trials 15-2116-02, 15-2119-01, and 15-2069-02 are not independent.
[9] Trials 15-2116-03 and 15-2069-03 are not independent.
[10] Trials 15-2116-04 and 15-2069-04 are not independent.
[12] Residues in corresponding control samples. Residues are not corrected for residues in controls.
[13] Trials S17-07939-01/GLP655-01 and S17-07939-02/GLP655-02 were accidentally overdosed.
[14] Trials S17-07939-03/GLP655-03 and S17-07939-04/GLP655-04 are not independent.
[15] Trials S18-07829-01 and S18-07829-02 are not independent.

FATE OF RESIDUES DURING STORAGE AND PROCESSING

Nature of the Residue Upon Processing

The Meeting received studies investigating radiolabelled isoflucypram, isoflucypram-desmethyl-propanol, and isoflucypram-propanol following temperatures and pH conditions simulating typical processing procedures.

Report No. EnSa-16-0135.

The Meeting received a study investigating the degradation of isoflucypram in water with a citrate-based buffer (Heinemann *et al.,* 2017). The radiolabelled test compounds [pyrazole-4-^{14}C] isoflucypram and [phenyl-UL-^{14}C] isoflucypram were used for the hydrolysis investigations.

The experiments were performed with a target concentration of 1 mg/L for the test compound. However, pre-tests showed that the target concentration could not be achieved when the ACN of the stock solution was evaporated before addition of the buffer solution. Therefore, both pyrazole- and phenyl-labelled test compounds were prepared as stock solution dissolved in ACN and aliquots containing the respective amount of test compound for the target concentration were added to a respective amount of buffer solution assuring for final test solutions with ACN content was <1 percent (actual ACN concentration was ≤ 0.9 percent).

Each test was carried out with sterilised, buffered drinking water at three different pH levels and three different temperatures: pH 4/90 °C, pH 5/100 °C and pH 6/120 °C (pH ± 0.1 and temperature ± 5 ˚C) to measure pasteurization, baking/brewing/boiling, and sterilization, respectively. The treatment duration was 20 min, 60 min, and 20 min for the three scenarios, respectively.

The pH value of each buffer and processed sample was measured after every significant step. All pH measurements were conducted at room temperature. An additional control vessel containing buffer solution alone was also prepared for each of the three pH/temperature combinations. The untreated control was used to monitor the temperature.

Before starting the incubation procedures, the pH value of all test solutions was measured and three aliquots of each test solution were subjected to LSC measurement to determine radioactivity. A further aliquot was taken for purity and stability analysis of the test compound by HPLC at test time before hydrolysis.

The tests at 90 °C and 100 °C were carried out using a heating/stirring module. The tests at 120 °C were performed in an autoclave. The temperature was recorded in a separate vial filled with 7 mL buffer. The vials were weighed before and after hydrolysis to correct for possible losses by evaporation of water.

The radioactivity measurement in liquid samples was determined by LSC. Recoveries for HPLC sample preparations determined for all test solutions ranged from 99.0 percent to 103.2 percent confirming that no radioactivity was lost during HPLC sample preparation. Isoflucypram and hydrolysis products were determined by HPLC with radiometric- and UV-detection with MS analysis.

Parent compound was identified in both radiolabeled studies in the tests at 100 °C by LC-MS/MS analysis and by HPLC co-chromatography with the non-radiolabeled reference compound. The assignment and identification of parent compound in the other tests was achieved by comparison of HPLC metabolite profiles.

The material balances for all tests were in the range of 104.2 percent to 110.5 percent and demonstrated that no radioactivity, in form of volatile degradation products, dissipated from the test systems. The amount of ACN in the test solutions accounted for 0.7 percent and 0.9 percent for the pyrazole- and phenyl-labels, respectively.

HPLC profiling of samples before and after processing showed that isoflucypram was predominantly stable under the tested conditions representative for food processing. For all tests, almost complete recovery of the parent compound was observed (≥ 98.0 percent). Identification of parent

compound was accomplished by LC-MS and LC-MS/MS analyses as well as by HPLC co-chromatography with non-radiolabelled reference compound.

Identification rates were in the range of 98.0 to 99.1 percent comprised fully of parent. Degradation products detected under the tested conditions representatively for food processing were very minor (≤ 0.51 percent; ≤ 0.005 mg/L) and were not further investigated (Table 144).

Table 144 High temperature hydrolysis of [Pyrazole-4-^{14}C] and [Phenyl-UL-^{14}C] isoflucypram

	Processing Conditions					
	pH 4/90°C/20 minutes		pH 5/100°C/60 minutes		pH 6/120°C/20 minutes	
Compound	% TRR	Concen-tration (mg/L)	% TRR	Concen-tration (mg/L)	% TRR	Concen-tration (mg/L)
[Pyrazole-4-^{14}C] isoflucypram						
Isoflucypram	99.10	0.962	98.66	0.959	99.05	0.971
Unknown	-	-	-	-	0.07	0.001
Unknown	-	-	0.12	0.001	0.17	0.002
Unknown	-	-	-	-	-	-
Unknown	0.10	0.001	0.24	0.002	0.20	0.002
Unknown	0.12	0.001	-	-	-	-
Unknown	0.05	0.001	-	-	-	-
Unknown	0.26	0.002	0.27	0.003	-	-
Unknown	-	-	0.24	0.002	-	-
Unknown	-	-	0.06	0.001	-	-
Unknown	0.12	0.001	0.08	0.001	-	-
Unknown	0.25	0.002	-	-	-	-
Unknown	-	-	0.34	0.003	0.51	0.005
Total identified	99.10	0.962	98.66	0.959	99.05	0.971
Total characterised	0.90	0.009	1.35	0.013	0.95	0.009
Accountability	100.0	0.970	100.0	0.972	100.0	0.981
[Phenyl-UL-^{14}C] Isoflucypram						
Isoflucypram	98.65	1.013	98.03	0.968	98.74	0.960
Unknown	-	-	0.05	0.001	-	-
Unknown	-	-	-	-	0.10	0.001
Unknown	-	-	0.14	0.001	0.15	0.001
Unknown	-	-	-	-	0.11	0.001
Unknown	-	-	0.08	0.001	-	-
Unknown	-	-	0.12	0.001	-	-
Unknown	0.07	0.001	0.12	0.001	-	-
Unknown	0.33	0.003	0.27	0.003	0.21	0.002
Unknown	0.22	0.002	0.31	0.003	-	-
Unknown	-	-	0.09	0.001	-	-
Unknown	0.28	0.003	0.32	0.003	0.19	0.002
Unknown	0.30	0.003	0.20	0.002	0.08	0.001
Unknown	0.15	0.002	-	-	-	-
Unknown	-	-	0.25	0.002	0.42	0.004
Total identified	98.65	1.013	98.03	0.968	98.74	0.960
Total characterised	1.35	0.014	1.95	0.019	1.26	0.012
Accountability	100.0	1.027	100.0	0.987	100.0	0.972

Report No. EnSa-20-0057.

The Meeting received a study investigating the hydrolytic transformation and degradation of isoflucypram-desmethyl-propanol in water in a citrate-based buffer (Lamshoeft *et al.,* 2020). The

radiolabelled test item [propane-1-^{14}C] isoflucypram-desmethyl-propanol was used for the hydrolysis investigations.

The experiments were performed with a target concentration of 1 mg/L of the test item. The test systems were incubated at three representative sets of hydrolysis conditions: 90 °C at pH 4 for 20 min (pasteurisation), 100°C at pH 5 for 60 min (baking/brewing/boiling), and 120 °C at pH 6 for 20 min (sterilisation).

An additional control vessel contacting buffer solution alone also underwent the simulated processing procedures. The untreated control was used to monitor the temperature.

Before starting the incubation procedures, the pH value of all test solution was measured (at room temperature) and three aliquots were subject to LSC to determine radioactivity. The radioactivity content was determined in the samples by LSC before and after undergoing the representative processing conditions.

The pasteurisation and baking/brewing/boiling experiments were carried out using a heating/stirring module. The sterilisation experiment was carried out with an autoclave. The vials were weighed before and after hydrolysis to correct for possible losses by evaporation of water.

Isoflucypram-desmethyl-propanol and hydrolysis products were quantitatively determined using HPLC with radiometric- and UV-detection. Isoflucypram-desmethyl-propanol was identified by LC-MS, LC-MS/MS, and NMR analysis as well as HPLC co-chromatography with non-radiolabelled isoflucypram-desmethyl-propanol. Isoflucypram-desmethyl-propanol-aldehyde was identified by LC-MS and LC-MS/MS analysis.

The stability of isoflucypram-desmethyl-propanol was investigated by HPLC analysis. The chromatogram after structure elucidation exhibited an additional peak which was not detected in the sample before structure elucidation. The additional substance could be identified as the formyl derivative of isoflucypram-desmethyl-propanol (by means of LC-MS/MS). The formation of the artefact can be explained by the addition of formic acid, an ingredient of the HPLC solvents.

The material balances for all tests were in the range of 96.7 percent to 99.7 percent and demonstrated that no radioactivity, in form of volatile degradation products, dissipated from the test systems. Under the tested conditions, the degradation of the test item increased with increasing pH values and temperature. Hydrolysis was very low following conditions representing pasteurisation, slight following conditions representing baking/brewing/boiling, and complete following conditions representing sterilisation.

Approximately 99 percent of the test item was recovered at the end of the pasteurisation experiment and approximately 34 percent following the baking/brewing/boiling experiment. Following the sterilisation experiment, isoflucypram-desmethyl-propanol was not detected. Isoflucypram-desmethyl-propanol-aldehyde was identified as main hydrolysis product following sterilisation conditions.

Identification of test item isoflucypram-desmethyl-propanol and isoflucypram-desmethyl-propanol-aldehyde was accomplished by LC-MS and LC-MS/MS analyses of isolated fractions in the baking/brewing/boiling experiment. Additionally, isoflucypram-desmethyl-propanol was identified by HPLC co-chromatography of the isolated fraction with the non-radiolabelled reference item.

Identification rates ranged between 98 percent and 100 percent with no unknown compound comprising ≥ 1.34 percent of the TRR (Table 145).

Table 145 High-Temperature hydrolysis of [Propane-1-^{14}C] isoflucypram-desmethyl-propanol

	Processing Conditions					
	pH 4/90 °C/20 Minutes		pH 5/100 °C/60 Minutes		pH 6/120 °C/20 Minutes	
Compound	% TRR	Concentration (mg/L)	% TRR	Concentration (mg/L)	% TRR	Concen-tration (mg/L)
Isoflucypram-desmethyl-propanol	98.99	0.952	34.05	0.330	-	-
Isoflucypram-desmethyl-propanol-aldehyde	0.88	0.008	66.15	0.631	97.68	0.938
Unknown	-	-	-	-	0.11	0.001
Unknown	-	-	-	-	0.13	0.001
Unknown	-	-	-	-	0.34	0.003
Unknown	0.14	0.001	-	-	-	-
Unknown	-	-	0.19	0.002	0.40	0.004
Unknown	-	-	0.61	0.006	1.34	0.013
Total identified	99.87	0.961	99.20	0.961	97.68	0.938
Total characterised	0.14	0.001	0.80	0.008	2.32	0.022
Accountability	100.01	0.962	100.00	0.969	100.00	0.960

Report No. EnSa-19-0734.

The Meeting received a study investigating the hydrolytic transformation and degradation of isoflucypram-propanol in drinking water with a citrate-based buffer (Lamshoeft *et al.,* 2020). The radiolabelled test item [pyrazole-4-^{14}C] isoflucypram-propanol was used for the hydrolysis investigations.

The experiments were performed with a target concentration of approximately 1 mg/L. The test systems were incubated at three representative sets of hydrolysis conditions: 90 °C at pH 4 for 20 min (pasteurisation), 100 °C at pH 5 for 60 min (baking/brewing/boiling), and 120 °C at pH 6 for 20 min (sterilisation).

The pH value of all test solutions was measured in three aliquots of each test solution and were subject to LSC measurements. The pasteurisation and baking/brewing/boiling experiments were carried out with a heating/stirring module and the sterilisation module was carried out with an autoclave. The temperature was recorded in a separate vial filled with 5 mL buffer. The vials were weighed before and after hydrolysis to measure losses due to evaporation.

The pH values of the samples were measured at room temperature. Three aliquots were taken from each test solution for the determination of the radioactivity content by LSC. A further aliquot of all individual test samples was taken for stability analysis of the test item by HPLC at the test time after hydrolysis.

For HPLC profiling of the test solutions at test termination, an aliquot of each test solution was analysed using HPLC. Recoveries from HPLC sample preparations ranged from 95.2 percent to 96.0 percent, confirming that no significant radioactivity was lost during HPLC sample preparation.

[Pyrazole-4-^{14}C] isoflucypram-propanol and possible hydrolysis products were quantitatively determined using HPLC with radiometric- and UV-detection. Identification was achieved by LC-MS and LC-MS/MS, and by HPLC co-chromatography with the non-radiolabelled reference compound. When performing the mass spectrometric analysis, the formic acid used in the HPLC caused the formation of an artifact, which was identified by LC-MS and LC-MS/MS. Reanalysis showed that this artifact is not a metabolite.

The material balances for all tests were in the range of 98.5 percent to 102.0 percent, demonstrating that little to no radioactivity, in form of volatile degradation products, dissipated from the test systems. HPLC profiling of samples before and after processing showed that isoflucypram-propanol was predominantly stable under the tested conditions representative for food processing. For all tests, almost complete recovery of parent compound was observed at approximately 98 percent. No unknown compound was found to comprise more than 0.43 percent TRR (Table 146).

Table 146 High-temperature hydrolysis of [Pyrazole-4-^{14}C] isoflucypram-propanol

	Processing Conditions					
	pH 4/90 °C/20 Minutes		pH 5/100 °C/60 Minutes		pH 6/120 °C/20 Minutes	
Compound	% TRR	Concentration (mg/L)	% TRR	Concentration (mg/L)	% TRR	Concentration (mg/L)
Isoflucypram-propanol	98.67	0.927	98.69	0.951	98.69	0.926
Unknown	0.40	0.004	0.30	0.003	0.34	0.003
Unknown	-	-	0.21	0.002	0.20	0.002
Unknown	-	-	0.12	0.001	-	-
Unknown	-	-	0.08	0.001	0.12	0.001
Unknown	0.39	0.004	0.26	0.002	0.22	0.002
Unknown	0.13	0.001	-	-	0.43	0.004
Unknown	0.21	0.002	0.22	0.002	-	-
Unknown	0.21	0.002	0.13	0.001	-	-
Total identified	98.67	0.927	98.69	0.951	98.69	0.926
Total characterised	1.34	0.013	1.32	0.013	1.31	0.012
Accountability	100.00	0.939	100.00	0.964	100.00	0.939

Residues upon processing

The Meeting received two studies investigating the behaviour of isoflucypram in processed wheat and barley commodities.

Barley

Report No. 15-3407

The Meeting received a study investigating residues of isoflucypram in barley grain and processed commodities (Freitag *et al.,* 2017). Two trials were submitted with residues evaluated in/on barley grain and the processed commodities for the processed commodities related to beer (malt sprouts, brewer's malt, brewer's grain, hops draff, brewer's yeast, and beer) and pearl barley (pearl barley rub off and pearl barley). Isoflucypram EC 050, an EC formulation containing 50 g/L, was applied to barley as a spray application at 0.375 kg ai/ha.

No irrigation was applied at either trial location. Barley was harvested 61 (Trial 01) and 58 (Trial 02) days after application. A minimum of 1.34 kg, 1.10 kg, 25.6 kg, and 5.26 kg of barley grain, stored barley grain (raw agricultural commodity [RAC]), barley harvested for processing into beer fractions, and barley harvested for processing into pearled barley, respectively.

Samples of barley stored grain (RAC) and all processed fractions were sent to the facility for cleansing (Gerichshain, Germany) under ambient conditions. At the cleansing facility, grain was separated from soil and contaminants.

Processing procedures simulated commercial practices. Material mass balances were provided in the study report. For processing into beer, samples first were sieved, steeped, allowed to germinate, and

kiln-dried. Brewer's malt and malt sprouts (germ following kiln-drying) were collected for analysis. Samples of brewer's malt was mashed (process of adding ground malt with water at 76 °C) and lautered (process of separating wort from insoluble malt components by filtration). The remaining solids (brewer's grain) were collected for analysis. The wort was boiled for approximately 90 minutes with hop pellets. The flocs (hops draff) were separated in a whirlpool causing the sludge to deposit on the bottom. The hops draff were collected for analysis. Yeast was added and the wort was allowed to ferment at 9 °C for approximately four weeks. During fermentation, the yeast deposits (i.e., brewer's yeast) on the tank bottom were sampled for analysis. The fermented beer was filtered and the mature beer was sampled for analysis.

For processing into pearl barley, the grain was conditioned to a moisture content of 14 percent. Samples were hulled until the stipulation abrasion for pearl barley (30–35 percent) was reached. Pearl barley and pearl barley rub off were sampled. Figure 13 summarises the processing procedures for beer and pearl barley processed commodities.

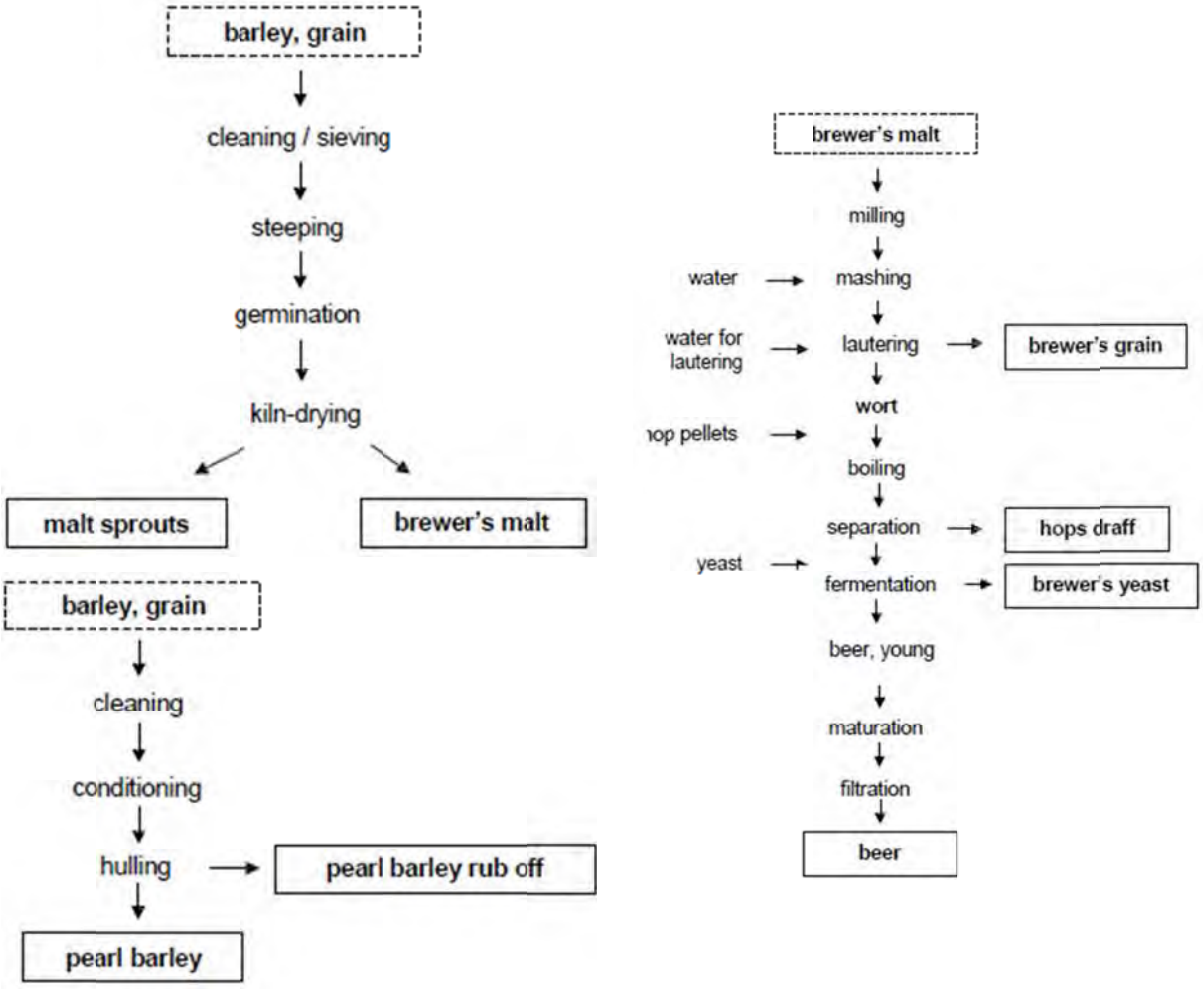


Figure 13 Barley processing procedure

Frozen samples were shipped to the laboratory for preparation (Monheim, Am Rhein) where they were homogenized by shredding with dry ice. Samples were then sent to the laboratory for analysis.

Samples of grain, stored grain (RAC), and processed fractions were maintained under ambient conditions for two days and 71 days for Trial 01 and Trial 02, respectively, prior to processing. After processing, samples were maintained frozen (<-18 °C) for a maximum period of 321 days (Table 147).

Table 147 Sample Weights and Duration of Frozen Stability for Barley RAC and Processed Commodities

Sample	Minimum sample weight (kg)	Maximum storage at ambient temperature (Days)	Maximum storage at -18 °C (Days)
Grain, stored (RAC)	1.07	105	321
Malt sprouts	0.54	239	161
Brewer's malt	0.61	239	161
Brewer's grain	1.10	251	150
Hops draff	0.32	251	160
Brewer's yeast	0.53	259	152
Beer	1.15	286	115
Pearl barley rub off	0.43	109	297
Pearl barley	1.06	109	297

Samples were analysed according to Method 01475. No residues were found in control samples. Therefore, the results were not corrected for concurrent recoveries. The results are shown in Table 148. Average processing factors were <0.67 for barley malt sprouts, brewer's malt, brewer's grain, hops draff, brewer's yeast, beer, and pearl barley. The average processing factor for pearl barley rub off is >1.9.

Table 148 Summary of Residue Data and Processing Factors for Isoflucypram on Barley Grain and Processed Commodities

Trial Number (Country)	Sample	DALA	Isoflucypram (mg/kg)	Processing Factor[1]
15-3407-01 (Netherlands)	Grain, stored (RAC)	61	0.018, 0.012 (0.015)	-
	Malt sprouts		<0.01	<0.67
	Brewer's malt		<0.01	<0.67
	Brewer's grain		<0.01	<0.67
	Hops draff		<0.01	<0.67
	Brewer's yeast		<0.01	<0.67
	Beer		<0.01	<0.67
	Grain, stored (RAC)		0.016, 0.013 (0.015)	-
	Pearl barley rub off		0.032	2.1
	Pearl barley		<0.01	<0.67
15-3407-02 (Spain)	Grain, stored (RAC)	58	<0.01, <0.01 (<0.01)	-
	Malt sprouts		<0.01	NC
	Brewer's malt		<0.01	NC
	Brewer's grain		<0.01	NC
	Hops draff		<0.01	NC
	Brewer's yeast		<0.01	NC
	Beer		<0.01	NC
	Grain, stored (RAC)		<0.01, <0.01 (<0.01)	-
	Pearl barley rub off		0.017	>1.7
	Pearl barley		<0.01	NC

Notes:

[1] Processing factor = [residue in processed commodity]/[residue in stored grain (RAC)]. Residues below the LOQ are calculated using the LOQ (0.01 mg/kg). Processing factors were not calculated (NC) when residues are below the LOQ in the RAC and processed commodity.

Wheat

Report No. RALNN137.

The Meeting received a study investigating isoflucypram residues in wheat processed commodities (Harbin, A.; 2017). Two trials were submitted evaluating residues in/on wheat and the processed commodities of bran, white flour, whole meal flour, germ, middlings, shorts, pasta (fresh), pasta (dry), pasta (cooked), pasta (dried and cooked), gluten, starch, aspirated grain fractions (AGF), cooking water, white bread, and whole meal bread. Isoflucypram EC 50, an EC formulation containing 50 g ai/L, was applied at a nominal rate of 0.375 g ai/ha. No adjuvants were added to the spray mixture.

A single composite sample of wheat grain was harvested from the control and treated plots. Samples were harvested at commercial maturity, corresponding to PHIs of 69 and 67 days for Trials C1101 and C1102, respectively. A minimum of 1.31 kg and 306 kg were collected for the RAC and for processed commodities, respectively. Samples for processing were sent under ambient conditions to the laboratory (max = 33 days). Samples of the RAC were frozen within 4 hours of harvest and stored up to 419 days before analysed. Storage duration of processed samples before analysis ranged from 83 to 109 days.

Samples were shipped to the laboratory for analysis at ≤-12 °C for homogenization and analysis. Samples of grain, bran, pasta (fresh, dry, and cooked) white bread, and whole meal bread were homogenized with dry ice. All other processed fractions were considered already homogenized. Samples were analysed according to Method LN-002-P16-01. No residues were found in control samples.

Processing procedure simulateing small-scale industrial procedures are descibed below and ilustrated in Figures 14 and 15.

Generation of Aspirated Grain Fraction.

Samples with a moisture content greater than 13.0 percent were dried in an oven until the moisture content reached 10.0–13.0 percent. Samples were placed in a dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. As the sample moved through the system, aspiration was used to remove light impurities (grain dust). Each sample was moved for 120 minutes. Material through the 2,360 micron sieve were removed and the ash content was determined

Wheat Milling-Cleaning

Following AGF generation, samples were cleaned by aspiration and screening. Light impurities were removed. After aspiration, samples were screened to separate large and small foreign particles (screenings) from the cleaned grain.

Germ Production

Cleaned wheat was moisture adjusted to 16 percent by adding reverse osmosis water and mixing. After tempering for 1–1.5 hours, the wheat was passed through a disc mill. Ground material was sifted with an 8, 14, and 30 mesh sieve. Material on top of the 30 mesh sieve was aspirated to remove bran from the germ fraction. Germ (with endosperm) was passed through the reduction side of the mill. Germ and reduced endosperm were separated with a sifter equipped with 18, 20, 24, and 28 mesh screens. Germ material was aspirated again to remove additional bran and milled/sieved to remove additional endosperm.

Flour Production

Cleaned wheat was moisture conditioned to 17.5 percent moisture content with a 24hour (+/- 30 minute) resting period before milling. Moisture-adjusted wheat was fed through the spout on a Chopin mill. Breaking of the wheat was accomplished by three break rolls. After passing through the break rolls, the material was fed onto the break sifter screen with sizes of 140 microns 800 microns. Material exiting the break rolls passed over the 800 micron screen first. Material passing through the 800 micron screen is "Break Flour." Material not passing through was conveyed over the 140 micron screen. Material passing through the 140 micron screen is middlings. Material not passing through was conveyed to the end of the sifter. Material exiting the end is bran.

Middlings were poured into the feed hopper of the reduction system. Reduction is achieved through two reduction rolls. After passing through the reduction rolls, the material was fed onto a number 160 micron reduction sifter screen. Material passing through the screen is reduction flour. Material not passing through and conveyed to the end of the sifter is shorts. Representative amounts of break and reduction flours were mixed to produce standard mill run (white flour). Remaining standard mill run flour was used in the production of white bread. Remaining break and reduction flour was used in the production of starch, gluten, and pasta.

Bran exiting the break sieve is placed in the reduction side of the mill but is not reduced with the rollers. The coarse bran is conveyed by beater bars over a number 128 micron screen. Material passing through the screen is "Shorts" and is added to "Shorts" from the reduction mill. Material passing over the screen and exiting the end is "Bran." Requested bran and shorts fractions were collected and placed into frozen storage.

Processing into white bread

Standard mill run flour, sugar, dry milk, salt, margarine, water, and dry yeast were placed in a bread machine. The machine automatically mixed the ingredients, let the dough rise, and baked the bread.

Processing into wholemeal flour and wholemeal bread

Cleaned wheat was ground in a pin mill. Ground material was whole meal flour. Remaining whole meal flour was used to produce "Whole Meal Bread".

Whole meal flour, brown sugar, salt, margarine, water, and dry yeast were placed in a bread machine. The machine automatically mixed the ingredients, let the dough rise and baked the bread.

Wheat Gluten and Starch Production

For vital wheat gluten and starch, break flour was mixed with water. The dough was allowed to rest for two hours. After resting, the dough was kneaded as water washed away the starch, leaving the gluten. This processes continued until the water ran clear, indicating all starch was removed, leaving gluten. Starch was separated from the water using centrifugation and dried in an oven until the moisture content was less than 15 percent. Gluten was dried with a steam heated drum dryer.

Pasta (Noodle) Production

Equal parts of break and reduction flour were mixed with water and salt to form dough. This dough was kneaded and allowed to rest for 20–40 minutes. Dough was fed into a pasta machine to produce a fresh Asian noodle. Requested fresh cooked noodles were produced by placing fresh noodles into boiling water (10:1 ratio water to noodles) for 1–4 minutes. Requested cooking water fractions were also collected.

A portion of the fresh noodles were dried for eight hours at 24–35 °C. Remaining dried noodles were placed into boiling water (10:1 ratio) for 1–4 minutes.

Residues found in RAC and processed commodities with the calculated processing factors are shown in Table 149. The processing factor for wheat flour, middlings, fresh pasta, dry pasta, cooked pasta, dried and cooked pasta, starch, cooking water, white bread, and whole meal bread is <0.63. The processing factor for whole meal flour is 0.67, for shorts is 0.84×, for gluten is 0.94, for germ is 1.13, and for bran is 1.18. The average processing factor for AFG is >119.9.

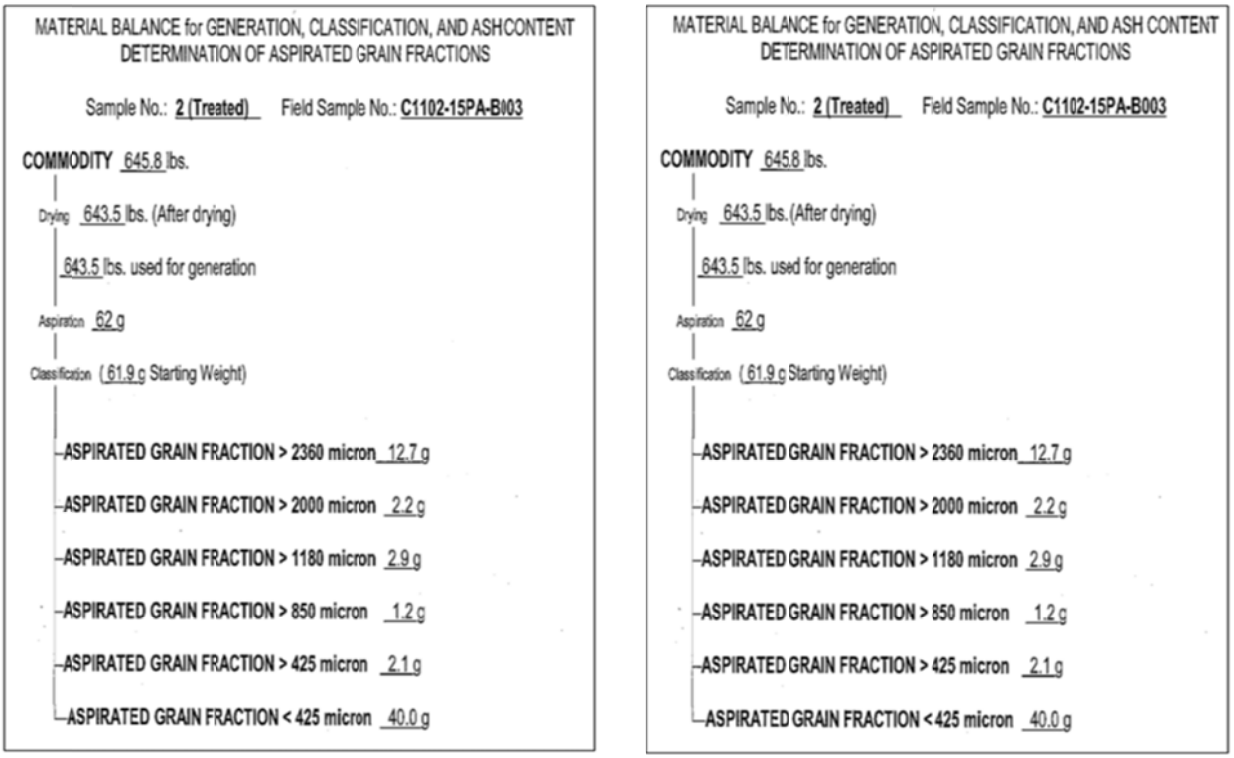


Figure 14 processing procedure and material balance for aspirated grain fractions

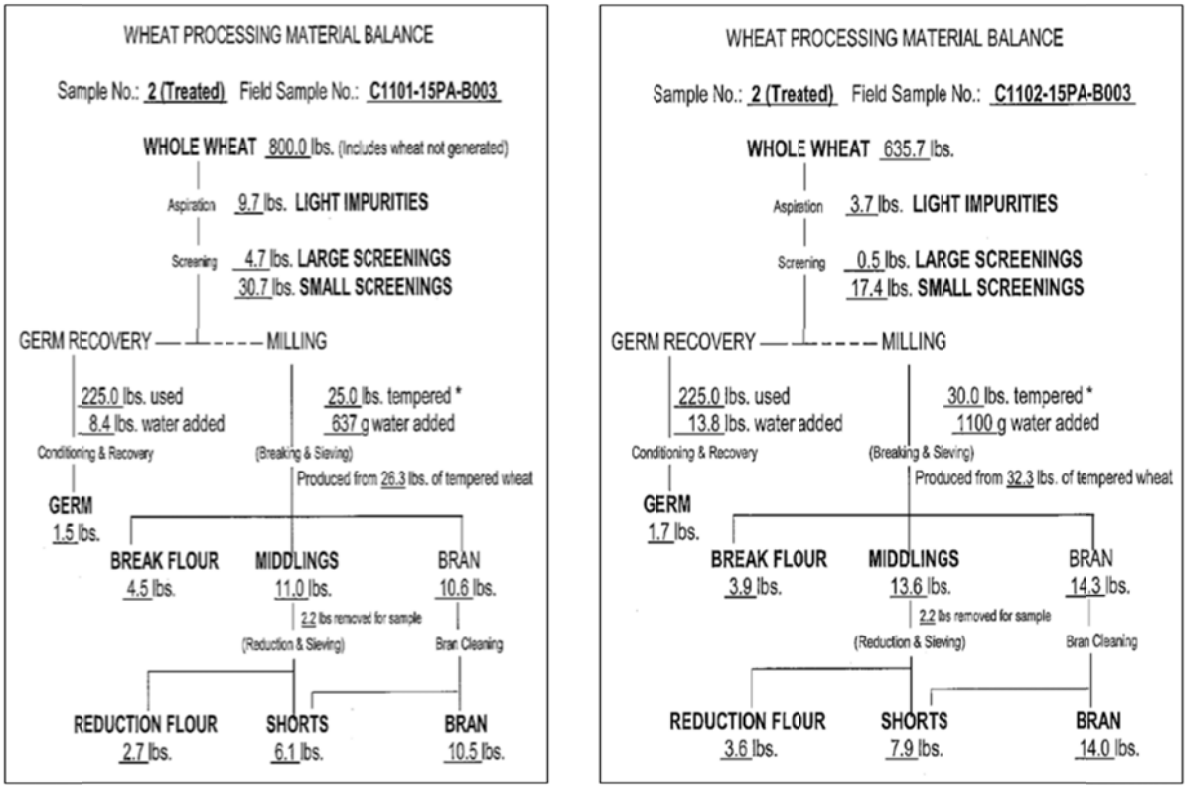


Figure 15 Processing procedure and material balance for wheat processed commodities.

Table 149 Summary of residue data and processing factors for isoflucypram on wheat grain and processed commodities

	Trial C1101-15PA, DALA= 69		Trial C1102-15PA, DALA = 67	
Sample	Isoflucypram Residue (mg/kg)	Processing Factor[1]	Isoflucypram (mg/kg)	Processing Factor[1]
Grain (RAC)	0.0159, 0.0163, 0.0157 (0.0160)	-	<<0.010, <0.010, <0.010 (<0.010)	NC
Bran	0.0182, 0.0192, 0.0194 (0.01890	1.18	<0.010, <0.010, <0.010 (<0.010)	NC
White flour	<0.010, <0.010, <0.010 (<0.010)	<0.63	<0.010, <0.010, <0.010 (<0.010)	NC
Whole meal flour	<0.010, 0.0110, 0.0111 (0.0107)	0.67	<0.010, <0.010, <0.010 (<0.010)	NC
Germ	0.0193, 0.0173, 0.0178 (0.0181)	1.13	<0.010, <0.010, <0.010 (<0.010)	NC
Middlings	<0.010, <0.010, <0.010 (<0.010)	<0.63	<0.010, <0.010, <0.010 (<0.010)	NC
Shorts	0.0159, 0.0115, 0.0128 (0.0134)	0.84	<0.010, <0.010, <0.010 (<0.010)	NC
Pasta, fresh	<0.010, <0.010, <0.010 (<0.010)	<0.63	<0.010, <0.010, <0.010 (<0.010)	NC
Pasta, dry	<0.010, <0.010, <0.010 (<0.010)	<0.63	<0.010, <0.010, <0.010 (<0.010)	NC
Pasta, cooked	<0.010, <0.010, <0.010 (<0.010)	<0.63	<0.010, <0.010, <0.010 (<0.010)	NC
Pasta, dried and cooked	<<0.010, <0.010, <0.010 (<0.010)	<0.63	<<0.010, <0.010, <0.010 (<0.010)	NC

	Trial C1101-15PA, DALA= 69		Trial C1102-15PA, DALA = 67	
Sample	Isoflucypram Residue (mg/kg)	Processing Factor[1]	Isoflucypram (mg/kg)	Processing Factor[1]
Gluten	0.0139, 0.0165, 0.0149 (0.0151	0.94	<<0.010, <0.010, <0.010 (<0.010)	NC
Starch	<0.010, <0.010, <0.010 (<0.010)	<0.63	<0.010, <0.010, <0.010 (<0.010)	NC
AGF	2.31, 2.37, 2.36 (2.35)	147.88	0.936, 0.921, 0.900 (0.919)	>91.9
Cooking water	<0.010, <0.010, <0.010 (<0.010)	<0.63	<0.010, <0.010, <0.010 (<0.010)	NC
White bread	<0.010, <0.010, <0.010 (<0.010)	<0.63	<0.010, <0.010, <0.010 (<0.010)	NC
Whole meal bread	<0.010, <0.010, <0.010 (<0.010)	<0.63	<0.010, <0.010, <0.010 (<0.010)	NC

Notes:
[1] Processing factor = [residue in processed commodity]/[residue in stored grain (RAC)]. Residues below the LOQ are calculated using the LOQ (0.01 mg/kg). Processing factors were not calculated (NC) when residues are below the LOQ in the RAC and processed commodity.

RESIDUES IN ANIMAL COMMODITIES

The Meeting received animal feeding studies with lactating cows and laying hens dosed with isoflucypram.

Dairy Cow

Report No. 17-8001.

The Meeting received a study investigating the magnitude of the free isoflucypram, isoflucypram-2-propanol, isoflucypram-carboxylic acid, isoflucypram-propanol, isoflucypram-desmethyl-propanol, and isoflucypram-desmethyl-carboxylic acid in milk, cream, whey, muscle, liver, kidney, and fat of dairy cows orally dosed with isoflucypram for 28 days (Glaubitz *et al.,* 2017). Doses were administered orally via gelatine capsule with a pill gun. Additionally, free plus conjugated isoflucypram-2-propanol and isoflucypram-propanol were analysed in liver and kidney.

Eighteen Holstein Frisian black dairy cows were selected with ages of approximately 2.0–2.7 years and weights ranging from 445–569 kg (determined during the acclimatization phase). Cows were allocated to a control group and 5 treated groups; each group comprised 3 animals. The target dose rates of isoflucypram were 0.05 mg/kg bw/day, 0.15 mg/kg bw/day, 0.5 mg/kg bw/day, 1.5 mg/kg bw/day, and 1.5 mg/kg bw/day for depuration. The amount of feed consumed was monitored daily and ranged from 10.0–21.3 kg/day dry matter. Based on the actual feed intake, the dose rates simulated residue concentrations in feed dry matter of about 1.61 ppm, 4.18 ppm, 15.54 ppm, 48.13 ppm, and 47.13 ppm (for depuration). Stability data were produced for isoflucypram in capsules at ambient room temperature, reflecting the conditions and duration of the study (Table 150).

Table 150 Stability of isoflucypram in capsules

Dose Group	Nominal content of isoflucypram (mg/L)	Storage duration	% remaining	Mean
1.61 ppm	45.69-57.39	0	91, 94, 79	88
4.18 ppm	134.8-168.7	0	101, 97, 101	100
15.54 ppm	467.8-522.1	0	96, 98, 91	95
48.13 ppm	739.7-811.3	0	104, 94, 93, 98, 87, 96	95

Dose Group	Nominal content of isoflucypram (mg/L)	Storage duration	% remaining	Mean
1.61 ppm	45.65-57.20	38	103, 79, 111, 109, 116	104
48.13 ppm	680.4-804.5	34-38	99, 98, 101, 100, 102	100

For the residue analysis, milk samples of from the control, 1.61 ppm, 4.18 ppm, 15.54 ppm, 48.13 ppm, and 47.13 ppm (depuration) groups were taken at various intervals during the acclimation phase, during and dosing phase, and during the depuration phase for the depuration group. Milk was sampled on Day 0-3 (except for the 48.13 ppm group), 4, 7, 9, 11, 14, 16, 18, 21, 23, 28-31, and 32 days (for the 48.13 ppm group only). Additionally, milk was collected from the control group on Day 29, 30, 31, 32, 35, 38, and 45; and from the 47.13 ppm depuration group on Day 32, 35, 38, and 45. Evening milking (prior to dose administration) and morning milk samples were combined for composite analysis. Milk from the control and 47.13 ppm depuration groups were separated into cream and whey by centrifugation on Day 31.

Animals were sacrificed within 24 hours after the final dose except for the 47.13 ppm depuration group, which were sacrificed 4, 7, or 14 days after the last dose. Liver, muscle, kidney, and fat (perirenal, subcutaneous, and mesenteric) were taken for analysis. Samples were either analysed immediately after collection or frozen at -18 °C within 24 hours of collection.

Samples were stored for a maximum of 30 days. Feedstuffs were analysed for isoflucypram according to Method 01475. All residues in feedstuffs were below the LOQ. Milk and tissues were analysed according to Method 01511. There were no residues in pre-dosing or control samples. The results are shown in Tables 151 to 154.

Table 151 Residues in milk.

Milk Sample Time (Day)	Residue[1] (mg eq/kg)					
	Isoflucypram	Isoflucypram-2-Propanol	Isoflucypram Carboxylic Acid	Isoflucypram-Propanol	Isoflucypram-Desmethyl-Propanol	Isoflucypram-Desmethyl-Carboxylic Acid
1.61 ppm						
1	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
4	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
7	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
9	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
11	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
14	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
16	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
18	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
21	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
23	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
29	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
4.18 ppm						
2	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
4	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
7	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
9	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
11	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
14	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
16	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
18	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
21	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
23	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
30	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
15.54 ppm						
3	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005

Milk Sample Time (Day)	Residue[1] (mg eq/kg)					
	Isoflucypram	Isoflucypram-2-Propanol	Isoflucypram Carboxylic Acid	Isoflucypram-Propanol	Isoflucypram-Desmethyl-Propanol	Isoflucypram-Desmethyl-Carboxylic Acid
4	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
7	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
9	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
11	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
14	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
16	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
18	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
21	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
23	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
31	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
48.13 ppm						
4	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
7	0.006, 0.006, 0.007 (0.006)	<0.005	<0.005	<0.005	<0.005	<0.005
9	0.008, 0.006, 0.008 (0.007)	<0.005	<0.005	<0.005	<0.005	<0.005
11	0.008, 0.005, 0.013 (0.009)	<0.005	<0.005	<0.005	<0.005	<0.005
14	0.010, 0.006, 0.011 (0.009)	<0.005	<0.005	<0.005	<0.005	<0.005
16	0.007, 0.006, 0.009 (0.007)	<0.005	<0.005	<0.005	<0.005	<0.005
18	0.008, 0.006, 0.009 (0.008)	<0.005	<0.005	<0.005	<0.005	<0.005
21	0.008, 0.007, 0.008 (0.008)	<0.005	<0.005	<0.005	<0.005	<0.005
23	0.008, 0.008, 0.008 (0.008)	<0.005	<0.005	<0.005	<0.005	<0.005
30	0.007, 0.006, 0.009 (0.007)	<0.005	<0.005	<0.005	<0.005	<0.005
32	0.008, 0.007, 0.009 (0.008)	<0.005	<0.005	<0.005	<0.005	<0.005
47.13 ppm (depuration)						
32	0.007, 0.009, 0.007	<0.005	<0.005	<0.005	<0.005	<0.005
35	<LOQ	<0.005	<0.005	<0.005	<0.005	<0.005
38	<0.005[2]	<0.005[2]	<0.005[2]	<0.005[2]	<0.005[2]	<0.005[2]
45	<0.005[3]	<0.005[3]	<0.005[3]	<0.005[3]	<0.005[3]	<0.005[3]

Notes:

[1] Mean of samples from three cows, unless otherwise specified.

[2] 2 Cows.

[3] 1 Cow.

Table 152 Separation of isoflucypram and metabolites in milk cream and whey (47.13 ppm depuration group, 31 days)

Sample Material	Residue (mg eq/kg)					
	Isoflucypram	Isoflucypram-2-propanol	Isoflucypram carboxylic acid	Isoflucypram-propanol	Isoflucypram-desmethyl-propanol	Isoflucypram-desmethyl-carboxylic acid
Milk	0.007	<0.005	<0.005	<0.005	<0.005	<0.005
Cream	0.11	0.006	<0.005	<0.005	<0.005	<0.005
Whey	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Milk	0.006	<0.005	<0.005	<0.005	<0.005	<0.005
Cream	0.11	0.006	<0.005	<0.005	<0.005	<0.005
Whey	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Milk	0.009	<0.005	<0.005	<0.005	<0.005	<0.005

Sample Material	Residue (mg eq/kg)					
	Isoflucypram	Isoflucypram-2-propanol	Isoflucypram carboxylic acid	Isoflucypram-propanol	Isoflucypram-desmethyl-propanol	Isoflucypram-desmethyl-carboxylic acid
Cream	0.15	0.009	<0.005	0.005	<0.005	<0.005
Whey	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005

Table 153 Residue Levels of Free Analytes in Tissues

Time (Days)	Sample Material	Residue Level (mg eq/kg)[1]					
		Isoflucypram	Isoflucypram-2-propanol	Isoflucypram carboxylic acid	Isoflucypram-propanol	Isoflucypram-desmethyl-propanol	Isoflucypram-esmethyl-carboxyli acid
1.61 ppm							
29	Muscle	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
29	Fat, Mesenteric	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
29	Fat, Perirenal	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
29	Fat, Subcutaneous	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
29	Kidney	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
29	Liver	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
4.18 ppm							
30	Muscle	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
30	Fat, Mesenteric	<0.01, <0.01, 0.010 (<0.010)	<0.01	<0.01	<0.01	<0.01	<0.01
30	Fat, Perirenal	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
30	Fat, Subcutaneous	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
30	Kidney	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
30	Liver	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
15.54 ppm							
30	Muscle	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
30	Fat, Mesenteric	0.037, 0.041, 0.020 (0.033)	<0.01	<0.01	<0.01	<0.01	<0.01
30	Fat, Perirenal	0.039, 0.041, 0.021 (0.034)	<0.01	<0.01	<0.01	<0.01	<0.01
30	Fat, Subcutaneous	0.032, 0.028, 0.013 (0.024)	<0.01	<0.01	<0.01	<0.01	<0.01
30	Kidney	<0.01	<0.01	0.024, 0.021, 0.021 (0.022)	<0.01	<0.01	<0.01
30	Liver	<LOQ, 0.013, <LOQ (<0.011	<0.01	<0.01	<0.01	<0.01	<0.01
48.13 ppm							
31	Muscle	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
31	Fat, Mesenteric	0.085, 0.077, 0.068 (0.077)	<0.01	<0.01	<0.01	<0.01	<0.01
31	Fat, Perirenal	0.080, 0.087, 0.075 (0.081)	<0.01	<0.01	<0.01	<0.01	<0.01
31	Fat, Subcutaneous	0.051, 0.057, 0.066 (0.058)	<0.01	<0.01	<0.01	<0.01	<0.01
31	Kidney	0.011, <0.01, <0.01 (<0.010)	<0.01	0.063, 0.074, 0.050 (0.062)	<0.01	<0.01	020, 0.023, 0.01 (0.019)
31	Liver	0.018, 0.020, <0.01 (<0.016	<0.01	0.032, 0.037, 0.016 (0.028)	<0.01	<0.01	0.014, 0.010, <0.01 (<0.011)
47.13 ppm (depuration)							
35, 38, 45	Muscle	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
35, 38, 45	Fat, Mesenteric	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Time (Days)	Sample Material	Residue Level (mg eq/kg)[1]					
		Isoflucypram	Isoflucypram-2-propanol	Isoflucypram carboxylic acid	Isoflucypram-propanol	Isoflucypram-desmethyl-propanol	Isoflucypram-esmethyl-carboxyli acid
35, 38, 45	Fat, Perirenal	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
35[2], 38, 45	Fat, Subcutaneous	0.014, <0.01, <0.01	<0.01	<0.01	<0.01	<0.01	<0.01
35, 38, 45	Kidney	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
35, 38, 45	Liver	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Notes:
[1] Mean of samples from three cows, unless otherwise specified.
[2] Value of 0.014 from 35 day cow.

Table 154 residue levels of free plus conjugated isoflucypram-2-propanol and isoflucypram-propanol in kidney and liver

Sampling Time (Days)	Sample Material	Isoflucypram-2-Propanol (mg eq/kg)[1]	Isoflucypram-Propanol (mg eq/kg)[1]
1.61 ppm			
29	Kidney	<0.01	<0.01
29	Liver	<0.01	<0.01
4.18 ppm			
30	Kidney	<0.01	<0.01
30	Liver	<0.01	<LOQ, 0.011, <0.01 (<0.010)
15.54 ppm			
30	Kidney	<0.01	<0.01
30	Liver	0.030, 0.031, 0.019 (0.027)	0.040, 0.033, 0.036 (0.036)
48.13 ppm			
31	Kidney	<0.01	0.016, 0.015, 0.016 (0.016)
31	Liver	0.091, 0.065, 0.052 (0.069)	0.12, 0.15, 0.064 (0.11)
47.13 ppm (depuration)			
35, 38, 45	Kidney	<0.01	<0.01
35, 38, 45	Liver	<0.01	<0.01

Notes:
[1] Mean of samples from three cows, unless otherwise specified.

Layer hen

Report No. 17-8002.

The Meeting received a study investigating the magnitude of the free isoflucypram, isoflucypram-2-propanol, isoflucypram-carboxylic acid, isoflucypram-propanol, isoflucypram-desmethyl-propanol, and isoflucypram-desmethyl-carboxylic acid in eggs (whole, white, and yolk), muscle, liver, and fat with skin of laying hens orally dosed with isoflucypram for 28 days. (Glaubitz, J., *et al*., 2017). Doses were administered orally via gelatine capsule. Additionally, free plus conjugated residues of isoflucypram-desmethyl-propanol were analysed in liver.

Forty-two *Gallus domesticus* layer hens were selected with weights ranging from 1.556–2.072 kg (determined during the acclimatization phase). Hens were allocated to a control group and 4 treated groups. Each group was contained two (control only) or three subgroups, each comprised of three animals. The target dose rates of isoflucypram were 0.03 mg/kg bw/day, 0.12 mg/kg bw/day, 0.48 mg/kg bw/day, and 0.48 mg/kg bw/day for depuration. The amount of feed consumed was monitored daily and ranged from 0.010–0.245 kg/day dry matter. Based on the actual feed intake, the dose rates

simulated residue concentrations in feed dry matter of ca. 0.530 ppm, 2.119 ppm, 8.698 ppm, and 8.140 ppm (for depuration).

Stability data were produced for isoflucypram in capsules at ambient room temperature, reflecting the conditions and duration of the study. Samples were analysed directly after preparation for all dose levels with recoveries ranging from 80–109 percent. Five capsules each from 0.530 ppm and 8.698 ppm dose groups were analysed at various days after preparation. Recoveries ranged from 89–90 percent on Day 8, 84–87 percent on Day 11, 83–85 percent on Day 13, and 81–86 percent on Day 17. Further, the stability of isoflucypram in the dosing solution was demonstrated under storage conditions at 2–8 °C for 41 days. At all storage intervals (8, 21, 31, and 41 days), two aliquots from the 0.530 and 8.698 ppm dose solutions were analysed. The recoveries from both dose groups were 97-113 percent on Day 8, 104–132 percent on Day 21, 85–93 percent on Day 31, and 84–111 percent on Day 41.

For the residue analysis, egg samples of the animals of the control, 0.530 ppm, 2.119 ppm, 8.698 ppm, and 8.140 ppm (for depuration) groups were taken at various intervals during the acclimation phase, during and dosing phase, and during the depuration phase for the 8.140 ppm depuration group. For the control, 0.530 ppm, 2.119 ppm, and 8.698 ppm dose groups, eggs were sampled on Day 0-2 (except for the 8.698 ppm group), 3, 4, 8, 9, 11, 14, 16, 21, 23, 28-30, and 31 (8.698 ppm group, only). Additionally, eggs from hens were collected from the control group on day 29, 30, 31, 35, 38, and 45; and from the 8.140 ppm (for depuration) group on Day 21, 23, 31, 35, 38, and 45. Eggs from the control and 8.698 ppm groups were separated into egg white and yolk samples.

Animals were sacrificed within 6 hours after the final dose except for the 8.140 ppm (depuration) group, which were sacrificed 4, 7, or 14 days after the last dose. Liver, muscle, and fat with adhering skin were taken for analysis. Samples were either analysed within 24 hours of collection or frozen at -18 °C within 24 hours of collection.

Feed was analysed for isoflucypram according to Method 01475. All residues in feedstuffs were below the LOQ. Eggs and tissues were analysed according to Method 01511. Samples were stored for a maximum of 6 days. The maximum time from extraction to analysis was 13 days. There were no residues in pre-dosing or control samples. The results are in Tables 155 to 158.

Table 155 Residues in eggs

Sample Time (Day)	Residue[1] (mg eq/kg)					
	Isoflucypram	Isoflucypram-2-propanol	Isoflucypram carboxylic acid	Isoflucypram-propanol	Isoflucypram-desmethyl-propanol	Isoflucypram-desmethyl-carboxylic acid
0.530 ppm						
1	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
3	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
4	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
8	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
9	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
11	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
14	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
16	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
21	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
23	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
29	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
2.119 ppm						
2	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
3	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
4	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Sample Time (Day)	Residue[1] (mg eq/kg)					
	Isoflucypram	Isoflucypram-2-propanol	Isoflucypram carboxylic acid	Isoflucypram-propanol	Isoflucypram-desmethyl-propanol	Isoflucypram-desmethyl-carboxylic acid
8	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
9	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
11	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
14	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
16	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
21	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
23	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
30	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
8.698 ppm						
3	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
4	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
8	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
9	<0.01	<0.01	<0.01	<0.01, <0.01, 0.015 (<0.012)	<0.01	<0.01
11	<0.01	<0.01	<0.01	0.014, 0.012, <0.01 (<0.012)	<0.01	<0.01
14	<0.01	<0.01	<0.01	<0.01, <0.01, 0.014 (<0.011	<0.01	<0.01
16	<0.01	<0.01	<0.01	<0.01, 0.020, 0.013 (<0.014)	<0.01	<0.01
21	<0.01	<0.01	<0.01	<0.01, 0.017, <0.01 (<0.012)	<0.01	<0.01
23	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
31	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
8.140 ppm (depuration)						
21	<0.01	<0.01	<0.01	<0.01, 0.011, <0.01 (<0.010)	<0.01	<0.01
23	<0.01	<0.01	<0.01	<0.01, 0.013, <0.01 (<0.011)	<0.01	<0.01
31	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
38[2]	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
45[3]	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Notes:
[1] Average of three samples.

Table 156 Separation of isoflucypram and metabolites in egg white and yolk (8.140 ppm depuration group)

Sample Time (Days)	Sample Material	Residue level (mg eq/kg)[1]					
		Isoflucypram	Isoflucypram-2-propanol	Isoflucypram carboxylic acid	Isoflucypram-propanol	Isoflucypram-desmethyl-propanol	Isoflucypram-desmethyl-carboxylic acid
30	Yolk	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
30	White	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
31	Whole egg	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Notes:
[1] Average of three samples.

Table 157 Residue Levels of Free Analytes in Tissues

Sample time (Days)	Sample Material	Residue Level (mg eq/kg)[1]					
		Isoflucypram	Isoflucypram-2-propanol	Isoflucypram carboxylic acid	Isoflucypram-propanol	Isoflucypram-desmethyl-propanol	Isoflucypram-desmethyl-carboxylic acid
0.530 ppm							
29	Fat with adhering skin	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
29	Liver	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
29	Muscle	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
2.119 ppm							
30	Fat with adhering skin	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
30	Liver	<0.01	<0.01	0.015, 0.012, <LOQ (<0.012)	<0.01	<0.01	0.045, 0.023, 0.010 (0.026)
30	Muscle	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
8.698 ppm							
31	Fat with adhering skin	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
31	Liver	<0.01	<0.01	0.040, 0.035, 0.019 (0.031)	<0.01	<0.01	0.097, 0.11, 0.061 (0.089)
31	Muscle	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
8.140 ppm (depuration)							
35, 38, 45	Fat with adhering skin	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
35, 38, 45	Liver	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
35, 38, 45	Muscle	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Notes:
[1] Average of three samples.

Table 158 residues of free plus conjugated isoflucypram-desmethyl-propanol in liver

Dose group and sampling time (Days)	Isoflucypram-desmethyl-propanol (mg eq/kg)
0.530 ppm (29)	<0.01
2.110 ppm (30)	0.012, 0.021, 0.015 (0.016)
8.698 ppm (31)	0.053, 0.059, 0.079 (0.064)
8.140 ppm (depuration) (35, 38, 45)	<0.01

APPRAISAL

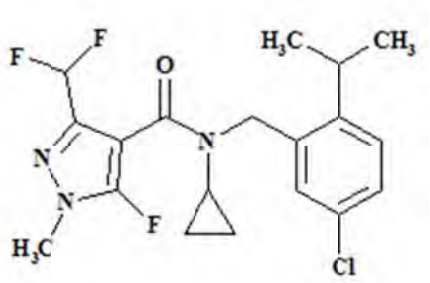
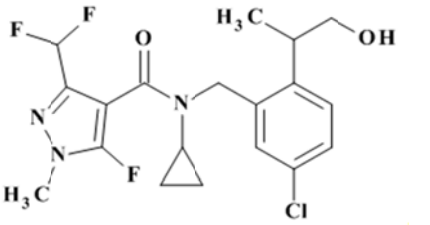
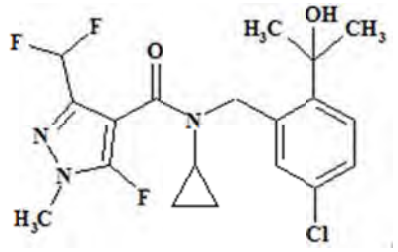
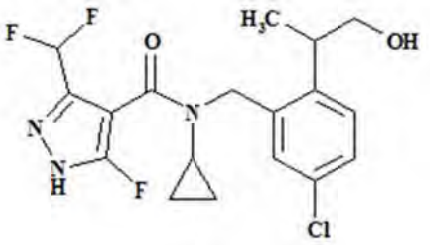
Isoflucypram is a novel broad-spectrum fungicide of the chemical class of N-cyclopropyl-N-benzyl-pyrazole-carboxamides. Isoflucypram is a succinate dehydrogenase (SDH) inhibitor.

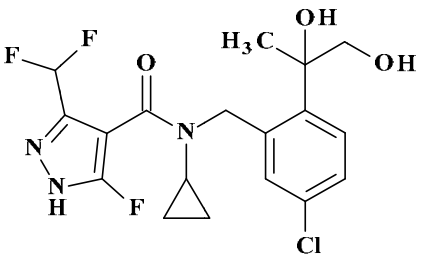
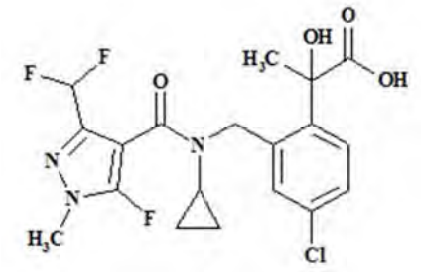
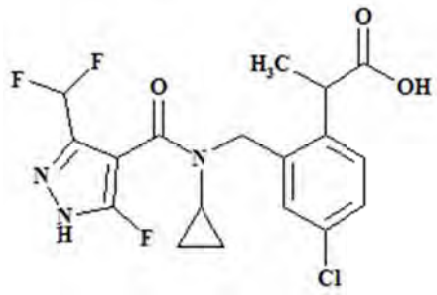
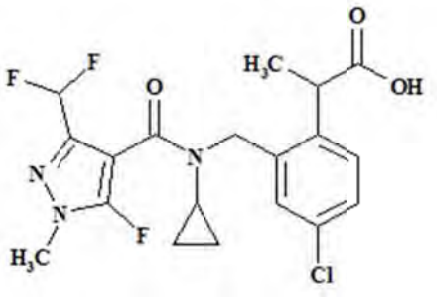
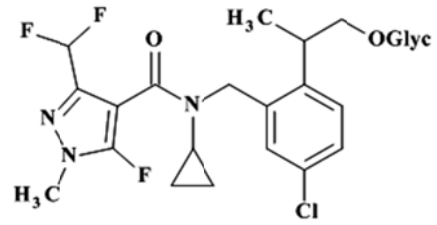
Isoflucypram was scheduled at the Fifty-first Session of the CCPR for evaluation as a new compound in 2020 and rescheduled to the 2022 JMPR. The 2022 Meeting established an ADI of 0–0.06 mg/kg bw/day and determined that an ARfD was unnecessary. Information on chemical identity,

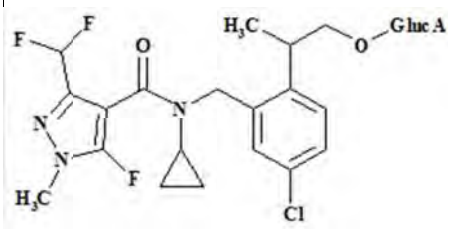
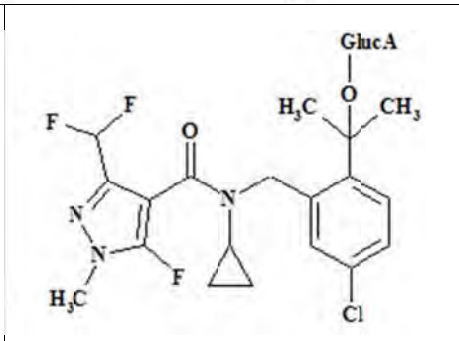
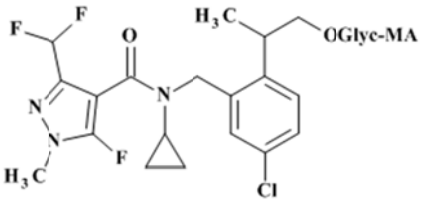
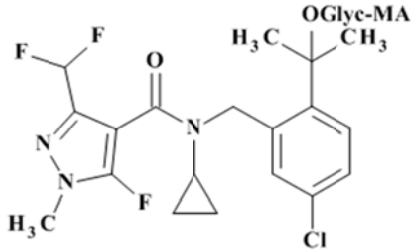
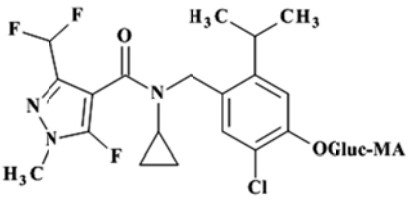
physical-chemical properties, metabolism and environmental fate, methods of residue analysis, storage stability, intended use patterns, supervised residue trials, fate of residues upon processing, and farm animal feeding studies were submitted for evaluation by the 2022 JMPR.

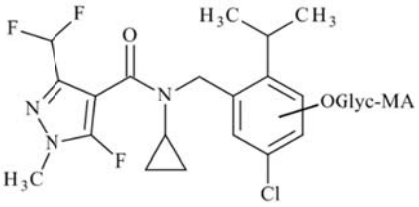
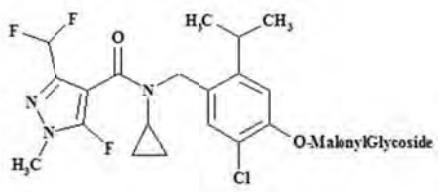
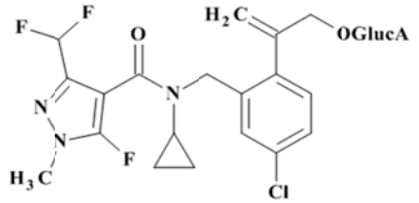
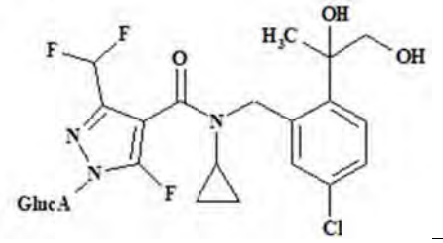
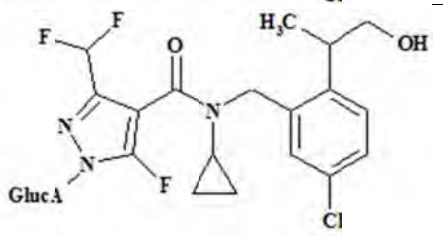
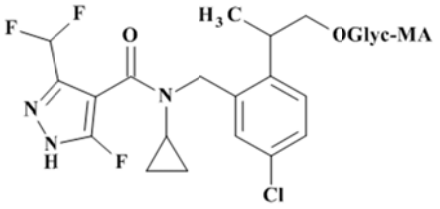
The chemical structures of isoflucypram and its metabolites/degradates relevant for the appraisal are shown in Talbe 159.

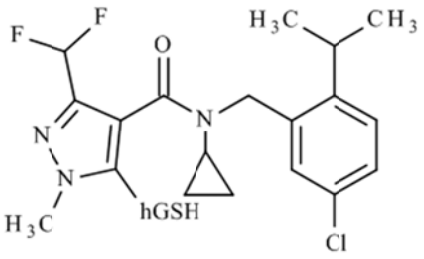
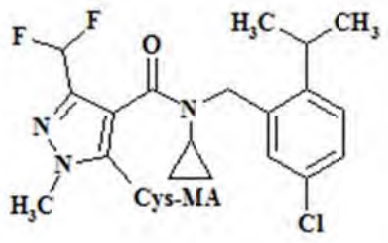
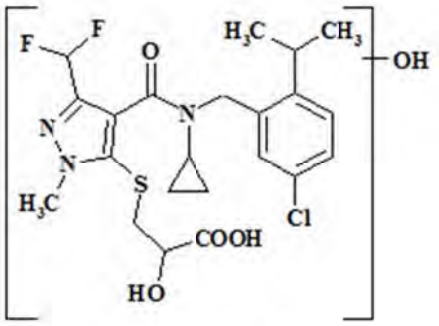
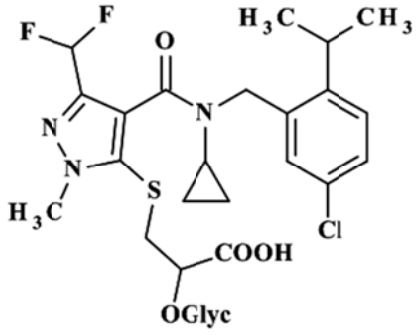
Table 159 Abbreviations used for relevant compounds referred to in the appraisal

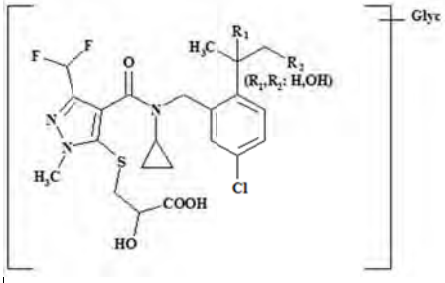
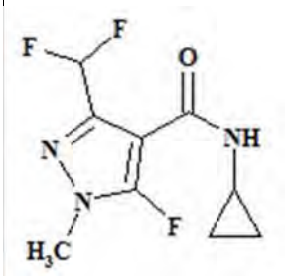
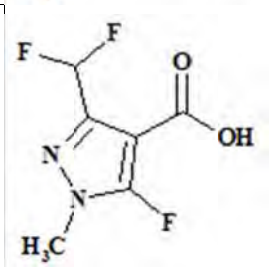
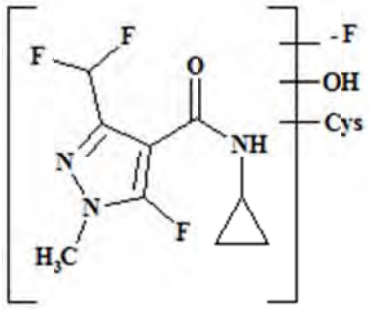
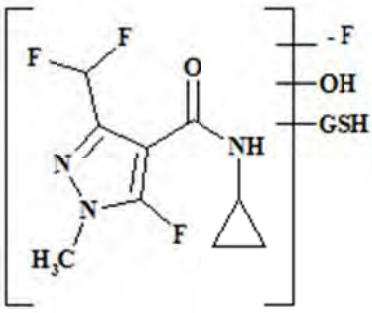
Code Name	Chemical Identity (IUPAC)	Structure
Isoflucypram	N-(5-chloro-2-isopropylbenzyl)-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide	
M01 Isoflucypram-propanol	N-[5-chloro-2-(1-hydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide	
M02 Isoflucypram-2-propanol	2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)-methyl]phenyl}propan-2-yl	
M06 Isoflucypram-desmethyl-propanol	N-[5-chloro-2-(1-hydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxamide	

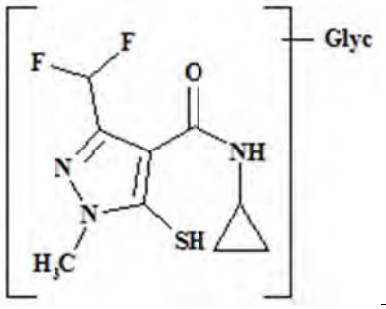
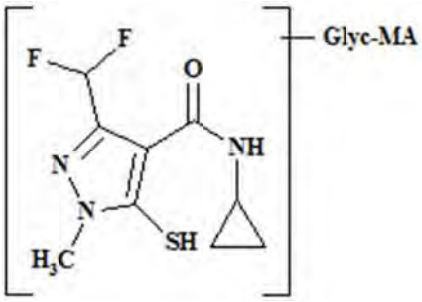
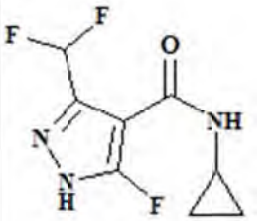
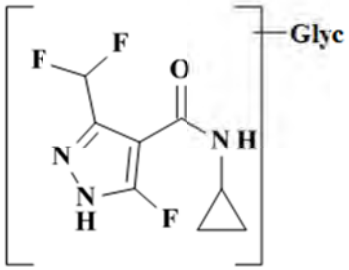
Code Name	Chemical Identity (IUPAC)	Structure
M07 Isoflucypram-desmethyl-1,2-propandiol	N-[5-chloro-2-(1,2-dihydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxamide	
M10 Isoflucypram-lactic acid	2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}-2-hydroxypropanoic acid	
M11 Isoflucypram-desmethyl-carboxylic acid	2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}propanoic acid	
M12 Isoflucypram-carboxylic acid	2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)-methyl]phenyl}-propanoic acid	
M18 Isoflucypram-propanol-Glyc	N-{5-chloro-2-[1-(hexopyranosyloxy)propan-2-yl]benzyl}-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide	

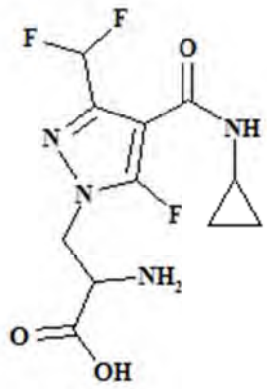
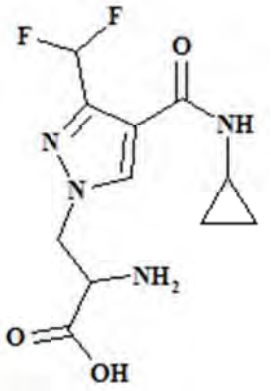
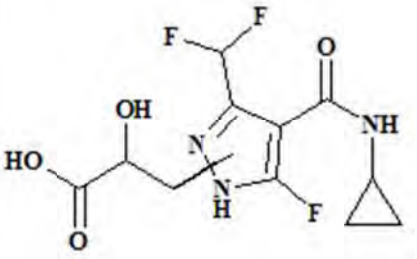
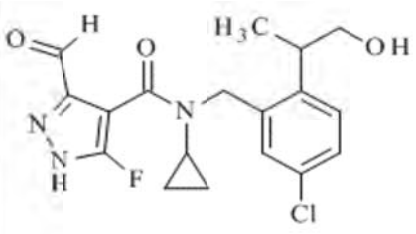
Code Name	Chemical Identity (IUPAC)	Structure
M19 Isoflucypram-propanol-GlucA (isomer 1 and 2)	2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)methyl]phenyl}propyl glucopyranosiduronic acid	
M20 Isoflucypram-2-propanol-GlucA	2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}propan-2-yl beta-D-glucopyranosiduronic acid	
M21 Isoflucypram-propanol-Glyc-MA	2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)methyl]phenyl}propyl 6-O-(carboxyacetyl)hexopyranoside	
M22 Isoflucypram-2-propanol-Glyc-MA	2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)-methyl]phenyl}propan-2-yl 6-O-(carboxy-acetyl)hexopyranoside	
M23 Isoflucypram-hydroxyphenyl-Gluc-MA	2-chloro-4-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)-methyl]-5-isopropyl-phenyl 6-O-(carboxyacetyl)-beta-D-glucopyranoside	

Code Name	Chemical Identity (IUPAC)	Structure
M23a Isoflucypram-OH-phenyl-Glyc-MA	-	
M24 Isoflucypram-hydroxyphenyl-Glyc-MA	2-chloro-4-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]-carbonyl}amino)-methyl]-5-isopropyl-phenyl 6-O-(carboxyacetyl)-hexopyranoside	
M25 Isoflucypram-propenol-GlucA	2-{4-chloro-2-[(cycloprop yl{[3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}prop-2-en-1-yl beta-D-glucopyranosiduronic acid	
M36 Isoflucypram-desmethyl-1,2-propandiol-N-GlucA	N-[5-chloro-2-(1,2-dihydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-(gluco-pyranuronosyl)-1H-pyrazole-4-carboxamide	
M37 Isoflucypram-desmethyl-propanol-N-GlucA	N-[5-chloro-2-(1-hydroxypropan-2-yl)benzyl]-N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-(gluco-pyranuronosyl)-1H-pyrazole-4-carboxamide	
M41 Isoflucypram-desmethyl-propanol-Glyc-MA	2-{4-chloro-2-[(cyclopropyl{[3-(difluoromethyl)-5-fluoro-1H-pyrazol-4-yl]carbonyl}amino)methyl]phenyl}propyl 6-O-(carboxyacetyl)hexopyranoside	

Code Name	Chemical Identity (IUPAC)	Structure
M44 Isoflucypram-desfluoro-homoGSH	gamma-glutamyl-S-{4-[(5-chloro-2-isopropylbenzyl)(cyclopropyl)carbamoyl]-3-(difluoromethyl)-1-methyl-1H-pyrazol-5-yl}cysteinyl-beta-alanine	
M45 Isoflucypram-desfluoro-Cys-MA	N-(carboxyacetyl)-S-{4-[(5-chloro-2-isopropylbenzyl)(cyclopropyl)carbamoyl]-3-(difluoromethyl)-1-methyl-1H-pyrazol-5-yl}cysteine	
M46 Isoflucypram-desfluoro-mercapto-lactic acid-OH	-	
M47 Isoflucypram-desfluoro-mercapto-lactic acid-Glyc	3-({4-[(5-chloro-2-isopropylbenzyl)(cyclopropyl)carbamoyl]-3-(difluoromethyl)-1-methyl-1H-pyrazol-5-yl}sulfanyl)-2-(hexopyranosyloxy)propanoic acid	

Code Name	Chemical Identity (IUPAC)	Structure
M48 Isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc	-	
M49 BCS-CR60082 Isoflucypram-N-methyl-cyclopropyl-pyrazole-carboxamide	N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1-methyl-1H-pyrazole-4-carboxamide	
M50 Isoflucypram-N-methyl-pyrazole-carboxylic acid	3-(difluoromethyl)-5-fluoro-1-methyl-pyrazole-4-carboxylic acid	
M52 Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys	-	
M54 Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH	-	

Code Name	Chemical Identity (IUPAC)	Structure
M56 Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc	-	
M57 Isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc-MA	-	
M58 Isoflucypram-cyclopropyl-pyrazole-carboxamide	N-cyclopropyl-3-(difluoromethyl)-5-fluoro-1H-pyrazole-4-carboxamide	
M62 Isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc (isomer 1 and 2)	-	

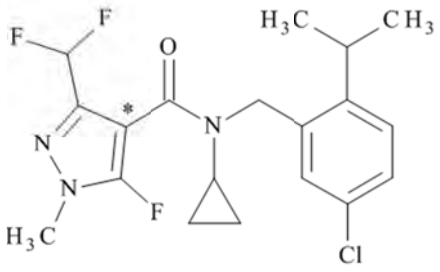
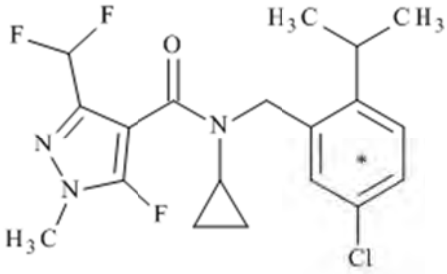
Code Name	Chemical Identity (IUPAC)	Structure
M66 Isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala	3-[4-(cyclopropylcarbamoyl)-3-(difluoromethyl)-5-fluoro-1H-pyrazol-1-yl]alanine	
M67 Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala	3-[4-(cyclopropylcarbamoyl)-3-(difluoromethyl)-1H-pyrazol-1-yl]alanine	
M69 Isoflucypram-cyclopropyl-pyrazole-carboxamide-OH-lactic acid (isomer 1 and 2)	-	
M77 Isoflucypram-desmethyl-propanol-aldehyde	N-[5-chloro-2-(1-hydroxypropan-2-yl)benzyl]-N-cyclopropyl-5-fluoro-3-formyl-1H-pyrazole-4-carboxamide	

Physical-chemical properties

Isoflucypram is regarded as not volatile, it has a higher solubility in organic solvents compared to its solubility in water, and the partition coefficient (logPow=4.0) indicates potential to sequester into fat.

Plant metabolism

The Meeting received studies describing the metabolism of isoflucypram in tomatoes, wheat, soya bean, and oilseed rape following foliar application, and in potatoes following application to potato seed pieces. The studies were conducted with isoflucypram radiolabelled at either the pyrazole or phenyl position, as shown below. All studies were conducted in a greenhouse and the compound was formulated as emulsifiable concentrate (EC).

[pyrazole-4-^{14}C]isoflucypram	[phenyl-UL-^{14}C]isoflucypram
	

(*): Position of ^{14}C-radiolabel.

Figure 19 Isoflucypram radiolabelled at either the pyrazole or phenyl position

Tomato

Isoflucypram was applied twice to tomatoes at BBCH 14–15 (78–79 g ai/ha) and BBCH 85–87 (78–89 g ai/ha). The interval between applications was 97–99 days. Tomato fruits were harvested 14 days after the last application.

The total radioactive residue (TRR) was 1.8× higher in the pyrazole study (0.17 mg eq/kg) as compared to the phenyl study (0.095 mg eq/kg). The main portion of the radioactivity was recovered in the surface wash, comprising 74–75 percent TRR (0.071–0.13 mg eq/kg). TRR in the acetonitrile (ACN)/water extract was 25–26 percent TRR (0.024–0.045 mg eq/kg), leaving ≤ 0.2 percent TRR (≤ 0.001 mg eq/kg) in the post-extraction solids (PES).

Isoflucypram was the only metabolite observed, comprising 97–98 percent TRR (0.094–0.17 mg/kg). There were no unknown peaks.

Wheat

Isoflucypram was applied twice to wheat, at BBCH 30 at 64–69 g ai/ha and at BBCH 69 at 66–67 g ai/ha. The interval between applications was 28–33 days. Wheat hay was harvested one day prior to the second application (BBCH 69) and allowed to dry for four days. Wheat straw and grain were harvested at maturity (BBCH 89) corresponding to 17–18 days between the final application and harvest.

Total radioactive residues (TRR) in both labels were similar and were highest in straw (16 mg eq/kg), hay (3.0–4.0 mg eq/kg) and grain (0.28–0.39 mg eq/kg). The conventional ACN/water extract released ≥ 94 percent TRR. The conventional extract of pyrazole-labelled wheat straw was subjected to microwave-assisted extraction, releasing an additional 4.7 percent TRR. The conventional extract of wheat hay and straw were hydrolysed with 1 mol/L hydrochloric acid (HCl) at 100 °C for 1 hour.

In wheat hay, parent isoflucypram was the major component representing 50–55 percent TRR (1.7–2.0 mg/kg). Identified metabolites included isoflucypram-propanol-Glyc-MA (7.5-10 percent TRR [0.23–0.41 mg eq/kg]), isoflucypram-desmethyl-propanol-Glyc-MA (2.5–2.7 percent TRR [0.081–0.10 mg eq/kg]), isoflucypram-propanol-Glyc (0.8–2.4 percent TRR [0.023–0.096 mg eq/kg]), and

isoflucypram-propanol (0.7 percent TRR [0.021–0.029 mg eq/kg]). Up to 23 unknown metabolites were characterised in the extracts by their chromatographic behaviour, individually accounting for ≤ 3.1 percent TRR (≤ 0.13 mg eq/kg). PES accounted for ≤ 4.2 percent TRR (≤ 0.17 mg eq/kg).

In hydrolysed extract of wheat hay, parent isoflucypram was the major component representing 44–50 percent TRR (1.5–1.8 mg/kg). Remaining identified metabolites included isoflucypram-propanol (21–22 percent TRR [0.64–0.90 mg eq/kg]), isoflucypram-desmethyl-propanol (6.7–6.9 percent TRR [0.20–0.28 mg eq/kg]), isoflucypram-propanol-Glyc-MA (0.90 percent TRR [0.036 mg eq/kg]), and isoflucypram-propanol-Glyc (0.60–0.80 percent TRR [0.019–0.031 mg eq/kg]).

In wheat straw, parent isoflucypram was the major component representing 62–64 percent TRR (9.9–10 mg/kg). Remaining identified metabolites included isoflucypram-propanol-Glyc-MA (5.0–6.7 percent TRR [0.808–1.042 mg eq/kg]), isoflucypram-propanol-Glyc (2.3–3.7 percent TRR [0.37–0.56 mg eq/kg]), isoflucypram-desmethyl-propanol-Glyc-MA (1.9–2.9 percent TRR [0.306–0.45 mg eq/kg), isoflucypram-propanol (0.9–1.7 percent TRR [0.15–0.27 mg eq/kg]), and isoflucypram-desmethyl-propanol (0.30–1.1 percent TRR [0.052–0.17 mg eq/kg]). Up to 39 unknown metabolites were characterised in the extracts by their chromatographic behaviour, individually accounting for ≤ 2.1 percent TRR (≤ 0.35 mg eq/kg). TRR was successfully extracted as demonstrated by ≤ 4.8 percent TRR (0.77 mg eq/kg) remaining in the PES.

In the hydrolysed extract of wheat straw, parent isoflucypram was the major component representing 61–67 percent TRR (9.8–10 mg/kg). Remaining identified metabolites included isoflucypram-propanol (11–13 percent TRR [1.6–2.0 mg eq/kg]), isoflucypram-desmethyl-propanol (3.6–4.0 percent TRR [0.56–0.64 mg eq/kg]), isoflucypram-desmethyl-propanol-Glyc-MA (0.30 percent TRR [0.054 mg eq/kg]), isoflucypram-propanol-Glyc (0.20–0.30 percent TRR [0.024–0.053 mg eq/kg]), and isoflucypram-propanol-Glyc-MA (0.10–0.30 percent TRR [0.022–0.054 mg eq/kg]).

Wheat grain contained only parent isoflucypram, representing 92–93 percent TRR (0.26–0.35 mg/kg). There were no unknown peaks. TRR was successfully extracted as demonstrated by ≤ 6.5 percent TRR (0.025 mg eq/kg) remaining in the PES.

Soya bean

Isoflucypram was applied three times to soya bean plants, at BBCH 14 (54–59 g ai/ha), BBCH 51 (56–57 g ai/ha) and BBCH 84–85 (65–66 g ai/ha). Application interval was 6–8 days between applications one and two and 62–69 days between applications two and three. Soya bean forage was harvested at BBCH 49, corresponding to 5–6 days after the first application. Soya bean hay was harvested at BBCH 77, corresponding to 38–39 days after the second application, and allowed to dry for four days. Soya bean straw and seed were harvested at BBCH 96, corresponding to 21 days after the final application. TRR in forage was similar for both labels, but was 2.1–3.3× higher in the pyrazole study for remaining soya bean commodities. Radioactivity (phenyl/pyrazole) was highest in straw (8.5/18 mg eq/kg), followed by forage (3.9/4.4 mg eq/kg), hay (1.4/4.7 mg eq/kg) and seed (0.015/0.035 mg eq/kg). For both studies, ≥ 87 percent TRR was extracted in the conventional ACN/water extract, except for phenyl-label seed where 70 percent was extracted, possibly as a result of low overall TRR (0.015 mg eq/kg). Microwave-assisted extraction of soya bean forage, hay, and straw successfully released an additional 2.5–6.9 percent TRR. Soya bean hay extract from microwave-assisted extraction underwent partitioning against ethyl acetate, resulting in complete transfer of the radioactivity to the organic phase.

In soya bean forage, isoflucypram accounted for 19 percent TRR (0.76–0.82 mg/kg). Identified metabolites included isoflucypram-desfluoro-homoGSH (20–23 percent TRR, 0.79–1.0 mg eq/kg), isoflucypram-desfluoro-mercapto-lactic acid-OH (9.5–17 percent TRR, 0.42–0.67 mg eq/kg),

isoflucypram-desfluoro-Cys-MA (8.2–9.2 percent TRR, 0.32–0.40 mg eq/kg), isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc (3.4–4.8 percent TRR, 0.15–0.19 mg eq/kg), and isoflucypram-desfluoro-mercapto-lactic acid-Glyc (2.7–3.0 percent TRR, 0.11–0.13 mg eq/kg). Up to 17 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 3.9 percent TRR (≤ 0.17 mg eq/kg). PES accounted for ≤ 3.4 percent TRR (≤ 0.13 mg eq/kg).

In soya bean hay, isoflucypram accounted for 10 percent TRR (0.14–0.49 mg/kg). Identified metabolites included isoflucypram-desfluoro-Cys-MA (15–21 percent TRR, 0.29–0.72 mg eq/kg), isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc (15–18 percent TRR, 0.25–0.71 mg eq/kg), isoflucypram-desfluoro-mercapto-lactic acid-Glyc (11 percent TRR, 0.15–0.52 mg eq/kg), isoflucypram-desfluoro-homoGSH (7.8 percent TRR, 0.11–0.37 mg eq/kg), and isoflucypram-desfluoro-mercapto-lactic acid-OH (2.8–3.2 percent TRR, 0.040–0.15 mg eq/kg). Up to 13 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 6.1 percent TRR (≤ 0.21 mg eq/kg). PES accounted for ≤ 5.7 percent TRR (≤ 0.27 mg eq/kg).

In soya bean straw, isoflucypram was the major component, representing 65–70 percent TRR (6.0–11 mg/kg). Remaining identified metabolites included isoflucypram-desfluoro-Cys-MA (4.3–4.6 percent TRR, 0.37–0.83 mg eq/kg), isoflucypram-desfluoro-homoGSH (2.8–4.8 percent TRR, 0.24–0.86 mg eq/kg), isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc (2.1–3.8 percent TRR, 0.18–0.69 mg eq/kg), isoflucypram-desfluoro-mercapto-lactic acid-Glyc (2.8–3.0 percent TRR, 0.24–0.53 mg eq/kg), and isoflucypram-desfluoro-mercapto-lactic acid-OH (1.9–2.5 percent TRR, 0.22–0.34 mg eq/kg). Up to 20 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 2.8 percent TRR (≤ 0.33 mg eq/kg). PES accounted for ≤ 4.1 percent TRR (≤ 0.35 mg eq/kg).

In soya bean seed, parent isoflucypram was the only identified component, representing 67–77 percent TRR (0.011–0.027 mg/kg). There were no unknown peaks. Radioactivity remaining in the PES was ≤ 30 percent TRR (≤ 0.005 mg eq/kg).

Oilseed rape

Isoflucypram was applied twice to oilseed rape plants, at BBCH 14 at 63–64 g ai/ha and at BBCH 77 at 62–63 g ai/ha. The interval between applications was 84 days. Oilseed rape was harvested at BBCH 30 for intermediate harvest (whole plant) corresponding to two days after the first application. Oilseed rape forage was harvested at BBCH 55, 40 days after the first application and the plants and seed were harvested at maturity (BBCH 89), 21 days after the final application. TRR was similar between both labels. TRR was highest in intermediate harvest (3.3 and 4.8 mg eq/kg) and plants (3.9 and 4.1 mg eq/kg), followed by seed (0.099 and 0.13 mg eq/kg), and forage (0.008 and 0.012 mg eq/kg). The conventional ACN/water extract released ≥ 71 percent TRR. For seed, an additional 9.8–11 percent TRR was subsequently extracted with microwave-assistance. The purified extract for whole plant intermediate harvest was hydrolysed with 10 mol/L HCl for 1 hour.

In oilseed rape whole plant intermediate harvest, isoflucypram was the major component, representing 82–84 percent TRR (2.8–3.9 mg/kg). Identified metabolites included and isoflucypram-hydroxyphenyl-Glyc-MA (3.1–3.8 percent TRR, 0.13–0.15 mg eq/kg), isoflucypram-hydroxyphenyl-Gluc-MA (2.3 percent TRR, 0.077–0.11 mg eq/kg), isoflucypram-propanol-Glyc-MA (2.2–2.8 percent TRR, 0.071–0.13 mg eq/kg), and isoflucypram-2-propanol-Glyc-MA (1.6–2.2 percent TRR, 0.052–0.11 mg eq/kg). Up to 23 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 1.5 percent TRR (≤ 0.072 mg eq/kg). Radioactivity remaining in the PES was ≤ 0.50 percent TRR (≤ 0.022 mg eq/kg).

Comparison of metabolic profiles before and after acid hydrolysis of mature plant intermediate harvest indicated cleavage of isoflucypram-hydroxyphenyl-Gluc-MA, isoflucypram-2-propanol-Glyc-MA, isoflucypram-propanol-Glyc-MA, and isoflucypram-hydroxyphenyl-Glyc-MA to less polar compounds.

In oilseed rape forage, no individual peak was observed above the background noise due to low radioactivity. Radioactivity remaining in the PES accounted for ≤ 23 percent TRR (≤ 0.002 mg eq/kg).

In oilseed rape mature plants, isoflucypram was the major component, representing 72–88 percent TRR (2.8–3.6 mg/kg). Identified metabolites included isoflucypram-hydroxyphenyl-Glyc-MA (1.0–3.1 percent TRR, 0.040–0.12 mg eq/kg), isoflucypram-2-propanol-Glyc-MA (0.90–4.6 percent TRR, 0.038–0.18 mg eq/kg), isoflucypram-hydroxyphenyl-Gluc-MA (0.70–2.2 percent TRR, 0.027–0.087 mg eq/kg), and isoflucypram-propanol-Glyc-MA (0.60–2.5 percent TRR, 0.025–0.097 mg eq/kg]. Up to 39 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 1.3 percent TRR (≤ 0.054 mg eq/kg). Radioactivity remaining in the PES was ≤ 3.8 percent TRR (≤ 0.15 mg eq/kg).

In oilseed rape seeds, parent was the only identified component, representing 71–74 percent TRR (0.070–0.093 mg/kg). There were no unknown peaks. Radioactivity remaining in the PES was ≤ 6.7 percent TRR (≤ 0.008 mg eq/kg).

Potatoes

Isoflucypram was applied to potato seed pieces prior to planting in at 28 g ai/ha (0.55 mg ai/ tuber and 50,000 plants/ha) for a low dose experiment and 274–280 g ai/ha (5.5 mg ai/tuber and 50,000 plants/ha) for a high dose experiment. Potato tubers and leaves were harvested at BBCH 97, corresponding to 119 days after planting.

TRR was higher in the pyrazole study by factors ranging from 1.5–7.5×. TRR was higher in leaves (0.050 and 0.37 mg eq/kg in the low dose experiment; 0.69 and 1.1 mg eq/kg in the high-dose experiment) than in tubers (0.002 and 0.009 mg eq/kg in the low dose experiment; 0.042 and 0.064 mg eq/kg in the high dose experiment). Radioactivity was not extracted in the low dose potato tuber experiments due to low TRRs. For all other matrices, conventional extraction with ACN/water released ≥ 82 percent TRR. Leaf extract from the low dose experiment and potato extract from the high dose experiment were further extracted with microwave assistance to release an additional 7.5 and 12.9 percent TRR, respectively.

In potato leaves, parent isoflucypram was a minor component accounting for 2.0–7.3 percent TRR (0.004–0.007 mg/kg) in the low dose experiment and 2.5–4.0 percent TRR (0.027 mg eq/kg) in the high dose experiment. Remaining identified metabolites included isoflucypram-2-propanol-Glyc-MA (14–29 percent TRR [0.014–0.053 mg eq/kg in the low dose experiment and 0.10–0.15 mg eq/kg in the high dose experiment]); isoflucypram-OH-phenyl-Glyc-MA (6.6–23 percent TRR [0.012–0.025 mg eq/kg in the low dose experiment and 0.10 mg eq/kg in the high dose experiment]); and isoflucypram-cyclopropyl-pyrazole-carboxamide (7.2–10.7 percent TRR [0.040 mg eq/kg in the low dose experiment and 0.077 mg eq/kg in the high dose experiment]). Up to 32 unknown metabolites were characterised based on chromatographic behaviour, individually accounting for ≤ 19.9 percent TRR (≤ 0.064 mg eq/kg). Radioactivity remaining in the PES was ≤ 7.4 percent TRR (≤ 0.073 mg eq/kg).

In potato tubers, parent isoflucypram was the main residue representing 69–86 percent TRR (0.029–0.056 mg/kg). The only remaining identified metabolite was isoflucypram-cyclopropyl-pyrazole-carboxamide (11 percent TRR [0.007 mg eq/kg]). There were no unidentified peaks and no radioactivity remaining in the PES.

Plant metabolism summary and conclusions

The metabolism of isoflucypram was evaluated in tomatoes, wheat, soya bean, and oilseed rape following 2–3 foliar directed applications and in potatoes following seed treatment.

Isoflucypram was the only residue observed in all food commodities (i.e., tomatoes, wheat grain, soya bean seed, oilseed rape seed, and potato tubers) representing 68–98 percent TRR. In oilseed rape intermediate harvest and mature plants, isoflucypram was partially metabolised (72–88 percent TRR) with low levels (up to 4.6 percent TRR) of glucose-malonic acid and glycine-malonic acid conjugates of isoflucypram-propanol, isoflucypram-2-propanol, and isoflucypram-hydroxyphenyl observed. Parent isoflucypram was metabolised to a further extent in wheat hay and straw (50–64 percent TRR) with isoflucypram-propanol and isoflucypram-desmethyl-propanol, as well as their glycine and glycine-malonic acid conjugates, observed up to 10 percent TRR. Hydrolysis data suggest that the conjugated metabolites in oilseed and wheat commodities are hydrolysed to their free forms.

In soya bean forage, hay, and straw, isoflucypram was metabolised to a further extent, representing 10–70 percent TRR. Isoflucypram-desfluoro-Cys-MA, isoflucypram-desfluoro-homoGSH, isoflucypram-desfluoro-mercapto-lactic acid-propyl-OH-Glyc, isoflucypram-desfluoro-mercapto-lactic acid-Glyc, and isoflucypram-desfluoro-mercapto-lactic acid-OH were observed in concentrations up to 23 percent TRR. All identified soya bean metabolites follow a similar pathway resulting in defluorination and conjugation with a sulfur containing moiety. The Meeting noted that hydrolysis data for the soya bean extracts would be informative for concluding whether or not the metabolites observed in soya bean feeds are of interest for animal dietary burden calculations.

When isoflucypram was applied to potato seed pieces prior to planting, near full metabolism of parent was observed (2–7 percent TRR remaining) with the glycine-malonic acid conjugates of isoflucypram-OH-phenyl and isoflucypram-2-propanol observed up to 29 percent TRR. Following application to potato seed pieces prior to planting, parent isoflucypram represented 69–86 percent TRR in mature tubers with isoflucypram-cyclopropyl-pyrazole-carboxamide accounting for up to 11 percent TRR.

Environmental fate

Hydrolytic Degradation

Hydrolytic degradation of pyrazole-labelled isoflucypram was investigated in closed sterile aqueous buffer solutions at pH 4, 7, and 8.9 in the dark at 50.0 °C for 7 days. Isoflucypram was stable at all pH values, and no DT_{50} values were calculated. Hydrolytic degradation is unlikely to contribute to the degradation of isoflucypram under typical environmental conditions.

Photodegradation

In the neutral buffer solution, isoflucypram degraded to 91 and 79 percent of the initial concentration after 150 days of irradiation

Aerobic degradation in soil

The route and rate of degradation of pyrazole (n=8) and phenyl (n=7) labelled isoflucypram were studied in laboratory soil under aerobic conditions in the dark at 20 °C for 120–125 days. The application rate was 75 g ai/ha for most trials, except for two of the pyrazole labelled trials (50 g ai/ha).Trials conducted at 75 g ai/ha were evaluated for degradation rate with the KinGUII software under the FOCUS guideline. Isoflucypram followed single first order (SFO) degradation in all trials, with DT_{50}s ranging from 223 to 875 days (geometric mean = 446 days).

Isoflucypram-carboxylic acid was the only prominent degradate identified, increasing with time to a maximum of 10.9 percent of the applied radioactivity (AR) 120 days after treatment (DAT).

The degradation of isoflucypram-carboxylic acid was investigated in five different laboratory soils under aerobic conditions in the dark at 20°C for 120 days at a nominal rate of 4.12 µg/100 g dry soil. Dissipation kinetics were modelled with the KinGUII software under the FOCUS guideline. Isoflucypram-carboxylic acid dissipated following double first order in parallel (DFOP; n=4) or SFO (n=1) kinetics with DT_{50}s ranging from 17 to 27 days (geometric mean = 22 days).

Terrestrial Field Dissipation

The Meeting received 12 terrestrial field dissipation studies. Isoflucypram was applied to bare soil plots one or two times at rates ranging from 100–500 g ai/ha. Soil samples were collected at various intervals from 749 to 832 days after application to a maximum depth of 60–120 cm. Collected soil was partitioned into 10–15 cm horizons. The rate of isoflucypram degradation was evaluated with the KinGUII software under the FOCUS guideline. The Meeting excluded three DT_{50} values due to low r^2 and/or high chi^2 error values. The remaining acceptable DT_{50} values (first and second applications calculated separately, where applicable) ranged from 9 to 177 days (geometric mean = 46 days). One trial was best fit by SFO, six trials were best fit by DFOP, and four trials were best fit by first order multi-component (FOMC) dissipation.

Isoflucypram-carboxylic acid comprised up to 3.6 percent of the applied dose. Isoflucypram-carboxylic acid was only quantifiable in the top soil segment and peaked in concentration at 30–241 days after the first application.

Confined rotational crop studies

Isoflucypram was applied to bare sandy loam soil as a single spray application at a rate of 198–210 g ai/ha (2.6–2.8× the seasonal cGAP for field crops). Turnips, Swiss chard, and wheat were sown into the treated soil 30, 140, and 287 days after application. Immature samples of Swiss chard (BBCH 45), wheat forage (BBCH 29), and wheat hay (BBCH 75–83) and mature samples of turnip roots, turnip tops, Swiss chard, wheat straw, and wheat grain were harvested and analysed for TRR.

TRR in pyrazole labelled studies was consistently higher than phenyl labelled studies by a factor of 1.1× (immature Swiss chard) to 6.2× (wheat straw). In general, TRR was highest at the second plant-back interval (PBI), followed by the third PBI, and lowest at the first PBI. One notable exception is pyrazole-labelled wheat straw which followed an increasing trend with increasing PBIs. Across both studies, TRR was lowest (0.003–0.011 mg eq/kg) in turnip roots, wheat grain, and phenyl-labelled turnip tops; slightly higher (0.015–0.078 mg eq/kg) in high-water components of crops (pyrazole-labelled turnip tops, Swiss chard, and wheat forage); and highest (0.036–0.34 mg eq/kg) in dry components of crops (wheat hay and straw). The following samples incurred low TRR and were not extracted for residue identification: turnip tops (phenyl label, all PBIs), turnip roots (all samples), wheat grain (phenyl label, all PBIs; pyrazole label, 30-day PBI).

ACN/water extraction released 53–54 percent TRR from wheat grain and 78–98 percent TRR from the other matrices. The PES of wheat hay, wheat straw, and wheat grain were further extracted with microwave assistance with optional dioxane and 5 mol/L HCl, releasing an additional 5.5–32 percent TRR.

In the phenyl study, parent isoflucypram was the only structure identified in extracts prior to hydrolysis, accounting for ≤ 17 percent TRR (≤ 0.004 mg/kg) and decreasing in concentration with increasing PHIs. Up to 17 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 31 percent TRR (≤ 0.009 mg eq/kg). Up to 11.7 percent TRR (≤ 0.007 mg eq/kg) remained in the PES.

In hydrolysed immature Swiss chard extract at the 30-day PBI, prominent metabolites included isoflucypram-propanol and isoflucypram-carboxylic acid accounting for 11 percent TRR (0.003 mg eq/kg) and 37 percent TRR (0.011 mg eq/kg), respectively. In hydrolysed mature Swiss chard extract at the 30-day PBI, prominent metabolites included isoflucypram, isoflucypram-propanol, and isoflucypram-carboxylic acid accounting for 17 percent TRR (0.003 mg eq/kg), 17 percent TRR (0.004 mg eq/kg), and 41 percent TRR (0.008 mg eq/kg), respectively. In hydrolysed wheat straw extract at the 30-day PBI, the only prominent metabolite was isoflucypram-carboxylic acid (11 percent TRR; 0.006 mg eq/kg).

In the pyrazole study, all identified metabolites contained only the pyrazole ring. Many metabolites shared structural similarities with sugar and/or amino acid conjugates. The Meeting did not receive hydrolysis data for the extracts. The Meeting assigned metabolites to one of the following groups based on structural similarities:

- Compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide structure (including isoflucypram-cyclopropyl-pyrazole-carboxamide-Ala and isoflucypram-cyclopropyl-pyrazole-carboxamide-Glyc isomers 1 and 2);
- Compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH structure (including isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys and isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH);
- Compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide-OH structure (including isoflucypram-cyclopropyl-pyrazole-carboxamide-OH-lactic acid isomers 1 and 2);
- Compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto structure (including isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc and isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto-Glyc-MA).

The following metabolites are not included in the above groups and are summarised individually:

- BCS-CR60082;
- Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala.

In turnip tops grown in rotation, parent isoflucypram was a minor residue representing 4.8 percent TRR (0.001 mg/kg) at the 30 day PBI and was not detected at other PBIs. Up to 9 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 9.5 percent TRR (≤ 0.003 mg eq/kg). Radioactivity remaining in the PES was ≤ 7.7 percent TRR (≤ 0.002 mg eq/kg).

In rotational immature and mature Swiss chard, isoflucypram was a minor component representing ≤ 6 percent TRR (≤ 0.002 mg/kg). Compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH structure accounted for 23–57 percent TRR (0.015–0.020 mg eq/kg in immature Swiss chard and 0.013–0.015 mg eq/kg in mature Swiss chard). Up to 22 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 11 percent TRR (≤ 0.007 mg eq/kg). Radioactivity remaining in the PES was ≤ 7.6 percent TRR (≤ 0.004 mg eq/kg).

In rotational wheat forage, parent isoflucypram was a minor component representing ≤ 7 percent TRR (≤ 0.003 mg/kg). Compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide structure accounted for 19–30 percent TRR (0.009–0.023 mg eq/kg) and compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH structure accounted for 8.3–13 percent TRR (0.004–0.010 mg eq/kg). Up to 12 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤9.0 percent TRR (≤ 0.007 mg eq/kg).

Radioactivity remaining in the PES was ≤9.0 percent TRR (≤ 0.007 mg eq/kg). In rotational wheat hay, isoflucypram was not detected. Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala accounted for ND-4.8 percent TRR (ND-0.011 mg eq/kg), compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto accounted for 5.5–7.1 percent TRR (0.008–0.012 mg eq/kg), compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide structure accounted for 15–23 percent TRR (0.023–0.051 mg eq/kg), compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH structure accounted for 5.2–12 percent TRR (0.006-0.026 mg eq/kg), and compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide-OH structure accounted for 3.2–12 percent TRR (0.007–0.022 mg eq/kg). Up to 17 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 4.3 percent TRR (≤ 0.010 mg eq/kg). Radioactivity remaining in the PES was ≤ 4.3 percent TRR (≤ 0.010 mg eq/kg).

In rotational wheat hay, parent isoflucypram was not recovered. Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala accounted for ND-4.8 percent TRR (ND-0.011 mg eq/kg), compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto accounted for 5.5–7.1 percent TRR (0.008–0.012 mg eq/kg), compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide structure accounted for 15–23 percent TRR (0.023–0.051 mg eq/kg), compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH structure accounted for 5.2–12 percent TRR (0.006–0.026 mg eq/kg), and compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide-OH structure accounted for 3.2–12 percent TRR (0.007–0.022 mg eq/kg). Up to 17 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤4.3 percent TRR (≤ 0.010 mg eq/kg). Radioactivity remaining in the PES was ≤ 4.3 percent TRR (≤ 0.010 mg eq/kg).

In rotational wheat straw, parent isoflucypram was not detected. BCS-CR60082 accounted for 2.8–7.0 percent TRR (0.007–0.020 mg eq/kg), isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto accounted for 4.9–9.2 percent TRR (0.012–0.018 mg eq/kg), compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide structure accounted for 8.4–16 percent TRR (0.011–0.039 mg eq/kg), compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH structure accounted for 9.2–11 percent TRR (0.012–0.035 mg eq/kg), and compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide-OH structure accounted for 5.9–12 percent TRR (0.015–0.037 mg eq/kg). Up to 18 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 6.4 percent TRR (≤ 0.014 mg eq/kg). Radioactivity remaining in the PES was ≤ 6.4 percent TRR (≤ 0.014 mg eq/kg).

In rotational wheat grain, parent isoflucypram was not detected. Isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala accounted for 13 percent TRR (0.002 mg eq/kg). Three unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 20 percent TRR (≤ 0.002 mg eq/kg). Radioactivity remaining in the PES was ≤ 20 percent TRR (≤ 0.002 mg eq/kg).

Field Rotational Crops

The Meeting received four independent rotational crop field trials following one broadcast application of an EC formulation of isoflucypram at 180 g ai/ha (2.4× the seasonal cGAP for cereal grains). Rotational crops of barley, carrot or turnips, and lettuce were planted in treated plots at PBIs of 21–34 days, 100–201 days, and 299–370 days and analysed for isoflucypram and BCS-CR60082. Soil samples (0–30 cm depth) were taken at from each plot at each PBI and analysed for isoflucypram and isoflucypram-carboxylic acid.

Average residues of isoflucypram were < 0.01 mg/kg in all samples, except carrot tops at one trial at the 106-day PBI (average residue of 0.066 mg/kg). Residues of BCS-CR60082 were < 0.01 mg eq/kg in all samples.

In soil samples, average residues of isoflucypram were between 0.011 and 0.049 mg/kg and average residues of isoflucypram-carboxylic acid were ≤ 0.002 mg eq/kg. Isoflucypram residues generally decreased with increased time between application and soil collection.

Environmental fate and rotational crops summary and conclusions

Isoflucypram is stable to hydrolytic degradation. In aerobic metabolism and terrestrial field dissipation studies, isoflucypram-carboxylic acid was observed at up to 10.9 percent AR or 3.6 percent, respectively. Neither isoflucypram nor isoflucypram-carboxylic acid are persistent in the soil, with geometric mean DT_{50}s of 46 days (field dissipation) and 22 days (aerobic metabolism), respectively.

While isoflucypram-carboxylic acid is a prominent soil metabolite, it is not observed in rotational crops as demonstrated by the confined rotational crops study.

In the confined rotational crops study, only the metabolite group containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH structure was detected at levels higher than 0.01 mg eq/kg in food commodities (up to 0.015 mg eq/kg in mature Swiss chard). The Meeting concluded that, at the cGAP considered by the current Meeting, this group of metabolites may be present at an estimated residue level of 0.0054 mg eq/kg in rotational leafy crops.

The metabolites or groups of metabolites that exceeded 0.01 mg eq/kg in livestock feed items were isoflucypram-desfluoro-cyclopropyl-pyrazole-carboxamide-Ala (up to 0.011 mg eq/kg), compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-mercapto structure (up to 0.018 mg/kg), compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide structure (up to 0.051 mg eq/kg), compounds containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH structure (up to 0.035 mg eq/kg), and compounds containing the isoflucypram-cyclopropyl-pyrazole-carboxamide-OH structure (up to 0.037 mg eq/kg). Because the confined rotational crop trials were conducted at approximately 2.7× the cGAP considered by the current Meeting, the Meeting concluded that residues of these metabolites or groups of metabolites would not be expected to contribute significantly to the livestock dietary burdens and are not expected to be detected in animal tissues.

The field rotational crops study analysed residues of isoflucypram and BCS-CR60082. Residues of BCS-CR60082 were < 0.01 mg eq/kg in all crops, except isoflucypram in carrot tops at one trial (0.066 mg/kg). The Meeting noted that the study report attributed this value to spray drift from an application to an adjacent field rather than uptake from the soil. The Meeting concluded that residues of isoflucypram and BCS-CR60082 are not expected in rotational crops.

The Meeting noted that, should a higher cGAP be received in the future, the expectation of residues of the pyrazole metabolites in rotational crops may need to be re-evaluated.

Animal metabolism

The Meeting received studies describing the metabolism of isoflucypram in laboratory rats, lactating goats and laying hens.

Rats

The metabolism of isoflucypram in rats was reviewed in the framework of the toxicological evaluation by the WHO Core Assessment Group of the 2022 JMPR.

Lactating goats

Lactating goats were orally dosed with pyrazole- and phenyl-labelled isoflucypram at 45 ppm or 21 ppm (dry feed) per day in the diet for five consecutive days. Approximately 0.03–0.06 percent of the administered dose (AD) was secreted with milk, correlating to TRRs ranging from 0.008–0.021 mg eq/kg. Per-day pooled milk samples reached a plateau after three days.

Approximately six hours after the final dose, animals were sacrificed and TRRs were determined in liver, kidney, muscle (pooled round and loin), and fat (pooled perirenal and omental). The sum of radioactive residues in edible fractions was 0.27–0.72 percent AD, correlating to TRRs of 0.35–0.72 mg eq/kg in liver, 0.18–0.19 mg eq/kg in kidney, 0.10 mg eq/kg in fat, and 0.011–0.038 mg eq/kg for muscle

Extraction with ACN/water and THF (for pyrazole-labelled milk only) released 89–100 percent TRR. Various matrices were partitioned against n-heptane resulting in low amounts of radioactivity in the organic phase (≤ 1.4 percent; ≤ 0.002 mg eq/kg). Liver and pyrazole-labelled muscle extracts were additionally extracted with microwave assistance and HCl (liver only), together releasing an additional 3.5–4.9 percent TRR.

In plateau-level milk, isoflucypram was the predominant residue, accounting for 33–34 percent TRR (0.004–0.005 mg/kg). The only prominent metabolite was isoflucypram-2-propanol (5.0–20 percent TRR [0.001–0.003 mg eq/kg]). Up to five unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 39 percent TRR (≤ 0.005 mg eq/kg). Radioactivity remaining in the PES was ≤ 1.5 percent TRR (< 0.001 mg eq/kg).

The majority of the radioactivity (approximately 87 percent TRR) in milk remained in the skimmed milk fraction in the pyrazole study, whereas it was evenly distributed between the skimmed milk and cream fractions in the phenyl study.

In muscle, isoflucypram was the predominant residue, accounting for 22 percent TRR (0.002–0.008 mg/kg). Prominent metabolites were isoflucypram-2-propanol (14–18 percent TRR, 0.002–0.006 mg eq/kg) and isoflucypram-propanol (9.0–10 percent TRR, 0.001–0.004 mg eq/kg). Up to 10 unknown peaks were characterised based on chromatographic behaviour between the conventional and exhaustive extract, individually accounting for ≤ 25 percent TRR (≤ 0.003 mg eq/kg). Radioactivity remaining in the PES was ≤ 6.4 percent TRR (≤ 0.002 mg eq/kg).

In fat, only the pyrazole label was studied due to lack of fat on the phenyl labelled goat, although no abnormality on feed consumption, weight, or common behaviour was observed. Isoflucypram was the predominant residue, at 59 percent TRR (0.061 mg/kg). The only prominent metabolite was isoflucypram-2-propanol, accounting for 17 percent TRR (0.017 mg eq/kg). Four unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 7.5 percent TRR (≤ 0.008 mg eq/kg). Radioactivity remaining in the PES was 1.7 percent TRR (0.002 mg eq/kg).

In liver, isoflucypram accounted for 3.5–5.3 percent TRR (0.018–0.025 mg/kg). Prominent metabolites were isoflucypram-2-propanol-GlucA (13–14 percent TRR, 0.045–0.099 mg eq/kg), isoflucypram-propanol-GlucA isomer 1 (8.8–13 percent TRR, 0.031–0.094 mg eq/kg), isoflucypram-propanol-GlucA isomer 2 (5.9–7.7 percent TRR, 0.021–0.055 mg eq/kg), isoflucypram-carboxylic acid (4.2–8.9 percent TRR, 0.015–0.064 mg eq/kg), isoflucypram-propanol (4.9–5.8 percent TRR, 0.017–

0.042 mg eq/kg), isoflucypram-2-propanol (2.6–2.8 percent TRR, 0.010–0.019 mg eq/kg), and isoflucypram-lactic acid (1.3–1.6 percent TRR, 0.005–0.011 mg eq/kg). Up to 32 unknown peaks were characterised based on chromatographic behaviour between the conventional, exhaustive ACN/water and HCl extract, individually accounting for ≤ 6.7 percent TRR (≤ 0.029 mg eq/kg). Radioactivity remaining in the PES was ≤ 8.2 percent TRR (≤ 0.036 mg eq/kg).

Liver extracts were enzymatically cleaved with ß-glucuronidase and arylsulfatase (20 hours at 37 °C) resulting in decreased concentrations of conjugates accompanied by increase concentrations of respective aglycones. Isoflucypram accounted for 4.7–6.1 percent TRR (0.016–0.044 mg/kg). Prominent metabolites were isoflucypram-propanol (18–21 percent TRR, 0.064–0.15 mg eq/kg), isoflucypram-2-propanol (12–13 percent TRR, 0.047–0.085 mg eq/kg), isoflucypram-carboxylic acid (10–12 percent TRR, 0.033–0.084 mg eq/kg), isoflucypram-desmethyl-propanol (4.3–4.6 percent TRR, 0.015–0.033 mg eq/kg), and isoflucypram-desmethyl-carboxylic acid (1.5–2.1 percent TRR, 0.005–0.016 mg eq/kg).

In kidney, isoflucypram was a minor compound accounting for 1.6–2.7 percent TRR (0.003–0.005 mg/kg). Prominent metabolites were isoflucypram-carboxylic acid (6.8–18 percent TRR, 0.012–0.034 mg eq/kg), isoflucypram-propanol-GlucA isomer 2 (7.0–8.6 percent TRR, 0.013–0.016 mg eq/kg), isoflucypram-lactic acid (4.2–6.1 percent TRR, 0.008–0.012 mg eq/kg), isoflucypram-propanol (2.5–5.6 percent TRR, 0.004–0.011 mg eq/kg), isoflucypram-propenol-GlucA (3.6–6.2 percent TRR, 0.007–0.011 mg eq/kg), and isoflucypram-N-methyl-pyrazole-carboxylic acid (5.8 percent TRR, 0.011 mg eq/kg). Up to 21 unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 9.1 percent TRR (≤ 0.017 mg eq/kg). Radioactivity remaining in the PES was ≤ 7.8 percent TRR (≤ 0.015 mg eq/kg).

Kidney extracts were enzymatically cleaved with ß-glucuronidase and arylsulfatase (20 hours at 37 °C) resulting in decreased concentrations of conjugates accompanied by increased concentrations of respective aglycones. Isoflucypram accounted for 1.3–2.6 percent TRR (0.002–0.005 mg/kg). Prominent metabolites were isoflucypram-carboxylic acid (13–23 percent TRR, 0.023–0.044 mg eq/kg), isoflucypram-propanol (11–20 percent TRR, 0.020–0.037 mg eq/kg), isoflucypram-2-propanol (5.5–6.7 percent TRR, 0.010–0.013 mg eq/kg), isoflucypram-desmethyl-carboxylic acid (4.2–6.4 percent TRR, 0.008–0.012 mg eq/kg) and isoflucypram-desmethyl-propanol (4.6–5.0 percent TRR, 0.009 mg eq/kg).

Laying Hen

Laying hens were orally dosed with labelled isoflucypram at 17 ppm and 18 ppm (dry feed) per day for 14 consecutive days. Eggs were collected daily and 0.12–0.14 percent of the AD was recovered corresponding to 0.029–0.066 mg eq/kg TRR. TRR in eggs plateaued on Day 4–6.

Hens were sacrificed approximately six hours after the final dose. Radioactive residues in the tissues were approximately 0.22–0.24 percent AD, corresponding to TRRs of 0.37 mg eq/kg in liver, 0.36–0.39 mg eq/kg in kidney, 0.075–0.11 mg eq/kg in skin, 0.042–0.047 mg eq/kg in subcutaneous fat, 0.029 mg eq/kg in leg muscle, and 0.017–0.018 mg eq/kg in thorax muscle.

Extraction with ACN/water released 84–93 percent TRR. Liver PES underwent extractions with microwave assistance and HCl which together released an additional 15–16 percent TRR. Extracts from various matrices were partitioned with n-heptane resulting in low amounts of radioactivity in the organic phase (≤1.3 percent TRR; 0.001 mg eq/kg).

In eggs, parent isoflucypram was a minor component accounting for 3.7–6.4 percent TRR (0.002–0.003 mg/kg). Prominent metabolites were isoflucypram-propanol (34–35 percent TRR [0.017–0.018 mg eq/kg]) and isoflucypram-desmethyl-propanol (22–23 percent TRR [0.011 mg eq/kg]). Up to five unknown peaks were characterised based on chromatographic behaviour, individually accounting for

≤ 7.7 percent TRR (≤ 0.004 mg eq/kg). Radioactivity remaining in the PES was ≤ 7.2 percent TRR (≤ 0.004 mg eq/kg).

In muscle (thorax and leg analysed separately), parent compound was a minor component accounting for ≤2.9 percent TRR (0.001 mg/kg). Remaining prominent metabolites included isoflucypram-desmethyl-propanol (21–30 percent [0.004–0.009 mg eq/kg]), isoflucypram-desmethyl-1,2-propandiol (14–22 percent TRR [0.003–0.004 mg eq/kg]), isoflucypram-desmethyl-carboxylic acid (12–20 percent TRR [0.002–0.006 mg eq/kg]), and isoflucypram-carboxylic acid (6.6–11 percent TRR [0.001–0.003 mg eq/kg]). Up to four unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 19.5 percent TRR (≤ 0.004 mg eq/kg). Radioactivity was successfully extracted as demonstrated by ≤ 8.2 percent TRR (≤ 0.002 mg eq/kg) remaining in the PES.

In fat, isoflucypram was the predominant component representing 20–24 percent TRR (0.009–0.010 mg/kg). Prominent metabolites included isoflucypram-propanol (6.5–12 percent TRR [0.003–0.005 mg eq/kg]) and isoflucypram-desmethyl-propanol (8.0–10 percent TRR [0.004 mg eq/kg]). Up to seven unknown peaks were characterised based on chromatographic behaviour, individually accounting for ≤ 11.2 percent TRR (≤ 0.005 mg eq/kg). Radioactivity remaining in the PES was ≤ 8.2 percent TRR (≤ 0.004 mg eq/kg).

Parent compound was not detected in the liver. Prominent metabolites were isoflucypram-desmethyl-carboxylic acid (14–22 percent TRR [0.053–0.082 mg eq/kg]), isoflucypram-carboxylic acid (5.8–12 percent TRR [0.022–0.044 mg eq/kg]), isoflucypram-desmethyl-1,2-propandiol-N-GlucA (5.4–9.2 percent TRR [0.020–0.034 mg eq/kg]), isoflucypram-desmethyl-propanol-N-GlucA (6.1–11 percent TRR [0.023–0.040 mg eq/kg]), isoflucypram-desmethyl-1,2-propandiol (5.6–6.9 percent TRR [0.021–0.025 mg eq/kg]), isoflucypram-desmethyl-propanol (2.7–5.3 percent TRR [0.010–0.020 mg eq/kg]), and isoflucypram-desmethyl-2-propanol-N-GlucA (2.5–3.0 percent TRR [0.009–0.011 mg eq/kg]). Up to 27 unknown peaks were identified based on chromatographic behaviour in the conventional extract, exhaustive ACN/water extract, and exhaustive ACN/HCl extract, individually accounting for ≤ 8.4 percent TRR (≤ 0.031 mg eq/kg). Radioactivity remaining in the PES was ≤ 0.1 percent TRR (< 0.001 mg eq/kg).

Liver extracts were enzymatically cleaved with β-glucuronidase and arylsulfatase (96 hours at 37 °C). Parent compound was not identified in the hydrolysed extract. Prominent metabolites included isoflucypram-desmethyl-carboxylic acid (17–26 percent TRR [0.064–0.096 mg eq/kg]), isoflucypram-desmethyl-propanol (14–15 percent TRR [0.053–0.054 mg eq/kg]), isoflucypram-carboxylic acid (9.0–17 percent TRR [0.033–0.062 mg eq/kg]), and isoflucypram-desmethyl-1,2-propandiol (11–12 percent TRR [0.041–0.043 mg eq/kg]),

Animal metabolism summary and conclusions

The distribution and elimination of isoflucypram appeared to be similar in rats, goat and hens. Radioactivity was consistently observed at highest levels in the liver, followed by lower (rat and goat) or similar (hen) levels in kidney, followed by fat. Muscle had the lowest level of radioactivity in the goat and hen.

Isoflucypram was metabolized to a further extent in hens than in goats in all tissues. In fat, muscle, milk, and eggs, low overall radioactivity was observed and metabolites were generally unconjugated. In these matrices, the metabolic pathways differed between the goat and hen species with formation of isoflucypram-propanol and isoflucypram-2-propanol observed in ruminant fat, muscle, and milk (up to 20 percent TRR), and formation of isoflucypram-propanol, isoflucypram-desmethyl-propanol, isoflucypram-carboxylic acid, isoflucypram-desmethyl-carboxlylic acid, and isoflucypram-desmethyl-1,2-propandiol (as well as its N-GlucA conjugate) observed in hen fat, muscle, and egg (up to 35 percent TRR).

Goat liver/kidney and hen liver contained little to no parent isoflucypram and a higher levels of conjugated metabolites. Isoflucypram-carboxylic acid was the only prominent metabolite common to all three matrices. Isoflucypram-propanol (free and/or GlucA conjugates) was prevalent in goat liver/kidney and trace in kidney liver. Isoflucypram-desmethyl-propanol (free and N-GlucA conjugate) and isoflucypram-desmethyl-carboxylic acid were prevalent in hen liver and trace in goat liver/kidney. In goat liver and kidney, common metabolites included isoflucypram-2-propanol (free and GlucA conjugate) and isoflucypram-lactic acid. The metabolites isoflucypram-N-methyl-pyrazole-carboxylic acid and isoflucypram-propenol-GlucA were found only in goat kidney. The metabolites isoflucypram-desmethyl-2-propanol-N-GlucA and isoflucyrapm-1,2-propandiol (free and N-GlucA conjugate) were found only in hen liver.

Methods of analysis

The Meeting received several analytical methods for quantitation of isoflucypram and various metabolites.

Plants

Method 01475 analysed isoflucypram and BCS-CR60082. Successful method validation was performed on tomato fruit, orange fruit, rape seed, wheat grain/straw, and dry bean seed. Samples are extracted twice with ACN/water (dry commodities left to soak in water for 20 minutes before addition of ACN in the first extraction). Internal standards are added and extracts are analysed by LC-MS/MS. The LOQ is 0.01 mg/kg for both analytes in all matrices. The ability of the method to extract incurred residues was successfully demonstrated based on comparison with samples from the primary and confined rotational crop metabolism studies.

Method LN-002-P16-01 uses the same extraction procedure as Method 01475 but only analyses isoflucypram. The method was successfully validated in tomato fruit, orange fruit, wheat grain, soya bean seed, and canola seed. The LOQ is 0.01 mg/kg in all matrices.

Method 01564 analyses isoflucypram, isoflucypram-desmethyl-propanol (free and conjugated), and isoflucypram-propanol (free and conjugated). Successful method validation was performed on wheat green material, grain, and straw. Samples are spiked with isotopic isoflucypram-desmethyl-propanol internal standard, 5 mol/L HCl is added, and samples are heated at 98 °C for 30 minutes followed by two extractions with ACN/water. Isotopic internal standards for isoflucypram and isoflucypram-propanol are added to the combined extracts for LC-MS/MS analysis. The LOQ is 0.01 mg/kg in all matrices. The ability of the method to extract residues and hydrolyse conjugates was successfully demonstrated based on comparison with samples from the wheat hay and straw metabolism studies.

Method 01520 uses the same extraction procedure as Method 01475; however, the only analyte is isoflucypram and the method does not use an internal standard. Successful method validation and Independent Laboratory Validation data were provided on tomato fruit, orange fruit, wheat grain, coffee green bean, oilseed rape seed, and dry bean seed. The LOQ is 0.01 mg/kg in all matrices.

The QuEChERS multi-residue successfully extracted incurred residues of isoflucypram in tomato fruit, soya been seed/straw, and oilseed rape intermediate harvest/seed matrices based on comparison with samples from the crop metabolism studies. Extraction efficiency of the QuEChERS multi-residue method was unsuccessful in wheat hay/straw/grain and soya bean forage/hay matrices.

Animals

Method 01511 analyses parent isoflucypram as well as free isoflucypram-2-propanol, isoflucypram-carboxylic acid, isoflucypram-propanol, isoflucypram-desmethyl-propanol, and isoflucypram-desmethyl-carboxylic acid, in animal matrices. The method also quantitates free and conjugated isoflucypram-propanol and isoflucypram-2-propanol in cow liver and kidney, and free and conjugated isoflucypram-desmethyl-propanol in hen liver.

For free analytes, samples are extracted with ACN/water, isotopic internal standards are added and samples are analysed by LC-MS/MS. Successful method validation was performed in eggs, milk, cow muscle/fat/liver/kidney, and hen liver. For free and conjugated analytes, samples are extracted with ACN/water, allowed to hydrolyse with β-glucoronidase and arylsulfatase at 37 °C, cleaned up by SPE, and analysed by LC-MS/MS. The LOQ is 0.005 mg eq/kg for milk, cream, and whey, and 0.01 mg/kg for all other matrices. The extraction solvent was the same as that used in the animal metabolism studies (≥ 92 percent TRR extracted).

The QuEChERS multi-residue method (Method 01300/M034) was successfully validated for milk, eggs, cattle muscle/fat/liver/kidney, and hen muscle. The LOQ is 0.005 mg eq/kg for milk, cream, and whey, and 0.01 mg/kg for all other matrices. Independent Laboratory Validation data were also submitted. The ability of the method to extract incurred residues was successfully demonstrated based on comparison with aged samples from the goat metabolism study.

In conclusion, methods are available for the analysis of isoflucypram and relevant metabolites in crop and animal matrices. The QuEChERS method is suitable for analysing isoflucypram in high-water and high-oil content crops and animal matrices but not suitable for high-starch content crops, cereal and oilseed animal feeds.

Stability of pesticide residues in stored analytical samples

The Meeting received two storage stability studies for isoflucypram and its metabolites in plants fortified at 0.1 or 0.2 mg/kg. Isoflucypram and BCS-CR60082 are stable during freezer storage (≤-18 °C) for a period of at least 24–25 months in tomato fruit, bean dry seed, wheat grain, rape seed, and orange fruit. Isoflucypram and BCS-CR60082 are also stable following frozen storage for at least 25–26 months at -18 °C followed by six days of storage at -1 ± 2 °C. Isoflucypram-desmethyl-propanol and isoflucypram-propanol are stable in/on wheat grain, green material, and straw under frozen storage conditions (<-18 °C) for at least 30 months. The storage stability data support the storage durations and conditions of the submitted studies. All plant samples in the submitted studies were stored frozen for durations supported by the submitted studies. Storage stability data were not submitted for animal matrices. As dairy cow and laying hen matrices from the farm animal feeding studies were stored frozen for a maximum of 30 days, storage stability data are not needed.

Definition of the residue

Plant commodities

In plant metabolism studies, parent isoflucypram was a major residue in tomato, wheat, soya bean, oilseed rape, and potato tubers grown from treated potato seed pieces (10–98 percent TRR).

A suitable analytical method using LC-MS/MS is available to determine residues of isoflucypram in plant commodities.

The Meeting concluded that isoflucypram was a suitable marker for enforcement purposes.

In deciding which additional compounds should be included in the residue definition for risk assessment, the Meeting considered the likely occurrence of the compounds and the toxicological properties of the candidates. In metabolism studies, no metabolites were identified in the tomato, wheat grain, soya bean seed, oilseed rape seed, or potato tubers. Isoflucypram-propanol (free and conjugated) and isoflucypram-desmethyl-propanol (free and conjugated) were considered based residues in the wheat plants in the metabolism study and quantifiable residues in the wheat grain in supervised residue trials. Additionally, the Meeting considered the group of metabolites containing the isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH structure based on residues > 0.01 mg eq/kg in the confined rotational crop metabolism study.

Based on toxicological properties, isoflucypram-propanol (free and conjugated) is assumed to be covered by the isoflucypram HBGVs. In supervised residue trials matching the cGAP, isoflucypram-propanol (free and conjugated) was observed in cereal grains in three of 39 trials at concentrations of 0.011, 0.014, and 0.019 mg eq/kg with corresponding isoflucypram residues of 0.019, 0.01, and 0.01 mg/kg. Due to low occurrence of quantifiable residues, the Meeting concluded that isoflucypram-propanol should be excluded from in the residue definition for dietary risk assessment.

The Meeting concluded that isoflucypram-desmethyl-propanol (free and conjugated) could be assessed using the threshold of toxicological concern (TTC) approach Cramer Class III (1.5 µg/kg bw/day), results discussed under *animal commodities*).

The Meeting concluded that the metabolites isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys and isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH could be assessed using the TTC approach for genotoxicity (0.0025 µg/kg bw/day). As the metabolites are conjugates of the same structure, the Meeting combined exposures from both metabolites for the TTC assessment.

The Meeting estimated dietary exposures for isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys and isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH of 0.0004–0.0107 µg/kg bw per day, above the TTC for a compound with potential of genotoxicity.

Animal commodities

In the cattle feeding study, isoflucypram was detected in all matrices except kidney. Although isoflucypram was not observed in the hen feeding study, it was observed in the laying hen metabolism study as a major component of fat (20–24 percent TRR) and a minor component of eggs and muscle (2.3–6.4 percent TRR).

A suitable analytical method using LC-MS/MS is available to determine residues of isoflucypram in animal commodities.

The Meeting concluded that isoflucypram was a suitable marker for enforcement purposes.

In deciding which additional compounds should be included in the residue definition for risk assessment, the Meeting considered the likely occurrence of the compounds and the toxicological properties of the candidates. The metabolites isoflucypram-propanol (free and conjugated), isoflucypram-desmethyl-propanol (free and conjugated), isoflucypram-2-propanol (free and conjugated), isoflucypram-carboxylic acid, isoflucypram-desmethyl-carboxylic acid, isoflucypram-lactic acid, isoflucypram-desmethyl-1,2-propandiol, isoflucypram-desmethyl-1,2-propandiol-N-GlucA, isoflucypram-N-methyl-pyrazole-carboxylic acid, isoflucypram-propenol-Gluc-A, and isoflucypram-desmethyl-2-propanol-N-GlucA were > 10 percent TRR and/or > 0.01 mg eq/kg in the lactating goat and laying hen metabolism studies.

Isoflucypram-carboxylic acid, isoflucypram-desmethyl-carboxylic acid, and isoflucypram-propanol (free and conjugated) are considered to be covered by the HBGV for isoflucypram. These metabolites were analysed in the cattle and hen feeding studies and each were quantifiable in at least two matrices, representing a significant portion of the residues covered by the HBGV of isoflucypram. The Meeting concluded that these metabolites should be considered in the residue definition for dietary risk assessment.

Toxicological data are not available for isoflucypram-lactic acid, isoflucypram-desmethyl-1,2-propandiol, isoflucypram-desmethyl-1,2-propandiol-N-GlucA, isoflucypram-N-methyl-pyrazole-carboxylic acid, isoflucypram-propenol-GlucA, and isoflucypram-desmethyl-2-propanol-N-GlucA. These metabolites were not analysed in the feeding studies. Isoflucypram-lactic acid was quantifiable in liver and kidney in the lactating goat metabolism studies (0.011 and 0.012 mg eq/kg, respectively), isoflucypram-desmethyl-1,2-propandiol was quantifiable in all matrices in the laying hen metabolism studies (0.002–0.025 mg eq/kg), isoflucypram-desmethyl-1,2-propandiol-N-GlucA was quantifiable in all matrices in the laying hen metabolism studies (0.001–0.034 mg eq/kg), isoflucypram-N-methyl-pyrazole-carboxylic acid was quantifiable in kidney in the goat metabolism study (0.011 mg eq/kg), isoflucypram-propenol-GlucA goat was quantifiable in kidney in the goat metabolism studies (0.007–0.01 mg eq/kg), and isoflucypram-desmethyl-2-propanol-N-GlucA was quantifiable in liver in the hen metabolism studies (0.009–0.011 mg eq/kg). Because these metabolites are present in low concentrations at the dietary burdens (≤ 0.00029 mg eq/kg), the Meeting concluded that they should be excluded from the residue definition for dietary risk assessment.

Isoflucypram-desmethyl-propanol (free and conjugated) and isoflucypram-2-propanol (free and conjugated) were quantifiable in at least one matrix in the feeding studies. The Meeting determined that these metabolites could be assessed using TTC Cramer Class III (1.5 µg/kg bw/day). The Meeting estimated dietary exposures for isoflucypram-desmethyl-propanol (free and conjugated) of 0.0212–0.1827 µg/kg bw/day and for isoflucypram-2-propanol of 0.0022–0.0194 µg/kg bw/day, below the TTC for Cramer Class III.

Based on the above, the Meeting recommended the following residue definition:

Definition of the residue for compliance with the MRL for plant and animal commodities: *Isoflucypram*.

Because dietary exposure to isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-Cys and isoflucypram-desfluoro-N-methyl-cyclopropyl-pyrazole-carboxamide-OH-GSH were above the TTC for a compound with potential of genotoxicity, the Meeting could not reach a conclusion on a residue definition for dietary risk assessment.

Definition of the residue for dietary risk assessment for plant and animal commodities: A *conclusion could not be reached.*

In deciding whether isoflucypram is fat-soluble, the Meeting noted that the mean residues of isoflucypram at the highest dose tested in the lactating cow feeding study were < 0.01 mg/kg in muscle and 0.081 mg/kg in perirenal fat while residues in cream were 0.11–0.15 mg/kg compared to < 0.005 mg/kg in whey. The Meeting considered the residue to be fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised field trial data to support isoflucypram uses on wheat and barley.

Cereal Grains

Supervised residue trials are available from a number of European countries and New Zealand.

Barley

The cGAP for barley grain from New Zealand is one application at 75 g ai/ha up to BBCH 61 (56-day pre-harvest interval (PHI)). In independent trials approximating the cGAP, residues of isoflucypram were (n=21): < 0.010(19), 0.013, and 0.10 mg/kg.

The Meeting estimated a maximum residue level of 0.1 mg/kg and a median value of 0.010 mg/kg for barley grain.

Wheat

The cGAP for wheat grain from New Zealand is one application at 75 g ai/ha up to BBCH 69 (42-day PHI). In independent trials approximating the cGAP, residues of isoflucypram were (n=42): < 0.01(39), 0.015, 0.019, and 0.042 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg and a median value of 0.010 mg/kg for wheat grain.

As the use pattern covers triticale, the Meeting decided to extrapolate the wheat grain maximum residue level and median value to triticale grain.

Residues in animal feeds

The barley and wheat supervised residue trials included residue data on commodities defined as "forage" and "green material."

The Meeting noted that the percent dry matter in these samples collected at the cGAP PHI ranged from 29–91 percent for wheat and from 43–85 percent for barley, well above what the Meeting considers forage. The Meeting therefore considers this crop hay as opposed to forage.

Barley hay and straw

The cGAP for barley hay from New Zealand is one application at 75 g ai/ha up to BBCH 61 (42-day PHI).

Residues in barley hay were adjusted for percent dry matter (dw) in the submitted studies.

In independent trials approximating the cGAP, isoflucypram residues were (n=8): 0.094, 0.11, 0.24, 0.25, 0.32, 0.37, 0.48, and 0.51 and mg/kg (dw).

In independent trials at 116 and 106 g ai/ha (1.5× the cGAP application rate), isoflucypram residues were (n=2): 0.23 and 0.14 mg/kg (dw). By applying a proportionality factor of 0.65 and 0.71, the residues were (n=2): 0.15 and 0.097 mg/kg.

Isoflucypram residues in hay (proportionality factors of 0.65–1.0× applied) were (n=10): 0.094, 0.097, 0.11, 0.15, 0.24, 0.32(2), 0.37, 0.47, and 0.50 mg/kg (dw).

The cGAP for barley straw/stubble from New Zealand is one application at 75 g ai/ha up to BBCH 61 (56-day PHI).

Residues from the trials were corrected with the reported percent dry matter. For trials for which this information was not available, a percent dry matter of 89 percent was assumed for correction.

In independent trials approximating the cGAP, residues of isoflucypram in straw were (n=20): 0.055, 0.088, 0.16, 0.18(3), 0.22, 0.26, 0.27(3), 0.33, 0.36, 0.38, 0.45, 0.46, 0.69, 0.96, 1.1(2) mg/kg (dw).

Based on the straw data, the Meeting estimated a median residue of 0.27 mg/kg and a highest residue of 1.1 mg/kg for isoflucypram in barley hay and straw.

Wheat hay and straw

The cGAP for wheat hay from New Zealand is one application at 75 g ai/ha up to BBCH 69 (28-day PHI).

For 34 of the trials conducted according to cGAP, percent dry matter was reported and residues were corrected accordingly. For the remaining eight trials, a percent dry matter of 54 percent was assumed and corrected by the Meeting, based on the average of the given percent dry matter from the 34 trials above.

In independent trials approximating the cGAP, isoflucypram residues were (n=42): < 0.010, 0.14(2), 0.16, 0.17, 0.19(2), 0.20(2), 0.22, 0.26, 0.29(2), 0.31(2), 0.33(3), 0.36, 0.38, 0.41, 0.44, 0.50, 0.62, 0.86, 0.90, 0.91, 0.95, 1.1(3), 1.2(4), 1.3(2), 1.5(3), 1.7, and 3.3 mg/kg (dw).

The cGAP for wheat straw/stubble from New Zealand is one application at 75 g ai/ha up to BBCH 69 (42-day PHI).

Residues from the trials were corrected with the reported percent dry matter. For trials for which this information was not available, a percent dry matter of 88 percent was assumed for correction.

In independent trials approximating the cGAP, residues of isoflucypram in straw were (n=37): < 0.010, 0.061, 0.081, 0.13, 0.14(3), 0.17, 0.22, 0.25(2), 0.27, 0.32, 0.35, 0.43, 0.44, 0.45, 0.47, 0.55, 0.60, 0.73, 0.93, 1.1, 1.3(2), 1.4, 1.5, 1.6(3), 1.8(2), 1.9, 2.2(2), 2.7, and 3.6 mg/kg (dw).

Based on straw data, the Meeting estimated a median residue of 0.55 mg/kg and a highest residue of 3.6 mg/kg for isoflucypram in wheat hay and straw.

'Barley hay and straw' and 'wheat hay and straw', as commodities moving in trade, may not always be readily distinguishable from each other. The Meeting agreed to use the wheat straw data (dw) for the maximum residue level estimation for both 'barley hay and/or straw (dw)' and 'wheat hay and/or straw (dw)'.

The Meeting estimated a maximum residue level of 5 mg/kg for isoflucypram in/on wheat hay and/or straw (dw), and barley hay and/or straw (dw).

As the use pattern covers triticale, the Meeting decided to extrapolate the estimated maximum residue level, median residue levels, and highest residue levels from wheat hay and straw to triticale hay and straw.

Fate of residues during processing

Nature of the residue in processed commodities

The Meeting received studies investigating radiolabelled isoflucypram, isoflucypram-desmethyl-propanol, and isoflucypram-propanol following temperatures and pH conditions simulating typical processing procedures.

Isoflucypram and isoflucypram-propanol were stable under conditions simulating pasteurisation, baking/brewing/boiling, and sterilisation (test compounds were ≥ 98.0 percent TRR following all simulated processing procedures).

Isoflucypram-desmethyl-propanol was stabile under conditions simulating pasteurisation (approximately 99 percent TRR). Under conditions simulating baking/brewing/boiling, approximately 34 percent TRR was recovered as isoflucypram-desmethyl-propanol and approximately 66 percent TRR was

identified as isoflucypram-desmethyl-propanol-aldehyde. Under conditions simulating sterilisation, isoflucypram-desmethyl-propanol was not recovered and approximately 98 percent TRR was recovered as isoflucypram-desmethyl-propanol-aldehyde.

Residues in processed commodities

The Meeting received studies evaluating the effect of processing on isoflucypram in barley and wheat. Isoflucypram was applied at 5× the cGAP application rate.

Calculated processing factors indicate with a '<' (less-than) sign when the residue in the processed commodity is below the LOQ of the analytical method. The calculation in these cases is based on the LOQ (0.01 mg/kg) of the analytical method and the residue concentration of the RAC. The STMR-P values are calculated by multiplying the PF with the RAC STMR value (Table 160).

Table 160 Processing factors for isoflucypram

Raw Commodity (median)	Processed Commodity	Processing Factors	Median-P = Median $_{RAC}$ X PF (mg/kg)
Barley grain (0.010 mg/kg)	Pearl Barley	< 0.67	0.0067
	Beer	< 0.67	0.0067
	Brewer's Grain	< 0.67	0.0067
Wheat grain (0.010 mg/kg)	Bran, processed	1.2	0.012
	White Flour	< 0.63	0.0063
	Whole Meal Flour	0.67	0.0067
	Germ	1.1	0.011
	Pasta, Dry	< 0.63	0.0063
	Pasta, Cooked	< 0.63	0.0063
	Gluten	0.94	0.0094
	Starch	< 0.63	0.0063
	White Bread	< 0.63	0.0063
	Whole Meal Bread	< 0.63	0.0063
	AGF	148	1.5

Residues in animal commodities

Dairy cow

The Meeting received a study investigating the magnitude of isoflucypram in milk, cream, whey, muscle, liver, kidney, and fat of dairy cows orally dosed with isoflucypram for 28 days. Doses were administered orally via gelatine capsule with a pill gun at 1.6 ppm, 4.2 ppm, 16 ppm, and 48 ppm (dry feed basis).

Animals were sacrificed within 24 hours after the final dose except for the depuration group, which were sacrificed 4, 7, or 14 days after the last dose. Residues of isoflucypram were < 0.005 mg/kg in milk from the 1.6, 4.2, and 16-ppm dosing levels and ranged from 0.006–0.009 mg/kg at the 48-ppm dose level. Isoflucypram partitioned into cream with residues ranging from 0.11–0.15 mg/kg compared to < 0.005 mg/kg in corresponding skim fraction.

Residues of isoflucypram were < 0.01 mg/kg in muscle and kidney at all dose levels. Residues of isoflucypram were < 0.01 mg/kg in fat at the 1.6 and 4.2-ppm dose levels, 0.034 mg/kg at the 16 ppm dose level, and 0.081 mg/kg at the 48 ppm dose level. Residues of isoflucypram were < 0.01 mg/kg in liver

at the 1.6 and 4.2-ppm dose levels, < 0.011 mg/kg at the 16-ppm dose level, and < 0.016 mg/kg at the 48-ppm dose level.

Laying hen

The Meeting received a study investigating the magnitude of isoflucypram in eggs (whole, white, and yolk), muscle, liver, and fat with skin of laying hens orally dosed with isoflucypram for 28 days. Doses were administered orally via gelatine capsule at 0.53 ppm, 2.1 ppm, and 8.7 ppm (dry feed basis).

Animals were sacrificed within six hours after the final dose except for the depuration group, which were sacrificed 4, 7, or 14 days after the last dose. Residues of isoflucypram were < 0.01 mg/kg in all matrices at all dose levels.

Farm animal dietary burdens

Dietary burdens were calculated for beef cattle, dairy cattle, broilers, and laying poultry based on feed items evaluated by the JMPR by the current Meeting. The dietary burdens, estimated using the most recent version of the OECD livestock dietary burden calculator, are presented in Annex 6 and summarised in Table 161.[2]

Table 161 Estimated maximum dietary burdens of farm animals

	Livestock dietary burden, isoflucypram, ppm of dry matter diet							
	Japan		United States-Canada		European Union		Australia	
	Max		Max		Max		Max	
Beef cattle	0.012		0.64		0.84		3.6❶	
Dairy cattle	0.011		0.73		0.84		2.5❷	
Poultry – broiler	0.002		0.012		0.012		0.012	
Poultry – layer	0.004		0.012		0.37❸		0.009	

Notes:

❶ Highest maximum beef cattle dietary burden suitable for MRL estimates for mammalian tissues.

❷ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk.

❸Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues and eggs.

Animal commodity estimations of maximum residue levels and dietary intake

Mammals (other than marine mammals)

The isoflucypram maximum dietary burdens for beef and dairy cattle were 3.6 and 2.5 ppm, respectively. Table 162 shows the anticipated isoflucypram residues in beef and dairy cattle for maximum residue level estimation.

Table 162 Residue of isoflucypram in mammals other than marine mammals for maximum residue level estimation

	Feed level (ppm) for milk residues	Isoflucypram residue (mg/kg) in milk	Feed level (ppm) for tissue residues	Isoflucypram residue (mg/kg) in tissues			
				Muscle	Liver	Kidney	Fat
Maximum residue level estimates for beef and dairy cattle							
Feeding study	4.18	< 0.005	4.18	< 0.01	< 0.01	< 0.01	< 0.01

[2] http://www.fao.org/agriculture/crops/core-themes/theme/pests/pm/jmpr/jmpr-docs/en/

	Feed level (ppm) for milk residues	Isoflucypram residue (mg/kg) in milk	Feed level (ppm) for tissue residues	Isoflucypram residue (mg/kg) in tissues			
				Muscle	Liver	Kidney	Fat
Dietary burden and residue estimate	2.50	< 0.005[a]	3.60	< 0.01	< 0.01	< 0.01	< 0.01

Notes:

[a] Residue of < 0.005 mg/kg at the 15.5 ppm feeding level was used for the determination of the residue in milk fats.

The Meeting estimated the following maximum residue levels: 0.005(*) mg/kg for milks and milk fats and 0.01(*) mg/kg for meat (from mammals other than marine mammals), fat (from mammals other than marine mammals), and edible offal (mammalian).

Poultry

The isoflucypram maximum dietary burden for poultry is 0.37 ppm.

Table 163 shows the anticipated isoflucypram residues in poultry for maximum residue level estimation.

Table 163 Residues of isoflucypram in poultry for maximum residue level estimation

	Feed level (ppm) for egg residues	Isoflucypram residue (mg/kg) in eggs	Feed level (ppm) for tissue residues	Isoflucypram residue (mg/kg) in tissues			
				Muscle	Liver	Kidney	Fat
Maximum residue level estimates for poultry							
Feeding study	0.53	< 0.01	0.53	< 0.01	< 0.01	< 0.01	< 0.01
Dietary burden and residue estimate	0.37	< 0.01	0.37	< 0.01	< 0.01	< 0.01	< 0.01

The Meeting estimated maximum residue levels of 0.01(*) mg/kg for eggs, poultry meat, poultry fat, and poultry edible offal.

RECOMMENDATIONS

Summary of recommendations for isoflucypram

Definition of the residue for compliance with the MRL for plant and animal commodities: *Isoflucypram*.

Definition of the residue for dietary risk assessment for plant and animal commodities: A *conclusion could not be reached.*

The residue is fat-soluble.

DIETARY RISK ASSESSMENT

As the Meeting was unable to recommend residue definitions for dietary risk assessment for plants and animal commodities, chronic and acute dietary risk assessments could not be conducted.

FURTHER WORK OR INFORMATION

Desirable information

- Cereal grain processing data for isoflucypram-desmethyl-propanol.
- Hydrolysis of the extracts from the soya bean metabolism studies.

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PA17/054	Ziemer, F.; Strunk, B.	2017	Isoflucypram, Technical Substance: Physical Characteristics Colour, Physical State and Odour
20140107.01	Krack, M.	2014	Isoflucypram, Pure Substance: Melting Point, Boiling Point, Thermal Stability
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EnSa-16-0959	Lamshoeft, M.	2017	Metabolism of [Pyrazole-4-^{14}C] Isoflucypram in Tomato
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S17-01394	Botterweck, J.	2018	Metabolism of [Pyrazole-4-^{14}C] Isoflucypram in Potato after Seed Treatment
S17-01392	Botterweck, J.	2018	Metabolism of [Phenyl-UL-^{14}C] Isoflucypram in Potato after Seed Treatment
S14-01087	Traub, M.	2018	Amendment No.1 to Final Report Metabolism of [Pyrazole-4-^{14}C] Isoflucypram in Wheat Plants

Report Number	Author(s)	Year	Study Title
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S14-01089	Traub, M.	2017	Metabolism of [Pyrazole-4-^{14}C] Isoflucypram in Soya bean Plants
S14-01090	Traub, M.	2017	Metabolism of [Phenyl-UL-^{14}C] Isoflucypram in Soya bean Plants
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EnSa-17-0308	Bongartz, R.; Luks, A.-K.; Doebbe, A.; Conrad, F.	2017	[Phenyl-UL-^{14}C] Isoflucypram - Metabolism in the Lactating Goat
EnSa-17-0307	Bongartz, R.; Luks, A.-K.; Conrad, F.	2017	[Pyrazole-4-^{14}C] Isoflucypram: Metabolism in the Laying Hen
EnSa-17-0306	Bongartz, R.; Doebbe, A.; Conrad, F.	2017	[Phenyl-UL-^{14}C] Isoflucypram: Metabolism in the Laying Hen
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Report Number	Author(s)	Year	Study Title
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16-2052	Kaussmann, M.	2017	Determination of the Residues of Isoflucypram and Prothioconazole in/on Barley After Spray Application of Prothioconazole & Isoflucypram EC 150 in Portugal, Southern France and Spain
17-2017	Noss, G.; Nayyar, B.	2019	Determination of the Residues of Isoflucypram, Prothioconazole and AE C656948 in/on Spring Barley After Spray Application of FLU & Isoflucypram & PTZ EC 234 in Germany, Denmark, the United Kingdom and Northern France
17-2018	Noss, G.; Nayyar, B.	2019	Determination of the Residues of Isoflucypram, Prothioconazole and AE C656948 in/on Barley After Spray Application of FLU & Isoflucypram & PTZ EC 234 in Southern France, Italy, Spain and Greece
PNZ16414	Neill, D.	2017	Determination of Residues of Isoflucypram in Cereals Following one Application of Isoflucypram & Prothioconazole EC 150
BAYERNZ /GLP/16/04/a	Ranchodbhai, T.	2018	Determination of the Residues of Isoflucypram in Barley and Wheat Following Spray Application of Isoflucypram & Prothioconazole EC 150 in New Zealand 2016/2017
S17-07996	Ranchodbhai, T.	2018	Determination of the Residues of Isoflucypram After Spray Application of Isoflucypram & Prothioconazole EC 150 at GS61 in Barley, New Zealand 2017/2018
GLP658	Jeannes, G.	2019	Determination of Residues of Isoflucypram-Desmethyl-Propanol and Isoflucypram-Propanol (Metabolites of Isoflucypram) After Spray Application of Isoflucypram + PTZ EC 150 at GS61 in Barley, New Zealand 2017/2018
S18-07828	Ranchodbhai, T.	2020	Determination of the Residues of Isoflucypram After Spray Application of Isoflucypram & Prothioconazole EC 150 at GS61 in Barley, New Zealand 2018/2019
15-2069	Schulte, G.	2017	Determination of the Residues of Isoflucypram in/on Wheat and Durum Wheat After Spray Application of Isoflucypram EC 050 in Portugal, Southern France and Spain
15-2111	Schulte, G.	2017	Determination of the Residues of Isoflucypram in/on Spring and Winter Wheat After Spray Application of Isoflucypram EC 050 in Northern France, the United Kingdom, the Netherlands and Germany
15-2115	Glaubitz, J.	2017	Determination of the Residues of Isoflucypram and Prothioconazole in/on Wheat After Spray Application of Prothioconazole & Isoflucypram EC 150 in Northern France, United Kingdom, the Netherlands and Germany
15-2116	Glaubitz, J.	2017	Determination of the Residues of Isoflucypram and Prothioconazole in/on Wheat After Spray Application of Prothioconazole & Isoflucypram EC 150 in the Field in Portugal, Southern France and Spain
15-2119	Noss, G.	2017	Determination of the Residues of Isoflucypram, Prothioconazole and Tebuconazole in/on Wheat and Durum Wheat After Spray Application of Prothioconazole & Tebuconazole & Isoflucypram EC 250 in Southern France, Spain, Portugal and Italy
15-2120	Noss, G.	2017	Determination of the Residues of Isoflucypram, Prothioconazole and Tebuconazole in/on Spring Wheat and Winter Wheat After Spray Application of Prothioconazole & Tebuconazole & Isoflucypram EC 250 in United Kingdom, Hungary, Northern France and Poland

Report Number	Author(s)	Year	Study Title
16-2053	Kaussmann, M.	2017	Determination of the Residues of Isoflucypram and Prothioconazole in/on Winter Wheat and Spring Wheat After Spray Application of Prothioconazole & Isoflucypram EC 150 in Northern France, Belgium, the Netherlands and Germany
16-2054	Kaussmann, M.	2017	Determination of the Residues of Isoflucypram and Prothioconazole in/on Wheat and Wheat, Durum After Spray Application of Prothioconazole & Isoflucypram EC 150 in Italy, Spain, Southern France and Greece
17-2019	Kaussmann, M.; Nayyar, B.	2019	Determination of the Residues of Isoflucypram, AE C656948 and Prothioconazole in/on Winter Wheat and Spring Wheat After Spray Application of FLU & Isoflucypram & PTZ EC 234 in Germany, Northern France and the Netherlands
17-2020	Kaussmann, M.; Nayyar, B.	2019	Determination of the Residues of Isoflucypram, AE C656948 and Prothioconazole in/on Wheat and Wheat, Durum After Spray Application of FLU & Isoflucypram & PTZ EC 234 in Southern France, Spain, Greece and Italy
18-2014	Noss, G.; Nayyar, B.	2019	Determination of the Residues of Isoflucypram and Prothioconazole in/on Wheat and Wheat, Durum After Spraying Application of Prothioconazole & Isoflucypram EC 150 in Southern France, Italy, Spain and Greece
18-2135	Kaussmann, M.; Kerkering, S.	2019	Determination of the Residues of Isoflucypram and Prothioconazole in/on Wheat and Spring Wheat After Spray Application of Prothioconazole & Isoflucypram EC 150 in Germany, Belgium, United Kingdom and the Netherlands
S17-07939	Ranchodbhai, T.	2018	Determination of the Residues of Isoflucypram After Spray Application of Isoflucypram & Prothioconazole EC 150 at GS69 in Wheat, New Zealand 2017/2018
P672186503	Kaussmann, M.	2019	Determination of Isoflucypram-Desmethyl-Propanol and Isoflucypram-Propanol in/on Wheat and Barley Straw Samples by HPLC-MS/MS
P672186504	Kaussmann, M.	2019	Determination of Isoflucypram-Desmethyl-Propanol and Isoflucypram-Propanol in/on Wheat and Barley Grain Samples by HPLC-MS/MS
GLP655	Jeannes, G.	2019	Determination of Residues of Isoflucypram-Desmethyl-Propanol and Isoflucypram-Propanol (Metabolites of Isoflucypram) After Spray Application of Isoflucypram + PTZ EC 150 at GS69 in Wheat, New Zealand 2017/2018
S18-07829	Ranchodbhai, T.	2020	Determination of the Residues of Isoflucypram After Spray Application of Isoflucypram & Prothioconazole EC 150 at GS69 in Wheat, New Zealand 2018/2019
E19RP054	Kaussmann, M.; Stolpe, C.	2020	Determination of the Residues of Isoflucypram in/on Winter Barley and Spring Barley After Spray Application of Prothioconazole & Isoflucypram EC 150 in Germany and Belgium
E19RP055	Kaussmann, M.; Wilbuer, J.	2020	Determination of the Residues of Isoflucypram in/on Barley After Spray Application of Prothioconazole & Isoflucypram EC 150 in Southern France and Italy
E19RP056	Kaussmann, M.; Fecker, L.	2020	Determination of the Residues of Isoflucypram, Prothioconazole and AE C656948 in/on Barley After Spray Application of FLU & Isoflucypram & PTZ EC 234 in Italy and Spain
Processing studies			
EnSa-16-0135	Heinemann, D.; Doebbe, A.	2017	Nature of the Residues of [Pyrazole-4-^{14}C] Isoflucypram and [Phenyl-UL-^{14}C] Isoflucypram in Processed Commodities – High Temperature Hydrolysis
EnSa-20-0057	Lamshoeft, M.; Luks, A.-K.	2020	Nature of the Residues of [Propane-1-^{14}C] Isoflucypram-Desmethyl-Propanol in Processed Commodities – High Temperature Hydrolysis
EnSa-19-0734	Lamshoeft, M.; Bartelsen, N.	2020	Nature of the Residues of [Pyrazole-4-^{14}C] Isoflucypram-Propanol in Processed Commodities - High Temperature Hydrolysis

Report Number	Author(s)	Year	Study Title
15-3407	Freitag, T.; Hoffmeister, R.	2017	Determination of the Residues of Isoflucypram in/on Barley and the Processed Fractions (Malt Sprouts; Brewer's Malt; Brewer's Grain; Hops Draff; Brewer's Yeast; Beer; Pearl Barley Rub Off and Pearl Barley) After Spray Application of Isoflucypram EC 050 in the Field in the Netherlands and Spain
RALNN137	Harbin, A. M.	2017	Isoflucypram: Magnitude of Residues in/on Wheat Processed Fractions Following Treatment with Isoflucypram EC50
Livestock feeding studies			
17-8002	Glaubitz, J.; Fichant, E.	2017	Isoflucypram: Feeding Study with Laying Hens
17-8001	Glaubitz, J.	2017	Isoflucypram: Feeding Study with Dairy Cows
Residue definition[1]			
M-612432-02-1	Diot, R.; Heinemann, D.; Shipp, E.	2018	Isoflucypram: Evaluation of Dietary Metabolites and Residue Definition Proposals

[1] Contents not summarised herein.

MANCOZEB (050)

First draft prepared by Dr M Doherty, Environmental Protection Agency, United States of America

EXPLANATION

Mancozeb is an ethylene-bis-dithiocarbamate (EBDC) fungicide that was evaluated for the first time by the JMPR in 1967 and underwent multiple subsequent assessments prior to periodic review in 1993. The 1993 Meeting established an acceptable daily intake (ADI) of 0–0.03 mg/kg bw for the group of EBDCs (mancozeb, maneb, metiram, and zineb; alone or in any combination). In addition, the Meeting has established an ADI of 0–0.004 mg/kg bw for the common EBDC metabolite ethylenethiourea (ETU). To date, no acute reference dose (ARfD) has been established by the Meeting for either the EBDC fungicides or ETU. The dithiocarbamates were last evaluated by the JMPR at its 2014 Meeting.

The definition of the residue for compliance with MRLs in plant and animal commodities is total dithiocarbamates, determined as CS_2, evolved during acid digestion and expressed as mg CS_2/kg. For the estimation of dietary intake in plant and animal commodities, the residue definition is mancozeb plus ethylenethiourea (ETU); in practice, this is done by assessing mancozeb toxicity-equivalent residues. Dithiocarbamate residues are not fat soluble.

The current Meeting received residue data from crop field trials in longan, soya bean, maize, rice, and cotton; processing studies for maize; and supporting information on analytical methods and storage stability.

METHODS OF RESIDUE ANALYSIS

Mancozeb: All analyses for mancozeb used a method similar to VR-036/17 (below), with variations in the temperature and time parameters for the conversion to CS_2. Concurrent recoveries are summarised in Table 2.

VR-036/17 (Magagnato, M. B. B., 2017, VR-036/17)

For this method, residues of mancozeb are extracted from the sample matrix and converted to CS_2 using a solution of stannous chloride/water/conc. HCl (1.5 g/20 mL/33 mL) + isooctane (4+1, v/v) in a sealed flask maintained at 95 °C for 30 minutes. After cooling to room temperature, an aliquot of the isooctane phase was taken for analysis by GC-MS (*m/z* = 76). The validated LOQ for the method is 0.1 mg/kg (0.056 mg/kg as CS_2).

Ethylenethiourea (ETU): Multiple methods were provided for the analysis of ETU and are summarized in Table 1. Concurrent recoveries are summarised in Table 2. Method validation data were not provided.

Table 1 Summary of analytical methods considered by the current Meeting for ETU

Method ID		QuEChERS	Not specified	Haines & Adler[1]	PRM-006 rev 1
Report No.		Annex Report Trial 1	BPL-JM-066-009 BPL-JM-066-002	34-89-21	AA950302
Extraction and clean-up	Crop	Longan	Soya bean, cotton	Maize	Maize
	Matrix	Berries	Seed	Kernels + processed commodities	Kernels + processed commodities
	Extraction	Acetonitrile with $MgSO_4$, NaCl	Methanol	Methanol	H_2O (pH 11-12 with NH_4OH) + NaCl + EtOH

[1] 1973. JAOAC 56:333-337

Method ID		QuEChERS	Not specified	Haines & Adler[1]	PRM-006 rev 1
Report No.		Annex Report Trial 1	BPL-JM-066-009 BPL-JM-066-002	34-89-21	AA950302
					+ Celite 545
	Column	--	None	Alumina	Alumina
	Eluent	--	None	Methanol	Chloroform:EtOH
	Additional	Filter (0.45µm)	Filter (0.45µm)	Derivitisation with 1-bromobutane	Filter (0.45µm)
Chromatography	Type	LC	LC	GC	HPLC
	Analytical column	not specified	C18	SE-30	Graphitized carbon
	Dimensions		50 mm × 2.1 mm	2 m × 4 mm	100 mm × 4.6 mm
	Sorbent size		1.8 µm	80-100 µm	7 µm
	Parameters		A= H_2O + 10 mM ammonium formate + 0.1 percent formic acid B= MeOH+ 10 mM ammonium formate + 0.1 percent formic acid Gradient 95 percent A to 95 percent B over 6 minutes	Injector: 210°C Column: 200°C Detector: 210°C	H_20 (0.022 M H_3PO_4):ACN, 98:2, v/v)
	Flow rate		0.2 mL/min	Air: 110 mL/min H: 150 mL/min O_2: 35 mL/min He: 60 mL/min	1 mL/min
	Injection volume		5 µL	Not specified	100 µL
	Instrument		Agilent	Barber-Coleman 5000	Not specified
Detection	Quantitative detection	MS/MS	MS/MS	Flame photometric	Amperometric
	LOQ	0.01	0.1	0.01	0.05
	Whole method linearity (r^2)	not provided	0.9999	not provided	not provided

Table 2 Summary of recovery of mancozeb and ETU from longan, soya bean seed, maize kernels, rice grain, and cotton seed

Crop	Matrix	Fortification level [mg/kg]	n	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Mancozeb (as CS_2)							
Longan	Berries	0.1	10	70-110	96	11	Annex Report Trials 1-6
		1	11	87-116	90	12	
		10	6	86-102	101	10	
		30	11	84-115	98	11	
Maize	Kernels	0.1	5	84-91	87	2.6	000672.034.164.12, 13790.034.029.14, 13790.034.030.14, 13790.034.093.14, 13790.034.094.14, 13790.034.095.14
		1	5	75-96	81	11	
		0.1	5	88-110	96	10	170129
		1	5	101-116	110	6.6	

Crop	Matrix	Fortification level [mg/kg]	n	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
	Kernels	0.05	1	97	97	--	34-89-21
		0.1	1	97	97	--	
	Steepwater concentrate	0.05	1	97	97	--	
		0.1	1	92	92	--	
	Steepwater distillate	0.05	1	97	97	--	
		0.1	1	97	97	--	
	Germ	0.05	1	92	92	--	
		0.1	1	94	94	--	
	Screenings	0.05	1	92	92	--	
		0.2	1	94	94	--	
	Hulls	0.05	1	92	92	--	
		0.1	1	89	89	--	
	Starch-gluten	0.05	1	97	97	--	
		0.1	1	89	89	--	
	Processing water	0.05	1	92	92	--	
		0.1	1	92	92	--	
	Gluten	0.05	1	92	92	--	
		0.1	1	89	89	--	
	Starch	0.05	1	87	87	--	
		0.1	1	87	87	--	
	Crude oil	0.05	1	87	87	--	
		0.1	1	92	92	--	
	Presscake	0.05	1	97	97	--	
		0.2	1	95	95	--	
	Refined oil	0.05	1	87	87	--	
		0.1	1	87	87	--	
	Solvent-extracted oil	0.05	1	92	92	--	
		0.1	1	92	92	--	
	Solvent-extracted presscake	0.05	1	98	98	--	
		0.1	1	92	92	--	
	Small grits	0.05	1	92	92	--	
		0.1	1	92	92	--	
	Medium grits	0.05	1	87	87	--	
		0.1	1	90	90	--	
	Large grits	0.05	1	92	92	--	
		0.1	1	95	95	--	
	Coarse meal	0.05	1	98	98	--	
		0.1	1	95	95	--	
	Flour	0.05	1	90	90	--	
		0.1	1	90	90	--	
	Meal	0.05	1	90	90	--	
		0.1	1	92	92	--	
	Kernels	0.05	2	101, 107	104	--	AA950302
		0.1	1	69	69	--	
		0.25	1	80	80	--	
		0.5	1	90	90	--	
	Grits	0.05	1	89	89	--	
		0.1	1	88	88	--	
		0.25	1	70	70	--	
	Meal	0.05	1	107	107	--	
		0.1	1	95	95	--	
		0.25	1	104	104	--	
	Flour	0.05	2	51, 129	85	--	
		0.1	1	78	78	--	

Crop	Matrix	Fortification level [mg/kg]	n	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
		0.25	1	97	97	--	
		0.5	1	101	101	--	
	Refined oil (dry milling)	0.05	2	47, 47	47	--	
		0 1	2	31, 34	32.5	--	
		0.25	2	34, 53	43.5	--	
	Refined oil (wet milling)	0.05	2	51, 64	57.5	--	
		0.1	2	30, 44	37	--	
		0.25	2	44, 57	50.5	--	
	Starch	0.05	1	79	79	--	
		0.1	1	95	95	--	
		0.25	1	73	73	--	
Rice	Grain	0.1	5	95-101	98	2.3	13790.034.152.17, 13790.034.150.17
		1	5	89-103	92	6.7	
		0.1	5	113-119	116	2.0	13790.034.085.14
		1	5	95-107	102	5.3	
		0.05	5	86-102	92	8.9	13790.034.086.14
		0.5	5	102-120	112	6.8	
		0.05	5	90-120	107	13	13790.034.218.15, 13790.034.217.15, 13790.034.053.16, 13790.034.052.16
		0.5	5	93-114	100	9.1	
		0.2	8	98-119	110	7.3	GHB-P 1046, GHB-P 1047, GHB-P 1048, GHB-P 1049, GHB-P 1146, GHB-P 1147
		1	4	75-78	77	3.7	
		4	6	77-91	83	7.2	
Soya bean	Seed	0.1	5	83-99	95	7.3	170130, 13790.034.127.18, 13790.034.126.18, 13790.034.124.18
		1	5	91-110	103	7.3	
		0.1	5	73-89	80	8.9	000672.034.165.12, 000672.034.156.12, 13790.034.032.14, 13790.034.031.14, 13790.034.329.14, 13790.034.330.14
		1	5	72-86	81	6.6	
		0.1	5	77-99	89	9.8	BPL-JM-066-007-19, BPL-JM-066-009-19, BPL-JM-037-032-16-RF
		1	5	87-92	89	2.3	
		0.1	5	81-88	84	3.6	RS-040020/15
		1	5	88-95	90	3.2	
Cotton	Seed	0.1	5	84-113	95	12	170131
		1	5	87-106	98	10	
		0.1	5	87-103	97	6.2	13790.034.199.15, 13790.034.213.15
		1	5	89-105	94	6.8	
		0.1	5	80-110	92	12	000672.034.163.12, 000672.034.153.12
		1	5	78-113	90	16	

Crop	Matrix	Fortification level [mg/kg]	n	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Ethylenethiourea							
Longan	Berries	0.01	5	74-106	97	14	Annex Report Trials 1-6
		0.02	2	75-100	88	20	
		0.05	5	72-104	90	15	
Soya bean	Seed	0.1	5	84-92	87	3.6	BPL-JM-066-009-19-RF BPL-JM-066-007-19-RF
		1	5	86-95	92	3.8	
Cotton	Seed	0.1	3	71-79	76	6	BPL-JM-066-112-16
		1	3	69-78	74	6	
Maize	Kernel	0.01	1	83	83	--	34-89-21
		0.1	1	81	81	--	
	Steepwater distillate	0.01	1	83	83	--	
		0.2	1	78	78	--	
	Germ	0.01	1	103	103	--	
		0.1	1	82	82	--	
	Hull	0.01	1	76	76	--	
		0.1	1	76	76	--	
	Starch-gluten	0.01	1	83	83	--	
		0.1	1	98	98	--	
	Processing water	0.01	1	97	97	--	
		0.1	1	89	89	--	
	Gluten	0.01	1	90	90	--	
		0.1	1	48	48	--	
	Starch	0.01	1	110	110	--	
		0.1	1	85	85	--	
	Presscake	0.01	1	116	116	--	
		0.1	1	86	86	--	
	Large grits	0.01	1	97	97	--	
		0.1	1	76	76	--	
	Coarse meal	0.01	1	97	97	--	
		0.1	1	77	77	--	
	Flour	0.01	1	85	85	--	
		0.1	1	81	81	--	
	Meal	0.01	1	130	130	--	
		0.1	1	82	82	--	
	Refined oil	0.01	1	85	85	--	
		0.1	1	86	86	--	
	Solvent-extracted oil	0.01	1	140	140	--	
		0.1	1	72	72	--	
	Solvent-extracted presscake	0.01	1	85	85	--	
		0.1	1	74	74	--	
	Small grits	0.01	1	97	97	--	
		0.1	1	68	68	--	
	Medium grits	0.01	1	85	85	--	
		0.1	1	83	83	--	
	Screenings	0.02	1	101	101	--	
		0.2	1	90	90	--	
	Steepwater concentrate	0.01	1	118	118	--	
		0.1	1	100	100	--	
	Crude oil	0.02	1	129	129	--	
		0.2	1	76	76	--	
	Soapstock	0.4	1	16	16	--	
	Kernel	0.01	1	91	91	--	AA950302
		0.02	1	73	73	--	

Crop	Matrix	Fortification level [mg/kg]	n	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
		0.05	1	64	64	--	
	Grits	0.01	1	89	89	--	
		0.02	1	80	80	--	
		0.05	1	90	90	--	
	Meal	0.01	1	66	66	--	
		0.02	1	96	96	--	
		0.05	1	86	86	--	
	Flour	0.01	1	60	60	--	
		0.02	1	66	66	--	
		0.05	1	68	68	--	
	Refined oil (dry milling)	0.01	1	88	88	--	
		0.02	1	94	94	--	
		0.05	1	69	69	--	
	Refined oil (wet milling)	0.01	1	64	64	--	
		0.02	1	99	99	--	
		0.05	1	63	63	--	
	Starch	0.01	1	94	94	--	
		0.02	1	82	82	--	
		0.05	1	82	82	--	

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

A study depicting the stability of mancozeb and ETU residues in peanut nutmeat was submitted to the Meeting (Viana de Moraes, M. 2017, Report BPL-JM-037-032-16-RF). Control samples of peanut nutmeat were spiked with mancozeb or ETU, each at 1 mg/kg, and placed into frozen storage (≤-20 °C). Samples were analysed after 0 and 160 days in storage. Mancozeb was analysed, as CS_2, using the method described above. For ETU, the method was the same as that described above for soya bean seed and cotton seed. Each method has a limit of quantitation of 0.1 mg/kg. Results are summarised in Table 3.

Data on the stability of mancozeb was generated concurrently with field trials for longan (Table 3). The majority of samples for other commodities evaluated by the 2022 Meeting were stored for *ca*. 30 days or less except maize (≤42 days), cotton (≤40 days), and rice (≤ 425 days).

Table 3 Summary of stability data for residues of mancozeb and ETU residues under frozen conditions

Matrix	Analyte	Storage time, days	Procedural recovery, percent	Fortification level, mg/kg	Residue remaining, mg/kg	Mean percent remaining	Reference
Peanut nutmeat	Mancozeb	0	96	1	0.78, 0.74, 0.78	77	BPL-JM-037-032-16-RF
		160	106	1	0.95, 0.81, 0.80	85	
	ETU	0	80	1	0.86, 0.79, 0.86	82	
		160	72	1	0.79, 0.84, 0.85	83	
Longan	Mancozeb	0	104	1	1.03, 1.01	102	Annex Report Trial 1
		30	98	1	0.89, 0.92	91	
		60	112	1	1.12, 1.13	113	
		180	111	1	1.12, 1.10	111	
		240	95	1	0.91, 0.99	95	
		300	93	1	0.87, 0.99	93	

Data submitted to the current meeting demonstrate stability, under frozen storage conditions, for mancozeb and ETU for at least 160 days in peanut nutmeat and for mancozeb for at least 300 days in longan berries.

USE PATTERN

Registered labels describing the use of mancozeb were submitted to the present Meeting for longan, soya bean, maize, rice, and cotton (Table 4).

Table 4 Registered uses of mancozeb submitted to the 2022 JMPR. All uses are foliar application

Use site	Country	Formulation		Application [a)]					PHI, days
		Conc., g/kg	Type	Rate, kg ai/ha/applic	Rate, kg ai/ha/year	Water, L/ha	Max No.	Interval, days	
Longan	Thailand	800	WP	0.24 kg ai/hL	n.s.	5 L/tree	3	7	14
Soya bean	Brazil	800	WP	0.8-2.4	n.s.	200	3	7-10	30
		750	WG	1.125-2.25	n.s.	100-300 (20-50 aerial)	3	7-14	30
		597	WG	1.19	n.s.	100-300 (20-50 aerial)	3	7-14	30
		700	WG	1.05-1.4	n.s.	100-300 (20-50 aerial)	3	7-14	30
Maize	Brazil	750	WG	1.125-2.25	n.s	100-300 (20-50 aerial)	3	7-10	30
		700	WG	1.05-1.4	n.s.	100-300 (20-50 aerial)	3	10	42
	United States	800	WP	1.35	13.45	n.s.	n.s.	4-14	40
Rice	Brazil	750	WG	1.5-2.25	n.s	100-300 (20-50 aerial)	3	10	30
		800	WP	1.6-2.8	n.s.	100-200 (20-50 aerial)	3	10	32
		800	WP	1.6-2.8	n.s.	100-300 (20-50 aerial)	3	10	32
		800	WP	1.6-3.6	n.s.	400-600 (30 aerial)	2	Start at booting, repeat at panicles or at flowering	32
		800	WP	1.6-3.6	n.s.	100-300 (20-50 aerial)	2	Start at booting, repeat at panicles or at flowering	32
		700	WG	1.05-1.4	n.s.	100-300 (20-50 aerial)	3	10	32
	United States	800	WP	1-2 g ai/kg seed	n.a.	n.a.	1	n.a.	n.a.
Cotton	Brazil	700	WG	1.12-1.68	n.s.	100-300 (20-50 aerial)	3	7-14	42
		750	WG	1.125-2.25	n.s	100-300 (20-50 aerial)	3	7-10	30
	United States	800	WP	1.5-3 g ai/kg seed	n.a.	n.a.	1	n.a.	n.a.

Note:

[a)] n.a. = not applicable; n.s. = not specified

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on longan, soya bean, maize, rice, and cotton.

The field trial reports included method validation data, as recoveries from spiked samples at levels reflecting those observed in the field trial samples; dates from critical events during the study, including application, harvest, storage, and analysis; as well as information on the field site and treatment parameters. Analytical reports were sufficiently detailed and included example chromatograms and

example calculations. Samples were analysed by the methods described above. All samples were analysed for residues of mancozeb (as CS_2). Some samples were analysed for ETU as indicated in the tables below.

The field trial study designs included control plots. Measured residues from control plots were <LOQ and are not included in the summary tables in this evaluation. All residues for mancozeb are reported as CS_2 in the tables below.

When calculating average residues, values below the LOQ were assumed to be at the LOQ. In the summary tables, residue values leading to maximum residue estimations and used for long-term dietary risk assessment are underlined.

Supervised trials for mancozeb:

Group	Commodity	Table
Tropical/sub-tropical fruit, inedible peel, small	Longan	Table 5
Dry beans	Soya bean	Table 6
Cereals	Maize	Table 7
	Rice	Table 8
Oilseeds	Cotton	Table 9

Longan

Six field trials were conducted in Thailand during the 2015 and 2016 growing seasons (Annex I Reports 1 through 6; full study reports were not provided) using a WP formulation. Treatment consisted of three foliar applications of ca. 0.24 kg ai/hL, on generally a 7-day interval. All trials were residue-decline trials with harvest occurring from 0 up to 42 days after the last application (DALA).

Following harvest, samples (2 kg) were frozen within 1 day of collection and shipped, frozen, to the analytical laboratory. Upon arrival at the facility, samples were put into frozen storage. Prior to analysis, samples were pitted and homogenized in the presence of dry ice and then returned to frozen storage. Samples were stored for a maximum of 300 days prior to analysis.

Samples were analysed for residues of mancozeb and ETU using the methods described above. Concurrent recovery data indicate that the method is suitable, with LOQs of 0.056 mg/kg for CS_2 and 0.01 mg/kg for ETU.

Table 5 Results of mancozeb residue trials in longan

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [mean]		Study report
		No. [interval, days]	Conc., kg ai/hL	L/ha			CS_2	ETU	
Critical GAP (TL)	--	3 [7]	0.24	5 L/tree	--	14	--	--	--
Kaeng Hang Maeo District, Chanthaburi Province, Thailand (002.16-01) 2015	Longan (Daw)	1 [--]	0.24	1000	Whole fruit minus pit	0	19	0.01	Annex I Report Trial 01
		2 [6]	0.24	1000		3	6.2	0.01	
		3 [8]	0.24	1000		7	6.7	0.01	
						14	<u>2.2</u>	<0.01	
						21	2.2	<0.01	
Nikhom Phatthana District, Rayong Province, Thailand	Longan (Daw)	1 [--]	0.24	991	Whole fruit minus pit	0	29	0.04	Annex I Report
		2 [7]	0.24	991					

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [mean]		Study report
		No. [interval, days]	Conc., kg ai/hL	L/ha			CS_2	ETU	
(002.16-02) 2015		3 [7]	0.24	991					Trial 02
						3	16	0.01	
						7	12	0.01	
						14	7.0	0.01	
						21	5.0	<0.01	
Makham District, Chanthaburi Province, Thailand (002.16-03) 2015	Longan (Daw)	1 [--] 2 [7] 3 [7]	0.24 0.24 0.24	1008 1008 1008	Whole fruit minus pit	0	14	0.02	Annex I Report Trial 03
						24	9.3	0.01	
						28	8.3	0.01	
						35	6.7	<0.01	
						42	4.6	<0.01	
Sanpatong District, Chaingmai Province, Thailand (002.16-04) 2016	Longan (Daw)	1 [--] 2 [8] 3 [7]	0.24 0.24 0.24	1002 1002 1002	Whole fruit minus pit	0	20	0.04	Annex I Report Trial 04
						3	14	0.05	
						7	8.8	0.03	
						14	6.0	0.01	
						21	3.6	0.01	
						26	2.4	0.01	
					Flesh	0	1.7	0.05	
						3	0.73	0.04	
						7	0.93	0.03	
						14	0.19	0.01	
						21	0.080	<0.01	
						26	0.10	<0.01	
					Peel	0	52	0.56	
						3	35	0.47	
						7	29	0.33	
						14	16	0.13	
						21	7.4	0.09	
						26	7.3	0.06	
Sanpatong District, Chaingmai Province, Thailand (002.16-05) 2016	Longan (Daw)	1 [--] 2 [7] 3 [7]	0.24 0.24 0.24	1003 1003 1003	Whole fruit minus pit	0	17, 16 [17]	0.02, 0.02 [0.02]	Annex I Report Trial 05
						3	12, 12 [12]	0.02, 0.03 [0.025]	
						7	12, 11 [11]	0.01, 0.01 [0.01]	
						14	5.1, 5.9 [5.5]	0.01, 0.01 [0.01]	
						21	3.1, 1.8 [2.5]	0.01, 0.01 [0.01]	
						26	2.2, 2.0 [2.1]	<0.01, <0.01	

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [mean]		Study report
		No. [interval, days]	Conc., kg ai/hL	L/ha			CS_2	ETU	
								[<0.01]	
					Flesh	0	2.4, 2.4 [2.4]	0.05, 0.04 [0.045]	
						3	0.42, 0.38 [0.40]	0.05, 0.02 [0.035]	
						7	0.50, 0.60 [0.55]	0.02, 0.02 [0.02]	
						14	0.43, 0.37 [0.40]	0.01, 0.01 [0.01]	
						21	0.040, 0.020 [0.030]	<0.01, <0.01 [<0.01]	
						26	0.080, 0.11 [0.095]	<0.01, <0.01 [<0.01]	
					Peel	0	36, 41 [39]	0.45, 0.47 [0.46]	
						3	33, 33 [33]	0.31, 0.32 [0.32]	
						7	39, 25 [32]	0.31, 0.27 [0.29]	
						14	14, 14 [14]	0.14, 0.13 [0.14]	
						21	13, 9.5 [11]	0.07, 0.09 [0.08]	
						26	9.8, 9.7 [9.8]	0.06, 0.07 [0.065]	
Wiang Nong Long District, Lamphun Province, Thailand (002.16-06) 2016	Longan (Daw)	1 [--] 2 [7] 3 [7]	0.24 0.24 0.24	1015 1015 1015	Whole fruit minus pit	0	13, 11 [12]	0.06 0.080 [0.07]	Annex I Report Trial 06
						3	12, 7.6 [9.6]	0.06, 0.05 [0.055]	
						7	8.9, 6.4 [7.6]	0.04, 0.03 [0.035]	
						14	4.1, 4.0 [4.0]	0.02, 0.02 [0.02]	
						21	1.0, 1.5 [1.3]	<0.01, 0.01	

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [mean]		Study report
		No. [interval, days]	Conc., kg ai/hL	L/ha			CS_2	ETU	
								[0.01]	
					Flesh	0	0.93, 0.36 [0.64]	0.05, 0.02 [0.035]	
						3	0.69, 0.42 [0.55]	0.04, 0.02 [0.03]	
						7	0.58, 0.39 [0.48]	0.01, 0.02 [0.015]	
						14	0.54, 0.14 [0.34]	0.01, 0.01 [0.01]	
						21	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	
					Peel	0	34, 33 [34]	0.58, 0.60 [0.59]	
						3	30, 28 [29]	0.67, 0.44 [0.56]	
						7	20, 20 [20]	0.26, 0.49 [0.38]	
						14	22, 17 [20]	0.18, 0.14 [0.16]	
						21	8.7, 5.8 [7.2]	0.06, 0.06 [0.06]	

Soya bean

Thirty-two field trials were conducted in Brazil during the 2013, 2014, 2015, 2017, and 2019 growing seasons (see references Table 6). Treatment generally consisted of three foliar applications of mancozeb (SC, WG, and WP formulations) ranging from ca. 1.2 to 2.6 kg ai/ha with a retreatment interval of 7 days. Harvest was targeted for 30 days after last application (DALA), with many trials including residue decline samples ranging from 25 to 40 DALA.

Following harvest, samples (≥ 1 kg) were frozen on the day of collection and transported, frozen, to the laboratory. In preparation for analysis, samples were homogenized in the presence of dry ice and returned to frozen storage. Samples were maintained in frozen storage for a maximum of 30 days prior to analysis.

Samples were analysed for residues of mancozeb (as CS_2) and ETU using the methods described above.

Table 6 Results of mancozeb residue trials in soya bean

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
Critical GAP (BR)	--	3 [7]	2.4	200	--	30	--	--	--
Engenheiro Coelho, Sao Paulo, Brazil (SP1) 2017	Soya bean (Power RR)	1 [--] 2 [7] 3 [7]	2.4 2.45 2.4	200 204 200	Seed	25	0.29, 0.24 [0.26]	--	170130
						30	0.06, 0.07 [0.06]	--	
						35	<0.056, <0.056 [<0.056]	--	
						40	<0.056, <0.056 [<0.056]	--	
Cabeceiras, Goias, Brazil (GO1) 2017	Soya bean (Syn 13610 IPRO)	1 [--] 2 [7] 3 [7]	2.4 2.4 2.4	200 200 200	Seed	25	0.10, 0.11 [0.11]	--	
						30	0.10, 0.11 [0.11]	--	
						35	<0.056, <0.056 [<0.056]	--	
						40	<0.056, <0.056 [<0.056]	--	
Estiva Gerbi, Sao Paulo, Brazil (SP2) 2017	Soya bean (Pioneer RR)	1 [--] 2 [7] 3 [7]	2.6 2.6 2.56	217 217 213	Seed	30	0.17, 0.16 [0.16]	--	
Primavera do Leste, Mato Grosso, Brazil (MT1) 2017	Soya bean (TMG 2185)	1 [--] 2 [7] 3 [7]	2.4 2.4 2.4	200 200 200	Seed	30	<0.056, <0.056 [<0.056]	--	
Londrina, Parana, Brazil (C14) 2013	Soya bean (BMX Power RR)	1 [--] 2 [7] 3 [7]	2.33 2.33 2.28	208 208 203	Seed	30	<0.056, <0.056 [<0.056]	--	000672.034.165.12
Ibiporã, Parana, Brazil (C42) 2013	Soya bean (BMX Power RR)	1 [--] 2 [7] 3 [7]	2.42 2.33 2.31	215 208 205	Seed	25	<0.056, <0.056 [<0.056]	--	
		1 [--] 2 [7] 3 [7]	2.36 2.33 2.31	210 208 205		30	<0.056, <0.056 [<0.056]	--	
		1 [--] 2 [7] 3 [7]	2.31 2.28 2.31	205 203 205		35	<0.056, <0.056 [<0.056]	--	
Tamarana, Parana, Brazil (ENSAIO 01) 2013	Soya bean (BMX RR Power)	1 [--] 2 [7] 3 [7]	2.28 2.23 2.25	203 198 200	Seed	35	0.20, 0.20 [0.20]	--	000672.034.156.12
		1 [--]	2.25	200		30	0.12, 0.13	--	

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
		2 [7] 3 [7]	2.24 2.24	199 199			[0.12]		
		1 [--] 2 [7] 3 [7]	2.27 2.26 2.27	202 201 202		25	0.21, 0.22 [0.22]	--	
Uberlandia, Minas Gerais, Brazil (ENSAIO 02) 2013	Soya bean (BMX RR Power)	1 [--] 2 [7] 3 [7]	2.27 2.30 2.27	202 204 202	Seed	30	<0.056, <0.056 [<0.056]	--	
Erebango, Rio Grande do Sul, Brazil (RA) 2014	Soya bean (NS 4823)	1 [--] 2 [7] 3 [7]	2.26 2.26 2.26	200 200 200	Seed	30	<0.056, <0.056 [<0.056]	--	13790.034. 032.14
Vera Cruz, Rio Grande do Sul, Brazil (RA) 2014	Soya bean (NK 7059 RR)	1 [--] 2 [7] 3 [7]	2.26 2.26 2.26	200 200 200	Seed	30	<0.056, <0.056 [<0.056]	--	
Cambe, Parana, Brazil (C101) 2014	Soya bean (TMG 7262 RR)	1 [--] 2 [7] 3 [7]	2.25 2.24 2.24	200 199 199	Seed	30	<0.056, <0.056 [<0.056]	--	13790.034. 031.14
Jagupita, Parana, Brazil (C107) 2014	Soya bean (BMX Tornado RR)	1 [--] 2 [7] 3 [7]	2.25 2.25 2.23	200 200 199	Seed	30	<0.056, <0.056 [<0.056]	--	
Paranapanema, Sao Paulo, Brazil (C1) 2019	Soya bean (M 6410 IPRO)	1 [--] 2 [7] 3 [7]	1.89 1.89 1.89	200 200 200	Seed	23	<0.056, <0.056, <0.056 [<0.056]	ND, ND, ND	BPL-JM-066-009-19
		1 [--] 2 [7] 3 [7]	1.89 1.89 1.89	200 200 200		30	<0.056, <0.056, <0.056 [<0.056]	ND, ND, ND	
		1 [--] 2 [7] 3 [7]	1.89 1.89 1.89	200 200 200		37	<0.056, <0.056, <0.056 [<0.056]	ND, ND, ND	
Bauru, Sao Paulo, Brazil (C2) 2019	Soya bean (Monsoy 7739 IPRO)	1 [--] 2 [7] 3 [7]	1.89 1.89 1.89	200 200 200	Seed	23	0.27, 0.26, 0.31 [0.28]	ND, ND, ND	
		1 [--] 2 [7] 3 [7]	1.89 1.89 1.89	200 200 200		30	0.17, 0.17, 0.18 [0.17]	ND, ND, ND	
		1 [--] 2 [7] 3 [7]	1.89 1.89 1.89	200 200 200		37	0.14, 0.13, 0.13 [0.13]	ND, ND, ND	
Andira, Parana, Brazil (P1) 2019	Soya bean (NA 5909 RG)	1 [--] 2 [7] 3 [7]	1.89 1.89 1.89	200 200 200	Seed	30	0.10, 0.12, 0.10 [0.11]	ND, ND, ND	

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
Campo Verde, Masto Grosso, Brazil (P2) 2019	Soya bean (W 842 RR)	1 [--] 2 [7] 3 [7]	1.89 1.89 1.89	200 200 200	Seed	30	<0.056, <0.056, <0.056 [<0.056]	ND, ND, ND	
Paranapanema, Sao Paulo, Brazil (C1) 2019	Soya bean (M 6410 IPRO)	1 [--] 2 [7] 3 [7]	1.69 1.69 1.69	200 200 200	Seed	23	<0.056, <0.056, <0.056 [<0.056]	--	BPL-JM-066-007-19
		1 [--] 2 [7] 3 [7]	1.69 1.69 1.69	200 200 200		30	<0.056, <0.056, <0.056 [<0.056]	--	
		1 [--] 2 [7] 3 [7]	1.69 1.69 1.69	200 200 200		37	<0.056, <0.056, <0.056 [<0.056]	--	
Bauru, Sao Paulo, Brazil (C2) 2019	Soya bean (Monsoy 7739 IPRO)	1 [--] 2 [7] 3 [7]	1.69 1.69 1.69	200 200 200	Seed	23	0.45, 0.42, 0.37 [0.41]	--	
		1 [--] 2 [7] 3 [7]	1.69 1.69 1.69	200 200 200		30	0.12, 0.11, 0.17 [0.13]	--	
		1 [--] 2 [7] 3 [7]	1.69 1.69 1.69	200 200 200		37	0.09, 0.08, 0.07 [0.08]	--	
Andira, Parana, Brazil (P1) 2019	Soya bean (NA 5909 RG)	1 [--] 2 [7] 3 [7]	1.69 1.69 1.69	200 200 200	Seed	30	<0.056, <0.056, <0.056 [<0.056]	--	
Campo Verde, Matto Grosso, Brazil (P2) 2019	Soya bean (W 842 RR)	1 [--] 2 [7] 3 [7]	1.69 1.69 1.69	200 200 200	Seed	30	<0.056, <0.056, <0.056 [<0.056]	--	
Paranaiba, Matto Grosso do Sul, Brazil (Ra) 2019	Soya bean (Pioneer 97R22 IPRO)	1 [--] 2 [15] 3 [15]	1.19 1.19 1.19	200 200 200	Seed	30	<0.056]	--	13790.034. 127.18
Santa Cruz do Sul, Rio Grande do Sul (CDb) 2019	Soya bean (BRS 5601 RR)	1 [--] 2 [15] 3 [15]	1.19 1.19 1.19	200 200 200	Seed	37	<0.056	--	
		1 [--] 2 [15] 3 [15]	1.19 1.19 1.19	200 200 200		30	<0.056	--	
		1 [--] 2 [15] 3 [15]	1.19 1.19 1.19	200 200 200		23	<0.056	--	
Taciba, Sao Paulo, Brazil	Soya bean (NS 6700)	1 [--] 2 [15]	1.19 1.19	199 200	Seed	30	0.24	--	13790.034. 126.18

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
(C129) 2019		3 [15]	1.19	201					
Cambe, Parana, Brazil (C120) 2019	Soya bean (TMG 7262)	1 [--] 2 [15] 3 [15]	1.19 1.19 1.19	200 199 200	Seed	23	0.33	--	
		1 [--] 2 [15] 3 [15]	1.19 1.19 1.19	200 200 199		30	0.15	--	
		1 [--] 2 [15] 3 [15]	1.19 1.19 1.19	200 199 200		37	<0.056	--	
Ourinhos, Sao Paulo, Brazil (C184) 2019	Soya bean (BMX RR Power)	1 [--] 2 [15] 3 [15]	1.41 1.37 1.44	208 203 213	Seed	30	<0.056	--	13790.034. 124.18
Cambe, Parana, Brazil (C43) 2019	Soya bean (TMG 7062 IPRO)	1 [--] 2 [15] 3 [15]	1.44 1.42 1.37	213 210 203	Seed	23	<0.056	--	
		1 [--] 2 [15] 3 [15]	1.38 1.37 1.44	205 203 213		30	<0.056	--	
		1 [--] 2 [15] 3 [15]	1.44 1.49 1.40	213 220 207		37	<0.056	--	
Santa Cruz do Sul, Rio Grande do Sul (Ra) 2015	Soya bean (Syn 1163)	1 [--] 2 [15] 3 [15]	1.34 1.34 1.34	200 200 200	Seed	30	<0.056, <0.056 [<0.056]	--	13790.034. 329.14
Rio Pardo, Rio Grand do Sul, Brazil (CDb) 2015	Soya bean (Syn 1163)	1 [--] 2 [15] 3 [15]	1.34 1.34 1.34	200 200 200	Seed	40	<0.056, <0.056 [<0.056]	--	
		1 [--] 2 [15] 3 [15]	1.34 1.34 1.34	200 200 200		30	<0.056, <0.056 [<0.056]	--	
		1 [--] 2 [15] 3 [15]	1.34 1.34 1.34	200 200 200		20	<0.056, <0.056 [<0.056]	--	
Londrina, Parana, Brazil (C02) 2015	Soya bean (BMX RR Power)	1 [--] 2 [15] 3 [15]	1.43 1.43 1.45	213 213 217	Seed	30	<0.056, <0.056 [<0.056]	--	13790.034. 330.14
Ibiporã, Parana, Brazil (C56) 2015	Soya bean (BMX RR Power)	1 [--] 2 [15] 3 [15]	1.41 1.44 1.41	210 215 210	Seed	40	<0.056, <0.056 [<0.056]	--	
		1 [--] 2 [15]	1.39 1.43	207 213		30	<0.056, <0.056 [<0.056]	--	

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
		3 [15]	1.43	213					
		1 [--] 2 [15] 3 [15]	1.41 1.45 1.43	210 217 213		20	<0.056, <0.056 [<0.056]	--	
Ponta Grossa, Parana, Brazil (152) 2015	Soya bean (NA 5909 RG)	1 [--] 2 [16] 3 [14]	2.25 2.25 2.25	200 200 200	Seed	30	<0.056, <0.056 [<0.056]	--	RS-040020/ 15
Castro, Parana, Brazil (174) 2015	Soya bean (NA 5909 RG)	1 [--] 2 [15] 3 [15]	2.25 2.25 2.25	200 200 200	Seed	20	0.09, 0.10 [0.09]	--	
		1 [--] 2 [15] 3 [15]	2.25 2.25 2.25	200 200 200		30	0.11, 0.11 [0.11]	--	
		1 [--] 2 [15] 3 [15]	2.25 2.25 2.25	200 200 200		40	0.12, 0.14 [0.13]	--	

Maize

Fifteen field trials were conducted in Brazil during the 2013, 2014, and 2017 growing seasons (see references Table 7). Treatment consisted of 3 foliar applications at either ca 1.4 or 2.4 kg ai/ha, on a 7- to 10-day interval. Samples were harvested 30 days after the last application.

Following harvest, samples (≥1 kg) were put into put into frozen storage on the day of collection. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 42 days prior to analysis.

Samples were analysed for residues of mancozeb (as CS_2) using the method described above.

Table 7 Residues of mancozeb in maize

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
Critical GAP (BR)	--	3 [7]	2.25	100	--	30	--	--	--
Ibipora, Parana, Brazil (C01) 2013	Maize (AI Pennant)	1 [-] 2 [7] 3 [7]	2.42 2.36 2.53	215 210 225	Kernels	25	<0.056, <0.056 [<0.056]	--	000672.034. 164.12
		1 [-] 2 [7] 3 [7]	2.42 2.59 2.42	215 230 215		30	<0.056, <0.056 [<0.056]	--	
		1 [-] 2 [7] 3 [7]	2.36 2.36 2.36	210 210 210		35	<0.056, <0.056 [<0.056]	--	

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
Bela Vista do Araiso, Prana, Brazil (C67) 2013	Maize (AG 9040)	1 [-] 2 [7] 3 [7]	2.59 2.31 2.36	230 205 210	Kernels	30	<0.056, <0.056 [<0.056]	--	
Cambe, Parana, Brazil (C101) 2014	Maize (Power Core)	1 [-] 2 [7] 3 [7]	2.25 2.29 2.25	200 203 200	Kernels	30	<0.056, <0.056 [<0.056]	--	13790.034. 029.14
Jaguapita, Parana, Brazil (C107) 2014	Maize (Power Core)	1 [-] 2 [7] 3 [7]	2.29 2.25 2.29	203 200 203	Kernels	30	0.07, 0.06 [0.07]	--	
Candelaria, Rio Grande do Sul, Brazil (Rb) 2014	Maize (Formula)	1 [-] 2 [7] 3 [7]	2.26 2.26 2.26	200 200 200	Kernels	30	<0.056, <0.056 [<0.056]	--	13790.034. 030.14
Rio Pardo, Rio Grande do Sul, Brazil (Rc) 2014	Maize (Formula)	1 [-] 2 [7] 3 [7]	2.26 2.26 2.26	200 200 200	Kernels	30	<0.056, <0.056 [<0.056]	--	
Engenheiro Coelho, Sao Paulo, Brazil (SP1) 2017	Maize (AL Bandeirantes)	1 [-] 2 [7] 3 [7]	2.51 2.52 2.51	209 210 209	Kernels	25	0.077	--	170129
						30	0.056	--	
						35	0.068	--	
						40	0.073	--	
Cabeceiras, Goias, Brazil (GO1) 2017	Maize (Supreme Viptera)	1 [-] 2 [7] 3 [7]	2.40 2.40 2.40	200 200 200	Kernels	25	0.095	--	
						30	0.089	--	
						35	0.056	--	
						40	0.064	--	
Estiva Gerbi, Sao Paulo, Brazil (SP2) 2017	Maize (AL Bandeirantes)	1 [-] 2 [8] 3 [7]	2.56 2.48 2.52	213 207 210	Kernels	30	0.11	--	
Primavera do Leste, Mato Grosso, Brazil (MT1) 2017	Maize (DKB 290)	1 [-] 2 [7] 3 [7]	2.40 2.40 2.40	200 200 200	Kernels	30	0.069	--	
Cambe, Parana, Brazil (C101) 2014	Maize (Power Core)	1 [-] 2 [10] 3 [10]	1.44 1.40 1.44	205 200 205	Kernels	20	0.10, 0.11 [0.10]	--	13790.034. 093.14

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
		1 [-] 2 [10] 3 [10]	1.41 1.42 1.39	202 203 198		30	<0.056, <0.056 [<0.056]	--	
		1 [-] 2 [10] 3 [10]	1.40 1.39 1.40	200 198 200		40	<0.056, <0.056 [<0.056]	--	
Jaguapita, Parana, Brazil (C107) 2014	Maize (Power Core)	1 [-] 2 [10] 3 [10]	1.40 1.39 1.40	200 198 200	Kernels	30	<0.056, <0.056 [<0.056]	--	
Ibipora, Prana, Brazil (C01) 2014	Maize (AL Bandeirantes)	1 [-] 2 [10] 3 [10]	1.51 1.51 1.49	215 215 213	Kernels	30	<0.056, <0.056 [<0.056]	--	13790.034. 094.14
Candelaria, Rio Grande do Sul, Brazil (Ra) 2014	Maize (Formula)	1 [-] 2 [10] 3 [10]	1.40 1.40 1.40	200 200 200	Kernels	30	0.34, 0.32 [0.33]	--	13790.034.095.14
Rio Pardo, Rio Grande do Sul, Brazil (CDb) 2014	Maize (Formula)	1 [-] 2 [10] 3 [10]	1.40 1.40 1.40	200 200 200	Kernels	20	<0.056, <0.056 [<0.056]	--	
		1 [-] 2 [10] 3 [10]	1.40 1.40 1.40	200 200 200		30	<0.056, <0.056 [<0.056]	--	
		1 [-] 2 [10] 3 [10]	1.40 1.40 1.40	200 200 200		40	<0.056, <0.056 [<0.056]	--	
Santa Cruz do Sul, Rio Grande do Sul (Ra) 2018	Maize (BG 6070)	1 [-] 2 [10] 3 [10]	2.025 2.025 2.025	200 200 200	Kernels	45	<0.056	--	13790.034.150.17
Rio Pardo, Rio Grande do Sul, Brazil (CDb) 2018	Maize (BG 6070 HR)	1 [-] 2 [10] 3 [10]	2.025 2.025 2.025	200 200 200	Kernels	38	<0.056	--	
		1 [-] 2 [10] 3 [10]	2.025 2.025 2.025	200 200 200		45	0.19	--	
		1 [-] 2 [10] 3 [10]	2.025 2.025 2.025	200 200 200		52	<0.056	--	

Rice

Nineteen field trials were conducted in Brazil during the 2003, 2004, and 2014 to 2018 growing seasons (see references Table 8). Treatment consisted of either 3 foliar applications ranging from ca. 1.19 to 2.16 kg ai/ha, on a 10- to 14-day interval or 2 foliar applications at ca. 2.4 or 4.8 kg ai/ha on a 3- or 8- to 9-day interval. Samples were harvested 18 to 46 DALA, with most harvests occurring around 32 DALA.

Following harvest, samples (≥1 kg) were put into put into frozen storage on the day of collection. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 425 days prior to analysis.

Samples were analysed for residues of mancozeb (as CS_2) using the method described above.

Table 8 Residues of mancozeb in rice

Location (Trial ID) Year	Crop (Variety)	Application			Portion [a)]	DALA	Residues (mg/kg) [Mean]		Study report
		No. [int. days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
Critical GAP (BR)	--	3 [10]	2.8	100	--	32	--	--	--
Sertanopolis, Parana, Brazil (C150) 2018	Rice (IPR 117)	1 [-] 2 [10] 3 [10]	2.16 2.13 2.11	213 210 208	Caryopsis	25	0.28	--	13790.034.152.17
		1 [-] 2 [10] 3 [10]	2.16 2.06 2.16	213 203 213		32	0.22	--	
		1 [-] 2 [10] 3 [10]	2.10 2.13 2.13	207 210 210		39	0.09	--	
Ibipora, Parana, Brazil (C01) 2014	Rice (IPR 117)	1 [-] 2 [10] 3 [10]	1.49 1.47 1.49	213 210 213	Caryopsis	32	0.30, 0.33 [0.32]	--	13790.034.085.14
Santa Maria, Rio Grande do Sul, Brazil (Ra) 2015	Rice (Irga 421)	1 [-] 2 [10] 3 [10]	1.40 1.40 1.40	200 200 200	Caryopsis	32	<0.028, <0.028 [<0.028]	--	13790.034.086.14
Candelaria, Rio Grande do Sul, Brazil (CDbb) 2015	Rice (Inta Puita CL)	1 [-] 2 [10] 3 [10]	1.41 1.41 1.41	200 200 200	Caryopsis	25	0.05, 0.05 [0.05]	--	
		1 [-] 2 [10] 3 [10]	1.41 1.41 1.41	200 200 200		32	<0.028, <0.028 [<0.028]	--	
		1 [-] 2 [10] 3 [10]	1.41 1.41 1.41	200 200 200		38	0.031, 0.027 [0.03]	--	
Santa Cuz do Sul, Rio Grande do Sul, Brazil (CDc) 2015	Rice (Irga 421)	1 [-] 2 [10] 3 [10]	1.41 1.41 1.41	200 200 200	Caryopsis	25	0.10, 0.10 [0.10]	--	
		1 [-] 2 [10] 3 [10]	1.41 1.41 1.41	200 200 200		32	<0.028, <0.028 [<0.028]	--	
		1 [-]	1.41	200		38	<0.028,	--	

Location (Trial ID) Year	Crop (Variety)	Application			Portion [a)]	DALA	Residues (mg/kg) [Mean]		Study report
		No. [int. days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
		2 [10] 3 [10]	1.41 1.41	200 200			<0.028 [<0.028]		
Santa Maria, Rio Grande do Sul, Brazil (01) 2016	Rice (Piuta)	1 [-] 2 [14] 3 [13]	1.47 1.48 1.45	206 207 203	Caryopsis	34	<0.028, <0.028 [<0.028]	--	13790.034.218.15
Engenheiro Coelho, Sao Paulo, Brazil (02) 2016	Rice (Guri Inta CL)	1 [-] 2 [14] 3 [13]	1.54 1.47 1.41	216 206 212	Caryopsis	29	0.73, 0.82 [0.78]	--	
		1 [-] 2 [14] 3 [13]	1.49 1.52 1.52	208 212 212		35	0.67, 0.72 [0.70]	--	
		1 [-] 2 [14] 3 [13]	1.47 1.46 1.47	205 204 206		42	0.45, 0.42 [0.44]	--	
Santa Cruz do Sul, Rio Grande do Sul, Brazil (RA) 2016	Rice (Puita Inta CL)	1 [-] 2 [14] 3 [14]	1.43 1.43 1.43	200 200 200	Caryopsis	35	0.20, 0.20 [0.20]	--	13790.034.217.15
Candelaria, Rio Grande do Sul, Brazil (CDb) 2016	Rice (Puita Inta CL)	1 [-] 2 [14] 3 [14]	1.43 1.43 1.43	200 200 200	Caryopsis	28	0.53, 0.53 [0.53]	--	
		1 [-] 2 [14] 3 [14]	1.43 1.43 1.43	200 200 200		35	0.25, 0.24 [0.25]	--	
		1 [-] 2 [14] 3 [14]	1.43 1.43 1.43	200 200 200		42	0.12, 0.10 [0.11]	--	
Engenheiro Coelho, Sao Paulo, Brazil (C104) 2017	Rice (IAC 300))	1 [-] 2 [14] 3 [14]	1.27 1.25 1.31	213 210 219	Caryopsis	40	0.31, 0.32 [0.32]	--	13790.034.053.16
					Rice without husk	40	0.07, 0.07 [0.07]		
Tamarana, Parana, Brazil (C94) 2017	Rice (IRGA 428)	1 [-] 2 [14] 3 [14]	1.24 1.23 1.31	208 207 220	Caryopsis	45	0.08, 0.06 [0.07]	--	
					Rice without husk	45	0.07, 0.07 [0.07]		
		1 [-] 2 [14] 3 [14]	1.31 1.26 1.25	220 212 210	Caryopsis	40	0.29, 0.29 [0.29]	--	
					Rice without husk	40	0.08, 0.09 [0.09]		
		1 [-] 2 [14] 3 [14]	1.21 1.21 1.31	203 203 220	Caryopsis	35	0.40, 0.36 [0.38]	--	
					Rice without husk	35	0.10, 0.11 [0.11]	--	

Location (Trial ID) Year	Crop (Variety)	Application			Portion [a)]	DALA	Residues (mg/kg) [Mean]		Study report
		No. [int. days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
Candelaria, Rio Grande do Sul, Brazil (Ra) 2017	Rice (Puita Inta CL)	1 [-] 2 [14] 3 [14]	1.19 1.19 1.19	200 200 200	Caryopsis	40	0.25, 0.24 [0.25]	--	13790.034. 052.16
					Rice without husk	40	0.13, 0.11 [0.12]	--	
Santa Cuz do Sul, Rio Grande do Sul, Brazil (CDb) 2017	Rice (Puita Inta CL)	1 [-] 2 [14] 3 [14]	1.19 1.19 1.19	200 200 200	Caryopsis	35	0.20, 0.17 [0.18]	--	
					Rice without husk	35	0.14, 0.15 [0.14]	--	
		1 [-] 2 [14] 3 [14]	1.19 1.19 1.19	200 200 200	Caryopsis	40	0.14, 0.13 [0.13]	--	
					Rice without husk	40	0.11, 0.11 [0.11]	--	
		1 [-] 2 [14] 3 [14]	1.19 1.19 1.19	200 200 200	Caryopsis	45	0.07, 0.06 [0.07]	--	
					Rice without husk	45	0.06, 0.06 [0.06]	--	
Dona Francisca, Rio Grande do Sul (WP Formulation) 2003	Rice (Irga 417)	1 [-] 2 [9]	2.40 2.40	200 200	Caryopsis	18	0.23	--	GHB-P 1046
					Caryopsis	25	<0.10	--	
					Caryopsis	32	0.40	--	
					Husked rice	32	<0.10	--	
					Caryopsis	39	0.40	--	
					Husked rice	39	<0.10	--	
					Caryopsis	46	0.38	--	
					Husked rice	46	<0.10	--	
		1 [-] 2 [9]	4.80 4.80	200 200	Caryopsis	18	0.23	--	
					Caryopsis	25	<0.10	--	
					Caryopsis	32	0.58	--	
					Husked rice	32	<0.10	--	
					Caryopsis	39	0.66	--	
					Husked rice	39	<0.10	--	
					Caryopsis	46	0.59	--	
					Husked rice	46	<0.10	--	
Dona Francisca, Rio Grande do Sul (DG Formulation) 2003	Rice (Irga 417)	1 [-] 2 [9]	2.25 2.25	200 200	Caryopsis	18	0.19	--	GHB-P 1047
						25	<0.10	--	
						32	0.12	--	
					Husked rice	32	<0.10	--	
					Caryopsis	39	0.13	--	
					Husked rice	39	<0.10	--	
					Caryopsis	46	0.12	--	

Location (Trial ID) Year	Crop (Variety)	Application			Portion [a)]	DALA	Residues (mg/kg) [Mean]		Study report
		No. [int. days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
					Husked rice	46	<0.10	--	
		1 [-] 2 [9]	4.5 4.5	200 200	Caryopsis	18	1.2	--	
						25	<0.10	--	
						32	0.96	--	
					Husked rice	32	<0.10	--	
					Caryopsis	39	0.75	--	
					Husked rice	39	<0.10	--	
					Caryopsis	46	0.77	--	
					Husked rice	46	<0.10	--	
Faxinal do Soturno, Rio Grande do Sul, Brazil (WP Formulation) 2003	Rice (Irga 417)	1 [-] 2 [8]	2.40 2.40	200 200	Caryopsis	32	0.44	--	GHB-P 1048
					Husked rice	32	<0.10	--	
		1 [-] 2 [8]	4.80 4.80	200 200	Caryopsis	32	0.90	--	
					Husked rice	32	<0.10	--	
Faxinal do Soturno, Rio Grande do Sul, Brazil (DG Formulation) 2003	Rice (Irga 417)	1 [-] 2 [8]	2.25 2.25	200 200	Caryopsis	32	0.31	--	GHB-P 1049
					Husked rice	32	<0.10	--	
		1 [-] 2 [8]	4.5 4.5	200 200	Caryopsis	32	2.0	--	
					Husked rice	32	<0.10	--	
Mogi Mirim, Sao Paulo, Brazil (WP Formulation) 2004	Rice (IAC 202)	1 [-] 2 [3]	2.40 2.40	200 200	Caryopsis	38	0.30	--	GHB-P 1146
					Husked rice	38	<0.10	--	
		1 [-] 2 [3]	4.80 4.80	200 200	Caryopsis	38	0.40	--	
					Husked rice	38	<0.10	--	
Mogi Mirim, Sao Paulo, Brazil (DG Formulation) 2004	Rice (IAC 202)	1 [-] 2 [3]	2.25 2.25	200 200	Caryopsis	38	0.20	--	GHB-P 1147
					Husked rice	38	<0.10	--	
		1 [-] 2 [3]	4.5 4.5	200 200	Caryopsis	38	0.30	--	
					Husked rice	38	<0.10	--	

Note:

[a)] Caryopsis = rice with husk

Cotton

Fourteen field trials were conducted in Brazil during the 2013, 2016, or 2017 seasons. Treatment consisted of three foliar applications, each at ca. 2.4 or 1.5 kg ai/ha, on a 7- or 14-day interval. Samples of cotton seeds were harvested 20 to 42 DALA, with most harvests occurring around 30 DALA. Samples of cotton seed were ginned to produce undelinted cotton seed. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 40 days prior to analysis.

Samples were analysed for residues of mancozeb (as CS_2) and ETU using the methods described above.

Table 9 Residues of mancozeb in undelinted cotton seed

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
Critical GAP (BR)	--	3 [7]	2.25	100	--	30	--	--	--
Ibipora, Parana, Brazil (C56) 2013	Cotton (Delta Opal)	1 [-] 2 [7] 3 [7]	2.3 2.3 2.3	204 208 207	Undelinted seed	25	<0.056, <0.056 [<0.056]	--	000672.034. 163.12
						30	<0.056, <0.056 [<0.056]	--	
						35	<0.056, <0.056 [<0.056]	--	
Bela Vista do Paraiso, Parana, Brazil (C67) 2013	Cotton (Delta Opal)	1 [-] 2 [7] 3 [7]	2.5 2.3 2.4	220 205 215		30	<0.056, <0.056 [<0.056]	--	
Montividiu, Goias, Brazil (n.s.) 2013	Cotton (FM 966 LL)	1 [-] 2 [7] 3 [7]	2.25 2.25 2.25	187 189 192	Undelinted seed	35	<0.056, <0.056 [<0.056]	--	000672.034. 153.12
		1 [-] 2 [7] 3 [7]	2.25 2.25 2.25	193 198 200		30	<0.056, <0.056 [<0.056]	--	
		1 [-] 2 [7] 3 [7]	2.25 2.25 2.25	192 190 199		25	<0.056, <0.056 [<0.056]	--	
Uberlandia, Minas Gerais, Brazil (n.s.) 2013	Cotton (DP 555 B6 RR)	1 [-] 2 [7] 3 [7]	2.25 2.25 2.25	198 202 212	Undelinted seed	30	0.14, 0.12 [0.13]	--	
Botucatu, Sao Paulo, Brazil (SP1) 2017	Cotton (Bollgard II RR Flex)	1 [-] 2 [7] 3 [7]	2.4 2.5 2.5	200 208 208	Undelinted seed	25	0.056	--	170131
						30	<0.056	--	
						35	0.23	--	
						40	0.24	--	

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
Planaltina, Distrito Federal, Brazil (DF1) 2017	Cotton (FM 975 WS)	1 [-] 2 [7] 3 [7]	2.4 2.4 2.4	200 200 200	Undelinted seed	25	<0.056	--	
						30	0.066	--	
						35	0.11	--	
						40	0.059	--	
Primavera do Leste, Mato Grosso, Brazil (MT1) 2017	Cotton (FM 975 WS)	1 [-] 2 [7] 3 [7]	2.4 2.4 2.4	200 200 200	Undelinted seed	30	0.097	--	
Sao Desiderio, Bahia, Brazil (BA1) 2017	Cotton (FM 975 WS)	1 [-] 2 [7] 3 [7]	2.4 2.4 2.4	200 200 200	Undelinted seed	30	<0.056	--	
Ibipora, Parana, Brazil (C56) 2016	Cotton (FM 982 GL)	1 [-] 2 [14] 3 [14]	1.8 1.8 1.8	213 210 213	Undelinted seed	42	<0.056, <0.056 [<0.056]	--	13790.034.199.15
		1 [-] 2 [14] 3 [14]	1.7 1.8 1.8	207 213 210		35	<0.056, <0.056 [<0.056]	--	
		1 [-] 2 [14] 3 [14]	1.8 1.7 1.8	213 207 213		28	<0.056, <0.056 [<0.056]	--	
Londrina, Parana, Brazil (C05) 2016	Cotton (FM 982 GL)	1 [-] 2 [14] 3 [14]	1.8 1.8 1.8	213 210 213	Undelinted seed	35	<0.056, <0.056 [<0.056]	--	
Ourinhos, Sao Paulo, Brazil (C184) 2016	Cotton (FM 982 GL)	1 [-] 2 [14] 3 [14]	1.6 1.6 1.6	213 210 213	Undelinted seed	30	0.12, 0.12 [0.12]	--	13790.034.213.15
Ibipora, Parana, Brazil (C56) 2016	Cotton (FM 982 GL)	1 [-] 2 [14] 3 [14]	1.5 1.5 1.6	207 207 210	Undelinted seed	20	<0.056, <0.056 [<0.056]	--	
		1 [-] 2 [14] 3 [14]	1.6 1.6 1.6	213 210 213		30	<0.056, <0.056 [<0.056]	--	
		1 [-] 2 [16] 3 [14]	1.6 1.6 1.6	210 213 213		40	<0.056, <0.056 [<0.056]	--	
Engenheiro Coelho, Sao Paulo, Brazil (C1) 2016	Cotton (FMT-707)	1 [-] 2 [14] 3 [14]	1.5 1.5 1.5	200 200 200	Undelinted seed	20	<0.056	<0.1	BPL-JM-066-002-16
						30	<0.056	<0.1	
						40	<0.056	<0.1	
Ribeirao Preto,	Cotton	1 [-]	1.5	200	Undelinted	30	0.39	<0.1	

Location (Trial ID) Year	Crop (Variety)	Application			Portion	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, kg ai/ha	L/ha			CS_2	ETU	
Sao Paulo, Brazil (P1) 2016	(FM 940 GLT)	2 [14] 3 [14]	1.5 1.5	200 200	seed				

FATE OF RESIDUES IN STORAGE AND PROCESSING

The Meeting received two studies examining residues of mancozeb and ETU in maize kernels and processed commodities.

In the first study (Schweitzer, M.G., 1989, Report 34-89-21), a single field trial was conducted in the United States. Mancozeb, as a WP formulation was broadcast applied to maize at rates of either 1.35 or 8.1 kg/ha. Six applications were made on a ca. 14-day interval. Maize ears were allowed to dry in the field and then harvested 41 DALA. Harvested ears were shelled and processed into grits, meal, flour refined oil (dry and wet milling), and starch (wet milling) within ca. 1 month of harvest using simulated commercial dry and wet milling practices (Figure 1, Figure 2).

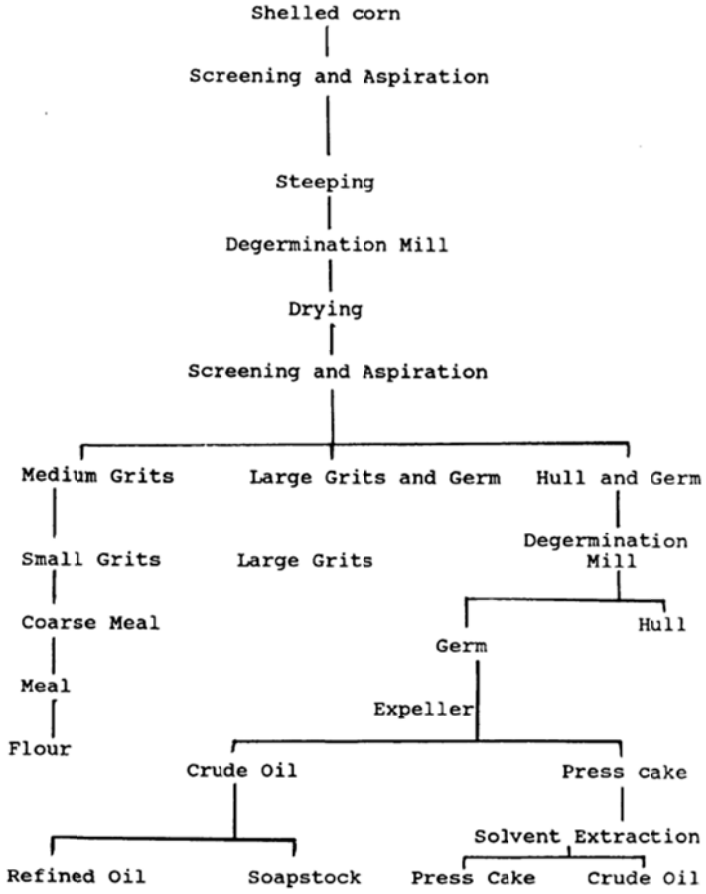

Figure 1 Schematic of maize dry milling processing (Report 34-89-21)

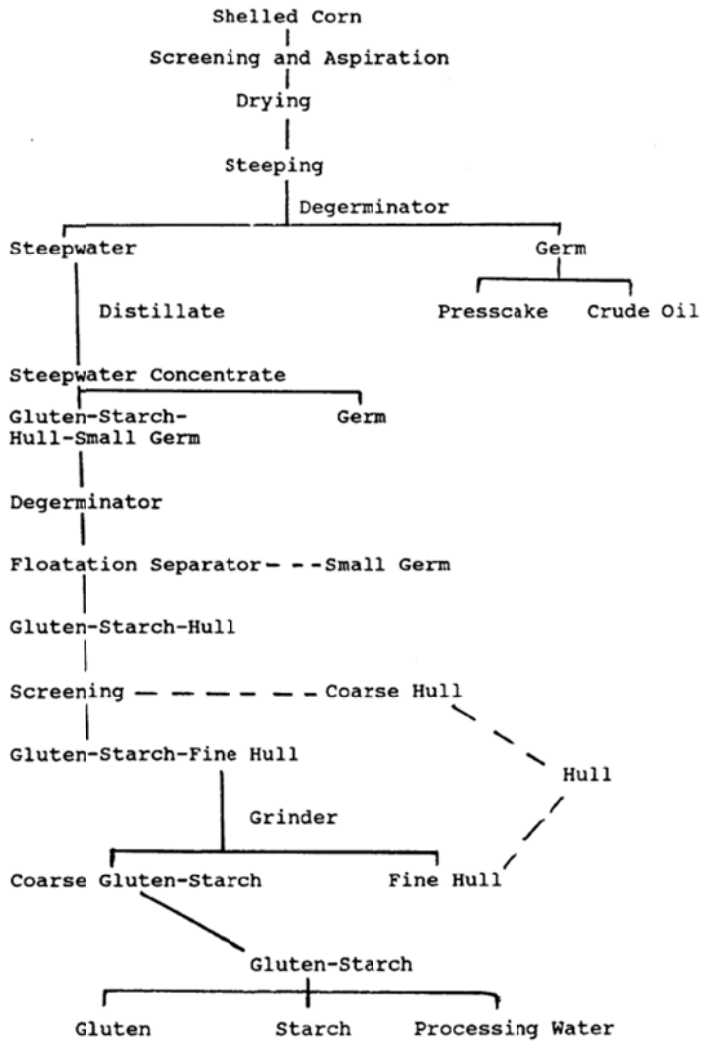

Figure 2 Schematic of maize wet milling processing (Report 34-89-21)

Residues of mancozeb were determined using the method for CS_2 described above, with the exception that a mixture of NaOH/benzene, rather than isooctane, was used to trap CS_2. Procedural recoveries (Table 2) ranged from 87 to 98 percent across all matrices. Residues of ETU were extracted into methanol, followed by clean-up and derivatisation, with analysis by GC-FPD. Procedural recoveries were variable but generally acceptable for all matrices except meal, solvent extracted oil, crude oil, and soapstock.

In the second study (Singer, G.M., 1996, Report AA920302), a single field trial was conducted in the United States. Mancozeb, as a DF formulation was broadcast applied to Pioneer 3489 variety of maize at rates of 6.7 to 6.8 kg/ha. Ten applications were made on a 6- to 13-day interval. Maize ears were harvested 40 DALA. Harvested kernels (ca. 10 kg) were placed into frozen storage within 45 minutes of sampling and shipped frozen to the processing facility. Samples were processed into grits, meal, flour refined oil (dry and wet milling), and starch (wet milling) within ca. 1 month of harvest using simulated commercial dry and wet milling practices (Figure 3, Figure 4).

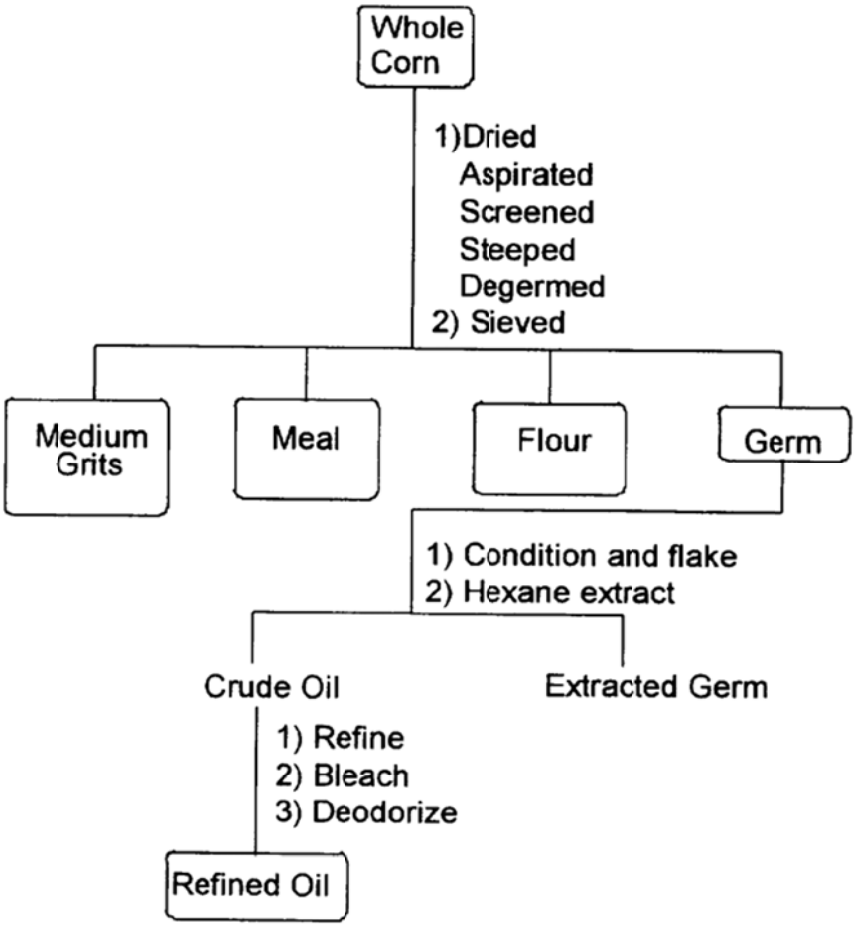

Figure 3 Schematic of maize dry milling processing (Report AA950302)

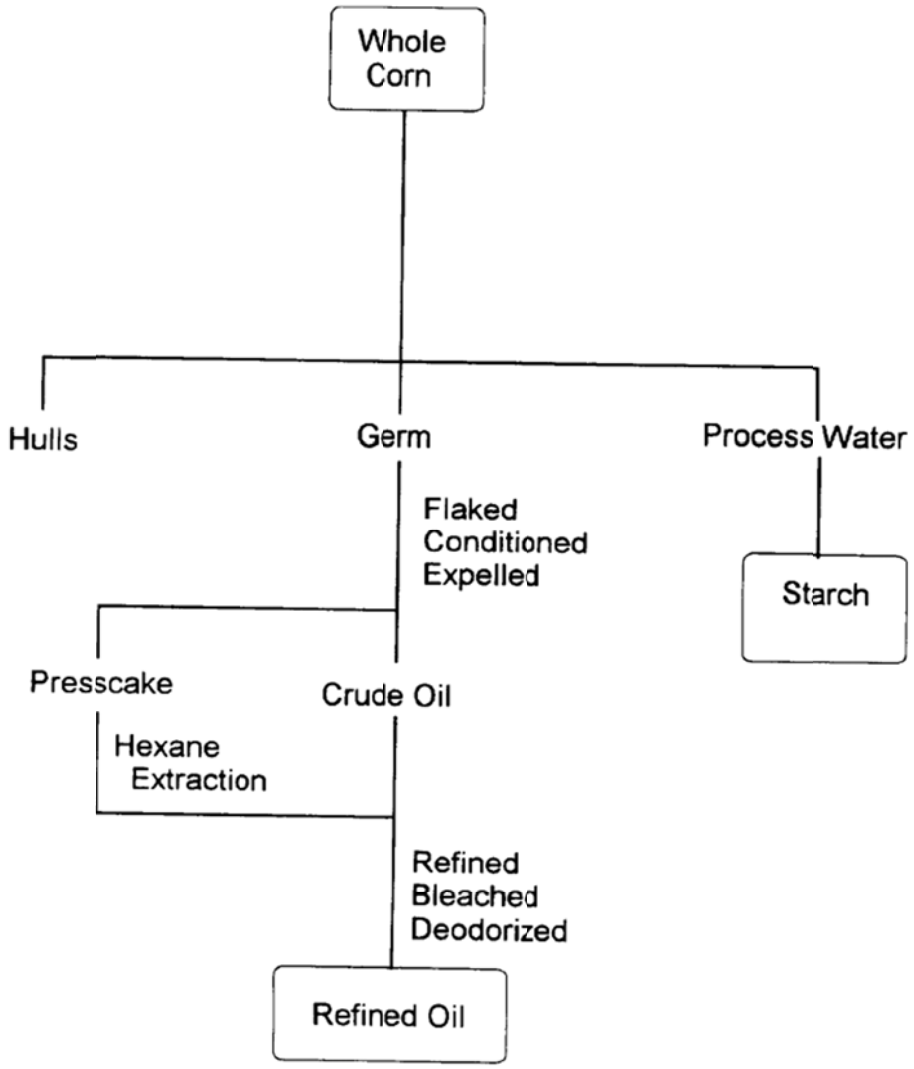

Figure 4 Schematic of maize wet milling processing (Report AA950302)

Residues of mancozeb were determined using the method for CS_2 described above. Procedural recoveries (Table 2) were generally acceptable for all matrices except in flour at the 0.05-mg/kg fortification level and in refined oil (both dry and wet milling, all fortification levels). Residues of ETU were extracted into alkaline solvent and analysed by HPLC-EC. Procedural recoveries were acceptable for all matrices.

Residues of mancozeb and ETU, and their associated processing factors, are summarised below for both studies. For the first study (34-89-21), residues of CS_2 and ETU were below the LOQ in kernels and in most processed commodities; only processed commodities with quantifiable residues at the higher rate are reported here.

Table 10 Residues and processing factors for mancozeb (as CS_2) and ETU in maize commodities

Crop	Commodity	Study Number	CS_2, mg/kg [mean]	CS_2 Processing factor	ETU, mg/kg	ETU Processing factor
Maize	Kernels	AA950302	0.38, 0.51, 0.35 [0.41]	--	0.05	--
		34-89-21	<0.028, <0.028	--	<0.01	--

Crop	Commodity	Study Number	CS_2, mg/kg [mean]	CS_2 Processing factor	ETU, mg/kg	ETU Processing factor
			[<0.028]			
	Grits	AA950302	0.084	0.205	<0.01	<0.2
	Meal	AA950302	0.358	0.873	<0.01	<0.2
		34-89-21	--	--	0.01	>1
	Flour	AA950302	0.49, 0.41, 0.43 [0.44]	1.07	<0.01	<0.2
		34-89-21	--	--	0.01	>1
	Refined oil (dry milling)	AA950302	<0.05	<0.122	<0.01	<0.2
	Refined oil (wet milling)	AA950302	<0.05	<0.122	<0.01	<0.2
	Starch	AA950302	<0.05	<0.122	<0.01	<0.2
	Screenings	34-89-21	0.067	>2.29	0.01	>1
	Presscake (dry milling)	34-89-21	0.135	>5.4	--	--
	Crude oil (dry milling)	34-89-21	0.028	>1	--	--
	Solvent-extracted crude oil (dry milling)	34-89-21	0.073	>2.61	--	--
	Processing water (wet milling)	34-89-21	0.028	>1	--	--
	Gluten (wet milling)	34-89-21	0.028	>1	--	--
	Steepwater concentrate (wet milling)	34-89-21	--	--	0.05	>5

RESIDUE AND ANALYTICAL ASPECTS

Mancozeb was evaluated in 1993 within the CCPR periodic review programme. Mancozeb was last evaluated for new MRLs by the 2014 Meeting.

Mancozeb was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional MRLs by the 2021 Extra JMPR and was re-scheduled to the 2022 JMPR.

The mancozeb residue definition for compliance with MRLs in plants and animals is defined as total dithiocarbamates, determined as CS_2, evolved during acid digestion and expressed as mg CS_2/kg. Dithiocarbamate residues are not fat soluble.

In 1993, the JMPR established a group (alone or in any combination) ADI of 0–0.03 mg/kg bw for ethylene-bis-dithiocarbamates (EBDCs: mancozeb, maneb, metiram and zineb) and an ADI of 0–0.004 mg/kg bw for their metabolite ethylenethiourea (ETU). The parent EBDC and ETU are defined as the residues for evaluating dietary intake in plant and animal commodities. The Meeting is assessing combined residues of mancozeb and ETU using the ratio of the ADIs (7.5) to express residues in terms of mancozeb-toxicity-equivalents (MTE).

At present, there are no ARfDs established by the JMPR for EBDCs or ETU.

The current Meeting received information on analytical methods, storage stability, supervised residue trials, and processing (maize only) to support new MRLs in longan, soya bean, maize, rice, and cottonseed.

Methods of analysis

The Meeting received method validation for analysis of mancozeb (as CS_2) in maize kernels as well as concurrent recovery data for other matrices for which field trials were provided. All methods for analysis of CS_2 use the same basic principles: extraction of mancozeb into an acidified stannous chloride solution with isooctane, *in-situ* conversion of mancozeb to CS_2 which is captured in the isooctane, and analysis of the residue by GC-MS. While validation data for the full method were not provided for all matrix types reviewed by the Meeting, the Meeting agreed that extraction and CS_2 conversion step has been previously validated and that the trapping of CS_2 in organic solvent with analysis by GC has been shown to be a reliable technique[2,3] for the analysis of CS_2. All methods were demonstrated to have adequate performance for recovery of mancozeb (as CS_2), with an LOQ of 0.056 mg/kg in all commodities tested, except for rice (0.028 mg/kg).

The Meeting also received concurrent recovery data for analysis of ETU in longan fruit, soya bean seed, maize, and cotton seed. Neither method validation nor radiovalidation data were provided.

Reported recoveries of ETU in longan fruit ranged from 91 to 104 percent at fortification levels ranging from 0.01 to 0.05 mg/kg, with RSD <7 percent. The description for the method used for the analysis of ETU in longan fruit cites a modified QuEChERS method[4] that uses alkaline acetonitrile as the extraction solvent. The information provided to the Meeting noted that QuEChERS was used for analysis of fruits but did not specif that alkaline acetonitrile was used in the analysis of ETU in longan fruit. Furthermore, there is no information available on the ability of the method to extract incurred residues of ETU. Noting that methods for the analysis of ETU reviewed by prior Meetings and found to be acceptable used non-polar solvents (dichloromethane) or alkaline polar solvents (aqueous ammonia (pH 11–12) + either methanol or ethanol, the Meeting agreed that it could not conclude on the suitability of the method for estimating residues of ETU in longan fruit.

Reported recoveries of ETU in soya bean seed and cotton seed ranged from 69 to 95 percent at fortification levels ranging from 0.1 to 1 mg/kg, with RSD <10 percent. Residues were extracted with methanol with analysis by LC-MS/MS. As noted above, methods previously considered adequate by the JMPR use alkaline methanol for the extraction of ETU. While suitable concurrent recoveries were obtained, there is no information available on the ability of the method to extract incurred residues of ETU. The Meeting agreed that it could not conclude on the suitability of the method for estimating residues of ETU in soya bean seed or cotton seed.

Stability of pesticide residues in stored analytical samples

Concurrent storage stability data were provided for residues of mancozeb in longan fruit. The data demonstrate that residues of mancozeb, analysed as CS_2, are stable in frozen longan fruit for at least 300 days. Samples from the longan trials were stored at ≤ 20 °C for a maximum of 300 days.

For other commodities considered by the present Meeting except rice, samples were kept in frozen storage for up to 42 days. The 1993 Meeting concluded that mancozeb residues are stable during

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[2] Woodrow, J. E., Seiber, J.N., Fitzell, D. 1995. Analytical Method for the Dithiocarbamate Fungicides Ziram and Mancozeb in Air: Preliminary Field Results. *J. Agric. Food Chem*. 43:1524-1539

[3] Abakerli, R.B., Sparrapan, R., Sawaya, A.C.H.F., Eberlin, M.N., Jara, J.L.P., Rodrigues, N.R., Fay, E.F., Luiz, A.J.B., Galvão, T.D.L., dos S. Martins, D., Yamanishi, O.K., Toledo, H.H.B. 2015. Carbon Disulfide Formation in Papaya Under Conditions of Dithiocarbamate Residue Analysis. *Food Chemistry*. 188:71-76

[4] Zhou, L., Liu, X., Kang, S, Zhang, F., Pan, C. 2013. A Rapid Determination Method for Ethylenethiourea in Potato and Cucumber by Modified QuEChERS – High Performance Liquid Chromatography-Tandem Mass Spectrometry. *Food Chemistry*. 138:1355-1359

frozen storage for a duration of at least 3 months in commodities similar to those being considered by the current Meeting. Rice samples were stored for up to 425 days. In a storage stability study in wheat reviewed by the 1993 JMPR, residues of mancozeb showed a slow decline beginning at six months and continuing until the end of the study at 24 months. The 1993 JMPR concluded that in wheat grain samples from a storage stability study, mancozeb stability was in the normally acceptable range for up to 2 years. The Current Meeting agreed the storage stability conclusion for wheat supported the storage duration for rice.

Results of supervised residue trials on crops

For estimating maximum residue levels, residues of mancozeb listed below are expressed as CS_2.

For estimating dietary exposure, combined residues of mancozeb and ETU (expressed as mancozeb toxicity-equivalents (MTE)) are calculated based on molecular weights and a potency factor for ETU of 7.5 (derived from the ratio of the ADI for mancozeb to that of ETU). The conversion factor for CS_2 to mancozeb is 1.777 (CS_2=76.1 g/mol; mancozeb = 541.0 g/mol; 4 molecules CS_2 per molecule mancozeb). However, the Meeting noted that in metabolism and field trials evaluated by previous Meetings, residues of ETU are consistently < 0.01 mg/kg and at least an order of magnitude less than residues of mancozeb (expressed as CS_2). Therefore, the Meeting agreed to assume residues of ETU in raw commodities were 0 mg/kg; thus, residues for dietary risk assessment for commodities that are consumed raw were derived directly from residues of mancozeb as 1.777×[CS_2]. Furthermore, the Meeting agreed that for RACs that are heated/cooked prior to consumption (i.e., soya bean, maize, rice, and cottonseed), all residues of mancozeb would be converted to ETU; therefore, STMRs were estimated as 7.5 times the median residue of mancozeb in the RAC (equivalent to 13.33×[CS_2]).

The Meeting received data from supervised residue trials and GAP information on longan, soya beans, maize, rice, and cotton.

Longan

The information provided for residues in longan fruit consisted of summary reports rather than complete study volumes. Nevertheless, sufficient details were provided to be able to evaluate the data for purposes of making recommendations.

The critical GAP for longan is from Thailand. The label allows three applications on a 7-day interval at a maximum spray concentration of 0.24 kg ai/hL and a spray rate of 5 L/tree, with a 14-day PHI.

Residues of CS_2 in the whole fruit (minus the pit) from independent trials matching the critical GAP were (n=5): 2.2, 4.0, 5.5, 6.0, and 7.0 mg/kg.

Based on the reported total sample size and weight (120 fruits, 2 kg) and on the reported weight of longan fruit pits (ca. 1.6 g), the Meeting estimated that the pit constitutes approximately 10 percent of the whole-fruit weight and agreed that an adjustment to the residues to account for weight of the pit is not necessary. The Meeting estimated a maximum residue level of 15 mg/kg for CS_2 in longan fruit.

The median residue of CS_2 was 5.5 mg/kg resulting in an STMR of 9.8 mg MTE/kg.

Soya bean

The critical GAP for soya beans is from Brazil and consists of three applications each at 2.4 kg ai/ha, on a 7-day interval, with a 30-day PHI.

Residues of CS_2 in seed from independent trials matching the critical GAP were (n=12): < 0.056 (8), 0.06, 0.11, 0.16, and 0.22 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for CS_2 in soya bean seed.

The median residue of CS_2 was < 0.056 mg/kg resulting in an STMR of 0.75 mg MTE/kg.

Maize

The critical GAP for maize is from Brazil and consists of three applications each at 2.25 kg ai/ha, on a 7-day interval, with a 30-day PHI.

Residues of CS_2 in kernels from independent trials matching the critical GAP were (n=10): < 0.056 (5), 0.069, 0.070, 0.073, 0.089, and 0.11 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg for CS_2 in maize kernels.

The median residue of CS_2 was 0.0625 mg/kg resulting in an STMR of 0.83 mg MTE/kg.

Rice

The critical GAP for rice is from Brazil and consists of three applications each at 2.8 kg ai/ha, on a 10-day interval, with a 32-day PHI.

Residues of CS_2 in rice grain (paddy rice) from independent trials matching the critical GAP for application and harvest timing but with applications rates ranging from 1.4 to 2.2 kg ai/ha were (n=5): < 0.028 (2), 0.030, 0.22, and 0.32 mg/kg.

After scaling based on application rate (scaling factor range: 1.3–1.99), residues in rice grain from trials with quantifiable residues were (n=3): 0.060, 0.29, and 0.61 mg/kg.

The Meeting agreed that three trials were not sufficient to make a recommendation for rice.

Additional trials were conducted with application rates ranging from 1.2 to 1.5 kg ai/ha at intervals of 14 days and with harvest 34–35 DALA. Quantifiable residues from those trials were (n=5): 0.18, 0.20, 0.25, 0.38, and 0.70 mg/kg.

After scaling based on application rate (scaling factor range: 1.95–2.25), residues in rice grain from trials with quantifiable residues were (n=5): 0.39, 0.42, 0.48, 0.85, and 1.4 mg/kg.

There are too few data points from the first set of trials listed above to complete a statistical comparison of the two data sets. Based on the perceived similarity in the data sets, the Meeting agreed to combine the scaled results from the two sets of trials (n=8): 0.06, 0.29, 0.39, 0.42, 0.48, 0.61, 0.85, and 1.4 mg/kg. The Meeting estimated a maximum residue level of 3 mg/kg for rice grain.

Analysis of husked rice was included for two sites from the second set of trials. Residues in husked rice and rice grain were, respectively, 0.11 mg/kg and 0.38 mg/kg (Trial C94)) and 0.14 mg/kg and 0.18 mg/kg (Trial CDb). The corresponding residue reduction factors were 0.289 and 0.778 (mean = 0.534). Applying the mean factor to the residues listed above for the consideration of the residues in rice grain gives the following residue estimates (n=8): 0.032, 0.15, 0.21, 0.22, 0.26, 0.33, 0.45, and 0.75 mg/kg for CS_2 in husked rice.

The Meeting estimated a maximum residue level of 1.5 mg/kg for CS_2 in husked rice.

The median residue of CS_2 was 0.24 mg/kg resulting in an STMR of 3.2 mg MTE/kg.

A processing study on rice is not available. The Meeting agreed to extrapolate the residue estimates from husked rice to polished rice. The Meeting estimated a maximum residue level of 1.5 mg/kg for CS_2 in polished rice and an STMR of 0.43 mg MTE/kg.

Cotton

The critical GAP for cotton is from Brazil and consists of 3 applications each at 2.25 kg ai/ha, on a 7-day interval, with a 30-day PHI.

Residues of CS_2 in undelinted seed from independent trials matching the critical GAP were (n=8): < 0.056 (4), 0.097, 0.11, 0.13, and 0.24 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg for CS_2 in cottonseed (undelinted).

The median residue of CS_2 was < 0.056 mg/kg resulting in an STMR of 0.75 mg MTE/kg.

Fate of residues during processing

The 1993 Meeting evaluated the effects of processing on residues in a number of commodities, including maize. Residues in raw and processed commodities were <LOQ for both CS_2 (< 0.03 mg/kg) and ETU (< 0.01 mg/kg), and processing factors could not be calculated.

In the processing study provided to the current meeting, residues of CS_2 were <LOQ in one RAC sample and 0.41 mg/kg in the second RAC sample. Based on that second sample, the Meeting was able to calculate CS_2 processing factors, summarized below, for maize.

Table 1 Processing factors (as CS_2)

Crop	Commodity	Processing factor (CS_2 only)
Maize	Kernels (max. residue level = 0.15 mg/kg; median = 0.11 mg MTE/kg)	--
	Grits	0.205
	Meal	0.873
	Flour	1.07
	Refined oil	< 0.122
	Starch	< 0.122

As no other processing data were available, the Meeting could not conclude on residue levels in processed commodities of soya bean, rice, or cotton.

The Meeting noted that processed commodities of maize, soya bean, rice, and cotton are heated during processing and/or prior to consumption. The Meeting therefore decided not to use empirical processing factors and to assume all residues of mancozeb in processed commodities would be converted to ETU. The Meeting agreed to derive STMRs for processed commodities based on the level of CS_2 in the RAC. The Meeting used the relative potency factor of 7.5 to convert STMRs in the RAC (as MTE) to STMRs in the processed commodities considered by the current Meeting.

The STMRs for processed commodities from RACs considered by the current meeting are:

Soya bean: 0.75 mg MTE/kg

Maize: 0.83 mg MTE/kg

Rice: 3.2 mg MTE/kg

Cotton: 0.75 mg MTE/kg

Residues in animal commodities

The 1993 Meeting recommended maximum residue levels for CS_2 in animal commodities as follows: milks 0.05(*) mg/kg, meat 0.02(*) mg/kg, mammalian edible offal 0.1 mg/kg, eggs 0.05(*) mg/kg, poultry meat 0.1 mg/kg, and poultry edible offal 0.1 mg/kg, noting that those recommendations should accommodate animals (ruminants and poultry) consuming up to 25 ppm CS_2 in their diet. A dietary burden was not provided by the 1993 Meeting. The recommendations from the 1993 Meeting included a large number of animal feed commodities, including cereal grains and fodders, with higher residues than those considered by the current Meeting. Therefore, the Meeting agreed that residues in soya bean, maize kernels, rice grain and cottonseed would not significantly change the dietary burdens for cattle or poultry. The Meeting confirmed is previous recommendations for residues in animal commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that it could not derive residues suitable for conducting dietary risk assessments. Based on the residue definitions, the residue levels listed in Annex 1 are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue for compliance with MRLs is total dithiocarbamates, determined as CS_2, evolved during acid digestion and expressed as mg CS_2/kg. Dithiocarbamate residues are not fat soluble.

Definition of the residue for dietary risk assessment is the parent EBDC and ETU. The Meeting is assessing combined residues of mancozeb and ETU using the ratio of the ADIs (7.5) to express residues in terms of mancozeb-toxicity-equivalents (MTE).

Dithiocarbamate residues are not fat-soluble

Table 12 Recommendations for residues of mancoaeb from the 2022 JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
SO 0691	Cottonseed	0.3		0.75	
FI 0342	Longan	15		9.8	
GC 0645	Maize	0.15		0.83	
GC 0649	Rice grain	3			
CM 0649	Rice, husked	1.5		3.2	
CM 1205	Rice, polished	1.5		3.2	
VD 0541	Soya bean (dry)	0.3		0.75	
	Soya bean – all processed commodities			0.75	
	Maize – all processed commodities			0.83	
	Rice – all processed commodities			3.2	
	Cottonseed – all processed commodities			0.75	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for EBDCs, including mancozeb, is 0–0.03 mg/kg bw. The ADI for ETU is 0–0.004 mg/kg bw (= 7.5 × mancozeb maximum ADI). The International Estimated Daily Intakes (IEDIs) for mancozeb (including ETU) were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 5–50 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of ethylene-bis-dithiocarbamates from uses considered by the 2014 and current JMPR is unlikely to present a public health concern.

Acute dietary exposure

An ARfD for the EBDCs has not been established. The Meeting noted that the dithiocarbamates were last evaluated in 1993, which was prior to the JMPR establishing ARfDs.

FURTHER WORK OR INFORMATION

Desirable information

- Radiovalidation studies for analysis of ETU by QuEChERS or methods using methanol or ethanol (unaltered) to extract residues.
- Processing studies with soya bean, rice, and cotton, including analysis of ETU

REFERENCES

Report Code	Author(s)	Year	Study Title
34-89-21	Schweitzer, M.G.	1989	Determination of the Magnitude of the Residue Due to Mancozeb and ETU in Corn Processed Components Prepared from Corn Treated with Mancozeb
AA950302	Singer, G.M.	1996	Magnitude of the Residue of Mancozeb in/on Processed Commodities from Field Corn Grain or Grain Grown for Hybrid Seed
GHB-P 1046 (020145)	Kalvan, H.C., Rubin, R., Oliveira, R.C.	2004	Residues of mancozeb in rice after multiple applications of Dithane PM NT, fungicide - Brazil, 2002-2003. Report No. GHB-P 1046 (020145) Non-GLP, Unpublished Study completion 10 Nov 2004
GHB-P 1047 (020145.01)	Kalvan, H.C., Rubin, R., Oliveira, R.C.	2004	Residues of mancozeb in rice after multiple applications of Dithane 75 DG NT, fungicide - Brazil, 2002-2003. Report No. GHB-P 1047 (020145.01) Non-GLP, Unpublished Study completion 10 Nov 2004
GHB-P 1048 (020145)	Kalvan, H.C., Rubin, R., Oliveira, R.C.	2004	Residues of mancozeb in rice after multiple applications of Dithane PM NT, fungicide - Brazil, 2002-2003. Report No. GHB-P 1048 (020145) Non-GLP, Unpublished Study completion 10 Nov 2004

Report Code	Author(s)	Year	Study Title
GHB-P 1049 (020145.01)	Kalvan, H.C., Rubin, R., Oliveira, R.C.	2004	Residues of mancozeb in rice after multiple applications of Dithane 75 DG NT, fungicide - Brazil, 2002-2003. Report No. GHB-P 1049 (020145.01) Non-GLP, Unpublished Study completion 10 Nov 2004
GHB-P 1146 (020145)	Kalvan, H.C., Cason, J.B., Rampazzo, P.E.	2005	2005 Residues of mancozeb in rice grains after multiple applications of Dithane PM NT, fungicide - Brazil, 2002-2003. Report No. GHB-P 1146 (020145) Non-GLP, Unpublished Study completion 10 Aug 2005
GHB-P 1147 (020145.01)	Kalvan, H.C., Cason, J.B., Rampazzo, P.E.	2005	Residues of mancozeb in rice grains after multiple applications of Dithane DG NT, fungicide - Brazil, 2002-2003. Report No. GHP-P 1147 (020145.01) Non-GLP, Unpublished Study completion 17 Aug 2005
000672.034.153.12	Lopez, N.M.R.	2013	Magnitude de resíduos de mancozebe após aplicação do produto comercial UNIZEB GOLD (Mancozeb 750 WG) em sementes de algodão. Report No. 000672.034.153.12 GLP, Unpublished Study completion 14 Aug 2013
000672.034.156.12	Lopez, N.M.R.	2013	Magnitude de residuos de mancozebe após aplicação do produto comercial UNIZEB GOLD (Mancozeb 750 WG) em grãos de soja. Report No. 000672.034.156.12 GLP, Unpublished Study completion 15 Jul 2013
000672.034.163.12	Lopez, N.M.R.	2013	Magnitude de resíduos de mancozebe após aplicação do produto comercial UNIZEB GOLD (Mancozeb 750 WG) em sementes de algodão. Report No. 000672.034.163.12 GLP, Unpublished Study completion 13 Aug 2013
000672.034.164.12	Lopez, N.M.R.	2013	Magnitude de resíduos de mancozebe após aplicação do produto comercial UNIZEB GOLD (Mancozeb 750 WG) em grãos de milho. Report No. 000672.034.164.12 GLP, Unpublished Study completion 06 Sep 2013
000672.034.165.12	Lopez, N.M.R.	2013	Magnitude de residuos de mancozebe após aplicação do produto comercial UNIZEB GOLD (Mancozeb 750 WG) em grãos de soja. Report No. 000672.034.165.12 GLP, Unpublished Study completion 24 Jul 2013
13790.034.093.14	Bassi, C.	2014	Magnitude de resíduos de Azoxystrobina + Mancozebe em grãos de milho após aplicação do produto de código UPL 207A FP. Report No. 13790.034.093.14 GLP, Unpublished Study completion 06 Aug 2014

Report Code	Author(s)	Year	Study Title
13790.034.094.14	Bassi, C.	2014	Magnitude de resíduos de Azoxystrobina + Mancozebe em grãos de milho após aplicação do produto de código UPL 207A FP. Report No. 13790.034.094.14 GLP, Unpublished Study completion 01 Aug 2014
13790.034.095.14	Bassi, C.	2014	Magnitude de resíduos de Azoxystrobina + Mancozebe em grãos de milho após aplicação do produto de código UPL 207A FP. Report No. 13790.034.095.14 GLP, Unpublished Study completion 07 Aug 2014
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13790.034.330.14	Bassi, C.	2015	Magnitude de residuos de Azoxystrobina e Z-azoxystrobin + Mancozebe + Tebuconazole em sementes de soja após aplicação do produto de código UPL 2000 FP. Report No. 13790.034.330.14 GLP, Unpublished Study completion 14 Aug 2015
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13790.034.029.14	Galesi, F.R.	2015	Magnitude de resíduos de Mancozebe em grãos de milho após aplicação do produto manzate WG. Report No. 13790.034.029.14 GLP, Unpublished Study completion 08 Jan 2015
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Report Code	Author(s)	Year	Study Title
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13790.034.329.14	Tomaz, M.L.	2015	Magnitude de residuos de Azoxystrobina e Z-azoxystrobin + Mancozebe + Tebuconazole em sementes de soja após aplicação do produto de código UPL 2000 FP. Report No. 13790.034.329.14 GLP, Unpublished Study completion 24 Aug 2015
BPL-JM-066-002-16-RF	Moraes, M.V.	2016	Relatório de estudo de resíduo em campo e laboratório do fungicida UPL 2000 FP (Azoxystrobina + Mancozebe + Tebuconazol) em sementes do algodão (Gossypium hirsutum L.) com adição de adjuvante. Report No. BPL-JM-066-002-16-RF GLP, Unpublished Study completion 03 Oct 2016
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13790.034.199.15	Rizzo, M.H.L.	2016	Magnitude de resíduos de azoxystrobina + Z-azoxystrobina e mancozebe em sementes de algodão após aplicação do produto UNIZEB GLORY. Report No. 13790.034.199.15 GLP, Unpublished Study completion 20 Oct 2016
13790.034.213.15	Rizzo, M.H.L.	2016	Magnitude de resíduos de azoxystrobina + Z-azoxystrobina + mancozebe + tebuconazole em sementes de algodão após aplicação do produto UPL 2000 FP. Report No. 13790.034.213.15 GLP, Unpublished Study completion 19 Oct 2016

Report Code	Author(s)	Year	Study Title
13790.034.217.15	Rizzo, M.H.L.	2016	Magnitude de resíduos de azoxystrobina, Z-azoxystrobina, mancozebe e tebuconazole em grãos de arroz após aplicação do produto UPL 2000 FP. Report No. 13790.034.217.15 GLP, Unpublished Study completion 29 Jul 2016
13790.034.218.15	Rizzo, M.H.L.	2016	Magnitude de resíduos de azoxystrobina + Z-azoxystrobina + mancozebe + tebuconazole em grãos de arroz após aplicação do produto UPL 2000 FP. Report No. 13790.034.218.15 GLP, Unpublished Study completion 19 Aug 2016
13790.034.052.16	Bassi, C.	2017	Magnitude de resíduos de azoxystrobina, z-azoxystrobina, mancozebe e tebuconazol em grãos de arroz após aplicação do produto UPL 2000 FP. Report No. 13790.034.052.16 GLP, Unpublished Study completion 16 Aug 2017
13790.034.053.16	Bassi, C.	2017	Magnitude de resíduos de azoxystrobina, z-azoxystrobina, mancozebe e tebuconazol em grãos de arroz após aplicação do produto UPL 2000 FP. Report No. 13790.034.053.16 GLP, Unpublished Study completion 28 Apr 2017
170131	Draetta, M.	2017	Resíduos de Mancozebe em sementes de algodgão após aplicação da formulação GF-2940, fungicida, Brasil, 2017. Report No. 170131 Non-GLP, Unpublished Study completion 06 Sep 2017
VR-036/17	Magagnato, M.B.B.	2017	Estudo de validação de metodologia analítica para análise de resíduo do ingrediente ativo Mancozebe em grãos de milho. GLP, Unpublished
BPL-JM-037-032-16-RF	Moraes, M.V.	2017	Relatório de estudo de estabilidade do ingrediente ativo mancozebe e seu metabólito em sementes de amendoim (Arachis hypogaea L.). GLP, Unpublished
170130	Obara, F., Magagnato, M.B.B.	2017	Magnitude of residues of mancozeb in soybean seed after application of GF-2940 formulation, fungicide, Brazil, 2017 Report No. 170130 GLP, Unpublished Study completion 30 May 2017
170129	Obaro, F.	2017	Resíduos de Mancozebe em grãos de milho após aplicação da formulação GF-2940, fungicida, Brasil, 2017. Report No. 170129 Non-GLP, Unpublished Study completion 30 May 2017
13790.034.152.17	Bassi, C.	2018	Magnitude de resíduos de azoxystrobina + mancozebe + ciproconazol em grãos de arroz após aplicação do produto UPL 216 FP. Report No. 13790.034.152.17 GLP, Unpublished Study completion 12 Jul 2018

Report Code	Author(s)	Year	Study Title
BPL-JM-066-007-19-RF	Lizier, T.M.	2019	Relatório de estudo de resíduo em campo e labora tório do fungicida e acaricida UNIZEB GOLD (mancozebe) em sementes de soja (Glycine max L.). Report No. BPL-JM-066-007-19-RF GLP, Unpublished Study completion 30 Aug 2019
BPL-JM-066-009-19-RF	Lizier, T.M.	2019	Relatório de estudo de resíduo em campo e labora tório do fungicida UPL 2037 FP (mancozebe e seu metabólito) em sementes de soja (Glycine max L.) com adição de adjuvante. Report No. BPL-JM-066-009-19-RF GLP, Unpublished Study completion 20 Aug 2019
13790.034.124.18	Magagnato, M.B.B.	2019	Magnitude de Residuos de Azoxystrobina, Z-azoxystrobina, Mancozebe e Ciproconazol em Sementes de soja após aplicação do produto UPL 216 FP. Report No. 13790.034.124.18 GLP, Unpublished Study completion 19 Jun 2019
13790.034.126.18	Magagnato, M.B.B.	2019	Magnitude de Residuos de Azoxystrobina, Z-azoxystrobina, Mancozebe e Tebuconazol em sementes de soja após aplicação do produto TRIDIUM. Report No. 13790.034.126.18 GLP, Unpublished Study completion 11 Jul 2019
13790.034.127.18	Magagnato, M.B.B.	2019	Magnitude de Residuos de Azoxystrobina, Z-azoxystrobina, Mancozebe e Tebuconazol em sementes de soja após aplicação do produto TRIDIUM. Report No. 13790.034.127.18 GLP, Unpublished Study completion 27 Aug 2019

MANDIPROPAMID (231)

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EXPLANATION

Mandipropamid is a fungicide that belongs to the subset mandelamides in the class carboxylic acid amides. It is intended for the control of Oomycete fungal pathogens in a range of crops.

Mandipropamid was first evaluated (toxicology and residues) by the JMPR in 2008. An ADI of 0–0.2 mg/kg bw was established and it was decided that an ARfD was unnecessary. The residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities is mandipropamid. The residue is not fat-soluble.

The Meeting received information on additional analytical methods, storage stability, high temperature hydrolysis, residues during processing (tomato), GAP information and residue trials for bulb onion, green onion, cucumber, summer squash, melon, tomato, ginseng and basil.

Methods of Analysis

Descriptions of analytical methods together with validation data for residues of mandipropamid in plant and animal matrices were submitted to the Meeting. The methods rely on an initial extraction, usually with acetonitrile/water. After column clean-up, mandipropamid is prepared for LC analysis. Their residues can be measured by mass spectrometric detector (MS/MS), to an LOQ of 0.01 mg/kg. Since the methods use standard extraction solvents and standard detection techniques, they have the potential to be incorporated into existing multi-residue methods.

Detailed descriptions of new analytical methods are presented below.

Plant matrices

High water content, high starch content, high oil content and high acid content category (VV-411985)			
Analyte:	Mandipropamid (m/z 412→328 for quantification, 412→125 for confirmation)	LC-MS/MS	GRM001.07A
LOQ:	0.01 mg/kg		
Description	Samples were extracted by homogenisation with acetonitrile/water (80/20, v/v). Extracts were centrifuged and aliquots (1 mL = 0.1 g) were diluted with water. Clean-up was performed by solid-phase extraction (SPE) using Oasis HLB cartridges. Final determination was by LC-MS/MS.		
High water content, high acid content, high starch content, high protein content, high oil content and dry matrices (VV-414187)			
Analyte:	Mandipropamid (m/z 412→328 for quantification, 412→356 for confirmation)	LC-MS/MS	QuEChERS
LOQ:	0.01 mg/kg		
Description	Residues of mandipropamid were extracted from crop matrices by shaking with acetonitrile, after the addition of a suitable volume of water, if necessary. After the addition of a mixture of magnesium sulphate, sodium chloride, and buffering citrate salts, the extracts were shaken and then centrifuged. In the case of cocoa bean matrices only, the fat was frozen out, and then an aliquot of each extract (for all matrices) was cleaned up using a pre-mixed, commercially available dispersive SPE PSA clean-up tube. For cocoa bean extracts only, a portion of C_{18} was also added prior to shaking with the dispersive SPE clean-up tube. After centrifugation, extracts were diluted to within the calibration range with acetonitrile/water (20/80, v/v, containing 0.1 percent formic acid). Final determination was with LC-MS/MS.		
Fresh ginseng, dried ginseng and red ginseng (JLND2020RS001-A)			
Analyte:	Mandipropamid (m/z 412→328 for quantification, 412→125 for confirmation)	LC-MS/MS	JLND2020RS001-A
LOQ:	0.01 mg/kg		

Description	Residues of mandipropamid were extracted from ginseng samples with acetonitrile and distilled water by homogenizing for 2 minutes, dried ginseng or red ginseng samples with acetonitrile and distilled water by shaking for 1 hour. After filtering under vacuum conditions by Buchner funnel, the filtrate was added with sodium chloride and shaking vigorously. An aliquot of upper layer was transfered to a centrifuge tube containing PSA and anhydrous $MgSO_4$. After dispersive solid-phase extraction, the purified extracts were filtered through a 0.22-μm syringe filter and analysed by LC-MS/MS.

Validation data for methods on plant matrices are summarized in Table 1.

Mean recoveries were within the range of 70–110 percent with RSD values of < 20 percent.

Table 1 Summary of Recovery Data for mandipropamid fortified into plant matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Savoy Cabbage (MV)	412→328	0.01	5	80–87	82	3.4	GRM001.07A
	Quantification	2.0	5	87–91	89	2.1	
	412→125	0.01	5	81–85	83	2.2	VV-411985
	Confirmation	2.0	5	86–92	90	2.8	Austin R, 2015
Broccoli (MV)	412→328	0.01	5	84–88	86	1.9	
	Quantification	2.0	5	90–97	94	2.7	
	412→125	0.01	5	86–96	90	5.0	
	Confirmation	2.0	5	90–96	94	2.6	
Cauliflower (MV)	412→328	0.01	5	83–86	84	1.5	
	Quantification	2.0	5	92–95	94	1.4	
	412→125	0.01	5	83–86	84	1.4	
	Confirmation	2.0	5	92–94	93	1.0	
Brussels sprouts (MV)	412→328	0.01	5	82–85	83	1.6	
	Quantification	2.0	5	81–89	87	4.0	
	412→125	0.01	5	84–87	85	1.4	
	Confirmation	2.0	5	82–91	88	4.2	
Artichoke (MV)	412→328	0.01	5	83–89	85	2.7	
	Quantification	2.0	5	92–99	95	2.7	
	412→125	0.01	5	82–88	84	3.0	
	Confirmation	2.0	5	91–96	94	2.2	
Kohlrabi (MV)	412→328	0.01	5	83–87	85	1.8	
	Quantification	2.0	5	91–98	95	2.7	
	412→125	0.01	5	82–88	84	2.6	
	Confirmation	2.0	5	90–98	95	3.1	
Radish (MV)	412→328	0.01	5	79–87	82	4.7	
	Quantification	0.5	5	91–93	92	0.9	
	412→125	0.01	5	81–88	84	3.4	
	Confirmation	0.5	5	92–94	93	1.0	
Pea, Fresh - Whole plant (MV)	412→328	0.01	5	77–86	83	4.5	
	Quantification	10	5	91–95	93	1.6	
	412→125	0.01	5	80–87	84	3.1	
	Confirmation	10	5	91–95	93	1.6	
Pea, Fresh - Immature pod (MV)	412→328	0.01	5	89–91	90	1.0	
	Quantification	3.0	5	97–102	99	1.8	
	412→125	0.01	5	87–93	90	2.4	
	Confirmation	3.0	5	97–102	99	1.9	
Pea, Fresh - Succulent green seeds (MV)	412→328	0.01	5	86–91	89	2.1	
	Quantification	0.5	5	90–102	96	4.5	
	412→125	0.01	5	86–95	90	3.6	
	Confirmation	0.5	5	92–101	96	3.5	

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Cacao - Fermented beans (MV)	412→328	0.01	5	90–92	91	0.9	
	Quantification	1.0	5	99–106	103	2.7	
	412→125	0.01	5	90–93	91	1.5	
	Confirmation	1.0	5	98–104	101	2.4	
Grape (MV)	412→328	0.01	5	86–96	90	4.0	
	Quantification	2.0	5	78–99	91	8.5	
	412→125	0.01	5	87–96	92	4.1	
	Confirmation	2.0	5	77–99	90	9.0	
Potato (MV)	412→328	0.01	5	100–105	103	2.0	GRM001.07A
	Quantification	0.8	5	95–102	98	2.9	GRM001.07B
	412→125	0.01	5	98–104	101	2.8	
	Confirmation	0.8	5	93–99	97	2.4	VV-619459
Lettuce (MV)	412→328	0.01	5	91–102	96	4.1	Allen L, 2019
	Quantification	13.0	5	88–94	90	2.5	
	412→125	0.01	5	93–102	97	3.9	
	Confirmation	13.0	5	88–95	91	2.8	
Broad Beans (MV)	412→328	0.01	5	101–108	103	2.6	
	Quantification	0.4	5	94–101	99	2.9	
	412→125	0.01	5	98–109	102	4.5	
	Confirmation	0.4	5	95–105	99	3.7	
Soybeans (MV)	412→328	0.01	5	92–98	95	2.3	
	Quantification	0.1	5	88–91	90	1.4	
	412→125	0.01	5	95–103	98	3.2	
	Confirmation	0.1	5	89–94	91	2.1	
Wheat (grain) (MV)	412→328	0.01	5	97–100	99	1.2	
	Quantification	0.1	5	90–94	92	1.7	
	412→125	0.01	5	95–102	99	3.2	
	Confirmation	0.1	5	90–95	92	2.2	
Wheat (straw) [1)] (MV)	412→328	0.01	5	81–89	85	3.8	
	Quantification	0.1	5	78–83	80	2.4	
	412→125	0.01	5	85–98	91	7.0	
	Confirmation	0.1	5	78–83	80	2.6	
Wheat (forage) (MV)	412→328	0.01	5	81–87	84	2.9	
	Quantification	0.1	5	78–82	80	2.2	
	412→125	0.01	5	88–92	90	2.0	
	Confirmation	0.1	5	78–81	79	1.8	
Carrot (MV)	412→328	0.01	5	95–102	98	3.1	
	Quantification	0.1	5	88–96	92	3.3	
	412→125	0.01	5	84–105	96	8.2	
	Confirmation	0.1	5	87–94	91	3.8	
Onion [1)] (MV)	412→328	0.01	5	91–104	97	5.0	
	Quantification	7.0	5	91–97	94	2.5	
	412→125	0.01	5	87–94	90	3.5	
	Confirmation	7.0	5	91–97	95	2.5	
Melon (MV)	412→328	0.01	5	87–95	91	3.9	
	Quantification	0.7	5	89–93	90	1.9	
	412→125	0.01	5	87–97	91	4.4	
	Confirmation	0.7	5	91–94	92	1.5	
Cucumber (MV)	412→328	0.01	5	85–88	86	1.3	
	Quantification	0.3	5	83–85	84	1.0	
	412→125	0.01	5	76–86	82	4.6	
	Confirmation	0.3	5	81–87	84	2.7	

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Tomato (whole fruit) (MV)	412→328	0.01	5	88–93	91	2.2	
	Quantification	2.0	5	86–90	88	1.7	
	412→125	0.01	5	89–94	92	2.0	
	Confirmation	2.0	5	86–91	89	2.3	
Tomato (peeled) (MV)	412→328	0.01	5	87–92	89	2.4	
	Quantification	0.1	5	83–90	88	3.3	
	412→125	0.01	5	81–96	88	6.1	
	Confirmation	0.1	5	87–92	89	2.3	
Tomato (canned) (MV)	412→328	0.01	5	82–91	87	3.8	
	Quantification	0.2	5	82–87	86	2.4	
	412→125	0.01	5	77–96	87	8.0	
	Confirmation	0.2	5	84–90	87	2.8	
Tomato (sundried) [1] (MV)	412→328	0.01	5	98–108	105	3.8	
	Quantification	0.1	5	100–105	102	2.0	
	412→125	0.01	5	97–107	103	3.8	
	Confirmation	0.1	5	101–104	103	1.1	
Tomato (juice) (MV)	412→328	0.01	5	89–95	91	3.0	
	Quantification	0.5	5	89–93	91	2.0	
	412→125	0.01	5	87–100	92	5.9	
	Confirmation	0.5	5	87–95	91	3.1	
Tomato (puree) (MV)	412→328	0.01	5	95–97	96	1.1	
	Quantification	0.5	5	97–98	97	0.6	
	412→125	0.01	5	91–98	94	3.0	
	Confirmation	0.5	5	96–98	97	0.9	
Tomato (dry pomace) [1] (MV)	412→328	0.01	5	104–110	108	2.3	
	Quantification	2.0	5	99–108	105	3.7	
	412→125	0.01	5	100–110	105	4.4	
	Confirmation	2.0	5	100–108	104	3.5	
Grape (juice) (MV)	412→328	0.01	5	101–111	107	3.6	
	Quantification	0.05	5	99–111	105	4.7	
	412→125	0.01	5	98–111	105	5.2	
	Confirmation	0.05	5	102–111	107	3.2	
Raisins (MV)	412→328	0.01	5	97–102	100	2.1	
	Quantification	3.0	5	98–103	101	1.9	
	412→125	0.01	5	92–102	97	4.3	
	Confirmation	3.0	5	99–102	101	1.3	
Wine (MV)	412→328	0.01	5	81–85	82	2.2	
	Quantification	0.1	5	80–85	83	2.5	
	412→125	0.01	5	85–92	88	3.2	
	Confirmation	0.1	5	81–84	82	1.4	
Grape (must) (MV)	412→328	0.01	5	91–98	94	3.2	
	Quantification	0.1	5	90–99	94	4.3	
	412→125	0.01	5	92–101	96	4.0	
	Confirmation	0.1	5	93–100	96	3.5	
Grape (seed oil) (MV)	412→328	0.01	5	94–99	97	2.2	
	Quantification	0.1	5	88–90	89	0.9	
	412→125	0.01	5	94–106	101	4.7	
	Confirmation	0.1	5	86–90	88	1.7	
Grape (dry pomace) (MV)	412→328	0.01	5	81–91	87	4.4	
	Quantification	0.7	5	81–95	90	7.1	
	412→125	0.01	5	82–92	87	4.5	
	Confirmation	0.7	5	83–95	91	5.3	

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Honey (MV)	412→328	0.01	5	76–83	78	3.7	
	Quantification	0.1	5	69–78	76	4.9	
	412→125	0.01	5	76–88	82	5.7	
	Confirmation	0.1	5	70–79	77	5.0	
Broccoli (MV)	412→328	0.01	5	107–111	109	1.5	QuEChERS
	Quantification	2.0	5	106–112	110	2.3	
	412→356	0.01	5	100–111	106	4.1	VV-414187
	Confirmation	2.0	5	106–112	109	2.0	Richter S,
Grape (MV)	412→328	0.01	5	97–109	104	4.8	Schmiedt S, 2015
	Quantification	2.0	5	84–103	95	8.1	
	412→356	0.01	5	97–111	105	5.0	
	Confirmation	2.0	5	82–98	92	7.4	
Wheat grain (MV)	412→328	0.01	5	103–117	110	4.7	
	Quantification	0.10	5	99–108	105	3.5	
	412→356	0.01	5	104–115	104	4.6	
	Confirmation	0.10	5	96–109	106	4.8	
Dried broad bean (MV)	412→328	0.01	5	93–104	101	4.8	
	Quantification	1.5	5	94–101	97	2.7	
	412→356	0.01	5	90–109	101	7.8	
	Confirmation	1.5	5	94–102	98	3.0	
Cocoa bean (MV)	412→328	0.01	5	99–110	105	4.5	
	Quantification	0.10	5	103–107	105	1.7	
	412→356	0.01	5	96–111	105	5.3	
	Confirmation	0.10	5	98–102	100	1.9	
Wheat straw (MV)	412→328	0.01	5	100–109	104	3.3	
	Quantification	2.0	5	89–101	95	5.7	
	412→356	0.01	5	97–107	100	4.3	
	Confirmation	2.0	5	87–97	91	4.8	
Broccoli (ILV)	412→328	0.01	5	97–102	100	2.3	QuEChERS
	Quantification	2.0	5	97–99	98	0.9	
	412→356	0.01	5	100–105	103	2.0	VV-414778
	Confirmation	2.0	5	96–99	98	1.3	Homazava N, 2015
Wheat straw (ILV)	412→328	0.01	5	82–89	84	3.5	
	Quantification	2.0	5	73–77	75	2.3	
	412→356	0.01	5	78–87	83	3.9	
	Confirmation	2.0	5	72–76	73	2.2	
Cocoa bean (ILV)	412→328	0.01	5	103–106	104	1.3	
	Quantification	0.10	5	102–103	103	0.4	
	412→356	0.01	5	102–106	104	1.4	
	Confirmation	0.10	5	104–104	103	0.9	
Fresh ginseng (MV)	412→328	0.010	5	83–106	90	10	JLND2020RS001-A
	Quantification	0.050	5	85–94	87	4.3	
		0.50	5	94–98	96	1.5	Zhiguang Hou,
Dried ginseng (MV)	412→328	0.010	5	94–98	96	1.5	2020
	Quantification	0.050	5	96–101	99	1.9	
		0.50	5	97–104	102	2.6	
		5.0	5	94–97	96	1.4	
Red ginseng (MV)	412→328	0.010	5	90–99	93	3.8	
	Quantification	0.050	5	96–104	99	3.1	
		0.50	5	98–102	100	1.5	
		5.0	5	96–103	100	2.7	

Notes:

MV: Method Validation, ILV: Independent Laboratory Validation.

1) Significant (> ±20 percent) suppression of detector response was observed for mandipropamid in the presence of wheat (straw), onions, tomato (sundried) and tomato (dry pomace). Matrix-matched standards were therefore used for quantification of these matrices during the validation study.

Animal matrices

Blood and animal matrices (milk, eggs, meat, liver, kidney and fat) (VV-402660)			
Analyte:	Mandipropamid (m/z 412→328 for quantification, 412→356 for confirmation)	LC-MS/MS	QuEChERS
LOQ:	0.01 mg/kg		
Description	Residues of mandipropamid were extracted from animal matrices by shaking with acetonitrile, after the addition of a suitable volume of water. For fat samples only, water was added and the samples heated in a water bath (40°C) and shaken to reduce clump building of fat; this was done prior to the addition of acetonitrile. After the addition of a mixture of magnesium sulphate, sodium chloride, and buffering citrate salts, the extracts were shaken and then centrifuged. After centrifugation, the acetonitrile phase was decanted and frozen out overnight. Extracts were centrifuged and an aliquot of each extract was cleaned-up using a pre-mixed, commercially available dispersive SPE PSA clean up tube. Extracts were diluted to within the calibration range with acetonitrile/water (1/1, v/v). Final determination was with LC-MS/MS.		

Validation data for methods on animal matrices are summarized in Table 2.

Mean recoveries were within the range of 70–110 percent with RSD values of <20 percent.

Table 2 Summary of Recovery Data for mandipropamid fortified into animal matrices

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Milk (MV)	412→328 Quantification	0.01	5	97–105	100	3.6	QuEChERS VV-402660 Class T, Göcer M., 2012
		0.10	5	92–106	102	5.6	
	412→356 Confirmation	0.01	5	97–104	100	3.1	
		0.10	5	93–105	101	4.8	
Eggs (MV)	412→328 Quantification	0.01	5	87–107	95	8.4	
		0.10	5	85–104	96	8.0	
	412→356 Confirmation	0.01	5	90–106	96	6.8	
		0.10	5	86–103	96	7.8	
Meat (MV)	412→328 Quantification	0.01	5	97–104	101	2.5	
		0.10	5	99–105	101	2.9	
	412→356 Confirmation	0.01	5	97–108	100	4.7	
		0.10	5	99–106	102	2.6	
Liver (MV)	412→328 Quantification	0.01	5	103–113	109	3.9	
		0.10	5	93–109	100	6.4	
	412→356 Confirmation	0.01	5	103–115	110	4.8	
		0.10	5	94–110	102	6.6	
Kidney (MV)	412→328 Quantification	0.01	5	99–104	102	2.4	
		0.10	5	105–114	110	3.1	
	412→356 Confirmation	0.01	5	100–103	102	1.4	
		0.10	5	105–114	110	3.3	
Fat (MV)	412→328 Quantification	0.01	5	88–100	93	5.5	
		0.10	5	81–95	88	7.0	
	412→356 Confirmation	0.01	5	89–99	95	5.1	
		0.10	5	84–96	90	5.8	
Whole blood (MV)	412→328 Quantification	0.01	5	101–113	107	5.0	
		0.10	5	92–96	94	1.6	
	412→356 Confirmation	0.01	5	98–114	107	5.8	
		0.10	5	92–95	93	1.2	

Commodity	Mass transition	Fortification mg/kg	N	Range Recovery (percent)	Mean recovery (percent)	percent RSD	Reference Method
Milk (ILV)	412→328	0.01	5	101–104	105	5.1	QuEChERS
	Quantification	0.1	5	97–115	107	7.6	
	412→356	0.01	5	100–111	104	4.1	VV-404305
	Confirmation	0.1	5	97–111	105	6.7	Amic S, 2013
Liver [1)] (ILV)	412→328	0.01	5	90–110	100	7.2	
	Quantification	0.1	5	94–103	98	3.7	
	412→356	0.01	5	90–106	98	6.6	
	Confirmation	0.1	5	92–102	97	4.1	
Fat (ILV)	412→328	0.01	5	90–97	93	2.8	
	Quantification	0.1	5	88–95	92	2.7	
	412→356	0.01	5	91–96	93	2.5	
	Confirmation	0.1	5	87–94	91	2.9	

Notes:

MV: Method Validation, ILV: Independent Laboratory Validation

[1)] Significant (> ±20 percent) matrix effect was observed in liver. Matrix-matched standards were therefore used for calibration and quantification for liver matrix during the validation study.

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

The Meeting received new storage stability data for dried beans, ginseng, basil and potato.

The storage stability of mandipropamid was tested in high protein crop commodity (Khoshab A, 2020: VV-725132). Samples of homogenised dried broad beans (10g) were fortified at 0.10 mg/kg with a known amount of mandipropamid. The solution was left to soak, and the samples were sealed and stored in a freezer at typically ≤-18°C. Duplicate samples were analysed after 1, 3, 6, 12 and 18 months of storage. Mandipropamid was analysed using method GRM001.07A by LC-MS/MS.

Table 3 Recovery of mandipropamid from stored fortified samples of dried broad beans

Storage interval (Actual)	Procedural (percent)	Residues (mg/kg)	Mean of percent remaining
Day 0	94, 97	0.0912, 0.0993, 0.0999	100
1 months (31 days)	104, 107	0.102, 0.103	105
3 months (84 days)	99, 100	0.0925, 0.0978	98
6 months (177 days)	109, 110	0.0857, 0.0875	90
12 months (356 days)	101, 107	0.0967, 0.0987	101
18 months (549 days)	98, 103	0.0150, 0.0155	15
18 months (561 days)	99, 100	0.0162, 0.0170	18

The storage stability of SYN500003 was tested in high starch crop commodity (Khoshab A, 2019: VV-725129). Samples of homogenised potato (10g) were fortified at 0.50 mg/kg with a known amount of SYN500003. The solution was left to soak, and the samples were sealed and stored in a freezer at typically <-18°C. Duplicate samples were analysed after 1, 3, 6 and 12 months of storage. SYN500003 was analysed using method GRM001.01A by LC-MS/MS.

Table 4 Recovery of SYN500003 from stored fortified samples of potato

Storage interval (Actual)	Procedural (percent)	Residues (mg/kg)	Mean of percent remaining
Day 0	106, 108	0.517, 0.514, 0.536	100

Storage interval (Actual)	Procedural (percent)	Residues (mg/kg)	Mean of percent remaining
1 months (30 days)	95, 98	0.475, 0.473	91
3 months (83 days)	107, 107	0.515, 0.509	98
6 months (180 days)	91, 95	0.440, 0.428	84
12 months (363 days)	103, 100	0.466, 0.461	89

The storage stability of mandipropamid was tested in fresh ginseng and dried/red ginseng (Zhiguang Hou, 2020: JLND2020RS001-S). Homogenised fresh ginseng samples and pulverized dried/red ginseng samples (each 5.0 g) destined for frozen storage were fortified with a known amount of mandipropamid in acetonitrile at a rate of 1.0 mg/kg. The solution was left to soak, and the samples were sealed and stored in a freezer at a nominal temperature of ≤-18°C. Duplicate samples were analysed after 0, 2, 3, 6 and 9 months of storage. Mandipropamid was analysed using method JLND2020RS001-A by LC-MS/MS. The LOQ was 0.01 mg/kg.

Table 5 Recovery of mandipropamid from stored fortified samples of fresh ginseng and dried/red ginseng

Storage interval (Actual)	Procedural (percent)	Residues (mg/kg)	Mean of percent remaining
Fresh ginseng			
Day 0	101, 98	0.90, 0.99	100
2 months (76 days)	98, 97	0.84, 0.82	87
3 months (91 days)	98, 96	0.92, 0.95	99
6 months (191 days)	107, 105	0.84, 0.82	87
9 months (277 days)	91, 92	0.97, 1.0	104
Dried ginseng			
Day 0	97, 99	0.90, 0.89	100
2 months (76 days)	98, 94	0.98, 0.96	107
3 months (91 days)	95, 98	0.98, 0.98	109
6 months (191 days)	89, 97	0.87, 0.87	97
9 months (277 days)	90, 86	1.1, 1.1	122
Red ginseng			
Day 0	93, 100	0.97, 0.98	100
2 months (76 days)	73, 73	0.81, 0.80	83
3 months (91 days)	94, 95	0.92, 0.93	95
6 months (191 days)	108, 94	0.87, 0.88	90
9 months (277 days)	90, 88	0.99, 0.97	100

The storage stability of mandipropamid and SYN500003 were tested in dried ginseng root (Corley J, 2012: VV-508245). Samples of dried ginseng root (10g) were fortified at 0.10 mg/kg with a known amount of mandipropamid and SYN500003. The storage stability samples were stored under the same storage conditions as field samples at <-20°C for 922 days. Mandipropamid and SYN500003 were analysed using method RAM 415/01 and GRM001.01A by LC-MS/MS.

Table 6 Recovery of mandipropamid and SYN500003 from stored fortified samples of dried ginseng root

Analyte	Storage interval	Procedural (percent)	percent remaining	Mean of percent remaining
Mandipropamid	922 days	99	96, 102, 119	105
SYN500003	922 days	84	90, 105, 105	100

The storage stability of mandipropamid was tested in fresh basil and dried basil (Corley J, 2012: VV-508246). Samples of basil (10g) were fortified at 1.0 mg/kg with a known amount of mandipropamid. The storage stability samples were stored under the same storage conditions as field samples at <-20 °C for 700 days. Mandipropamid was analysed using method RAM 415/01 by LC-MS/MS.

Table 7 Recovery of mandipropamid from stored fortified samples of basil

Commodity	Storage interval	Procedural (percent)	percent remaining	Mean of percent remaining
Basil (fresh)	700 days	105	82, 95, 98	92
Basil (dried)	700 days	85	98, 98, 98	98

USE PATTERN

Mandipropamid belongs to the mandelamide chemical class of funficides and is a synthetic fungicide intended for the control of Oomycete fungal pathogens in a range of crops. The Meeting received labels in China and the United States. The information available to Meeting on registered uses of mandipropamid is summarized in Table below.

Table 8 Registered uses of mandipropamid for crops

Crop	Country	Formulation		Application					PHI, days
		Type	Conc.	Method	Rate kg ai/ha	Water L/ha	No. max	Interval, days	
Bulb Vegetables Dry bulb	United States	SC	250 g/L	Spray	0.15 Max/season 0.59	≥93[1)] ≥47[2)]	-	7-10	7
Bulb Vegetables Green Onion	United States	SC	250 g/L	Spray	0.15 Max/season 0.44	≥93[1)] ≥47[2)]	-	7-10	7
Cucurbits	United States	SC	250 g/L	Spray	0.15 Max/season 0.59	≥93[1)] ≥47[2)]	-	7-10	0
Fruiting Vegetables (except tomatoes)	United States	SC	250 g/L	Spray	0.15 Max/season 0.59	≥93[1)] ≥47[2)]	-	7-10	1
Tomato	United States	SC	250 g/L	Spray	0.15 Max/season 0.59 (Max/year 2.3 for multiple croppings)	≥93[1)] ≥47[2)]	-	7-10	1
Ginseng	United States	SC	250 g/L	Spray	0.15 Max/season 0.59	≥93[1)] ≥47[2)]	-	7-10	2
Ginseng	China	SC	23.4 percent	Spray	0.14-0.21		1		21
Basil (fresh and dried)	United States	SC	250 g/L	Spray	0.15 Max/season 0.59	≥93[1)] ≥47[2)]	-	7-10	1

Notes:

[1)] Ground application.

[2)] Aerial application.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on mandipropamid supervised field trials for the following crops.

Group	Commodity	Table
Bulb vegetables	Bulb onion	Table 9, 10
	Spring onion	Table 11, 12

Fruiting vegetables, Cucurbits	Cucumber	Table 13, 14
	Summer squash	Table 15
	Melon	Table 16, 17
Fruiting vegetables, other than Cucurbits	Tomato	Table 18, 19
	Peppers	Table 20
Root and tuber vegetables	Ginseng	Table 21
	Dried ginseng	Table 22, 23
Herbs	Basil	Table 24
	Dried basil	Table 25

Mandipropamid formulation was applied for foliar treatment. Each of the field trial sites generally consisted of untreated control plot and treated plot. Application rates and residue concentrations have generally been rounded to two significant figures.

Residue values from the trials, which have been used for the estimation of maximum residue levels, STMRs and HRs, are underlined.

Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Date of analyses and duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except when residues were found in samples from control plots. Residue data are not corrected for percent recovery.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most field reports provided data on the applicators used, plot size, field sample size and sampling date.

Bulb vegetables

Bulb Onions, Subgroup of

The Meeting received eight trials on dry bulb onion which were conducted in the United States (Rice F, 2012: VV-507780). All trials received four foliar applications of an SC formulation (250 g ai/L) at a nominal rate of 0.15 kg ai/ha, with an application interval of 5 ± 1 days. All applications were made in tank-mix with an adjuvant, non-ionioc surfactant (NIS) or silicone-based. At each trial, bulbs were taken 7 days after last application (DALA).

Samples were analysed for residues of mandipropamid following analytical method RAM 415/01. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for mandipropamid in matrix were within 70–120 percent. Onion bulb samples were stored at ca -20 °C for a maximum of 10 months between sampling and analysis.

Table 9 Residues of mandipropamid on bulb onion from supervised trials in the United States

Onion country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93[2)]* *≥47[3)]*		*7-10*	*4*	*7*		
United States, 2011 Germansville, PA (Stuttgarter)	SC	0.15 0.15 0.15 0.15	234 234 234 234	BBCH 109/19 BBCH 43/403 BBCH 45-47 BBCH 48	5 5 5	4	7	0.061, 0.063 (0.062)	VV-507780 Mean recovery

Onion country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
Outdoor									for mandipropamid: 94 ± 11 percent (n=3) at 0.01 mg/kg 103 ± 4.6 percent (n=3) at 25 mg/kg Sampling to analysis: 33-307 days
United States, 2011 Moorhead, MN (Infinity) Outdoor	SC	0.15 0.15 0.15 0.15	234 234 234 234	BBCH 47 BBCH 47 BBCH 48 BBCH 49	 5 6 5	4	7	0.052, 0.079 (0.066)	
United States, 2012 Raymondville, TX (Yellow Granex F1) Outdoor	SC	0.15 0.15 0.15 0.15	187 187 187 187	BBCH 47 BBCH 47 BBCH 47 BBCH 47	 5 7 4	4	7	0.028, 0.036 (0.032)	
United States, 2011 Uvalde, TX (Leona) Outdoor	SC	0.15 0.15 0.15 0.15	140 140 187 140	BBCH 47 BBCH 47 BBCH 48 BBCH 49	 6 4 5	4	7	0.011, 0.013 (0.012)	
United States, 2011 Guadalupe, CA (Arcero) Outdoor	SC	0.15 0.15 0.15 0.15	290 280 290 299	BBCH 47 BBCH 47 BBCH 48 BBCH 49	 4 5 5	4	7	0.20, 0.22 (0.21)	
United States, 2011 King City, CA (Red Wing) Outdoor	SC	0.15 0.14 0.15 0.15	299 280 299 299	BBCH 48 BBCH 48 BBCH 49 BBCH 49	 4 5 5	4	7	0.079, 0.14 (0.11)	
United States, 2011 Ephrata, WA (Colorado #6) Outdoor	SC	0.15 0.15 0.15 0.15	280 280 280 280	BBCH 41 BBCH 43 BBCH 45 BBCH 47	 5 5 5	4	7	0.037, 0.13 (0.082)	
United States, 2011 Portland, OR (Yellow Danver) Outdoor	SC	0.15 0.15 0.15 0.15	206 206 196 206	BBCH 45 BBCH 45 BBCH 48 BBCH 48	 5 5 5	4	7	0.064, 0.072 (0.068)	

Notes:

Portion analysed: bulb.

RTI: Re-treatment interval.

[1)] Mean of replicate field samples is given in parenthesis.

[2)] Ground application.

[3)] Aerial application.

The data on dry bulb onion conducted in the United States which were submitted in 2008 JMPR were presented below.

Table 10 Residues of mandipropamid on onion from supervised trials in the United States [provided to 2008 JMPR]

Onion country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93[2)]* *≥47[3)]*		*7-10*	*4*	*7*		
United States, 2004 Hudson, NY (White Sweet Spanish) Outdoor	SC	0.15 0.15 0.15 0.15	393 368 376 376	BBCH 43 BBCH 47 BBCH 47 BBCH 49	 7 7 7	4	0 3 5 7 9 14 16	<0.01, 0.02 (0.02) 0.03, 0.03 (0.03) <0.01, <0.01 (<0.01) <0.01, <0.01 (<u>≤0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	VV-502811 Mean recovery for mandipropamid: 84 ± 13 percent (n=18) at 0.01 mg/kg 86 ± 4.2 percent (n=6) at 0.05 mg/kg 83 ± 14 percent (n=3) at 0.1 mg/kg 84 ± 13 percent (n=3) at 0.2 mg/kg 84 ± 16 percent (n=4) at 0.5 mg/kg Sampling to analysis: 67-336 days
United States, 2004 Champaign, IL (Yellow Sweet Spanish) Outdoor	SC	0.17 0.16 0.16 0.16	29 29 29 28	BBCH 47 BBCH 79 BBCH 80 BBCH 89	 7 7 7	4	5 14	<0.01, <0.01 (<u>≤0.01</u>) <0.01, <0.01 (<0.01)	
United States, 2004 LaPryor, TX (Ebano) Outdoor	SC	0.15 0.15 0.15 0.15	173 174 161 166	Bulbs 1-3″ Bulbs 2-3″ Bulbs 3″ Bulbs 3″	 7 7 6	4	8 15	0.02, 0.04 (<u>0.03</u>) <0.01, <0.01 (<0.01)	
United States, 2004 LaSalle, CO (Candy) Outdoor	SC	0.15 0.16 0.15 0.15	151 148 148 123	Bulbing Bulbing Bulb development Bulb development	 7 7 7	4	7 14	<0.01, <0.01 (<u>≤0.01</u>) <0.01, <0.01 (<0.01)	
United States, 2004 Madera, CA (Fresno White) Outdoor	SC	0.15 0.15 0.15 0.15	280 281 283 282	Maturing onions 2-3″ bulbs BBCH 47 Mature onions	 7 7 7	4	7 14	<0.01, 0.01 (<u>0.01</u>) <0.01, <0.01 (<0.01)	
United States, 2004 Parma, ID (Vaquero) Outdoor	SC	0.15 0.15 0.15 0.15	235 235 235 235	5 percent down 10 percent down 50 percent down Vegetative	 7 7 7	4	7 14	<0.01, <0.01 (<u>≤0.01</u>) <0.01, <0.01 (<0.01)	
United States, 2004 Burlington, WA (Walla Walla Sweet) Outdoor	SC	0.15 0.15 0.15 0.15	150 150 149 147	BBCH 43 BBCH 43 BBCH 45 BBCH 47	 7 7 7	4	7 14	<0.01,0.02 (<u>0.02</u>) <0.01, <0.01 (<0.01)	
United States, 2004 Hughson, CA (Stockton	SC	0.15 0.15 0.15 0.15	233 235 235 235	BBCH 47 BBCH 47 BBCH 47 BBCH 49	 8 6 7	4	10 14	<0.01, <0.01 (<u>≤0.01</u>) <0.01, <0.01 (<0.01)	

Onion country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
Early Red) Outdoor									

Notes:

Portion analysed: bulb.

RTI: Re-treatment interval.

[1)] Mean of replicate field samples is given in parenthesis.

[2)] ground application, 3) aerial application.

Green Onions, Subgroup of

The Meeting received three trials on green onion which were conducted in the United States (Rice F, 2012: VV-507780). All trials received three foliar applications of an SC formulation (250 g ai/L) at a nominal rate of 0.15 kg ai/ha, with an application interval of 5 ± 1 days. All applications were made in tank-mix with an adjuvant, NIS or silicone-based. At each trial, green onions were taken 7 DALA.

Samples were analysed for residues of mandipropamid following analytical method RAM 415/01. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for mandipropamid in matrix were within 70–120 percent. Green onion samples were stored at ca -20 °C for a maximum of 8.9 months between sampling and analysis.

Table 11 Residues of mandipropamid on spring onion from supervised trials in United States

Spring onion country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93[2)] ≥47[3)]*		*7-10*	*3*	*7*		
United States, 2011 Oviedo, FL (Yellow Sweet Spanish) Outdoor	SC	0.15 0.14 0.15	281 281 281	BBCH 49 BBCH 49 BBCH 49	 5 5	3	7	1.5, 1.7 (1.6)	VV-507780 Mean recovery for mandipropamid: 90 percent (n=1) at 0.01 mg/kg 104 percent (n=1) at 25 mg/kg Sampling to analysis: 90-270 days
United States, 2011 Raymondville, TX (Evergreen White Bunching) Outdoor	SC	0.15 0.15 0.15	187 187 187	BBCH 13 BBCH 14 BBCH 14	 6 5	3	7	3.7, 3.8 (3.7)	
United States, 2011 Salinas, CA (Emerald Isle) Outdoor	SC	0.15 0.15 0.15	187 187 187	BBCH 15 BBCH 15 BBCH 15	 5 5	3	7	0.69, 0.77 (0.73)	

Notes:

Portion analysed: whole plant.

RTI: Re-treatment interval.

[1)] Mean of replicate field samples is given in parenthesis.

[2)] Ground application, 3) aerial application.

The data on spring onion conducted in the United States which were submitted in 2008 JMPR were presented below.

Table 12 Residues of mandipropamid on spring onion from supervised trials in the United States [provided to 2008 JMPR]

Spring onion country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93[2)] ≥47[3)]*		*7-10*	*3*	*7*		
United States, 2004 Chula, GA (Yellow Granex Sweet Onions) Outdoor	SC	0.15 0.15 0.15	169 170 163	¼ - ½ inch bulbs ¾ inch bulbs ¾ - 1 inch bulbs	 7 6	3	7	0.31, 0.48 (0.40)	VV-502811 (previously evaluated by the 2008 JMPR) Mean recovery for mandipropamid: 82 ± 11 percent (n=6) at 0.01 mg/kg 72 percent (n=2) at 0.05 mg/kg 80 percent (n=2) at 0.1 mg/kg 87 percent (n=2) at 0.2 mg/kg 90 percent (n=1) at 0.5 mg/kg Sampling to analysis: 155-188 days
United States, 2004 Uvalde, TX (Texas Early White) Outdoor	SC	0.15 0.15 0.15	167 170 167	3 leaf 4 leaf, bulbs 0.75 inch 1 inch bulbs	 7 7	3	7	1.2, 1.7 (1.4)	
United States, 2004 Visalia, CA (Southport White 404) Outdoor	SC	0.15 0.15 0.15	296 294 297	BBCH 13 BBCH 13 BBCH 47	 8 6	3	0 3 5 7 9	1.2, 1.5 (1.4) 0.57, 0.73 (0.65) 0.35, 0.59 (0.47) 0.20, 0.25 (0.23) 0.10, 0.20 (0.15)	

Notes:

Portion analysed: whole plant.

RTI: Re-treatment interval.

[1)] Mean of replicate field samples is given in parenthesis.

[2)] Ground application, 3) aerial application.

Fruiting vegetables, Cucurbits

Fruiting vegetables, Cucurbits–Cucumbers and Summer squashes, Subfroup of Cucumber

The Meeting received 10 trials on cucumber which were conducted in southern Europe (Spain and Italy) (Anderson L: VV-333902, VV-331995, VV-331994 (2004) and VV333645 (2005), Elliott A: VV333082 and VV333998 (2005), Lakaschus S: VV338985 (2007) and VV394509 (2010)). All trials received four foliar applications of an SC formulation (250 g ai/L) at a nominal rate of 0.15 kg ai/ha, with an application interval of 7 ± 1 days. At each trial, fruits were collected from all treated plots 0, 1, 3, 7 and 14 days after the final application. In some trials, additional fruit samples were taken just before the 2nd application, just before the 3rd application and just before the 4th application.

Samples were analysed for residues of mandipropamid following analytical method RAM 415/01. Additionally, in the 2010 trials, samples were analysed for residues of SYN500003 following analytical method GRM001.01A. The LOQs for both mandipropamid and SYN500003 were 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for mandipropamid and SYN500003 in matrix were within 70–120 percent. Cucumber fruit samples were stored at or below -18 °C for a maximum of 10 months between sampling and analysis.

Table 13 Residues of mandipropamid and SYN500003 on cucumber from supervised trials in southern Europe

Cucumber country, year (variety)	Application						DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.		Parent	SYN500003	
GAP, United States	*SC*	*0.15*	*≥93*[1)] *≥47*[2)]		*7-10*	*4*	*0*			
Spain, 2003 Murcia (Anico) Outdoor	SC	0.15 0.15 0.15 0.15	1000 1006 994 1000	81 81 81 81	 8 6 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.01 <0.01 <0.01 <u>0.07</u> 0.05 0.02 <0.01 <0.01	NA	VV-333902 Recovery for mandipropamid: 94 percent at 0.01 mg/kg 94 percent at 0.10 mg/kg Sampling to analysis: 292 days
Italy, 2003 Vicenza (Bounty) Outdoor	SC	0.15 0.15 0.15 0.14	892 925 908 857	61 71 69-79 732-734	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	<0.01 <0.01 <0.01 <u>0.06</u> 0.03 0.01 <0.01 <0.01	NA	VV-331995 Recovery for mandipropamid: 92 percent at 0.01 mg/kg 94 percent at 0.10 mg/kg Sampling to analysis: 288 days
Spain, 2003 Sevilla (Dasher) Outdoor	SC	0.15 0.15 0.16 0.15	818 790 1033 1004	73 79 79 79	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.01 <0.01 0.01 <u>0.08</u> 0.06 0.03 0.02 <0.01	NA	VV-331994 Recovery for mandipropamid: 91 percent at 0.01 mg/kg 97 percent at 0.10 mg/kg Sampling to analysis: 317 days
Italy, 2003 Foggia (Marketmore) Outdoor	SC	0.15 0.15 0.15 0.15	997 1017 1007 1003	631-713 631-802 633-804 806	 6 7 6	4	0 DBA2 0 DBA3 0 DBLA 0 1 3	<0.01 <0.01 0.01 <u>0.03</u> 0.02 0.02	NA	VV-333645 Recovery for mandipropamid: 99 percent at 0.01 mg/kg

Cucumber country, year (variety)	Application						DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.		Parent	SYN500003	
							8 15	<0.01 <0.01		94 percent at 0.10 mg/kg Sampling to analysis: 281 days
Spain, 2004 Valencia (Speak Head) Outdoor	SC	0.15 0.15 0.14 0.14	805 803 771 733	75-79 76-80 77-80 77-80	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 13	<0.01 <0.01 0.01 0.06 <u>0.08</u> 0.02 0.01 <0.01	NA	VV-333082 Recovery for mandipropamid: 88 percent at 0.01 mg/kg 91 percent at 0.10 mg/kg Sampling to analysis: 222 days
Spain, 2004 Huelva (Suso) Outdoor	SC	0.14 0.15 0.15 0.15	1125 1228 1171 1203	71 72 74 74	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.01 <0.01 <0.01 <u>0.05</u> 0.04 0.03 <0.01 <0.01	NA	VV-333998 Recovery for mandipropamid: 94, 103 percent at 0.01 mg/kg 91, 92 percent at 0.10 mg/kg
Spain, 2004 Seville (Dasher) Outdoor	SC	0.15 0.15 0.15 0.15	1074 1171 1190 1231	73 73 74 75	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	<0.01 0.02 0.01 <u>0.06</u> 0.05 0.03 0.01 <0.01	NA	Sampling to analysis: 147-198 days
Spain, 2006 Seville (Suzo) Outdoor	SC	0.16 0.16 0.15 0.15	828 836 807 818	22 63 63-65 65	 7 7 7	4	0 DBLA 0 1 3 7 14	<0.01 <u>0.02</u> <0.01 0.01 <0.01 <0.01	NA	VV-338985 Recovery for mandipropamid: 111 percent at 0.01 mg/kg 105 percent at 0.10 mg/kg Sampling to analysis: 318 days
Spain, 2009 Cadiz (Alanis) Outdoor	SC	0.15 0.15 0.16 0.15	802 827 838 803	81 82-83 83-84 85-86	 7 7 7	4	0 1 3 7 14	<u>0.14</u> 0.12 0.05 0.02 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	VV-394509 Recovery for mandipropamid: 89 percent at 0.01 mg/kg 98 percent at 0.10 mg/kg

Cucumber country, year (variety)	Application						DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.		Parent	SYN500003	
Spain, 2009 Seville (Dasher) Outdoor	SC	0.15 0.16 0.15 0.15	793 858 784 803	62-63 68-69 69-71 72-74	 7 7 7	4	0 1 3 7 14	0.07 0.08 0.02 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	for SYN50003: 78 percent at 0.01 mg/kg 98 percent at 0.10 mg/kg Sampling to analysis: 135-192 days

Notes:

NA: not analysed.

Portion analysed: fruit.

RTI: Re-treatment interval.

DBA: days before application, DBLA: days before last application.

[1)] Ground application.

[2)] Aerial application.

The data on cucumber conducted in the United States which were submitted in 2008 JMPR were presented below.

Table 14 Residues of mandipropamid on cucumber from supervised trials in United States [provided to 2008 JMPR]

Cucumber country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93[2)] ≥47[3)]*		*7-10*	*4*	*0*		
United States, 2004 Chula, GA (Straight 8) Outdoor	SC	0.16 0.16 0.15 0.15	124 125 116 117	BBCH 61 BBCH 65 BBCH 45 BBCH 49	 7 7 7	4	0 7 	0.022, 0.045 (0.033) 0.011, 0.015 (0.013)	VV-501780 Mean recovery for mandipropamid: 93 ± 22 percent (n=9) at 0.01 mg/kg 79 percent (n=2) at 0.05 mg/kg 92 ± 13 percent (n=3) at 0.1 mg/kg 79 ± 4.4 percent (n=3) at 0.5 mg/kg 94 percent (n=1) at 1.0 mg/kg Sampling to analysis: 92-303
United States, 2004 Rose Hill, NC (Poinsett 76) Outdoor	SC	0.15 0.15 0.15 0.15	147 142 245 157	1st bloom Fruit set Fruiting Mature	 6 7 8	4	0 7 	0.010, 0.018 (0.014) <0.01, <0.01 (<0.01)	
United States, 2004/2005 Vero Beach, FL (Straight 8) Outdoor	SC	0.15 0.15 0.15 0.15	282 287 285 286	BBCH 51-59 BBCH 51-61 BBCH 65-69 BBCH 71-77	 7 7 9	4	0 7 	0.068, 0.071 (0.069) <0.01, <0.01 (<0.01)	
United States, 2004 Conklin, MI (Marketmore 76) Outdoor	SC	0.15 0.15 0.15 0.15	19 19 19 19	Fruiting Fruiting Fruiting Mature	 8 6 8	4	0 7 	<0.01, 0.016 (0.013) 0.012, 0.012 (0.012)	
United States, 2004 Fitchburg, WI (Marketmore 76)	SC	0.15 0.16 0.15 0.15	202 219 218 207	BBCH 65 BBCH 73 BBCH 74 BBCH 78	 6 7 7	4	0 6 	<0.01, 0.012 (0.011) <0.01, <0.01 (<0.01)	

Cucumber country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
Outdoor									days
United States, 2004 Uvalde, TX (Turbo) Outdoor	SC	0.15 0.15 0.15 0.15	164 171 166 173	Male flowers present Fruiting Fruiting (80 percent full) Normal harvest	 7 7 7	4	0 7 	<0.01, 0.017 (0.013) <0.01, <0.01 (<0.01)	
United States, 2004 Madera, CA (Armenian yard long) Outdoor	SC	0.15 0.15 0.15 0.15	281 285 284 282	BBCH 59 Flowering Medium fruit BBCH 79	 7 9 9	4	0 3 5 7 9	0.036, 0.054 (0.045) 0.025, 0.032 (0.028) 0.019, 0.026 (0.022) <0.01, <0.01 (<0.01) 0.011, 0.011 (0.011)	

Notes:

Portion analysed: fruit.

RTI: Re-treatment interval.

[1)] Mean of replicate field samples is given in parenthesis.

[2)] Ground application.

[3)] aerial application.

Summer Squash

The data on summer squash conducted in United States which were submitted in 2008 JMPR were presented below.

Table 15 Residues of mandipropamid on summer squash from supervised trials in United States [provided to 2008 JMPR]

Summer squash country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	For m	kg ai/h a	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93[2)] ≥47[3)]*		*7-10*	*4*	*0*		
United States, 2004 Hudson, NY (Yellow Straight) Outdoor	SC	0.15 0.15 0.15 0.15	391 383 386 367	BBCH 41 BBCH 45 BBCH 47 BBCH 49	 7 7 7	4	0 7 	0.022, 0.039 (0.030) <0.01, <0.01 (<0.01)	VV-501780 Mean recovery for mandipropamid: 91 ± 18 percent (n=9) at 0.01 mg/kg 79 percent (n=2) at 0.05 mg/kg 86 ± 3.5 percent (n=3) at
United States, 2004 Elko, SC (Lemondrop L) Outdoor	SC	0.15 0.15 0.15 0.15	301 301 300 301	Vegetative Vegetative/early bloom Fruiting Fruiting/mature	 6 5 5	4	0 6 	0.062, 0.079 (0.070) <0.01, 0.013 (0.011)	
United States, 2005 Vero Beach, FL	SC	0.16 0.15 0.15	283 280 273	BBCH 61-63 BBCH 61-65 BBCH 63-65	 6 8	4	0 7	0.047, 0.068 (0.058) <0.01, <0.01	

Summer squash country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
(Early Yellow Crookneck) Outdoor		0.16	288	BBCH 65-71	7			(<0.01)	0.1 mg/kg 88 percent (n=1) at 0.2 mg/kg 89 percent (n=1) at 0.5 mg/kg 98 percent (n=2) at 1.0 mg/kg Sampling to analysis: 25-308 days
United States, 2004 Champaign, IL (Ambassador) Outdoor	SC	0.15 0.15 0.15 0.16	23 23 23 24	BBCH 65 BBCH 73 BBCH 75 BBCH 77	 7 5 9	4	0 7 	0.017, 0.030 (0.023) <0.01, <0.01 (<0.01)	
United States, 2004 Hickman, CA (Yellow Crookneck) Outdoor	SC	0.15 0.15 0.15 0.15	233 234 235 235	BBCH 51 BBCH 65 BBCH 67 BBCH 73	 6 8 5	4	0 3 5 7 9	<0.01, 0.015 (0.013) <0.01, 0.017 (0.014) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	

Notes:

Portion analysed: fruit.

RTI: Re-treatment interval is given in parenthesis.

[1)] Mean of replicate field samples is given in parenthesis.

[2)] Ground application.

[3)] aerial application.

Fruiting vegetables, Cucurbits–Melons, Pumpkins and Winter squashes Melons, Subgroup of

The Meeting received 8 trials on melon which were conducted in Europe (France, Spain and Italy) (Gill P: VV-332608 and VV332609 (2005), Boxwell C: VV-333097 and VV33098 (2005), Gardinal P & Gill P: VV333790 and VV333791 (2005)). All trials received four foliar applications of an SC formulation (250 g ai/L) at a nominal rate of 0.15 kg ai/ha, with an application interval of 7 ± 1 days. At each trial, fruits were collected from all treated plots immediately before the 2nd application, immediately before the 3rd application, immediately before the last application, then nominally 0, 1, 3, 7 and 14 days after the final application.

Samples were analysed for residues of mandipropamid following analytical method RAM 415/01. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for mandipropamid in matrix were within 70–120 percent. Melon samples were stored at or below -18 °C for a maximum of 9 months between sampling and analysis.

Table 16 Residues of mandipropamid on melon from supervised trials in Europe

Melon country, year (variety)	Application						DALA Days	Commodity	Residues, mg/kg	Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.				
GAP, United States	*SC*	*0.15*	*≥93[2)]* *≥47[3)]*		*7-10*	*4*	*0*			

Melon country, year (variety)	Application						DALA Days	Commodity	Residues, mg/kg	Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.				
France, 2003 Mirabel (Cezame) Outdoor	SC	0.15 0.15 0.15 0.15	1000 1000 1000 1000	71 71-71 72-73 82-83	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Whole fruit[1)]	0.01 0.03 0.03 <u>0.08</u> 0.08 0.05 0.03 0.02	VV-332608 Recovery for mandipropamid in Flesh: 105 percent at 0.01 mg/kg 95 percent at 0.02 mg/kg in Peel: 98 percent at 0.01 mg/kg 94 percent at 0.1 mg/kg Sampling to analysis: 253-259 days
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Peel	0.01 0.05 0.05 0.18 0.20 0.13 0.07 0.04	
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Flesh	<0.01 <0.01 <0.01 <u><0.01</u> <0.01 <0.01 <0.01 <0.01	
Spain, 2003 Palos de la Frontera, Huelva (Sancho) Outdoor	SC	0.15 0.16 0.15 0.15	492 525 512 487	69 69-71 81 84	 6 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Whole fruit[1)]	0.01 0.02 0.02 <u>0.05</u> 0.04 0.03 0.03 0.01	VV-333791 Recovery for mandipropamid in Flesh: 99 percent at 0.01 mg/kg 94 percent at 0.02 mg/kg in Peel: 92 percent at 0.01 mg/kg 90 percent at 0.1 mg/kg Sampling to analysis: 252-253 days
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Peel	0.01 0.04 0.05 0.15 0.11 0.07 0.08 0.03	
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Flesh	<0.01 <0.01 <0.01 <u><0.01</u> <0.01 <0.01 <0.01 <0.01	
Italy, 2004 Serravalle a Po, MN (Bingo) Outdoor	SC	0.15 0.15 0.15 0.15	1016 1003 1016 1011	68-78 69-81 69-83 69-86	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7	Whole fruit[1)]	0.03 0.04 0.05 <u>0.11</u> 0.11 0.10 0.07	VV-333098 Recovery for mandipropamid in Flesh: 91, 93 percent at 0.01 mg/kg

Melon country, year (variety)	Application						DALA Days	Commodity	Residues, mg/kg	Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.				
							14		0.04	94, 96 percent at 0.05 mg/kg in Peel: 71, 88 percent at 0.01 mg/kg 91, 95 percent at 0.5 mg/kg Sampling to analysis: 210-212 days
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Peel	0.08 0.09 0.13 0.32 0.36 0.32 0.22 0.10	
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Flesh	<0.01 <0.01 <0.01 <u><0.01</u> <0.01 <0.01 <0.01 <0.01	
Italy, 2004 Ferrara, FE (Baggio) Outdoor	SC	0.15 0.15 0.15 0.15	1005 986 995 992	69-72 72-74 76-77 81-82	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Whole fruit[1)]	0.03 0.14 0.06 <u>0.13</u> 0.10 0.13 0.10 0.04	
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Peel	0.08 0.37 0.16 0.37 0.26 0.36 0.24 0.08	
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Flesh	<0.01 <0.01 <0.01 <u><0.01</u> <0.01 <0.01 <0.01 <0.01	
France, 2004 Monteux (Anasta) Outdoor	SC	0.15 0.15 0.15 015	800 800 800 800	73 75 79 81	 8 6 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Whole fruit[1)]	0.01 0.03 0.03 0.04 <u>0.06</u> 0.03 0.04 0.02	VV-333097 Recovery for mandipropamid in Flesh: 93, 94 percent at 0.01 mg/kg 99, 101 percent at 0.05 mg/kg in Peel: 100, 110 percent at 0.01 mg/kg
							0 DBA2 0 DBA3 0 DBLA 0 1	Peel	0.02 0.06 0.09 0.12 0.17	

Melon country, year (variety)	Application						DALA Days	Commodity	Residues, mg/kg	Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.				
							3 7 14		0.08 0.10 0.05	92, 98 percent at 0.5 mg/kg Sampling to analysis: 119-135 days
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Flesh	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	
France, 2004 Grisolles (Cezanne) Outdoor	SC	0.16 0.15 0.15 0.15	800 800 800 800	71 71 73 75	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Whole fruit[1)]	0.02 0.03 0.04 0.06 <0.07 0.04 0.04 0.01	
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Peel	0.05 0.07 0.13 0.19 0.22 0.13 0.12 0.03	
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Flesh	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	
Spain, 2003 Villalba del Alcor, Huelva (Sancho) Outdoor	SC	0.15 0.16 0.14 0.16	716 730 660 757	69-71 70-75 75-80 76-81	 6 6 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Whole fruit[1)]	0.02 0.02 0.02 <0.03 0.03 0.03 0.02 0.02	VV-333790 Recovery for mandipropamid in Flesh: 87 percent at 0.01 mg/kg 91 percent at 0.02 mg/kg in Peel: 96 percent at 0.01 mg/kg 88 percent at 0.1 mg/kg Sampling to analysis: 266-267 days
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Peel	0.04 0.06 0.04 0.08 0.08 0.08 0.05 0.06	
							0 DBA2 0 DBA3 0 DBLA 0	Flesh	<0.01 <0.01 <0.01 <0.01	

Melon country, year (variety)	Application						DALA Days	Commodity	Residues, mg/kg	Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.				
							1 3 7 14		<0.01 <0.01 <0.01 <0.01	
France, 2003 Senas (Escrito) Outdoor	SC	0.15 0.15 0.15 0.15	800 800 800 800	85-86 86-88 86-88 86-88	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Whole fruit[1)]	0.01 0.01 0.02 <u>0.06</u> 0.03 0.04 0.03 0.02	VV-332609 Recovery for mandipropamid in Flesh: 74 percent at 0.01 mg/kg 101 percent at 0.02 mg/kg in Peel: 99 percent at 0.01 mg/kg 100 percent at 0.1 mg/kg Sampling to analysis: 266-268 days
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Peel	0.02 0.02 0.05 0.20 0.06 0.09 0.06 0.03	
							0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	Flesh	<0.01 <0.01 <0.01 <u><0.01</u> <0.01 <0.01 <0.01 <0.01	

Notes:

RTI: Re-treatment interval.

DBA: days before application, DBLA: days before last application.

[1)] Whole fruit residues are calculated from individual peel and fresh residues, using the weights of the separated crop parts.

[2)] Ground application.

[3)] Aerial application.

The data on cantaloupe conducted in United States which were submitted in 2008 JMPR were presented below.

Table 17: Residues of mandipropamid on cantaloupe from supervised trials in United States [provided to 2008 JMPR]

Cantaloupe country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*<u>≥93</u>[2)] <u>≥47</u>[3)]*		*7-10*	*4*	*0*		
United States, 2004 Chula, GA (Athena) Outdoor	SC	0.15 0.15 0.15 0.15	259 261 265 269	Fruiting (70 percent) Fruiting (80 percent) 1 percent fruit ripe	 7 6 7	4	0 7	0.018, 0.072 (<u>0.045</u>) 0.015, 0.023 (0.019)	VV-501780 Mean recovery for mandipropamid:

Cantaloupe country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
				Mature fruit					82 ± 6.7 percent (n=8) at 0.01 mg/kg 74 percent (n=1) at 0.05 mg/kg 80 ± 13 percent (n=3) at 0.1 mg/kg 81 percent (n=1) at 0.2 mg/kg 84 percent (n=2) at 0.5 mg/kg 94 percent (n=2) at 1.0 mg/kg Sampling to analysis: 196-308 days
United States, 2004 Champaign, IL (Eclipse) Outdoor	SC	0.14 0.15 0.16 0.15	108 150 144 136	BBCH 73 BBCH 71 BBCH 75 BBCH 77	 8 6 8	4	0 5	0.16, 0.19 (0.17) 0.072, 0.075 (0.073)	
United States, 2004/2005 Uvalde, TX (Caravelle) Outdoor	SC	0.15 0.15 0.15 0.15	194 203 197 193	Fruiting 70 percent 60 percent full size fruit 60 percent fruit ripe Mature fruit	 7 7 7	4	0 7	0.084, 0.11 (0.097) 0.060, 0.061 (0.060)	
United States, 2004 Visalia, CA (Hale's Best Jumbo) Outdoor	SC	0.15 0.15 0.15 0.15	131 131 137 134	BBCH 73 BBCH 77 BBCH 81 BBCH 85	 7 7 7	4	0 3 5 7 9	0.091, 0.12 (0.11) 0.031, 0.095 (0.063) 0.031, 0.034 (0.032) 0.034, 0.048 (0.041) 0.031, 0.040 (0.036)	
United States, 2004 Live Oak, CA[a)] (Durango) Outdoor	SC	0.15 0.15 0.15 0.15	141 143 141 141	Fruit enlargement Fruit enlargement Fruit enlargement Mature fruit	 6 7 7	4	0 7	0.034, 0.054 (0.044) 0.053, 0.062 (0.057)	
United States, 2004 Live Oak, CA[b)] (Top Mark) Outdoor	SC	0.15 0.15 0.15 0.15	142 140 142 141	Fruit enlargement Fruit enlargement Fruit sizing Mature fruit	 7 7 7	4	0 7	0.20, 0.26 (0.23) 0.022, 0.071 (0.046)	

Notes:

Portion analysed: fruit

RTI: Re-treatment interval

1) Mean of replicate field samples is given in parenthesis.

2) ground application, 3) aerial application.

a) Address: not reported, Application dates (1st): 08/18/2004.

b) Address: not reported, Application dates (1st): 08/24/2004.

Fruiting vegetables, other than Cucurbits

Tomatoes, Subgroup of

The Meeting received 14 trials on protected tomato which were conducted in Europe (Richards S: VV-331426, VV-331742, VV331744, VV-331862, VV332991 and VV333934 (2004), Gill JP: VV333205, VV-333389, VV-333398, VV333777, VV333878 and VV-334287 (2005), Simon P: VV333427 (2005)). All trials received four foliar applications of an SC formulation (250 g ai/L) at a nominal rate of 0.15 kg ai/ha, with

an application interval of 7 ± 1 days. At each trial, fruits were collected from all treated plots immediately before the 2nd application, immediately before the 3rd application, immediately before the last application, then nominally 0, 1, 3, 7 and 14 days after the final application. In some trials, additional fruit samples were taken just after the 1st application, just after the 2nd application and just after the 3rd application.

Samples were analysed for residues of mandipropamid following analytical method RAM 415/01. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for mandipropamid in matrix were within 70–120 percent. Tomato fruit samples were stored at or below -18 °C for a maximum of 12 months between sampling and analysis.

Table 18 Residues of mandipropamid on protected tomato from supervised trials in Europe

Tomato country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93[1)] ≥47[2)]*		*7-10*	*4*	*1*		
Tomato (medium/large)									
Spain, 2004 Granada (Trimiti) Protected (greenhouse)	SC	0.15 0.15 0.15 0.15	795 848 963 1025	71-72 72-73 73-74 74-75	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	<0.01 0.02 0.07 0.06 <u>0.08</u> 0.04 0.03 0.03	VV-333878 Recovery for mandipropamid: 91, 93 percent at 0.05 mg/kg 94, 94 percent at 0.50 mg/kg Sampling to analysis: 357 days
	SC	0.15 0.15 0.15 0.15	803 853 965 1013	71-72 72-73 73-74 74-75	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	<0.01 0.03 0.06 0.04 0.04 0.04 0.04 0.04	
France, 2003 Longue Jumelles (Palmiro) Protected (tunnel)	SC	0.15 0.15 0.14 0.17	1000 1000 1000 1000	74 75 76 76	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.02 0.04 0.08 0.13 0.12 <u>0.14</u> 0.13 0.09	VV-332991 Recovery for mandipropamid: 94 percent at 0.01 mg/kg 92 percent at 0.30 mg/kg Sampling to analysis: 199 days
Spain, 2003 Granada (Caramba) Protected	SC	0.14 0.14 0.14 0.14	809 810 1009 1004	71-72 72-73 73-74 74-75	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.02 0.05 0.07 0.11 0.12 <u>0.13</u> 0.07 0.06	VV-331742 Recovery for mandipropamid: 95 percent at 0.01 mg/kg 88 percent at 0.10 mg/kg

Tomato country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.			
									Sampling to analysis: 205 days
Italy, 2004 Carpino, FG (Naxos) Protected (polytunnel)	SC	0.15 0.15 0.15 0.15	1214 1221 1227 1179	73 74-80 81 85	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.06 0.09 0.14 0.26 <u>0.28</u> 0.24 0.25 0.25	VV-333389 Recovery for mandipropamid: 73, 85 percent at 0.01 mg/kg 83, 84 percent at 0.10 mg/kg
	SC	0.15 0.15 0.16 0.15	1214 1229 1243 1207	73 74-80 81 85	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.07 0.13 0.20 0.28 0.28 0.28 0.25 0.23	 Sampling to analysis: 325 days
Switzerland, 2003 Fully, VS (Petula) Glasshouse	SC	0.15 0.15 0.16 0.16	909 889 1095 1095	68-71 68-81 69-82 75-84	 7 7 7	4	0 DAA1 0 DBA2 0 DAA2 0 DBA3 0 DAA3 0 DBLA 0 1 3 7 14	0.03 <0.01 0.02 0.03 0.06 0.06 0.05 0.07 <u>0.13</u> 0.11 0.11	VV-331862 Recovery for mandipropamid: 88 percent at 0.01 mg/kg 87 percent at 0.10 mg/kg Sampling to analysis: 182 days
Spain, 2003 Almeria (Marisma) Protected (glasshouse)	SC	0.15 0.16 0.14 0.15	975 1032 1048 1084	73 74 75 75	 7 7 6	4	0 DAA1 0 DBA2 0 DAA2 0 DBA3 0 DAA3 0 DBLA 0 1 3 7 14	<0.01 0.01 0.04 0.04 0.07 0.10 0.14 <u>0.24</u> 0.15 0.19 0.19	VV-331426 Recovery for mandipropamid: 87 percent at 0.01 mg/kg 95 percent at 0.10 mg/kg Sampling to analysis: 203 days
France, 2004 Vancluse (Myriade) Indoor	SC	0.15 0.15 0.15 0.15	1000 1000 1000 1000	71 73-74 74-81 82	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.02 0.05 0.14 0.11 0.09 0.09 0.15 <u>0.17</u>	VV-333205 Recovery for mandipropamid: 100 ± 4.6 percent (n=3) at 0.01 mg/kg 98 ± 1.2 percent (n=3)at 0.10 mg/kg Sampling to

Tomato country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.			
									analysis: 352 days
Switzerland, 2004 Fully, VS (Petula) Protected (glasshouse)	SC	0.16 0.16 0.15 0.15	1046 1033 1007 1020	68-81 68-82 69-84 69-85	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.05 0.09 0.13 0.19 <u>0.18</u> 0.16 0.11 0.14	VV-333777 Recovery for mandipropamid: 87, 91 percent at 0.01 mg/kg 97, 103 percent at 0.10 mg/kg
	SC	0.16 0.15 0.15 0.15	1056 1014 1000 972	68-81 68-82 69-84 69-85	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.05 0.09 0.14 0.16 0.16 0.09 0.12 0.10	Sampling to analysis: 315 days
Germany, 2004 Dresden (Vanessa) Glasshouse	SC	0.15 0.15 0.15 0.15	1000 1000 1000 1000	73 82 83 84	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.08 0.28 0.32 0.59 <u>0.45</u> 0.40 0.35 0.27	VV-333427 Recovery for mandipropamid: 95 percent at 0.05 mg/kg 94 percent at 0.50 mg/kg
	SC	0.15 0.15 0.15 0.15	1000 1000 1000 1000	73 82 83 84	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.09 0.13 0.22 0.49 0.44 0.35 0.38 0.25	Sampling to analysis: 295 days
Cherry tomato									
Italy, 2004 Manfredonia, FG (Rubino Top) Protected (plastic tunnel)	SC	0.15 0.15 0.15 0.15	1226 1215 1172 1185	74-80 74-81 82 86	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.15 0.32 0.42 0.59 <u>0.65</u> 0.52 0.42 0.29	VV-334287 Recovery for mandipropamid: 95 percent at 0.01 mg/kg 90 percent at 0.50 mg/kg Sampling to analysis: 312 days
Italy, 2003 Carpino, FC (Naomi) Protected (plastic tunnel)	SC	0.15 0.15 0.15 0.15	1010 1005 1006 967	87-89 87-89 89 89	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 6 14	0.16 0.16 0.32 0.55 0.50 <u>0.60</u> 0.48 0.37	VV-333934 Recovery for mandipropamid: 105 percent at 0.01 mg/kg 91 percent at 0.10 mg/kg

Tomato country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	L/ha	BBCH	RTI days	no.			
									Sampling to analysis: 150 days
Italy, 2003 Zapponeta, FG (Piccadilly) Protected (plastic tunnel)	SC	0.15 0.15 0.15 0.15	976 1012 1018 1000	81 82-83 84-85 87	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.08 0.14 0.27 0.37 0.29 0.29 <u>0.30</u> 0.25	VV-331744 Recovery for mandipropamid: 96 percent at 0.01 mg/kg 89 percent at 0.10 mg/kg 85 percent at 1.0 mg/kg Sampling to analysis: 148 days
Spain, 2004 Almeria (Natcha) Protected	SC	0.15 0.14 0.16 0.15	1040 1070 1160 1250	74 74 75 77	 7 6 8	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 6 15	0.25 0.15 0.10 0.30 0.27 <u>0.33</u> 0.32 0.23	VV-333398 Recovery for mandipropamid: 85 percent (n=2) at 0.01 mg/kg 91 percent (n=1) at 0.05 mg/kg 94 ± 4.6 percent (n=3)at 0.50 mg/kg Sampling to analysis: 131-199 days
Spain, 2004 Granada (Lupita) Protected	SC	0.15 0.15 0.15 0.15	813 853 1025 1098	71-72 72-73 73-74 74-75	 7 7 7	4	0 DBA2 0 DBA3 0 DBLA 0 1 3 7 14	0.05 0.18 0.25 0.30 0.27 0.28 <u>0.34</u> 0.29	

Notes:

Portion analysed: fruit.

RTI: Re-treatment interval.

DBA: days before application, DAA: days after application, DBLA: days before last application.

[1)] Ground application.

[2)] Aerial application.

The data on tomato conducted in United States which were submitted in 2008 JMPR were presented below.

Table 19 Residues of mandipropamid on tomato from supervised trials in United States [provided to 2008 JMPR]

Tomato country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93*[2)]		*7-10*	*4*	*1*		

Tomato country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
			≥47[3)]						
United States, 2003 Livingston, NY (Sebring) Outdoor	SC	0.15 0.15 0.15 0.15	283 280 286 284	BBCH 65-71 BBCH 65-75 BBCH 73 BBCH 75-77	 8 6 7	4	1 3 	0.015, 0.017 (0.016) <0.01, 0.014 (0.012)	VV-503530 Mean recovery for mandipropamid: 89 ± 10 percent (n=27) at 0.01 mg/kg 92 ± 14 percent (n=10) at 0.05 mg/kg 99 ± 8.8 percent (n=10) at 0.1 mg/kg 92 ± 11 percent (n=10) at 0.5 mg/kg Sampling to analysis: 98-261 days
United States, 2003 Champaign, IL (Roma) Outdoor	SC	0.16 0.16 0.15 0.16	156 174 163 157	BBCH 73 BBCH 73 BBCH 73 BBCH 77	 7 6 8	4	1 3 	0.055, 0.056 (0.056) 0.015, 0.022 (0.019)	
United States, 2003 Elko, SC (Celebrity) Outdoor	SC	0.15 0.15 0.15 0.15	323 332 330 331	Fruiting Fruiting Fruiting Ripening	 7 6 6	4	0 1 2 3 4 	0.099, 0.10 (0.10) 0.081, 0.12 (0.10) 0.020, 0.066 (0.043) 0.045, 0.048 (0.047) 0.022, 0.057 (0.040)	
United States, 2004 Vero Beach, FL (Grande) Outdoor	SC	0.15 0.15 0.15 0.15	437 426 427 425	BBCH 69-73 BBCH 69-75 BBCH 79-81 BBCH 79-89	 7 7 6	4	1 3 	0.16, 0.20 (0.18) 0.057, 0.085 (0.071)	
United States, 2003 Vero Beach, FL (Florida 47) Outdoor	SC	0.15 0.14 0.15 0.15	353 338 349 349	BBCH 67-73 BBCH 67-73 BBCH 75-77 BBCH 77-81	 7 7 7	4	1 3 	0.073, 0.091 (0.082) 0.012, 0.022 (0.017)	
United States, 2003 Brawley, CA (Mountain Fresh) Outdoor	SC	0.15 0.15 0.15 0.15	189 185 189 186	Ripening Ripening Mature Mature	 8 8 7	4	1 3 	0.032, 0.032 (0.032) 0.018, 0.033 (0.026)	
United States, 2003 Hickman, CA (Bobcat) Outdoor	SC	0.15 0.15 0.15 0.15	187 185 189 187	BBCH 77 BBCH 79 BBCH 79 BBCH 79	 7 7 7	4	1 3 	0.032, 0.062 (0.047) 0.023, 0.035 (0.029)	
United States, 2003 Visalia, CA (Roma) Outdoor	SC	0.15 0.15 0.15 0.16	201 201 197 207	BBCH 77 BBCH 79 BBCH 82 BBCH 88	 7 8 7	4	1 3 	0.023, 0.032 (0.028) 0.052, 0.058 (0.056)	
United States, 2003 Le Grand, CA (U-941) Outdoor	SC	0.15 0.14 0.15 0.15	672 634 699 657	BBCH 47 BBCH 88 BBCH 88 BBCH 88	 7 7 7	4	1 3 	0.042, 0.061 (0.052) 0.035, 0.052 (0.044)	
United States, 2003 Maxwell, CA[a)]	SC	0.15 0.15 0.15	186 187 186	Ripening Ripening Ripening /mature Ripening /mature	 7 7	4	1 3 	0.040, 0.056 (0.048) 0.050, 0.079	

Tomato country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
(9888) Outdoor		0.15	188		7			(0.065)	
United States, 2003 Maxwell, CA [b)] (410) Outdoor	SC	0.15 0.15 0.15 0.15	185 186 187 186	Ripening Ripening Ripening /mature Ripening /mature	 7 7 7	4	0 1 2 3 4	0.025, 0.026 (0.026) 0.032, 0.033 (0.033) 0.015, 0.020 (0.018) 0.015, 0.019 (0.017) <0.01, <0.01 (<0.01)	

Notes:

Portion analysed: fruit.

RTI: Re-treatment interval.

[1)] Mean of replicate field samples is given in parenthesis.

[2)] ground application, 3) aerial application.

[a)] Address: not reported, Application dates (1st): 06/20/2003.

[b)] Address: not reported, Application dates (1st): 06/13/2003.

Peppers, Subgroup of

Peppers

The data on peppers conducted in United States which were submitted in 2008 JMPR were presented below.

Table 20 Residues of mandipropamid on peppers from supervised trials in United States [provided to 2008 JMPR]

Peppers country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	For m	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93[2)] ≥47[3)]*		*7-10*	*4*	*1*		
United States, 2004 Visalia, CA (Habanero) Peppers, Chili Outdoor	SC	0.15 0.15 0.15 0.16	301 295 300 307	BBCH 85 BBCH 89 BBCH 89 BBCH 89	 6 7 7	4	1 3	0.11, 0.22 (0.16) 0.084, 0.14 (0.11)	VV-503530 Mean recovery for mandipropamid: 95± 12 percent (n=19) at 0.01 mg/kg 107 ± 11 percent (n=4) at 0.05 mg/kg 95 ± 9.8 percent (n=9) at 0.1 mg/kg 95 ± 8.5 percent
United States, 2003 Champaign, IL (Capistrano) Peppers, Sweet Outdoor	SC	0.15 0.13 0.14 0.15	156 146 144 148	BBCH 73 BBCH 73 BBCH 77 BBCH 79	 6 8 6	4	1 3	0.038, 0.043 (0.041) 0.026, 0.031 (0.029)	
United States, 2003 Uvalde, TX (Taurus)	SC	0.16 0.15 0.15 0.15	148 148 188 142	Fruiting Fruiting Fruiting Mature	 7 7 7	4	1 3	0.040, 0.067 (0.054) 0.045, 0.054 (0.050)	

Peppers country, year (variety)	Application						DALA Days	Residues, mg/kg[1)]	Ref
	For m	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
Peppers, Sweet Outdoor									(n=5) at 0.5 mg/kg
United States, 2003 Uvalde, TX (Jalapeno M) Peppers, Chili Outdoor	SC	0.15 0.15 0.15 0.15	194 189 196 189	Full bloom Full bloom/ mature Full bloom/ mature Mature	 7 7 8	4	1 3 	0.37, 0.38 (0.37) 0.22, 0.26 (0.24) 	Sampling to analysis: 64-217 days
United States, 2003 Rincon, NM (Big Jim) Peppers, Chili Outdoor	SC	0.15 0.15 0.15 0.15	183 209 232 246	Fruiting Fruiting Fruiting Fruiting	 7 7 7	4	1 3 	0.055, 0.11 (0.083) 0.048, 0.074 (0.061) 	
United States, 2003 Rose Hilly, NC (HMX 1640 F1) Peppers, Sweet Outdoor	SC	0.15 0.15 0.15 0.15	19 19 19 19	Bloom/ fruiting Fruiting Fruiting Mature	 7 7 7	4	1 3 	0.092, 0.17 (0.13) 0.058, 0.078 (0.068) 	
United States, 2003 Vero Beach, FL (Brigadier) Peppers, Sweet Outdoor	SC	0.15 0.15 0.15 0.16	347 344 347 369	BBCH 67-75 BBCH 67-75 BBCH 75-77 BBCH 75-79	 7 7 7	4	1 3 	0.32, 0.33 (0.33) 0.26, 0.29 (0.28) 	
United States, 2003 Gilroy, CA (Cal 300) Peppers, Sweet Outdoor	SC	0.15 0.15 0.15 0.15	314 314 299 301	BBCH 75 BBCH 77 BBCH 85 BBCH 79	 7 7 6	4	1 3 	0.087, 0.092 (0.090) 0.060, 0.080 (0.070) 	
United States, 2003 Visalia, CA (Valiant) Peppers, Sweet Outdoor	SC	0.15 0.15 0.15 0.15	218 216 210 201	BBCH 69 BBCH 71 BBCH 72 BBCH 74	 7 8 6	4	0 1 2 3 4	0.050, 0.12 (0.083) 0.027, 0.031 (0.029) 0.052, 0.068 (0.060) 0.060, 0.060 (0.060) 0.035, 0.089 (0.062)	

Notes:

Portion analysed: fruit.

RTI: Re-treatment interval.

[1)] Mean of replicate field samples is given in parenthesis.

[2)] Ground application.

[3)] Aerial application.

Ginseng

The Meeting received five trials on fresh ginseng which were conducted in China (Zhiguang Hou, 2020: JLND2020RS001). In each of these trials, an SC formulation (23.4 percent) was applied with dosages of 0.21 kg ai/ha for one time using a foliar treatment. At each trial, fresh ginsengs were taken 14, 21 and 28 DALA. In the decline trials additional samples were collected at 7 and 35 DALA.

Samples were analysed for residues of mandipropamid following analytical method JLND2020RS001-A. The LOQ was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for mandipropamid in matrix were within 70–120 percent. Ginseng samples were stored at -18 °C or below for 18-20 days between sampling and analysis.

Table 21 Residues of mandipropamid on ginseng from supervised trials in China

Ginseng country, year (variety)	Application				DALA Days	Residues, mg/kg[1)]	Ref
	Form	kg ai /ha	L/ha	no.			
GAP, China	*SC*	*0.14-0.21*		*1*	*21*		
China, 2020 Yanji, Jilin (-) Outdoor	SC	0.21 0.21 0.21	604 606 602	1 1 1	14 21 28	<0.010, <0.010 (<0.010) <0.010, <0.010 (<u><0.010</u>) <0.010, <0.010 (<0.010)	JLND2020RS001 Mean recovery for mandipropamid: 90 ± 3.0 percent (n=10) at 0.05 mg/kg for fresh ginseng, Sampling to analysis: 18-20 days for fresh ginseng,
China, 2020 Baishan, Jilin (-) Outdoor	SC	0.21 0.21 0.21 0.21 0.21	604 604 602 606 602	1 1 1 1 1	7 14 21 28 35	<0.010, <0.010 (<0.010) <0.010, <0.010 (<0.010) <0.010, <0.010 (<u><0.010</u>) <0.010, <0.010 (<0.010) <0.010, <0.010 (<0.010)	
China, 2020 Huanren, Liaonign (-) Outdoor	SC	0.21 0.21 0.21 0.21 0.21	602 606 602 606 604	1 1 1 1 1	7 14 21 28 35	0.019, 0.020 (0.020) 0.067, 0.19 (0.13) 0.033, 0.033 (0.033) 0.068, 0.069 (0.069) 0.076, 0.078 (<u>0.077</u>)	
China, 2020 Fusong, Jilin (-) Outdoor	SC	0.21 0.21 0.21 0.21 0.21	606 602 604 602 606	1 1 1 1 1	8 15 22 29 36	<0.010, <0.010 (<0.010) <0.010, <0.010 (<0.010) <0.010, <0.010 (<u><0.010</u>) <0.010, <0.010 (<0.010) <0.010, <0.010 (<0.010)	
China, 2020 Ji'an, Jilin (-) Outdoor	SC	0.21 0.21 0.21	602 606 604	1 1 1	14 21 28	0.034, 0.035 (0.035) <0.010, <0.010 (<0.010) <0.010, 0.016 (<u>0.013</u>)	

Notes:

Portion analysed: fresh ginseng.

1) Mean of replicate field samples is given in parenthesis.

Ginseng, dried including red ginseng

The Meeting received five trials on dried ginseng which were conducted in China (Zhiguang Hou, 2020: JLND2020RS001). In each of these trials, an SC formulation (23.4 percent) was applied with dosages of 0.21 kg ai/ha for one time using a foliar treatment. At each trial, fresh ginsengs were taken 14, 21 and 28 DALA. In the decline trials additional samples were collected at 7 and 35 DALA. A portion of fresh ginseng samples were processed into dried ginseng and red ginseng with simulation of industrial practices as closely as possible.

Dried ginseng: fresh samples were baked at 55 °C for 24 h.

Red ginseng: fresh samples were steamed with boiling water for 2.5 ~ 3 hours, and cooled and dried at 70 °C for 6 hours, then were infiltrated with water and dried again at 55 °C for 24 hours.

Samples were analysed for residues of mandipropamid following analytical method JLND2020RS001-A. The LOQ was 0.01 mg/kg for both commodities. The overall mean recoveries from concurrent fortifications for mandipropamid in each matrix were within 70–120 percent. Ginseng samples were stored at -18 °C or below for 18–20 days between sampling and analysis.

Table 22 Residues of mandipropamid on dried ginseng from supervised trials in China

Ginseng country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg[1)]	Ref
	Form	kg ai /ha	L/ha	no.				
GAP, China	*SC*	*0.14-0.21*		*1*	*21*			
China, 2020 Yanji, Jilin (-) Outdoor	SC	0.21	604	1	14	Dried ginseng Red ginseng	0.012, 0.012 (0.012) 0.085, 0.091 (0.088)	JLND2020RS001 Mean recovery for mandipropamid: 97 ± 5.3 percent (n=10) at 0.05 mg/kg for dried ginseng, 97 ± 2.6 percent (n=10) at 0.05 mg/kg for red ginseng Sampling to analysis: 23-25 days for dried ginseng, 28-30 days for red ginseng
		0.21	606	1	21	Dried ginseng Red ginseng	0.011, 0.011 (0.011) 0.16, 0.29 (0.23)	
		0.21	602	1	28	Dried ginseng Red ginseng	<0.010, <0.010 (<0.010) 0.11, 0.12 (0.12)	
China, 2020 Baishan, Jilin (-) Outdoor	SC	0.21	604	1	7	Dried ginseng Red ginseng	0.016, 0.023 (0.020) 0.80, 0.81 (0.81)	
		0.21	604	1	14	Dried ginseng Red ginseng	0.017, 0.019 (0.018) 0.24, 0.26 (0.25)	
		0.21	602	1	21	Dried ginseng Red ginseng	0.031, 0.035 (0.033) 0.30, 0.31 (0.31)	
		0.21	606	1	28	Dried ginseng Red ginseng	0.017, 0.018 (0.018) 0.48, 0.49 (0.49)	
		0.21	602	1	35	Dried ginseng Red ginseng	0.033, 0.039 (0.036) 0.36, 0.37 (0.37)	
China, 2020 Huanren, Liaonign (-) Outdoor	SC	0.21	602	1	7	Dried ginseng Red ginseng	0.13, 0.13 (0.13) 2.5, 2.5 (2.5)	
		0.21	606	1	14	Dried ginseng Red ginseng	0.22, 0.23 (0.23) 1.9, 1.9 (1.9)	
		0.21	602	1	21	Dried ginseng Red ginseng	0.16, 0.16 (0.16) 1.5, 1.5 (1.5)	
		0.21	606	1	28	Dried ginseng Red ginseng	0.21, 0.27 (0.24) 1.4, 1.5 (1.5)	
		0.21	604	1	35	Dried ginseng Red ginseng	0.38, 0.47 (0.43) 2.1, 2.2 (2.2)	
China, 2020 Fusong, Jilin (-) Outdoor	SC	0.21	606	1	8	Dried ginseng Red ginseng	<0.010, <0.010 (<0.010) 0.14, 0.15 (0.15)	
		0.21	602	1	15	Dried ginseng Red ginseng	<0.010, 0.012 (0.011) 0.092, 0.10 (0.096)	
		0.21	604	1	22	Dried ginseng Red ginseng	0.015, 0.015 (0.015) 0.18, 0.18 (0.18)	
		0.21	602	1	29	Dried ginseng Red ginseng	0.016, 0.018 (0.017) 0.13, 0.15 (0.14)	
		0.21	606	1	36	Dried ginseng Red ginseng	0.029, 0.031 (0.030) 0.19, 0.19 (0.19)	
China, 2020 Ji'an, Jilin (-) Outdoor	SC	0.21	602	1	14	Dried ginseng Red ginseng	0.099, 0.10 (0.10) 0.24, 0.30 (0.27)	
		0.21	606	1	21	Dried ginseng Red ginseng	0.024, 0.024 (0.024) 0.38, 0.39 (0.39)	
		0.21	604	1	28	Dried ginseng	0.021, 0.021 (0.021)	

Ginseng country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg[1)]	Ref
	Form	kg ai /ha	L/ha	no.				
						Red ginseng	0.46, 0.46 (0.46)	

Note:

[1)] Mean of replicate field samples is given in parenthesis.

The Meeting received four trials on dried ginseng which were conducted in United States and Canada (Corley J, 2012: VV-508245). All trials received four foliar applications of an SC formulation (250 g ai/L) at a nominal rate of 0.15 kg ai/ha, with an application interval of 6–9 days. All applications were made in tank-mix with an adjuvant, NIS. At each trial, fresh roots were taken 2 DALA. In one trial additional samples were collected at 0, 7, 15 and 21 DALA to provide residue decline data. The roots were rinsed in clean water and dried for 17 or19 days in a commercial dryer at temperatures ranging from 7.2 to 48 °C.

Samples were analysed for residues of mandipropamid and SYN500003 following analytical method RAM 415/01 and GRM001.01A. However modifications were made to improve the performance of the method. Extracts were diluted with ACN:H_2O (20:80, v/v), to produce a solution of ACN:H_2O (1:1, v/v), and analysed for mandipropamid by LC-LC/MS/MS coupled with online cleanup. The LOQ for mandipropamid was 0.022 mg/kg and for SYN500003 was 0.0056 mg/kg. The overall mean recoveries from concurrent fortifications for mandipropamid and SYN500003 in matrix were within 70–120 percent. Dried ginseng root samples were stored at or below -20 °C for a maximum of 31 months between sampling and analysis.

Table 23 Residues of mandipropamid and SYN500003 on dried ginseng from supervised trials in Canada and the United States

Ginseng country, year (variety)	Application						DALA Days	Residues[1)], mg/kg		Ref
	For m	kg ai/ha	L/ha	Growth Stage	RTI days	no.		Parent	SYN500003	
GAP, USA	*SC*	*0.15*	*≥93[2)] ≥47[3)]*		*7-10*	*4*	*2*			
USA, 2008 Wausau, WI (American) Outdoor	SC	0.15 0.16 0.15 0.15	528 557 543 537	Fruiting Fruiting Fruiting Fruiting	 8 6 6	4	2	0.070, 0.17 (0.12)	<0.005, <0.005 (<0.005)	VV-508245 Recovery for mandipropamid: 105 ± 2.1 percent (n=4) at 0.1 mg/kg for SYN500003: 117 ± 9.2 percent (n=3) at 0.1 mg/kg Sampling to
USA, 2008 Athens, WI (American) Outdoor	SC	0.15 0.15 0.15 0.15	716 709 730 728	Fruiting Fruiting Fruiting Fruiting	 8 6 6	4	2	0.070, 0.14 (0.11)	<0.005, <0.005 (<0.005)	
USA, 2008 Poniatowski, WI (American) Outdoor	SC	0.14 0.14 0.15 0.15	884 884 902 905	Fruiting Fruiting Fruiting Fruiting	 8 6 6	4	2	0.022, 0.048 (0.035)	<0.005, <0.005 (<0.005)	

Ginseng country, year (variety)	Application						DALA Days	Residues[1], mg/kg		Ref
	For m	kg ai/ha	L/ha	Growth Stage	RTI days	no.		Parent	SYN500003	
Canada, 2008 Coldstream, BC (Unknown) Outdoor	SC	0.15 0.16 0.15 0.15	725 935 911 913	Ripening Ripening Ripe berries Ripe berries	 9 6 6	4	0 2 7 15 21	0.027, 0.033 (0.030) 0.023, 0.031 (0.027) 0.021, 0.021 (0.021) 0.020, 0.047 (0.033) 0.021, 0.027 (0.024)	<0.005, <0.005 (<0.005) <0.005, <0.005 (<0.005) <0.005, <0.005 (<0.005) <0.005, <0.005 (<0.005) <0.005, <0.005 (<0.005)	analysis: 918-939 days

Notes:

Notes:

Portion analysed: dried root.

[1)] Re-treatment interval is given in parenthesis.

[2)] Mean of replicate field samples is given in parenthesis.

[3)] Ground application,

[4)] Aerial application.

Basil (fresh)

The Meeting received six trials (four in the field and two in the greenhouse) on basil which were conducted in Canada and the United States (Corley J, 2012: VV-508246). All trials received four foliar applications of an SC formulation (250 g ai/L) at a nominal rate of 0.15 kg ai/ha, with an application interval of 6 days. All applications were made in tank-mix with an adjuvant, NIS. At each trial, fresh basil stems and leaves were taken 1 DALA. In one trial additional samples were collected at 3, 7 and 11 DALA to provide residue decline data.

Samples were analysed for residues of mandipropamid following analytical method RAM 415/01. However modifications were made to improve the performance of the method. Extracts were diluted with ACN:H_2O (20:80, v/v), to produce a solution of ACN:H_2O (1:1, v/v), and analysed for mandipropamid by LC-LC/MS/MS coupled with online cleanup.The LOQ for mandipropamid was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for mandipropamid in matrix were within 70–20 percent. Fresh basil samples were stored at or below -20 °C for a maximum of 24 months between sampling and analysis.

Table 24 Residues of mandipropamid on fresh basil from supervised trials in Canada and the United States

Basil country, year (variety)	Application						DALA Days	Residues[1], mg/kg	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93[2)]* *≥47[3)]*		*7-10*	*4*	*1*		

Basil country, year (variety)	Application						DALA Days	Residues[1], mg/kg	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
United States, 2009 Arlington, WI (Sweet) Outdoor	SC	0.15 0.15 0.15 0.15	270 256 255 253	Vegetative Budding/bloom Budding/bloom Budding/bloom	 7 6 7	4	1	6.3, 6.7 (6.5)	VV-508246 Recovery for mandipropamid: 97 ± 4.9 percent (n=4) at 1.0 mg/kg, 106 ± 10 percent (n=3) at 10 mg/kg Sampling to analysis: 299-723 days
United States, 2009 Clinton, NC (Genovese) Outdoor	SC	0.15 0.15 0.15 0.15	100 101 100 101	Vegetative Vegetative Vegetative/budding Vegetative/budding	 6 6 6	4	1 3 7 11	18, 19 (19) 6.8, 7.5 (7.2) 1.2, 1.4 (1.3) 0.17, 0.20 (0.19)	
United States, 2009 Salinas, CA (Italian Large Leaf) Outdoor	SC	0.14 0.15 0.14 0.15	197 203 214 213	Vegetative Vegetative Maturing Mature	 8 7 6	4	1	7.1, 9.9 (8.5)	
Canada, 2009 Agassiz, BC (DiGenova) Outdoor	SC	0.14 0.15 0.14 0.15	259 259 267 269	Vegetative Vegetative Vegetative Vegetative/budding	 6 8 6	4	1	8.7, 9.3 (9.0)	
United States, 2009 Salisbury, MD (Genovese Compact Improved) Greenhouse	SC	0.16 0.15 0.14 0.15	131 123 121 123	5th set of true leaves Pre-bloom Pre-bloom Pre-bloom	 7 7 6	4	1	3.5, 3.6 (3.6)	
United States, 2008 Citra, FL (Genova) Greenhouse	SC	0.14 0.15 0.15 0.15	227 337 233 234	Vegetative Vegetative Vegetative Vegetative	 6 7 7	4	1	9.4, 9.8 (9.6)	

Notes:

Portion analysed: fresh stems and leaves.

RTI: Re-treatment interval.

[1] Mean of replicate field samples is given in parenthesis.

[2] Ground application.

[3] Aerial application.

Basil (dried)

The Meeting received 6 trials (four in the field and two in the greenhouse) on basil which were conducted in Canada and the United States (Corley J, 2012: VV-508246). All trials received four foliar applications of an SC formulation (250 g ai/L) at a nominal rate of 0.15 kg ai/ha, with an application interval of 6–8 days. All applications were made in tank-mix with an adjuvant, NIS. At each trial, basil for drying was taken 1 DALA and dried for up to 3 days according to local commercial practices.

Samples were analysed for residues of mandipropamid following analytical method RAM 415/01. However modifications were made to improve the performance of the method. Extracts were diluted with ACN:H_2O (20:80, v/v), to produce a solution of ACN:H_2O (1:1, v/v), and analysed for mandipropamid by LC-

LC/MS/MS coupled with online cleanup.The LOQ for mandipropamid was 0.01 mg/kg. The overall mean recoveries from concurrent fortifications for mandipropamid in matrix were within 70–120 percent. Dried basil samples were stored at or below -20 °C for a maximum of 22 months between sampling and analysis.

Table 25 Residues of mandipropamid on dried basil from supervised trials in Canada and the United States

Basil country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	L/ha	Growth Stage	RTI days	no.			
GAP, United States	*SC*	*0.15*	*≥93*[1)] *≥47*[2)]		*7-10*	*4*	*1*		
United States, 2009 Arlington, WI (Sweet) Outdoor	SC	0.15 0.15 0.15 0.15	270 256 255 253	Vegetative Budding/bloom Budding/bloom Budding/bloom	 7 6 7	4	1	63	VV-508246 Recovery for mandipropamid: 100 ± 11 percent (n=4) at 1.0 mg/kg, 97, 103 percent (n=2) at 10 mg/kg Sampling to analysis: 296-671 days
United States, 2009 Clinton, NC (Genovese) Outdoor	SC	0.15 0.15 0.15 0.15	100 101 100 101	Vegetative Vegetative Vegetative/budding Vegetative/budding	 6 6 6	4	1	36	
United States, 2009 Salinas, CA (Italian Large Leaf) Outdoor	SC	0.14 0.15 0.14 0.15	197 203 214 213	Vegetative Vegetative Maturing Mature	 8 7 6	4	1	62	
Canada, 2009 Agassiz, BC (DiGenova) Outdoor	SC	0.14 0.15 0.14 0.15	259 259 267 269	Vegetative Vegetative Vegetative Vegetative/budding	 6 8 6	4	1	91	
United States, 2009 Salisbury, MD (Genovese Compact Improved) Greenhouse	SC	0.16 0.15 0.14 0.15	131 123 121 123	5th set of true leaves Pre-bloom Pre-bloom Pre-bloom	 7 7 6	4	1	48	
United States, 2008 Citra, FL (Genova) Greenhouse	SC	0.14 0.15 0.15 0.15	227 337 233 234	Vegetative Vegetative Vegetative Vegetative	 6 7 7	4	1	78	

Notes:

Portion analysed: dried stems and leaves.

RTI: Re-treatment interval.

[1)] Ground application.

[2)] Aerial application.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting received information on high temperature hydrolysis of SYN50003 and the fate of mandipropamid residues during the processing of tomato.

High temperature hydrolysis

The hydrolytic stability of [phenyl-U-^{14}C]-SYN500003 was investigated under conditions representative of food processing (Kennedy J, 2019: VV-732906). The conditions used are summarized below.

Temperature, °C	Time, min	pH	Process Represented
90	20	4	Pasteurisation
100	60	5	Baking, Brewing, Boiling
120	20	6	Sterilisation

Test solutions were prepared by adding an aliquot of the radiochemical stock solution to each buffer (0.01 M citrate buffers were prepared at pH 4, 5 and 6) to give a final test concentration of 1.0 mg/L. Test solutions were dispensed into glass vials (which were capable of withstanding the high temperature and pressures expected during incubation) and samples were incubated as per the conditions detailed above. The radioactivity content of incubates sampled prior to and following incubation was determined by LSC analysis. Radioactivity content in incubated samples was compared to control samples.

The total recovery of applied radioactivity ranged from 99.5–101.5 percent for all hydrolysis experiments. Characterisation and identification of the radiolabelled components in treated solutions in this study was carried out by co-chromatography against reference standards using TLC and HPLC. Quantification of the degradates was conducted using the HPLC data.

Table 26 Identification of radioactivity under the conditions for processing simulation

Identified components	Recovery of applied [phenyl-U-^{14}C]-SYN500003 [1.0 mg/L]					
	pH 4, 90 °C, 20min		pH 5, 100 °C, 60 min		pH 6, 120 °C, 20 min	
	% TRR	mg/L	% TRR	mg/L	% TRR	mg/L
SYN500003	98.0, 98.6	1.04, 1.05	98.7, 99.0	1.01, 1.00	98.9, 98.6	1.05, 1.04
Unidentified	2.0, 1.4	0.02, 0.01	1.3, 1.0	<0.01, <0.01	1.1, 1.4	0.01, 0.01
Total	100, 100	1.06, 1.06	100, 100	1.02, 1.01	100, 100	1.06, 1.05

Note:

% TRR: Results are based on the assumption that the chromatographed radioactivity represents 100 percent of the applied radioactivity.

[phenyl-U-^{14}C]-SYN500003 was found to be hydrolytically stable in buffer solutions at pH 4, 5 and 6 at temperatures simulating pasteurisation (90 °C), baking/brewing/boiling (100 °C) and sterilisation (120 °C) respectively.

Tomato

One processing trial on tomato was conducted in Southern France during 2004. Four foliar applications of an SC formulation containing 250 g/L mandipropamid were applied at 0.50 kg ai/ha separated by a 7 day interval. Treated tomato samples were collected at 3 DALA (Gill JP, 2005: WW-333625). Samples were

shipped frozen to the analytical facility for residue analysis and transferred by car directly to the processing facility.

Fresh whole tomatoes were processed into washed fruit, wet and dry pomace, juice, puree and canned tomatoes according to procedures representative of commercial practice. One full balance study and three follow-up studies were carried out for each process.

Washing

Samples were washed with water (500 mL/kg fruit) and strained. Samples of washing water and washed tomatoes were collected and stored for analysis.

Tomato juice processing

Samples of unwashed tomatoes were crushed and sieved to separate juice from the peel and seeds (pomace). The sugar content of the juice was measured and salt (ca. 7 g/kg) and citric acid (to ca. pH 3.5) were added to produce "raw" juice. A sub-sample of raw juice was retained for the canning phase. The remaining juice was pasteurised for 1 minute at 82–85 °C and placed into glass jars. Wet pomace was oven dried at 60 °C for 1–2 days to produce dry pomace.

Tomato puree processing

Samples of unwashed tomatoes were crushed and reduced in a saucepan. Reduction was stopped when the sugar content reached 12 percent. The tomatoes were sieved to remove peel and seeds (pomace), salt (approximately 4 g/kg) and citric acid (to about pH 3.5) were then added to produce "raw" puree. The puree was then sterilised in glass jars for 10 minutes at 115 °C.

Tomato canning

Samples of unwashed tomatoes were blanched and then immediately plunged into cold water to loosen the peel, which was then removed with a knife. The peeled tomatoes were placed into glass jars and the jars filled with "raw" tomato juice at a ratio of two thirds tomatoes to one third juice. The jars were sealed and sterilised for 10 minutes at 115 °C.

Samples of pre-processed whole fruit and processed commodities were analysed for residues of mandipropamid using analytical method RAM 415/01 with an LOQ of 0.01 mg/kg for all commodities. The overall mean recoveries from concurrent fortifications for mandipropamid in each matrix were within 70–120 percent.

Table 27 Residues of mandipropamid in tomato and tomato processed commodities

country, year (variety)	Application		DALA	Commodity	Residues, mg/kg	Processing factor
	kg ai/ha	no.	Days			
France, 2004	0.49	4	3	Fruit	0.38, 0.37, 0.34, 0.35	
Herault	0.50				Mean 0.36	-
(Isola)	0.51			Washed tomatoes	0.07, 0.10, 0.09, 0.18	
	0.50				Mean 0.11	0.31
				Wash water	0.30	0.83
				Crushed tomatoes	0.39	1.1
				Wet pomace	0.33, 0.46, 0.35, 0.30	
					Mean 0.36	1.0
				Raw juice	0.37	1.0
				Dry pomace	2.0, 1.9, 1.3, 1.2	
					Mean 1.6	4.4

country, year (variety)	Application kg ai/ha	Application no.	DALA Days	Commodity	Residues, mg/kg	Processing factor
				Finished juice	0.33, 0.45, 0.31, 0.37 Mean 0.37	1.0
				Crushed tomatoes	0.48	1.3
				Fruit after reduction	0.87	2.4
				Sieved tomatoes	0.38	1.1
				Wet pomace	3.8	11
				Raw puree (before sterilisation)	0.38	1.1
				Finished puree	0.41, 0.34, 0.49, 0.41 Mean 0.41	1.1
				Blanch water	0.22	0.61
				Cooling water	0.01	0.028
				Peeled fruit	<0.01	0.028
				Peel	0.50	1.4
				Canned tomatoes (before sterilisation)	0.13	0.36
				Canned tomatoes	0.12, 0.18, 0.11, 0.14 Mean 0.14	0.39

Notes:

Processing Factor = Mandipropamid residues in processed commodity/ Mandipropamid residues in tomato fruits (prior to processing)

For calculations, 0.01 mg/kg was used for fractions with <LOQ residues.

Processing trial on tomato was conducted in the United States during 2003. Four foliar applications of an SC formulation containing 250 g/L mandipropamid were applied at 0.15 kg ai/ha with a 7 day interval between applications. Applications were also made at 5× rate for processing. Treated tomato samples were collected at 1 DALA (Joseph & Hamilton, 2005: VV-503530).

Samples were analysed for residues of mandipropamid using analytical method RAM 415/01 with an LOQ of 0.01 mg/kg. Samples were fortified between 0.01 and 0.50 mg/kg and recoveries ranged from 73–111 percent.

Table 28 Residues of mandipropamid in tomato and tomato processed commodities

Tomato country, year (variety)	Application kg ai/ha	Application no.	DALA Days		Residues, mg/kg	Processing factor
United States, 2003 Visalia, CA (Roma)	0.15 0.15 0.15 0.15	4	1	Fruit (unwashed) Puree Paste	0.034, 0.056 Mean 0.045 0.11, 0.074 Mean 0.090 0.19, 0.21 Mean 0.20	 2.0 4.4
	0.75 0.75 0.75 0.75	4	1	Fruit (unwashed) Puree Paste	0.38, 0.37 Mean 0.37 0.37, 0.27 Mean 0.32 0.62, 0.81 Mean 0.71	 0.86 1.9
United States, 2003 Maxwell, CA[a)] (9888)	0.15 0.15 0.15 0.15	4	1	Fruit (unwashed) Puree Paste	0.019, 0.015 Mean 0.017 0.033, 0.018 Mean 0.026 0.12, 0.083 Mean 0.10	 1.5 5.9

Tomato country, year (variety)	Application kg ai/ha	Application no.	DALA Days		Residues, mg/kg	Processing factor
	0.75	4	1	Fruit (unwashed)	0.24, 0.28 Mean 0.26	
	0.75			Puree	0.28, 0.20 Mean 0.24	0.92
	0.75			Paste	0.65, 0.68 Mean 0.67	2.6
	0.75					

Notes:

Processing Factor = Mandipropamid residues in processed commodity/ Mandipropamid residues in tomato fruits (prior to processing)

APPRAISAL

Mandipropamid is a fungicide that belongs to the subset mandelamides in the class carboxylic acid amides. It is intended for the control of Oomycete fungal pathogens in a range of crops. It was first evaluated by JMPR in 2008. An ADI of 0–0.2 mg/kg bw was established and decided that an ARfD was unnecessary. The 2018 JMPR further concluded that metabolite SYN500003 should be assessed by TTC as Cramer Class III. The residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities is mandipropamid. The residue is not fat-soluble. Additional uses were evaluated by the 2013, 2018 and 2021 (extra) JMPR.

Mandipropamid was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses in the 2021 JMPR and rescheduled to the 2022 JMPR. The Meeting received information on additional analytical methods, storage stability, high temperature hydrolysis, residues during processing (tomato), GAP information and residue trials for bulb onion, green onion, cucumber, summer squash, melon, tomato, ginseng and basil.

Methods of analysis

The Meeting received descriptive and validation data of analytical methods for residue of mandipropamid in plant and animal commodities.

In the method for determination of mandipropamid in plant, homogenized samples were extracted with acetonitrile: water (80:20 v/v), with clean-up with a solid phase extraction, residues were determined by LC-MS/MS. The method of analysis was validated at various fortification levels with an LOQ of 0.01 mg/kg for mandipropamid.

QuEChERS method was also validated for mandipropamid residue in plant and animal commodities with an LOQ of 0.01 mg/kg.

Stability of residues in stored analytical samples

The Meeting received information on the freezer storage stability of mandipropamid in plant (dried beans, ginseng and basil) and SYN500003 in plant (ginseng and potato). Storage stability results indicated that residues of mandipropamid were stable for up to 12 months in dried broad beans, and for at least 9 months in fresh ginseng, 30 months in dried ginseng and 23 months in basil under freezer conditions at about -18 °C or below.

The Meeting agreed that the demonstrated storage stability for mandipropamid covered the residue sample storage intervals used in the field trials.

Results of supervised residue trials on crops

The Meeting received supervised trial data for the foliar application of mandipropamid on bulb onion, spring onion, cucumber, melon, tomato, ginseng and basil. Residue trial data was made available from Canada, China, France, Germany, Italy, Spain, Switzerland and the United States.

Labels were available from China and the United States describing the registered uses of mandipropamid.

Bulb vegetables

The 2008 JMPR evaluated residue data for mandipropamid on bulb onion and spring onion conducted in the United States. The Meeting received new residue data for bulb onion and spring onion.

Onion, Bulb

Data were available from supervised trials on bulb onion in the United States.

The critical GAP for dry bulb vegetables in the United States (same GAP as that submitted to the 2008 JMPR) allows a maximum seasonal rate of 0.59 kg ai/ha (maximum of 0.15 kg ai/ha per application) with a re-treatment interval (RTI) of 7 days and a PHI of 7 days.

Mandipropamid residues in bulb onion from independent trials (RTI: 6-8 days) conducted in 2004 (submitted to the 2008 JMPR) in the United States matching the US GAP were (n=8): <0.01 (5), 0.01, 0.02 and 0.03 mg/kg.

Mandipropamid residues in bulb onion from independent trials with a RTI of 4-6 days conducted in 2011 in the United States were (n=8): 0.012, 0.032, 0.062, 0.066, 0.068, 0.082, 0.11 and 0.21 mg/kg.

The trials in 2011 were conducted with a shorter RTI than that of the US GAP. The residue populations from trials with a RTI of 5 ± 1 days were significantly different from those from trials with a RTI of 7 ± 1 days according to statistical test (Mann-Whitney U-test).

Based on the residues in bulb onion from trials conducted in 2004 in the United States, the Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR value of 0.01 mg/kg for the subgroup of bulb onions.

The Meeting withdrew the previous recommendation for onion, bulb of 0.1 mg/kg.

Spring Onion

Data were available from supervised trials on spring onion in the United States.

The critical GAP for green onions in the United States (same GAP as that submitted to the 2008 JMPR) allows a maximum seasonal rate of 0.44 kg ai/ha (maximum 0.15 kg ai/ha per application) with a RTI of 7 days and a PHI of 7 days.

Mandipropamid residues in spring onion from independent trials conducted in 2004 (submitted to the 2008 JMPR) in the United States matching the US GAP were (n=3): 0.23, 0.40 and 1.4 mg/kg.

Mandipropamid residues in spring onion from independent trials with a RTI of 5 days conducted in 2011 in the United States were (n=3): 0.73, 1.6 and 3.7 mg/kg.

The trials in 2011 were conducted with a shorter RTI than that of the US GAP. The residue populations from trials with a RTI of 5 ± 1 days were significantly different from those from trials with a RTI of 7 ± 1 days according to statistical test (Mann-Whitney U-test).

The trials for spring onion in the United States were insufficient to estimate a maximum residue level for the commodity, since minimum of five trials need for spring onion. The Meeting could not estimate a maximum residue level for spring onion.

The Meeting withdrew the previous recommendation for spring onion of 7 mg/kg.

Fruiting vegetables, Cucurbits

Fruiting vegetables, Cucurbits–Cucumbers and Summer squashes

The critical GAP for cucurbits in the United States (same GAP as that submitted to the 2008 JMPR) allows a maximum seasonal rate of 0.59 kg ai/ha (maximum of 0.15 kg ai/ha per application) with a RTI of 7 days and a PHI of 0 day.

Cucumber

Data were available from supervised trials on cucumber in Italy, Spain and the United States.

Mandipropamid residues in cucumber from independent trials in Italy, Spain and the United States matching the US GAP were (n=17): 0.011, 0.013(2), 0.014, 0.02, 0.03, 0.033, 0.045, 0.05, 0.06 (2), 0.069, 0.07, 0.08 (3) and 0.14 mg/kg.

Squash, summer

Data were available from supervised trials on summer squash in the United States.

Mandipropamid residues in summer squash from independent trials in the United States matching the US GAP were (n=5): 0.014, 0.023, 0.030, 0.058 and 0.070 mg/kg.

To consider a maximum residue level for a group, residues in individual crops should be similar (e.g. medians should not differ by more than 5×). The Meeting agreed to estimate a maximum residue level for the subgroup of fruiting vegetables, cucurbits–cucumbers and summer squashes. In considering whether to combine data to estimate a maximum residue level, the Meeting recognized that the residue populations from trials on cucumber and summer squash were not different according to statistical test (Mann-Whitney U-test). Therefore, the Meeting decided to combine the data from cucumber and summer squash to estimate a maximum residue level for fruiting vegetables, cucurbits–cucumber and summer squashes.

The combined mandipropamid residues in cucumber and summer squash were in rank order (n=22): 0.011, 0.013(2), 0.014 (2), 0.02, 0.023, 0.03 (2), 0.033, 0.045, 0.05, 0.058, 0.06 (2), 0.069, 0.07 (2), 0.08 (3) and 0.14 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR value of 0.0475 mg/kg for the subgroup of fruiting vegetables, cucurbits–cucumber and summer squashes.

The Meeting withdrew the previous recommendations for cucumber of 0.2 mg/kg and summer squash of 0.2 mg/kg.

Fruiting vegetables, Cucurbits–Melons, Pumpkins and Winter Squashes

Data were available from supervised trials on melons in France, Italy, Spain and the United States.

The critical GAP for cucurbits in the United States (same GAP as that submitted to the 2008 JMPR) allows a maximum seasonal rate of 0.59 kg ai/ha (max 0.15 kg ai/ha per application) with a RTI of 7 days and a PHI of 0 day.

Mandipropamid residues in melons (whole fruit) from independent trials in France, Italy, Spain and the United States matching the US GAP were (n=13): 0.03, 0.045, 0.05, 0.06 (2), 0.07, 0.08, 0.097, 0.11 (2), 0.13, 0.17 and 0.23 mg/kg.

Mandipropamid residues in melons (flesh) from independent trials in France, Italy and Spain matching the US GAP were (n=8): <0.01 (8) mg/kg.

Based on the residues in melon (whole fruit) from trials, the Meeting estimated a maximum residue level of 0.4 mg/kg, and based on the residues in melon (flesh) from trials the Meeting estimated an STMR value of 0.01 mg/kg for the subgroup of fruiting vegetables, cucurbits–melons, pumpkins and winter squashes.

The Meeting withdrew the previous recommendations for melons except watermelon of 0.5 mg/kg.

Fruiting vegetables, other than Cucurbits

The critical GAP for fruiting vegetables in the United States (same GAP as that submitted to the 2008 JMPR) allows a maximum seasonal rate of 0.59 kg ai/ha (max 0.15 kg ai/ha per application) with a RTI of 7 days and a PHI of 1 day.

Peppers

Data were available from supervised trials on peppers in the United States (submitted to the 2008 JMPR).

Mandipropamid residues in bell pepper and non-bell pepper from independent outdoor trials in the United States matching the US GAP were (n=6+3): 0.041, 0.054, 0.062, 0.090, 0.13 and 0.33 mg/kg for bell pepper and 0.083, 0.16 and 0.37 mg/kg for non-bell pepper.

Based on the residues in peppers from trials in the United States, the Meeting estimated a maximum residue level of 0.7 mg/kg and an STMR value of 0.090 mg/kg for the subgroup of peppers (except Martynia, Okra and Roselle) to replace the previous recommendation for peppers of 1 mg/kg.

On the basis of the maximum residue level and the STMR for subgroup of peppers and default dehydration factor of 10, the Meeting estimated a maximum residue level and an STMR value of 7 and 0.9 mg/kg to replace the previous recommendation for peppers, chili, dried.

Tomato

The critical GAP for fruiting vegetables for indoor use tomatoes in the United States (same GAP as that submitted to the 2008 JMPR) allows a maximum seasonal rate of 0.59 kg ai/ha (max 0.15 kg ai/ha per application) with a RTI of 7 days and a PHI of 1 day.

Data were available from supervised trials on tomatoes in France, Germany, Italy, Spain, Switzerland and the United States.

Mandipropamid residues in tomatoes from independent indoor trials in France, Germany, Italy, Spain and Switzerland matching the US GAP were (n=14): 0.08, 0.13 (2), 0.14, 0.17, 0.18, 0.24, 0.28, 0.30, 0.33, 0.34, 0.45, 0.60 and 0.65 mg/kg.

Mandipropamid residues in tomato from independent outdoor trials in the United States matching the US GAP were (n=10): 0.016, 0.032, 0.047, 0.052, 0.056 (2), 0.065, 0.082, 0.10 and 0.18 mg/kg.

The residue populations from trials indoor and outdoor were significantly different according to statistical test (Mann-Whitney U-test).

Based on the residues in tomatoes from indoor trials, the Meeting estimated a maximum residue level of 1 mg/kg and an STMR value of 0.26 mg/kg for the subgroup of tomatoes.

The Meeting withdrew the previous recommendation for tomato of 0.3 mg/kg.

Eggplant

In comparison with the residues in peppers and tomatoes from outdoor trials, the dataset from peppers leads to the highest maximum residue level.

The Meeting agreed that the maximum residue level for the subgroup of peppers could be extrapolated to that of the subgroup of eggplants. The Meeting estimated a maximum residue level of 0.7 mg/kg and an STMR value of 0.090 mg/kg for the subgroup of eggplants.

Root and tuber vegetables

Ginseng and Ginseng, dried including red ginseng

Data were available from supervised trials on ginseng and dried ginseng in China.

The GAP for ginseng in China allows one application of 0.21 kg ai/ha with a PHI of 21 days.

Mandipropamid residues in fresh ginseng from independent trials in China matching Chinese GAP were (n=5): <0.010 (3), 0.013 and 0.077 mg/kg.

Mandipropamid residues in dried and red ginseng from independent trials in China matching Chinese GAP were (n=5): 0.19, 0.23, 0.46, 0.49 and 2.2 mg/kg.

Data were available from supervised trials on dried ginseng in Canada and the United States.

The GAP for ginseng in the United States allows a maximum seasonal rate of 0.59 kg ai/ha (maximum of 0.15 kg ai/ha per application) with a RTI of 7 days and a PHI of 2 days.

Mandipropamid residues in dried ginseng from independent trials in Canada and the United States matching the US GAP were (n=4): 0.033, 0.035, 0.11 and 0.12 mg/kg.

Based on the residues in fresh ginseng from trials in China, the Meeting estimated a maximum residue level of 0.15 mg/kg and an STMR value of 0.010 mg/kg for ginseng.

Based on the residues in dried ginseng from trials in China, the Meeting estimated a maximum residue level of 4 mg/kg and an STMR value of 0.46 mg/kg for ginseng, dried including red ginseng.

Herbs and spices

Basil and Basil leaves, dried

Data were available from supervised trials on basil and dried basil in Canada and the United States.

The critical GAP for basil in the United States allows a maximum seasonal rate of 0.59 kg ai/ha (max 0.15 kg ai/ha per application) with a RTI of 7 days and a PHI of 1 day.

Mandipropamid residues in fresh basil from independent trials in Canada and the United States matching the US GAP were (n=6): 3.6, 6.5, 8.5, 9.0, 9.6 and 19 mg/kg.

Mandipropamid residues in dried basil from independent trials in Canada and the United States matching the US GAP were (n=6): 36, 48, 62, 63, 78 and 91 mg/kg.

Based on the residues in fresh basil from trials in Canada and the United States, the Meeting estimated a maximum residue level of 30 mg/kg and an STMR value of 8.75 mg/kg for basil, leaves.

Based on the residues in dried basil from trials in Canada and the United States, the Meeting estimated a maximum residue level of 200 mg/kg and an STMR value of 62.5 mg/kg for basil leaves, dried.

Fate of residues during processing

High temperature hydrolysis

The hydrolytic stability of [phenyl-U-^{14}C]-SYN500003 was studied under conditions at high temperature in sterile aqueous buffers at pH 4, 5 and 6 for periods of up to 60 minutes so as to simulate common processing practices (pasteurization, baking/brewing/boiling, and sterilization). SYN500003 was detected ranged from 97.5 to 100.4 percent of applied radioactivity at the investigated pH and temperature ranges. SYN500003 is considered stable under hydrolytic conditions at high temperatures.

Residues in processed commodities

The fate of mandipropamid residues has been examined in tomato processing studies. Estimated processing factors and the derived STMR-Ps are summarized in the Table below.

Table 1 Calculated STMR-Ps for processed food and feed commodities

Raw commodity [STMR]	Processed commodity	Processing factors # [Mean or best estimate processing factor]	STMR-P = $STMR_{RAC}$ × PF (mg/kg)
Tomato [0.26 mg/kg]	Wet pomace	1.0	0.26
	Dry pomace	4.4	1.14
	Juice	1.0	0.26
	Puree	0.86, 0.92, 1.1, 1.5, 2.0 [1.1]	0.286
	Canned	0.39	0.101
	Paste	1.9, 2.6, 4.4, 5.9 [3.5]	0.91

Note:

The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

Residues in animal commodities

The 2022 JMPR evaluated residues of mandipropamid in tomato wet pomace, which is listed in the OECD feeding table. The Meeting noted that the estimation did not result in a change of the dietary burdens of farm animals (20.8 ppm for cattle and 2 ppm for poultry). The previous recommendations of maximum residue level for animal commodities were maintained.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for estimating maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for plant and animal commodities (for compliance with the MRL and for estimation of dietary exposure): *mandipropamid*

The residue is not fat-soluble

Table 2 Residue levels suitable for establishing maximum residue limits and for IEDI assessments

Commodity		Recommended MRL, mg/kg		STMR, mg/kg
CCN	Name	New	Previous	
HH 0722	Basil, leaves	30		8.75
DH 0722	Basil leaves, dried	200		62.5
VA 2031	Bulb Onions, Subgroup of	0.05		0.01
VC 0424	Cucumber	W	0.2	
VO 2046	Eggplants, Subgroup of	0.7		0.090
VC 2039	Fruiting vegetables, Cucurbits–Cucumber and Summer squashes, Subgroup of	0.2		0.0475
VC 2040	Fruiting vegetables, Cucurbits–Melons, Pumpkins and Winter squashes, Subgroup of	0.4		0.01
VR 0604	Ginseng	0.15		0.010
DV 0604	Ginseng, dried including red ginseng	4		0.46
VC 0046	Melon, except watermelon	W	0.5	
VA 0385	Onion, bulb	W	0.1	
VO 0051	Peppers (except Martynia, Okra and Roselle), Subgroup of	0.7	1	0.090
HS 0444	Peppers, Chili, dried	7	10	0.9
VA 0389	Spring onion	W	7	
VC 0431	Squash, summer	W	0.2	
VO 0448	Tomato	W	0.3	
VO 2045	Tomatoes, Subgroup of	1		0.26
	Tomato, canned			0.101
JF 0448	Tomato juice			0.26
	Tomato paste			0.91
DM 0448	Tomato puree			0.286
DM 3525	Tomato pomace			0.26

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for mandipropamid is 0–0.2 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for mandipropamid were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report. The IEDIs ranged from 1–8 percent of the maximum ADI.

The Meeting concluded that long-term dietary exposure to residues of mandipropamid from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2008 JMPR decided that an ARfD for mandipropamid was unnecessary. Therefore, the Meeting concluded that the acute dietary exposure to residues of mandipropamid from the uses considered is unlikely to present a public health concern.

Threshold of toxicological concern (TTC) consideration for metabolite SYN500003

At the 2018 JMPR, the Meeting agreed to utilize the TTC approach to assess the metabolite SYN500003 (Cramer Class III). The 2018 JMPR concluded that residues of SYN500003 were only expected in root and tubers vegetables.

In the Canadian and United States trials for dried ginseng, SYN500003 residues were <0.005 mg/kg in all samples. The Meeting agreed that no additional exposure to SYN500003 is expected from ginseng and dried ginseng.

The Meeting concluded that SYN500003 is unlikely to present a public health concern.

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MEFENTRIFLUCONAZOLE (320)

First draft prepared by M Thomas and M Le, Pest Management Regulatory Agency, Canada

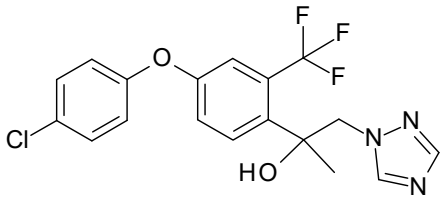
EXPLANATION

Mefentrifluconazole, (2RS)-2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-ol, is a triazole fungicide belonging to the group of the sterol biosynthesis inhibitors.

Mefentrifluconazole was scheduled at the Fifty-first Session of the CCPR for Evaluation for Residues and Toxicology by the 2020 JMPR. However, the Toxicology evaluation was conducted by the WHO Core Assessment Group at the 2021 JMPR Meeting, where an ADI of 0–0.04 mg/kg bw and an ARfD of 0.3 mg/kg bw were established. The Residue evaluation was rescheduled to the 2022 JMPR Meeting.

The Meeting received information from the manufacturer on physical and chemical properties, metabolism studies on plants and animals, environmental fate in soil, analytical method and stability in stored analytical samples, use patterns and supervised residue trials, processing studies and livestock feeding studies.

IDENTITY

ISO common Name:	Mefentrifluconazole
IUPAC Name:	(2RS)-2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-ol
Chemical Abstracts Name:	α-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-α-methyl-1*H*-1,2,4-triazole-1-ethanol
CAS No.:	1417782-03-6
CIPAC No.:	Not assigned
Synonyms:	BAS 750F
Molecular Formula:	$C_{18}H_{15}ClF_3N_3O_2$
Structural Formula:	
Molecular Weight:	397.78 g/mol

Specifications

Specifications for mefentrifluconazole have not been developed by the FAO.

Physical and chemical properties

Table 1 Physical and chemical properties of the pure active (98 percent purity)

Parameters	Value		Reference
Appearance	White, odourless crystalline powder		Kroehl, 2014
Vapour pressure	Temperature (°C)	Pa	Kroehl, 2014
	20	3.2×10^{-6}	
	25	6.5×10^{-6}	
Melting point	190 °C		Kroehl, 2014
Partition coefficient	pH	Log Kow	Wilbrand, 2013

Parameters	Value		Reference
n-octanol / water (20 °C)	4	3.4	
	7	3.4	
	7 (buffered)	3.3	
	9 (buffered)	3.4	
Solubility in water (22 °C)	Solvent	mg/L	Wilbrand, 2013
	Distilled water	0.81	
	pH 4	0.66	
	pH 7	0.71	
Solubility in organic solvents (20 °C)	Solvent	Solubility (g/L)	Wilbrand, 2013
	n-heptane	0.094	
	xylene	8	
	acetonitrile	50	
	methanol	71	
	acetone	94	
	ethyl acetate	116	
	1,2-dichloroethane	55	
Hydrolysis in water	Stable in water for at least 30 days at 25 °C in sterile aqueous buffer at pH 4, 5, 7, and 9		Hassink, 2015
Photolysis in sterile water	pH	DT_{50} in irradiated sterile buffer	Yan, 2015
	7	2.3 days	
	Irradiation of radiolabelled mefentrifluconazole resulted in the formation of the major photodegradation products M750F005 (≤32 percent applied radioactivity [AR]), M750F006 (≤31 percent AR), M750F007 (≤44 percent AR) and M750F008 (≤7 percent AR)		
Dissociation constant (20 °C)	3.0		Wilbrand, 2013

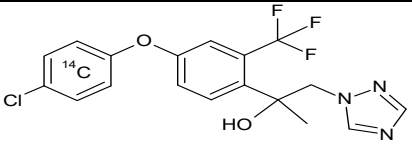
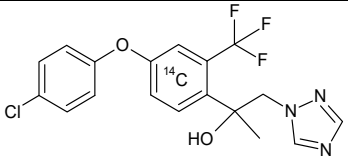
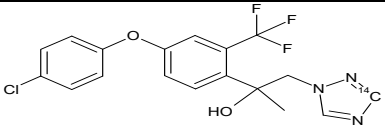
Mefentrifluconazole appears to be slightly soluble in water and moderately soluble in non-polar solvents. It is likely to sequester to fatty matrices based on its octanol/water partition coefficient. It has low potential for volatilization. Hydrolysis and aqueous photolysis are unlikely to be important routes of degradation at environmentally relevant pH levels.

Formulations

Mefentrifluconazole is available as soluble concentrate or emulsifiable concentrate formulations containing up to 400 g mefentrifluconazole/L.

METABOLISM AND ENVIRONMENTAL FATE

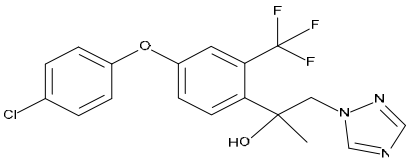
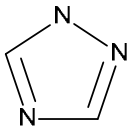
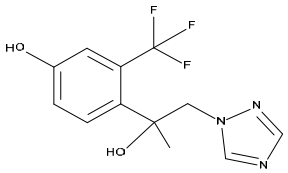
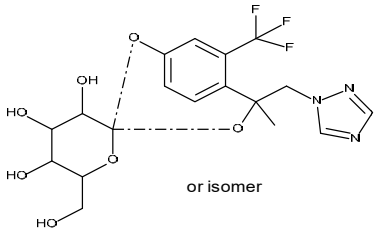
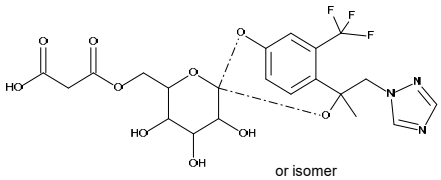
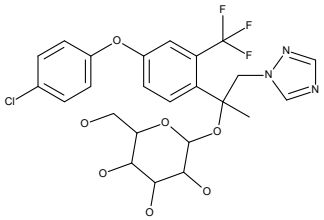
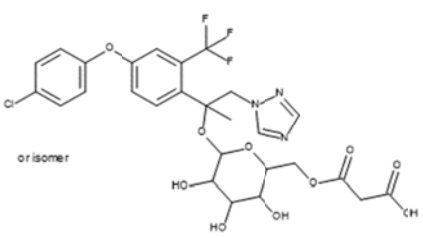
The fate and behaviour of mefentrifluconazole in animals, plants, and the environment was investigated using [^{14}C]-mefentrifluconazole as shown below:

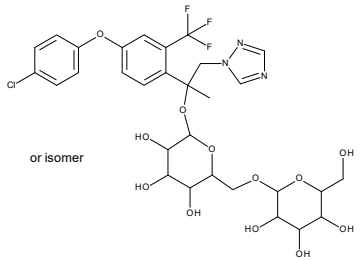
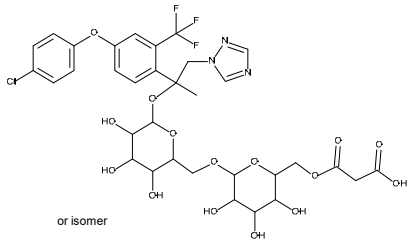
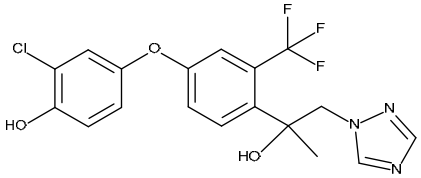
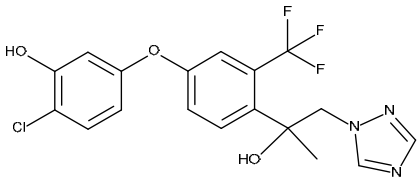
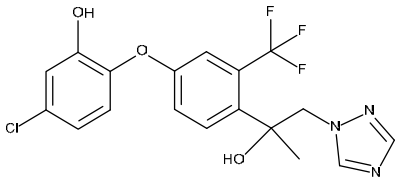
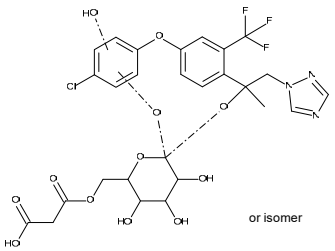
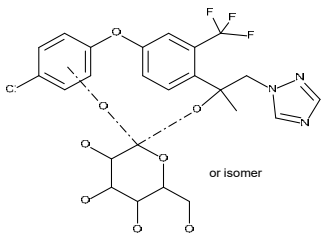
[Chlorophenyl-U-^{14}C]-mefentrifluconazole	[Trifluoromethylphenyl-U-^{14}C]-mefentrifluconazole	[Triazole-3(5)-^{14}C]- mefentrifluconazole
		

Chemical names, structures and code names of metabolites and degradation products of

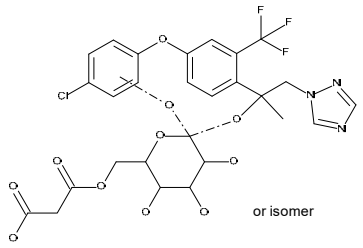
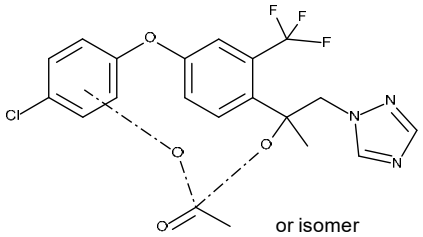
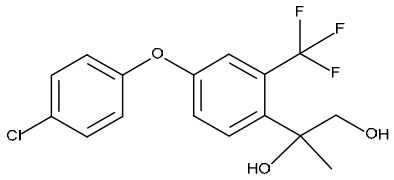
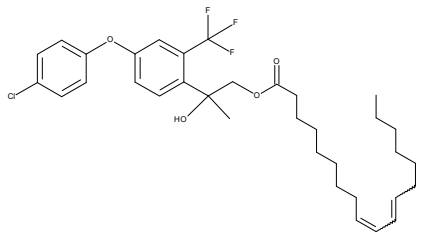
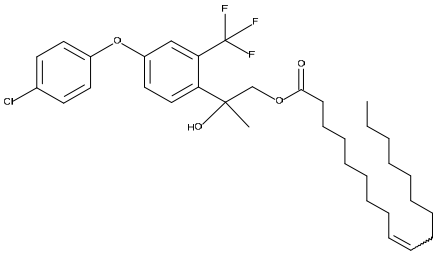
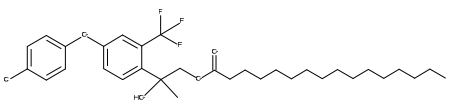
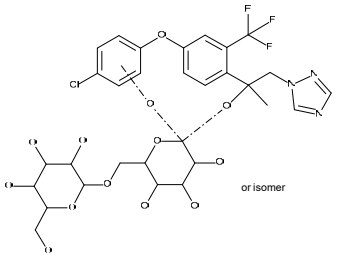
mefentrifluconazole are summarized in Table 2. Compounds are referred to primarily by the code name.

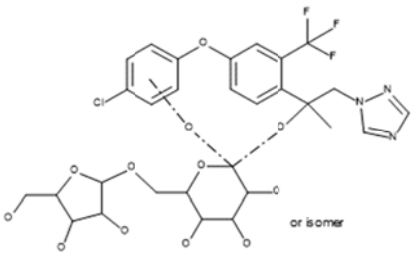
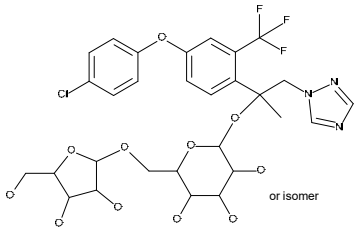
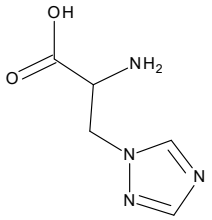
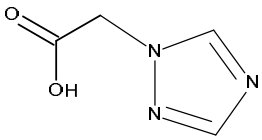
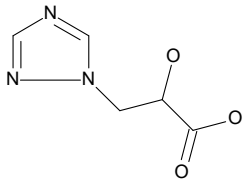
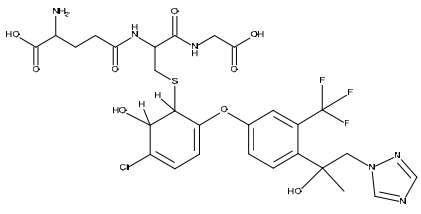
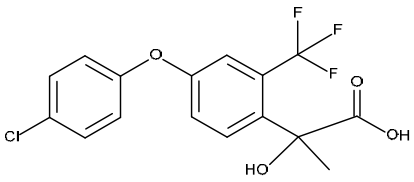
Table 2 Code names, chemical names and structures of mefentrifluconazole related substances

Code Number (Reg. Number)	Chemical Name	Molecular Structure	Rat	Hen & Goat	Wheat, Grape & Soya bean	Rotational Crops
Mefentrifluconazole (5834378)	(2RS)-2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-ol			X	X	
M750F001 (87084) 1,2,4-triazole	1,2,4-(1*H*)-triazole		X	X	X	X
M750F003 (5924326)	4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenol			X		
M750F009					X	
M750F010					X	
M750F011	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl hexopyranoside				X	
M750F012	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl 6-O-(carboxyacetyl)hexopyranoside				X	

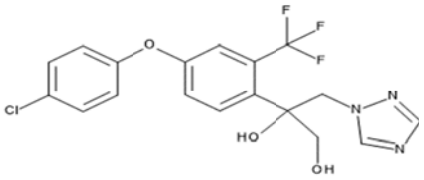
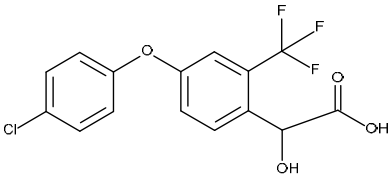
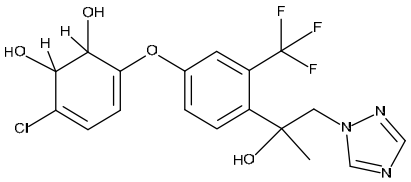
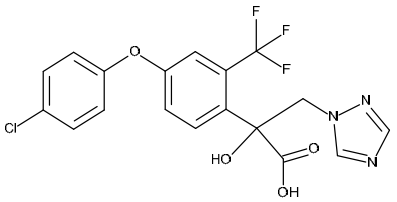
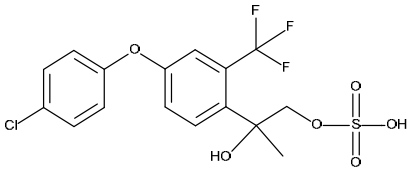
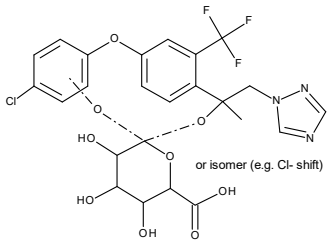
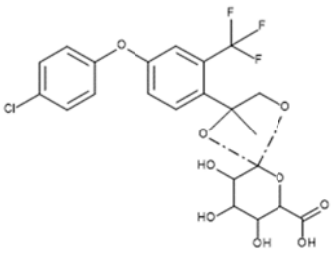
Code Number (Reg. Number)	Chemical Name	Molecular Structure	Rat	Hen & Goat	Wheat, Grape & Soya bean	Rotational Crops
M750F013	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl 6-O-hexopyranosylhexopyranoside				X	
M750F014	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl 6-O-[6-O-(carboxyacetyl)hexopyranosyl]hexopyranoside				X	
M750F015 (6011549)	2-chloro-4-{4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenoxy}phenol			X		
M750F016 (6010140)	2-chloro-5-{4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenoxy}phenol			X		
M750F017 (6010139)	5-chloro-2-{4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenoxy}phenol			X		
M750F018					X	
M750F019					X	

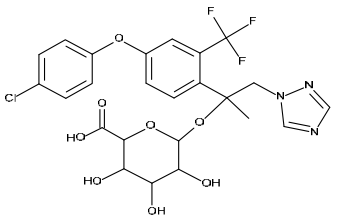
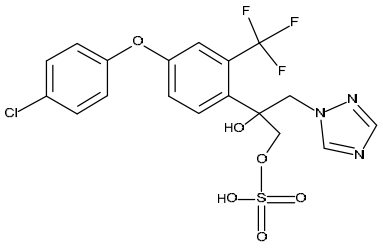
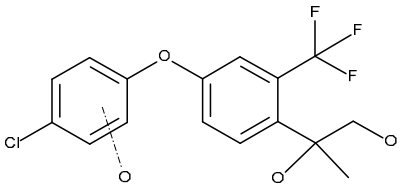
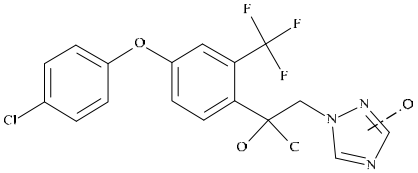
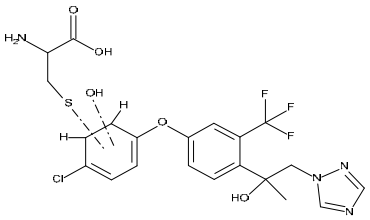
Mefentrifluconazole

Code Number (Reg. Number)	Chemical Name	Molecular Structure	Rat	Hen & Goat	Wheat, Grape & Soya bean	Rotational Crops
M750F020					X	
M750F021					X	
M750F022	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl] propane-1,2-diol			X		
M750F023	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl] -2-hydroxypropyl (9Z,11E)-octadeca-9,11-dienoate			X		
M750F024	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl] -2-hydroxypropyl (9Z)-octadec-9-enoate			X		
M750F025 (6056452)	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl] -2-hydroxypropyl hexadecanoate			X		
M750F026					X	

Code Number (Reg. Number)	Chemical Name	Molecular Structure	Rat	Hen & Goat	Wheat, Grape & Soya bean	Rotational Crops
M750F027					X	
M750F028	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1H-1,2,4-triazol-1-yl)propan-2-yl 6-O-pentofuranosylhexopyranoside				X	
M750F029 (270412) Triazole alanine (TA)	2-amino-3-(1*H*-1,2,4-triazol-1-yl)propionic acid				X	
M750F030 (137281) Triazole acetic acid (TAA)	(1*H*-1,2,4-triazol-1-yl)acetic acid				X	
M750F031 (5050862) Triazole lactic acid, Triazole hydroxypropionic acid (TLA)	2-hydroxy-3-(1*H*-1,2,4-triazol-1-yl)propanoic acid				X	
M750F034	gamma-glutamyl-S-(5-chloro-6-hydroxy-2-{4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenoxy}cyclohexa-2,4-dien-1-yl)cysteinylglycine			X		
M750F038	(2R)-2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxypropanoic acid			X		

Mefentrifluconazole

Code Number (Reg. Number)	Chemical Name	Molecular Structure	Rat	Hen & Goat	Wheat, Grape & Soya bean	Rotational Crops
M750F039	(2S)-2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-3-(1*H*-1,2,4-triazol-1-yl)propane-1,2-diol			X		
M750F040	(2S)-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl](hydroxy)acetic acid		X	X		
M750F041	3-chloro-6-{4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenoxy}cyclohexa-3,5-diene-1,2-diol			X		
M750F042	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxy-3-(1*H*-1,2,4-triazol-1-yl)propanoic acid			X		
M750F043	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxypropyl hydrogen sulfate		X	X		
M750F063				X		
M750F064				X		

Code Number (Reg. Number)	Chemical Name	Molecular Structure	Rat	Hen & Goat	Wheat, Grape & Soya bean	Rotational Crops
M750F068	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl hexopyranosiduronic acid			X		
M750F072	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxy-3-(1*H*-1,2,4-triazol-1-yl)propyl hydrogen sulfate			X		
M750F078		 or isomer (e.g. Cl- shift)		X		
M750F086				X		
M750F091				X		

Plant metabolism

Mefentrifluconazole metabolism data were submitted for grape, soya bean, and wheat.

Grape

Three grapevines (variety *Müller-Thurgau*), grown outdoors under natural climatic conditions, were treated three times foliarly with either a 1:1 mixture of [^{14}C-U-chlorophenyl:^{13}C-U-chlorophenyl]-mefentrifluconazole or a 2:1 mixture of [^{14}C-3(5)-triazole:^{13}C-3(5)-triazole]-mefentrifluconazole at a rate of 150 g ai/ha with re-treatment intervals of 10–11 days (Birk *et al*., 2015, BASF DocID 2015_1073822). Grape leaves and clusters were harvested early, 21 days after the first application (just prior to the third application) and at maturity (BBCH growth stage 89), 12 days following the last application. Grape clusters were manually separated into berries and stalks.

Total radioactive residues (TRR) were analysed following combustion by means of an oxidizer.

For the quantitation of radioactive residues in liquid samples a liquid scintillation counter (LSC) was used. TRRs were also calculated by summing extracted and unextracted residues. TRRs were highest in the leaves, followed by the stalks and berries (Table 3). Similar TRRs were observed between measured and calculated TRRs and for both labels.

Table 3 Total radioactive residues in grape matrices following application of chlorophenyl and triazole radiolabelled mefentrifluconazole (3 × 150 g ai/ha)

Matrix	TRR measured by combustion,mg eq/kg	TRR calculated from extracted and unextracted residues[1] , mg eq/kg
Chlorophenyl label		
Berries (DAT 12)	0.435	0.349 (first extraction)[2] 0.380 (second extraction)[2]
Leaves (DAT 12)	8.860	7.371
Stalks (DAT 12)	0.674	0.648
Triazole label		
Berries (DAT 12)	0.400	0.428 (first extraction)[2] 0.363 (second extraction)[2]
Leaves (DAT 12)	7.245	7.311
Stalks (DAT 12)	1.214	1.135

Notes:

[1] Calculated as the sum of the solvent extracted and unextracted radioactive residues.

[2] Grape berries were extracted twice (for each label) given that the first extraction produced limited amounts of sample material to conduct combustion analysis of the unextracted residues. The first extraction was used for the identification/characterization of the extracted and unextracted residues. The second extraction was used for the determination of the unextracted TRRs by combustion and to investigate storage stability.

Grape berries, leaves, and stalks were extracted three times with methanol and twice with water. After each extraction step, solid material was separated from the extract by centrifugation and filtration. The filtered supernatants (methanol extracts and water extracts) were individually radioassayed. The unextracted residues in each matrix (both labels) were dried, homogenized, and aliquots thereof were radioassayed. Solvent extracted radioactivity ranged between 87–90 percent TRR, 89–91 percent TRR, and 93–94 percent TRR in grape berries, leaves, and stalks, respectively (Table 4).

Table 4 Distribution and extractability of radioactive residues from grape matrices following application of chlorophenyl and triazole radiolabelled mefentrifluconazole (3 × 150 g ai/ha)

Matrix	Calculated TRR mg eq/kg	Methanol Extract mg eq/kg (% TRR)	Water Extract mg eq/kg (% TRR)	Total Solvent Extracted Residues mg eq/kg (% TRR)	Unextracted Residues mg eq/kg (% TRR)
Chlorophenyl label					
Berries (DALA 12)	0.349 (first extraction)	0.308 (88.3)	0.001 (0.3)	0.309 (88.5)	--[1]
	0.380 (second extraction)	0.331 (87.1)	0.001 (0.3)	0.332 (87.4)	0.048 (12.6)
Leaves (DALA 12)	7.371	6.456 (87.6)	0.102 (1.4)	6.558 (89.0)	0.813 (11.0)
Stalks (DALA 12)	0.648	0.606 (93.5)	0.004 (0.6)	0.610 (94.1)	0.038 (5.9)
Triazole label					
Berries (DALA 12)	0.428 (first extraction)	0.384 (89.7)	0.002 (0.5)	0.386 (90.2)	--[1]

Matrix	Calculated TRR mg eq/kg	Methanol Extract mg eq/kg (% TRR)	Water Extract mg eq/kg (% TRR)	Total Solvent Extracted Residues mg eq/kg (% TRR)	Unextracted Residues mg eq/kg (% TRR)
	0.363 (second extraction)	0.316 (87.1)	0.001 (0.3)	0.318 (87.6)	0.045 (12.4)
Leaves (DALA 12)	7.311	6.539 (89.4)	0.119 (1.6)	6.658 (91.1)	0.654 (8.9)
Stalks (DALA 12)	1.135	1.042 (91.8)	0.009 (0.8)	1.051 (92.5)	0.084 (7.4)

Notes:

[1] Not enough sample material for combustion analysis.

The methanol (all matrices) and water (leaves only) extracts for both labels were further purified using an SPE cartridge and analysed by HPLC-MS and/or by comparison of HPLC retention time against standards. For the identification of metabolites, the methanol extracts of the leaves (both labels) were partitioned with isohexane, dichloromethane, and ethyl acetate and the combined phases were fractionated using flash chromatography, and analysed by HPLC-MS and/or by comparison of HPLC retention time against standards.

The unextracted residues were solubilized by sequential treatment with 1 percent ammonia, maceroenzyme, α-amylase:β-amylase:amyloglucosidase (3:2:3), β-glucosidase:hesperidinase (1:2 for berries, 3:4 for leaves, 1:1 for stalks), laccase:tyrosinase (5:6 for berries, 5:8 for leaves, 1:1 for stalks), and protease. The radioactivity remaining after solubilisation were dried, homogenized, and aliquots thereof were radioassayed. As the results from the immature grape samples (treated 21 days after the first treatment) confirmed those from mature sampled grapes, the results from the immature grapes were not reported in the study.

The distribution of radioactivity in grape berries, leaves, and stalks is presented in Tables 5 and 6. Parent mefentrifluconazole (free) was the major identified residue in all matrices accounting for 64–70 percent TRR (0.224–0.301 mg eq/kg) in berries, 60–70 percent TRR (4.431–5.110 mg eq/kg) in leaves, and 86–92 percent TRR (0.556–1.039 mg eq/kg) in stalks. Metabolite M750F019 (free) was identified in berries, leaves, and stalks at 6.1–6.9 percent TRR (0.024–0.026 mg eq/kg), 14.5–21.1 percent TRR (1.058–1.554 mg eq/kg), and 2.3 percent TRR (0.015 mg eq/kg), respectively. Metabolite M750F026 (free) was identified as a minor metabolite in leaves (chlorophenyl-label only) at 1.3 percent TRR (0.097 mg eq/kg).

Table 5 Characterisation and identification of C-Label residues in grapes

Fraction	Berry [TRR = 0.349 mg eq/kg]		Leaves [TRR = 7.371 mg eq/kg]		Stalks [TRR = 0.648 mg eq/kg]	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total Solvent Extracted	88.5	0.309	89.0	6.558	94.1	0.610
Methanol	88.3	0.308	87.6	6.456	93.5	0.606
Mefentrifluconazole (free)	*64.2*	*0.224*	*60.0*	*4.422*	*85.8*	*0.556*
M750F019 (free)	*6.9*	*0.024*	*20.7*	*1.524*	*2.3*	*0.015*
M750F026 (free)	-	-	*1.3*	*0.097*	-	-
Unknowns	*5.2*	*0.018*	*2.6*	*0.193*	-	-
Water	0.3	0.001	1.4	0.102	0.6	0.004
Mefentrifluconazole (free)	-	-	*0.1*	*0.009*	*Not analysed*	

Fraction	Berry [TRR = 0.349 mg eq/kg]		Leaves [TRR = 7.371 mg eq/kg]		Stalks [TRR = 0.648 mg eq/kg]	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
M750F019 (free)	-	-	*0.4*	*0.03*		
Unknowns	-	-	*0.1*	*0.011*		
Post Extraction Solids	-	-[1]	11.0	0.813	5.9	0.038
Ammonia Hydrosylate	*0.9*	*0.003*	*1.5*	*0.108*	*0.8*	*0.005*
Maceroenzyme Hydrosylate	*0.3*	*0.001*	*0.7*	*0.051*	*0.5*	*0.003*
Amylase/Amyloglucosidase Hydrosylate	*0.3*	*0.001*	*0.4*	*0.028*	*0.2*	*0.001*
Glucosidase/Hesperidinase Hydrosylate	*0.3*	*0.001*	*0.3*	*0.023*	*0.2*	*0.001*
Tyrosinase/Laccase Hydrosylate	*0.3*	*0.001*	*0.5*	*0.038*	*0.3*	*0.002*
Protease Hydrosylate	*<0.3*	*<0.001*	*1.5*	*0.108*	*0.8*	*0.002*
Final Residues Remaining	*9.2*	*0.032*	*6.1*	*0.448*	*2.9*	*0.019*
Total Identified	71.1	0.248	82.5	6.082	88.1	0.571
Mefentrifluconazole (free)	*64.2*	*0.224*	*60.1*	*4.431*	*85.8*	*0.556*
M750F019 (free)	*6.9*	*0.024*	*21.1*	*1.554*	*2.3*	*0.015*
M750F026 (free)	-	-	*1.3*	*0.097*	-	-
Total Characterized[2]	7.7	0.027	6.6	0.487	2.2	0.014
Accountability[3]	88	0.307	95.2	7.09	93.2	0.604

Notes:

[1] Not enough sample material for combustion analysis.

[2] Total characterized from solvent extractable radioactive residues and hydrosylates from the unextracted residues.

[3] Accountability = Total Identified + Total Characterized + Final Residues Remaining after enzyme hydrolysis/TRR * 100.

Table 6 Characterisation and identification of T-Label residues in grapes

Fraction	Berry [TRR = 0.428 mg eq/kg]		Leaves [TRR = 7.312 mg eq/kg]		Stalks [TRR = 1.136 mg eq/kg]	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total Solvent Extracted	90.2	0.386	91.1	6.658	92.5	1.051
Methanol	89.7	0.384	89.4	6.539	91.7	1.042
Mefentrifluconazole (free)	*70.3*	*0.301*	*69.8*	*5.102*	*91.5*	*1.039*
M750F019 (free)	*6.1*	*0.026*	*14.5*	*1.058*	-	-
M750F026 (free)	-	-	-	-	-	-
Unknowns	*7.5*	*0.032*	*3.0*	*0.221*	-	-
Water	0.5	0.002	1.6	0.119	0.8	0.009
Mefentrifluconazole (free)	-	-	*0.1*	*0.008*	*Not analysed*	
M750F019 (free)	-	-	-	-		
Unknowns	-	-	*0.9*	*0.069*		
Post Extraction Solids	-	-[1]	8.9	0.654	7.4	0.084
Ammonia Hydrosylate	*1.2*	*0.005*	*1.1*	*0.079*	*1.1*	*0.013*
Maceroenzyme Hydrosylate	*0.5*	*0.002*	*0.5*	*0.04*	*0.6*	*0.007*
Amylase/Amyloglucosidase Hydrosylate	*0.2*	*0.001*	*0.4*	*0.028*	*0.4*	*0.004*
Glucosidase/Hesperidinase Hydrosylate	*0.2*	*0.001*	*0.2*	*0.018*	*0.4*	*0.004*

Fraction	Berry [TRR = 0.428 mg eq/kg]		Leaves [TRR = 7.312 mg eq/kg]		Stalks [TRR = 1.136 mg eq/kg]	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Tyrosinase/Laccase Hydrosylate	*0.2*	*0.001*	*0.4*	*0.027*	*0.4*	*0.004*
Protease Hydrosylate	*<0.2*	*<0.001*	*0.4*	*0.028*	*0.3*	*0.003*
Final Residues Remaining	*7.5*	*0.032*	*5.1*	*0.375*	*3.3*	*0.038*
Total Identified	76.4	0.327	84.4	6.168	91.5	1.039
Mefentrifluconazole (free)	*70.3*	*0.301*	*69.9*	*5.110*	*91.5*	*1.039*
M750F019 (free)	*6.1*	*0.026*	*14.5*	*1.058*	-	-
M750F026 (free)	-	-	-	-	-	-
Total Characterized[2]	2.6	<0.011	3.0	0.22	3.9	0.044
Accountability[3]	86.3	0.37	92.5	6.763	98.7	1.121

Notes:

[1] Not enough sample material for combustion analysis.

[2] Total characterized from solvent extractable radioactive residues and hydrosylates from the unextracted residues.

[3] Accountability = Total Identified + Total Characterized + Final Residues Remaining after enzyme hydrolysis/TRR * 100

The application formulation and methanol extracts from grape berries and leaves were further analysed by HPLC to determine the enantiomer ratios of mefentrifluconazole. The relative amounts of the two enantiomers were approximately 1:1 in the application formulations and each matrix (Table 7).

Table 7 Enantiomer ratios from the treatment of grapes with chlorophenyl and triazole radiolabelled mefentrifluconazole

Matrix	Enantiomer 1 (% ROI)	Enantiomer 2 (% ROI)
Chlorophenyl label		
Application Formulation	48.9	51.1
Berries	46.7	53.3
Triazole label		
Application Formulation	47.4	52.6
Leaves	48.1	52.0

Notes:
ROI = region of interest (on the chromatogram).

All samples were extracted 55–57 days after sampling, and were then analysed 14–27 days after extraction. Total time between sampling to analysis was 70–84 days. The stability of the radioactive residues in grape matrices (berries and leaves stored at ≤-18 °C) and extracts (berries and leaves, storage in fridge) was investigated using homogenates and extracts from both labels. Comparison of the chromatographic profiles between matrices extracted 55–57 days after sampling and re-extracted 203–252 days after sampling showed no significant changes to the composition and amounts of radioactivity for each matrix. Similarly, the chromatographic profiles between extracts analysed after 14–27 days of storage and re-analysed after 224–231 days of storage showed no significant changes to the composition and amounts of radioactivity for each extract.

Soya bean

Ten containers of soya bean plants (variety *Sultana*, 13 plants/container), grown in climatic chambers (phytotrons, simulating natural climatic conditions), were treated three times foliarly with either a 1:1 mixture of [^{14}C-U-chlorophenyl:^{13}C-U-chlorophenyl]-mefentrifluconazole or a 2:1 mixture of [^{14}C-3(5)-

triazole:^{13}C-3(5)-triazole]-mefentrifluconazole at a rate of 125 g ai/ha with re-treatment intervals of 17-19 days (Thiaener *et al*., 2015, BASF DocID 2014_1224012). Soya bean forage was harvested 19 days after the first application (just before the second application; BBCH growth stage of 71–72). At harvest, 47–48 days after the final application at BBCH growth stage 89, the leaves were removed from the plants and collected (matrix: rest of plant). The mature pods were removed and manually opened in order to separate seeds from hulls. In addition green pods were also harvested and the remaining stems were cut off and combined with the collected leaves (matrix: rest of plant).

TRRs were analysed following combustion by means of an oxidizer. For the quantitation of radioactive residues in liquid samples a liquid scintillation counter (LSC) was used. TRR were also calculated by summing extracted and unextracted residues. TRR levels found were highest in the rest of plant, followed by the green pods, forage, hull, and seed (Table 8). Similar TRR levels were observed between measured and calculated TRR and for both labels.

Table 8 Total radioactive residues in soya beans matrices following application of chlorophenyl and triazole radiolabelled mefentrifluconazole (3 × 125 g ai/ha)

Matrix	TRR by combustion, mg eq/kg	TRR calculated from extracted and unextracted residues, mg eq/kg
Chlorophenyl label		
Forage (DALA 19[1])	6.516	6.575
Seed (DALA 47)	0.109	0.129
Hull (DALA 47)	3.735	3.838
Rest of Plant (DALA 47)	16.016	16.459
Green Pods (DALA 47)	8.857	8.721
Triazole label		
Forage (DAT 19[1])	4.416	4.609
Seed (DAT 48)	2.592	3.063
Hull (DAT 48)	3.890	4.122
Rest of Plant (DAT 48)	19.934	19.264
Green Pods (DAT 48)	16.005	16.006

Notes:

[1] Days after the first application. Forage was harvested just before the second application.

Forage, hull, and rest of plant samples were extracted three times with methanol and twice with water. After each extraction step, solid material was separated from the extract by centrifugation and filtration. The filtered supernatants (methanol extracts and water extracts) were each pooled; the methanol extracts were purified by SPE fractionation. Soya bean seed and green pod samples were extracted three times with acetonitrile:isohexane (1:1) and twice with water. Solid material was separated from the extract by centrifugation and filtration. The residue after solvent extraction was dried, homogenized, and aliquots thereof were radioassayed. The filtered supernatants (acetonitrile, isohexane, and water extracts) were each pooled.

Solvent extracted radioactivity ranged between 91–93 percent, 69–74 percent, and 87–88 percent in soya bean forage, hull, and rest of plant (Table 9) and between 56–76 percent and 78–83 percent in soya bean seed and green pods (Table 10).

Table 9 Distribution of radioactive residues from soya bean forage, hull, and rest of plant following of chlorophenyl and triazole radiolabelled mefentrifluconazole (3 × 125 g ai/ha)

Matrix	Calculated TRR, mg eq/kg	Methanol Extract	Water Extract	Total Solvent Extracted Residues	Unextracted Residues
		mg eq/kg (% TRR)	mg eq/kg (% TRR)	mg eq/kg (% TRR)	mg eq/kg (% TRR)
Chlorophenyl label					
Forage (DALA 19[1])	6.575	5.898 (89.7)	0.090 (1.4)	5.988 (91.1)	0.587 (8.9)
Hull (DALA 47)	3.838	2.558 (66.6)	0.078 (2.0)	2.636 (68.7)	1.201 (31.3)
Rest of Plant (DALA 47)	16.459	13.732 (83.4)	0.601 (3.7)	14.333 (87.1)	2.126 (12.9)
Triazole label					
Forage (DALA 19[1])	4.609	4.249 (92.2)	0.054 (1.2)	4.303 (93.4)	0.307 (6.7)
Hull (DALA 48)	4.122	2.903 (70.4)	0.156 (3.8)	3.059 (74.2)	1.063 (25.8)
Rest of Plant (DALA 48)	19.264	16.165(83.9)	0.757 (3.9)	16.922 (87.8)	2.342 (12.2)

Notes:

[1] Days after the first application. Forage was harvested just before the second application.

Table 10 Distribution of radioactive residues from soya bean seed and green pods following application of chlorophenyl and triazole radiolabelled mefentrifluconazole (3 × 125 g ai/ha)

Matrix	Calculated TRR, mg eq/kg	Acetonitrile Phase	Isohexane Phase	Water Extract	Total Solvent Extracted Residues	Unextracted Residues
		mg eq/kg (% TRR)	mg eq/kg (% TRR)	mg eq/kg (% TRR)	mg eq/kg (% TRR)	mg eq/kg (% TRR)
Chlorophenyl label						
Seed (DALA 47)	0.129	0.005 (3.9)	0.022 (17.1)	0.045 (34.9)	0.072 (55.8)	0.056 (43.4)
Green Pods (DALA 47)	8.721	6.503 (74.6)	0.108 (1.2)	0.660 (7.6)	7.271 (83.4)	1.451 (16.6)
Triazole label						
Seed (DALA 47)	3.063	0.019 (0.6)	0.025 (0.8)	2.272 (74.2)	2.316 (75.6)	0.747 (24.4)
Green Pods (DALA 47)	16.006	10.782 (67.4)	0.141 (0.9)	1.564 (9.8)	12.487 (78.0)	3.518 (22.0)

The forage, hull, and rest of plant unextracted residues were solubilized by sequential treatment with maceroenzyme:cellulase (10:1 for forage and rest of plant, 5:1 for hull), β-glucosidase:hesperidinase (1:1), α-amylase:β-amylase:amyloglucosidase (1:1:1), laccase:tyrosinase (5:16), and protease. The seed unextracted residues were solubilized by sequential treatment with maceroenzyme:cellulase (10:1 or 15:1), β-glucosidase:hesperidinase (1:1), α-amylase:β-amylase:amyloglucosidase (1:1:1), and protease. The maceroenzyme:cellulase hydrosylates for forage, rest of plant, and seed samples were purified using SPE fractionation. The identification and characterization of the components in the extracts was carried out by HPLC-MS, co-chromatography and comparison of retention times.

Sequential enzyme treatment of the unextracted residues released an additional 3.5-33 percent of the TRR in each matrix except for green pods where the post-extraction residues were not further hydrolysed (Tables 11 to 14).

Parent mefentrifluconazole (free and conjugated) was a major residue in forage, hulls, and rest of plant, accounting for 79–80 percent TRR (3.648–5.257 mg eq/kg) in forage, 79–83 percent TRR (3.179–3.257 mg eq/kg) in hulls, and 60–71 percent TRR (9.848–13.698 mg eq/kg) in rest of plant. Mefentrifluconazole (free) was a major residue in green pods 69 percent TRR (5.978 mg eq/kg). Mefentrifluconazole (free) was a minor residue in seed accounting for only 0.4–3.9 percent TRR (0.005–0.013 mg eq/kg). Triazole alanine (free and conjugated) was a major metabolite found only in soya bean

seeds accounting for 48 percent TRR (1.461 mg eq/kg). The minor metabolites, M70F018/M750F020 and M750F012, free and conjugated, were identified in forage, hull, rest of plant, and green pods at levels of 0.03–4.5 percent TRR (0.001–0.748 mg eq/kg) and 0.4–5.9 percent TRR (0.014–0.971 mg eq/kg), respectively. Triazole lactic acid (free) and 1,2,4-triazole (free) were identified as minor metabolites in seed at levels of 1.3 percent TRR (0.04 mg eq/kg) and 0.3 percent TRR (0.008 mg eq/kg), respectively.

Table 11 Characterisation and identification of C-Label residues in soya bean forage, hull, and rest of plant

Fraction	Forage [TRR = 6.575 mg eq/kg]		Hull [TRR = 3.838 mg eq/kg]		Rest of plant [TRR = 16.459 mg eq/kg]	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total Solvent Extracted	91.1	5.988	68.7	2.636	74.4	14.333
Methanol	89.7	5.898	66.6	2.558	83.4	13.732
Mefentrifluconazole (free)	*76.7*	*5.042*	*66.7*	*2.559*	*52.9*	*8.706*
M750F012 (free)	*3.8*	*0.247*	-	-	*5.1*	*0.834*
M750F018/M750F020 (free)	*1.9*	*0.123*	-	-	*4.3*	*0.705*
Unknowns	*1.0*	*0.068*	-	-	*12.8*	*2.109*
Water	1.4	0.09	2.0	0.078	3.7	0.601
Mefentrifluconazole (free)	*0.5*	*0.036*	*0.3*	*0.012*	*1.1*	*0.18*
M750F012 (free)	*0.1*	*0.006*	*0.4*	*0.014*	*0.4*	*0.062*
M750F018/M750F020 (free)	-	-	*0.03*	*0.001*	*0.3*	*0.043*
Unknowns	*0.7*	*0.049*	*1.3*	*0.051*	*1.9*	*0.315*
Post Extraction Solids	8.9	0.587	31.3	1.201	12.9	2.126
Maceroenzyme/Cellulase Hydrosylate	1.1	0.075	9.0	0.346	2.1	0.352
Mefentrifluconazole (conjugated)	*0.5*	*0.033*	*6.0*	*0.232*	*0.7*	*0.117*
M750F012 (conjugated)	*0.1*	*0.005*	-	-	*0.2*	*0.031*
Unknowns	*0.4*	*0.024*	*3.0*	*0.114*	*0.8*	*0.126*
Glucosidase/Hesperidinase Hydrosylate	0.5	0.032	3.6	0.137	2.5	0.407
Mefentrifluconazole (conjugated)	*0.3*	*0.021*	*3.6*	*0.137*	*1.8*	*0.299*
M750F012 (conjugated)	*<0.1*	*0.001*	-	-	*0.1*	*0.024*
M750F018/M750F020 (conjugated)	*<0.1*	*<0.001*	-	-	-	-
Unknowns	*0.2*	*0.01*	-	-	*0.5*	*0.084*
Amylase/Amyloglucosidase Hydrosylate	0.4	0.026	2.8	0.106	1.0	0.17
Mefentrifluconazole (conjugated)	*0.4*	*0.026*	*2.8*	*0.106*	*0.9*	*0.15*
Unknowns	-	-	-	-	*0.1*	*0.02*
Tyrosinase/Laccase Hydrosylate	0.3	0.023	1.5	0.059	0.7	0.113
Mefentrifluconazole (conjugated)	*0.3*	*0.023*	*1.5*	*0.059*	*0.6*	*0.106*
Unknowns	-	-	-	-	*0.04*	*0.007*
Protease Hydrosylate	1.2	0.076	1.9	0.074	2.0	0.332
Mefentrifluconazole (conjugated)	*1.2*	*0.076*	*1.9*	*0.074*	*1.8*	*0.29*
M750F012 (conjugated)	-	-	-	-	*0.1*	*0.02*
Unknowns	-	-	-	-	0.1	*0.022*
Final Residues Remaining	4.8	0.316	10.9	0.420	5.8	0.954
Total Identified	85.8	5.64	83.2	3.194	70.3	11.567
Mefentrifluconazole (free and conjugated)	*80.0*	*5.257*	*82.8*	*3.179*	*59.8*	*9.848*
M750F012	*3.9*	*0.259 (F) (free + conj)*	*0.03*	*0.014 (F+C)*	*5.9*	*0.971 (F)*
M750F018/M750F020	*1.9*	*<0.124 (F)*	*0.4*	*0.001 (F+C)*	*4.5*	*0.748 (F+C)*
Total Characterized[1]	2.3	0.151	4.3	0.165	16.3	2.683
Accountability[2]	90.9	6.107	98.5	3.779	92.4	15.204

Notes:

F= free, C = conjugated.

[1] Total characterized from solvent extractable radioactive residues and hydrosylates from the unextracted residues.

[2] Accountability = Total Identified + Total Characterized + Final Residues Remaining after enzyme hydrolysis/TRR * 100.

Table 12 Characterisation and identification of C-Label residues in soya bean seed and green pods

Fraction	Seed [TRR = 0.129 mg eq/kg]		Green pods [TRR = 8.721 mg eq/kg]	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Total Solvent Extracted	55.8	0.072	83.4	7.271
Acetonitrile	3.9	0.005	74.6	6.503
Mefentrifluconazole (free)	*3.9*	*0.005*	*68.5*	*5.978*
M750F012 (free)	-	-	*2.2*	*0.188*
M750F018/M750F020 (free)	-	-	*3.9*	*0.338*
Isohexane	17.1	0.022	1.2	0.108
Water	34.9	0.045	7.6	0.66
Supernatant from acetone precipitate	10.9	0.014	*No hydrolysis*	
Acetone precipitate	23.3	0.030		
Protease hydrolysate	19.4	0.025		
Unknowns	*17.1*	*0.022*		
Protease residue	2.3	0.003		
Post Extraction Solids	43.4	0.056	16.6	1.451
Maceroenzyme/Cellulase Hydrosylate	15.5	0.02	*No hydrolysis*	
Unknowns	*14.7*	*0.019*		
Glucosidase/Hesperidinase Hydrosylate	4.7	0.006		
Unknowns	*4.7*	*0.006*		
Amylase/Amyloglucosidase Hydrosylate	3.9	0.005		
Protease Hydrosylate	8.5	0.011		
Final Residues Remaining	5.4	0.007		
Total Identified	3.9	0.005	74.6	6.504
Mefentrifluconazole (free)	*3.9*	*0.005*	*68.5*	*5.978*
M750F012 (free)	-	-	*2.2*	*0.188*
M750F018/M750F020 (free)	-	-	*3.9*	*0.338*
Total Characterized[1]	79.1	0.102	3.9	0.338
Accountability[2]	88.4	0.114	100	8.723

Notes:

[1] Total characterized from solvent extractable radioactive residues and hydrosylates from the unextracted residues.

[2] Accountability = Total Identified + Total Characterized + Final Residues Remaining after enzyme hydrolysis (or for green pods, total unextracted by solvent)/TRR * 100.

Table 13 Characterisation and identification of T-Label residues in soya bean forage, hull, and rest of plant

Fraction	Forage [TRR =4.609 mg eq/kg]		Hull [TRR = 4.122 mg eq/kg]		Rest of plant [TRR = 19.264 mg eq/kg]	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Total Solvent Extracted	93.4	4.303	74.2	3.059	87.8	16.922
Methanol	92.2	4.249	70.4	2.903	83.9	16.165
Mefentrifluconazole (free)	*76.2*	*3.513*	*69.9*	*2.882*	*75.6*	*12.448*
M750F012 (free)	*3.4*	*0.155*	-	-	*3.0*	*0.497*
M750F018/M750F020 (free)	*2.0*	*0.091*	-	-	*4.0*	*0.653*
Unknowns	*1.7*	*0.08*	-	-	*7.4*	*1.427*
Water	1.2	0.054	3.8	0.156	3.9	0.757
Mefentrifluconazole (free)	*0.4*	*0.017*	*0.2*	*0.010*	*1.6*	*0.258*

Fraction	Forage [TRR =4.609 mg eq/kg]		Hull [TRR = 4.122 mg eq/kg]		Rest of plant [TRR = 19.264 mg eq/kg]	
	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
M750F012 (free)	*0.1*	*0.006*	-	-	*0.4*	*0.064*
M750F018/M750F020 (free)	*0.1*	*0.005*	-	-	*0.5*	*0.082*
Unknowns	0.5	*0.025*	*3.5*	*0.145*	*1.8*	*0.353*
Post Extraction Solids	6.7	0.307	25.8	1.063	12.2	2.342
Maceroenzyme/Cellulase Hydrosylate	0.7	0.033	11.0	0.452	1.8	0.34
Mefentrifluconazole (conjugated)	*0.5*	*0.024*	*2.3*	*0.093*	*0.8*	*0.162*
M750F012 (conjugated)	-	-	*1.3*	*0.052*	*0.1*	*0.017*
Unknowns	*0.07*	*0.003*	*7.4*	*0.307*	*0.6*	*0.11*
Glucosidase/Hesperidinase Hydrosylate	0.5	0.023	2.6	0.109	1.4	0.264
Mefentrifluconazole (conjugated)	*0.4*	*0.020*	*2.2*	*0.09*	*0.6*	*0.124*
M750F012 (conjugated)	*0.02*	*0.001*	-	-	*0.10*	*0.019*
Unknowns	*0.07*	*0.003*	*0.5*	*0.019*	*0.3*	*0.062*
Amylase/Amyloglucosidase Hydrosylate	0.3	0.016	2.6	0.108	0.8	0.148
Mefentrifluconazole (conjugated)	*0.3*	*0.016*	*2.6*	*0.108*	*0.6*	*0.124*
Unknowns	-	-	-	-	*0.1*	*0.025*
Tyrosinase/Laccase Hydrosylate	*0.3*	0.016	1.0	0.042	0.7	0.141
Mefentrifluconazole (conjugated)	*0.3*	*0.014*	*1.0*	*0.042*	*0.5*	*0.088*
Unknowns	-	-	-	-	*0.3*	*0.054*
Protease Hydrosylate	1.0	0.044	0.8	0.032	2.3	0.434
Mefentrifluconazole (conjugated)	*1.0*	*0.044*	*0.8*	*0.032*	*2.3*	*0.434*
Final Residues Remaining	3.5	0.16	7.5	0.309	5.7	1.096
Total Identified	84.7	3.906	80.3	3.309	78.0	15.030
Mefentrifluconazole (free and conjugated)	*79.1*	*3.648*	79.0	*3.257*	*71.1*	*13.698*
M750F012 (free and conjugated)	*3.5*	*0.162*	1.3	*0.052*	*3.1*	*0.597*
M750F018/M750F020 (free)	*2.1*	*0.096*	-	-	3.8	*0.735*
Total Characterized[1]	2.4	0.111	11.4	0.471	10.5	2.031
Accountability[2]	90.6	4.177	99.2	4.089	94.3	18.157

Notes:

[1] Total characterized from solvent extractable radioactive residues and hydrosylates from the unextracted residues.

[2] Accountability = Total Identified + Total Characterized + Final Residues Remaining after enzyme hydrolysis/TRR * 100.

Table 14 Characterisation and identification of T-Label residues in soya bean seed and green pods

Fraction	Seed TRR = 3.063 mg eq/kg		Green pods TRR = 16.006 mg eq/kg	
	% TRR	m eq/kg	% TRR	mg eq/kg
Total Solvent Extracted	75.6	2.316	78.0	12.487
Acetonitrile	0.6	0.019	67.4	10.782
Mefentrifluconazole (free)	*0.4*	*0.013*	*Not analysed*	
Unknowns	*0.2*	*0.006*		
Isohexane	0.8	0.025	0.9	0.141
Water	74.2	2.272	9.8	1.564
Supernatant from acetone precipitate	9.0	0.277	*Not analysed*	
Triazole Alanine (free)	*3.1*	*0.096*		
1,2,4-triazole (free)	*0.3*	*0.008*		
Triazole Lactic Acid (free)	*1.3*	*0.04*		
Unknowns	*4.5*	*0.137*		
Acetone precipitate	59.1	1.811		
Protease hydrolysate	57.3	1.756		
Triazole Alanine (conjugated)	*27.9*	*0.855*		
Unknowns	*29.4*	*0.901*		

Fraction	Seed TRR = 3.063 mg eq/kg		Green pods TRR = 16.006 mg eq/kg	
	% TRR	m eq/kg	% TRR	mg eq/kg
Protease residue	0.7	0.02		
Post Extraction Solids	24.4	0.747	22.0	3.518
Maceroenzyme/Cellulase Hydrosylate	23.0	0.706	*No hydrolysis*	
Triazole Alanine (conjugated)	*16.7*	*0.51*		
Unknowns	*4.4*	*0.136*		
Glucosidase/Hesperidinase Hydrosylate	3.0	0.093		
Unknowns	*3.0*	*0.093*		
Amylase/Amyloglucosidase Hydrosylate	0.5	0.016		
Unknowns	*0.5*	*0.016*		
Protease Hydrosylate	0.3	0.009		
Final Residues Remaining	0.8	0.025		
Total Identified	49.7	1.522	*Not analysed*	
Mefentrifluconazole (free)	*0.4*	*0.013*		
Triazole Alanine (free and conjugated)	*47.7*	*1.461*		
1,2,4-triazole (free)	*0.3*	*0.008*		
Triazole Lactic Acid (free)	*1.3*	*0.04*		
Total Characterized[1]	42.9	1.314	78.0	12.487
Accountability[2]	93.4	2.861	100	16.005

Notes:

[1] Total characterized from solvent extractable radioactive residues and hydrosylates from the unextracted residues.

[2] Accountability = Total Identified + Total Characterized + Final Residues Remaining after enzyme hydrolysis/TRR * 100 (for green pods Accountability = Total Characterized + total unextracted by solvent/TRR * 100).

The application formulation and methanol extracts from soya bean forage, hull, and rest of plant were further analysed by HPLC to determine the enantiomer ratios of mefentrifluconazole. Chiral analysis was not conducted for seeds due to the low amounts of mefentrifluconazole found. The relative amounts of the two enantiomers were approximately 1:1 in the application formulations and in each matrix tested (Table 15).

Table 15 Enantiomer ratios from the treatment of soya beans with chlorophenyl and triazole radiolabelled mefentrifluconazole

Matrix	Enantiomer 1 (% ROI)	Enantiomer 2 (% ROI)
Chlorophenyl label		
Application Formulation	50.5	49.5
Forage	45.5	54.5
Hull	45.6	54.4
Rest of Plant	46.6	53.4
Triazole label		
Forage	51.3	48.7
Hull	48.1	51.9
Rest of Plant	42.7	57.3

Notes:

ROI = region of interest (on the chromatogram).

All samples were extracted 21–86 days after sampling, and were then analysed 17–85 days after extraction. Total time between sampling to analysis was 49–114 days. The stability of the radioactive residues in soya bean matrices (forage and rest of plant stored at ≤-18 °C) and extracts (soya bean forage, rest of plant, and seed, storage in fridge) was investigated using homogenates and extracts from both

labels. Comparison of the chromatographic profiles between matrices extracted 21–86 days after sampling and re-extracted 350–374 days after sampling showed no significant changes to the composition and amounts of radioactivity for each matrix. Similarly, the chromatographic profiles between extracts analysed after 17–85 days of storage and re-analysed after 294–330 days of storage showed no significant changes to the composition and amounts of radioactivity for each extract.

Wheat

Twenty containers of spring wheat (variety *Thassos*, 10 containers per label), grown in phytotrons simulating natural climatic conditions, were treated twice foliarly with either a 1:1 mixture of [^{14}C-U-chlorophenyl:^{13}C-U-chlorophenyl]-mefentrifluconazole or a 2:1 mixture of [^{14}C-3(5)-triazole:^{13}C-3(5)-triazole]-mefentrifluconazole at a rate of 150 g ai/ha with a re-treatment interval of 21 days(Thiaener *et al.*, 2015, BASF DocID 2015_1001872). Wheat forage was harvested 15 days after the first application (just before the second application; BBCH growth stage 61). Straw and grain were harvested 35 days after the final application at BBCH 89.

Total radioactive residues (TRR) were analysed following combustion by means of an oxidizer. For the quantitation of radioactive residues in liquid samples a liquid scintillation counter (LSC) was used. TRR were also calculated by summing extracted and unextracted residues.

TRR levels found were highest in straw, followed by forage and grain (Table 16). Similar TRR levels were observed between measured and calculated TRR and for both labels.

Table 16 Total radioactive residues in wheat matrices following application of chlorophenyl and triazole radiolabelled mefentrifluconazole (2 × 150 g ai/ha)

Matrix	TRR by combustion in mg eq/kg	TRR calculated from extracted and unextracted residues[1], mg eq/kg
Chlorophenyl label		
Forage (DALA 15[2])	2.472	2.378
Grain (DALA 35)	0.065	0.062
Straw (DAT 35)	24.305	24.380
Triazole label		
Forage (DALA 15[2])	2.634	2.310
Grain (DALA 35)	0.619	0.620
Straw (DALA 35)	14.339	13.984

Notes:

[1] Calculated as the sum of the solvent extracted and unextracted radioactive residues.

[2.] Days after the first application. Forage was harvested just before the second application.

Forage and straw samples were extracted three times with methanol and twice with water. After each extraction step, solid material was separated from the extract by centrifugation and filtration. The filtered supernatants (methanol extracts and water extracts) were each pooled.

Wheat grain was extracted three times with acetonitrile:isohexane (1:1) and two times with water. Solid material was separated from the extract by centrifugation and filtration. The filtered supernatants (acetonitrile, isohexane, and water extracts) were each pooled. The residue after solvent extraction was dried, homogenized, and aliquots thereof were radioassayed.

Solvent extracted radioactivity ranged between 95–96 percent, 44–78 percent, and 83–86 percent in wheat forage, grain, and straw, respectively (Table 17).

Table 17 Distribution and extractability of radioactive residues from wheat forage and straw following

application of chlorophenyl and triazole radiolabelled mefentrifluconazole (2 × 150 g ai/ha)

Matrix	Calculated TRR, mg eq/kg	Methanol Extract	Water Extract	Total Solvent Extracted Residues	Unextracted Residues
		mg eq/kg (% TRR)	mg eq/kg (% TRR)	mg eq/kg (% TRR)	mg eq/kg (% TRR)
Chlorophenyl label					
Forage (DALA 15[1])	2.378	2.236 (94.0)	0.029 (1.2)	2.265 (95.2)	0.114 (4.8)
Straw (DALA 35)	24.380	17.986 (73.8)	2.255 (9.2)	20.241 (83.0)	4.139 (17.0)
Triazole label					
Forage (DALA 15[1])	2.310	2.188 (94.7)	0.030 (1.3)	2.218 (96.0)	0.092 (4.0)
Straw (DALA 35)	13.984	10.869 (77.7)	1.213 (8.7)	12.082 (86.4)	1.901 (13.6)

Notes:

[1] Days after the first application. Forage was harvested just before the second application.

Table 18 Distribution and extractability of radioactive residues from wheat grain following application of chlorophenyl and triazole radiolabelled mefentrifluconazole (2 × 150 g ai/ha)

Matrix	Calculated TRR, mg eq/kg	Acetonitrile Phase	Isohexane Phase	Water Extract	Total Solvent Extracted Residues	Unextracted Residues
		mg eq/kg (% TRR)	mg eq/kg (% TRR)	mg eq/kg (% TRR)	mg eq/kg (% TRR)	mg eq/kg (% TRR)
Chlorophenyl label						
Grain (DALA 35)	0.062	0.011 (17.7)	0.001 (1.6)	0.015 (24.2)	0.027 (43.5)	0.035 (56.5)
Triazole label						
Grain (DALA 35)	0.620	0.022 (3.5)	0.001 (0.2)	0.460 (74.2)	0.483 (77.9)	0.137 (22.1)

The water extract of the grain samples was adjusted to pH 4 with formic acid and precipitated with acetone. The acetone precipitate was sequentially treated with protease, α-amylase:β-amylase:amyloglucosidase (2:1:1), 1 percent ammonia, and again with α-amylase:β-amylase:amyloglucosidase (2:1:1).

The forage and straw unextracted residues were solubilized by sequential treatment with 1 percent ammonia, maceroenzyme, α-amylase:β-amylase:amyloglucosidase (1:1:1), β-glucosidase:hesperidinase (1:2), and tyrosinase:laccase (5:1).

The identification and characterization of the components in the extracts and hydrolysates was carried out by HPLC-MS, co-chromatography and comparison of retention times.

The distribution of radioactivity in wheat forage, grain, and straw is presented in Tables 19 and Table 20. Parent mefentrifluconazole was a major identified residue in forage and straw accounting for 84–89 percent TRR (2.007–2.063 mg eq/kg, free) in forage and 59–69 percent TRR (9.574–14.298 mg eq/kg, free and conjugated) in straw. Mefentrifluconazole was not found in grain. Triazole alanine (free and conjugated) and triazole acetic acid (free and conjugated) were major metabolites found only in wheat grain accounting for 46 percent TRR (0.282 mg eq/kg) and 22 percent (0.133 mg eq/kg), respectively. As minor metabolites, M750F009, M750F012, M750F019, and M750F018/M750F020 were identified in both forage (all metabolites were in free form) and straw (free and conjugated metabolites) at levels of 1.1–1.3 percent TRR (0.025–0.178 mg eq/kg), 0.1–2.1 percent TRR (0.008–0.158 mg eq/kg), 0.1–5.8 percent TRR (0.003–1.406 mg eq/kg), and 0.1–6.9 percent TRR (0.037–1.682 mg eq/kg), respectively. Minor metabolites M750F010 (free), M750F018 (free and conjugated), and M750F012/M750F021 (free and conjugated) were identified in only straw at levels of 1.3 percent TRR (0.178 mg eq/kg), 2.9–5.5 percent TRR (0.717–0.766 mg eq/kg), and 3.4–4.9 percent TRR (0.47–

1.184 mg eq/kg), respectively. 1,2,4–Triazole (free) was identified as minor metabolite in grain at level of 1.0 percent TRR (0.006 mg eq/kg). Sequential enzyme treatment released an additional 1.9–42 percent of the TRR in each matrix

Table 19 Characterisation and identification of C-Label and T-Label residues in wheat forage, and straw

Fraction	C-Label				T-Label			
	Forage TRR=2.378 mg eq/kg		Straw TRR=24.380 mg eq/kg		Forage TRR=2.310 mg eq/kg		Straw TRR=13.984 mg eq/kg	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total Solvent Extracted	95.2	2.265	83.0	20.241	96.0	2.218	86.4	12.082
Methanol	94.0	2.236	73.8	17.986	94.7	2.188	77.7	10.869
Mefentrifluconazole (F)	*83.5*	*1.985*	*55.3*	*13.484*	*88.9*	*2.054*	*66.4*	*9.285*
M750F009 (F)	-	-	-	-	*1.1*	*0.025*	*0.8*	*0.117*
M750F010 (F)	-	-	-	-	-	-	*1.0*	*0.133*
M750F012 (F)	*2.1*	*0.049*	*0.6*	*0.158*	-	-	-	-
M750F018 (F)	-	-	*0.6*	*0.146*	-	-	*1.0*	*0.133*
M750F019 (F)	-	-	*2.8*	*0.688*	-	-	*1.0*	*0.145*
M750F018/M750F020 (F)	*2.1*	*0.037*	*4.3*	*1.057*	*1.6*	*0.037*	*3.4*	*0.473*
M750F012/M750F021 (F)	-	-	*3.3*	*0.807*	-	-	*2.5*	*0.345*
Unknowns	*2.9*	*0.07*	*1.3*	*0.322*	-	-	*0.5*	*0.070*
Water	1.2	0.029	9.2	2.255	1.3	0.03	8.7	1.213
Mefentrifluconazole (F)	*0.9*	*0.022*	*1.3*	*0.314*	*0.4*	*0.009*	*0.8*	*0.116*
M750F010 (F)	-	-	-	-	-	-	*0.3*	*0.047*
M750F012 (F)	-	-	-	-	-	-	-	-
M750F018 (F)	-	-	*1.9*	*0.471*	-	-	*2.3*	*0.328*
M750F019 (F)	*0.1*	*0.003*	*1.6*	*0.388*	-	-	*2.8*	*0.386*
M750F018/M750F020 (F)	-	-	*2.6*	*0.625*	-	-	*0.9*	*0.13*
M750F012/M750F021 (F)	-	-	*0.8*	*0.205*	-	-	*0.4*	*0.053*
Unknowns	-	-	*0.6*	*0.145*	*0.8*	*0.018*	*0.6*	*0.081*
Post Extraction Solids	4.8	0.114	17.0	4.139	4.0	0.092	13.6	1.901
Ammonia hydrosylate	1.0	0.023	4.2	1.026	0.7	0.017	3.6	0.506
Mefentrifluconazole (C)	-	-	*0.1*	*0.027*	-	-	*0.1*	*0.019*
M750F009 (C)	-	-	-	-	-	-	*0.4*	*0.061*
M750F018 (C)	-	-	*0.4*	*0.087*	-	-	*0.9*	*0.122*
M750F019 (C)	-	-	*1.2*	*0.299*	-	-	*1.2*	*0.164*
M750F012/M750F021 (C)	-	-	*0.1*	*0.073*	-	-	*0.2*	*0.025*
Unknowns	-	-	*2.2*	*0.540*	*0.7*	*0.017*	*0.8*	*0.115*
M/C Hydrosylate[1]	0.5	0.012	1.7	0.421	0.4	0.01	1.3	0.184
Mefentrifluconazole (C)	-	-	*0.5*	*0.119*	-	-	*0.2*	*0.029*
M750F012 (C)	-	-	-	-	-	-	*0.1*	*0.008*
M750F019 (C)	-	-	*0.4*	*0.087*	-	-	*0.5*	*0.076*
M750F012/M750F021 (C)	-	-	*0.4*	*0.099*	-	-	*0.3*	*0.047*
Unknowns	-	-	*0.7*	*0.172*	-	-	*0.2*	*0.023*
A/G Hydrosylate[1]	0.3	0.008	0.8	0.198	0.3	0.006	0.7	0.098
Mefentrifluconazole (C)	-	-	*0.1*	*0.034*	-	-	*0.2*	*0.032*
M750F018 (C)	-	-	*0.1*	*0.013*	-	-	*0.2*	*0.027*
Unknowns	-	-	*0.6*	*0.151*	-	-	*0.3*	*0.04*
G/H Hydrosylate[1]	0.3	0.007	0.7	0.167	0.2	0.005	1.1	0.148
Mefentrifluconazole (C)	-	-	*0.5*	*0.129*	-	-	*0.6*	*0.08*
M750F018 (C)	-	-	-	-	-	-	*0.2*	*0.028*
Unknowns	-	-	*0.2*	*0.039*	-	-	*0.3*	*0.04*
T/L Hydrosylate[1]	0.3	0.008	0.8	0.191	0.3	0.006	0.5	0.07
Mefentrifluconazole (C)	-	-	*0.8*	*0.191*	-	-	*0.1*	*0.013*
M750F018 (C)	-	-	-	-	-	-	*0.3*	*0.04*
Unknowns	-	-	-	-	-	-	*0.1*	*0.016*

Fraction	C-Label				T-Label			
	Forage TRR=2.378 mg eq/kg		Straw TRR=24.380 mg eq/kg		Forage TRR=2.310 mg eq/kg		Straw TRR=13.984 mg eq/kg	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Final Residues Remaining	2.0	0.047	7.9	1.924	1.9	0.043	5.7	0.804
Total Identified	88.1	2.096	79.8	19.445	92.0	2.125	89.0	12.449
Mefentrifluconazole	*84.4*	*2.007 (F)*	*58.6*	*14.298 (F+C)*	*89.3*	*2.063 (F)*	*68.5*	*9.574 (F+C)*
M750F009	-	-	-	-	*1.1*	*0.025 (F)*	*1.3*	*0.178 (F+C)*
M750F010	-	-	-	-	-	-	*1.3*	*0.18 (F)*
M750F012	*2.1*	*0.049 (F)*	*0.6*	*0.158 (F)*	-	-	*0.1*	*0.008 (C)*
M750F018	-	-	*2.9*	*0.717 (F+C)*	-	-	*5.5*	*0.766 (F+C)*
M750F019	*0.1*	*0.003 (F)*	*5.8*	*1.406 (F+C)*	-	-	*4.8*	*0.67 (F+C)*
M750F018/M750F020	*1.6*	*0.037 (F)*	*6.9*	*1.682 (F)*	*1.6*	*0.037 (F)*	*4.3*	*0.603 (F)*
M750F012/M750F021	-	-	*4.9*	*1.184 (F+C)*	-	-	*3.4*	*0.47 (F+C)*
Total Characterized[2]	5.4	0.128	5.6	1.369	2.7	0.062	2.8	0.385
Accountability[3]	95.5	2.271	93.3	22.738	96.5	2.23	97.5	13.638

Notes:

F = free, C = conjugated.

[1] M/C = macerozyme/cellulase, A/G = amylase/amyloglucosidase, G/H glucosidase/hesperidinasse, T/L tyrosinase/laccase.

[2] Total characterized from solvent extractable radioactive residues and hydrosylates from the unextracted residues.

[3] Accountability = Total Identified + Total Characterized + Final Residues Remaining after enzyme hydrolysis/TRR * 100.

Table 20 Characterisation and identification of C-Label and T-Label residues in wheat grain

Fraction	C-Label Grain [TRR = 0.062 mg eq/kg]		T-Label Grain [TRR = 0.620 mg eq/kg]	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Total Solvent Extracted	43.5	0.027	77.9	0.483
Acetonitrile	17.7	0.011	3.5	0.022
Isohexane	1.6	0.001	0.2	0.001
Water	24.2	0.015		
Acetone supernatant	8.1	0.005	9.8	0.061
Triazole Alanine (F)	-	-	*1.1*	*0.007*
1,2,4-triazole (F)	-	-	*1.0*	*0.006*
Triazole Acetic Acid (F)	-	-	*6.8*	*0.042*
Unknowns	*14.5*	*0.005*	*0.6*	*0.004*
Protease hydrosylate	14.5	0.009	59.0	0.366
Triazole Alanine (C)	-	-	*44.4*	*0.275*
Triazole Acetic Acid (C)	-	-	*14.7*	*0.091*
A/G[1] hydrosylate	<1.6	<0.001	1.0	0.006
A/G residue	<1.6	<0.001	0.2	0.001
Post Extraction Solids	56.5	0.035	22.1	0.137
Ammonia hydrosylate	17.7	0.011	15.3	0.095
A/G hydrosylate	24.2	0.015	4.5	0.028
Final Residues Remaining	16.1	0.01	1.9	0.012
Total Identified	0.0	0.0	67.9	0.421
Triazole Alanine	-	-	*45.5*	*0.282 (F+C)*
1,2,4-triazole	-	-	*1.0*	*0.006 (F)*
Triazole Acetic Acid	-	-	*21.5*	*0.133 (F+C)*

Fraction	C-Label Grain [TRR = 0.062 mg eq/kg]		T-Label Grain [TRR = 0.620 mg eq/kg]	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Total Characterized[2]	85.5	0.053	25.3	0.157
Accountability[3]	102	0.063	95.2	0.59

Notes:

F = free, C = conjugated.

[1] A/G = amylase/amyloglucosidase.

[2] Total characterized from solvent extractable radioactive residues and hydrosylates from the unextracted residues.

[3] Accountability = Total Identified + Total Characterized + Final Residues Remaining after enzyme hydrolysis/TRR * 100.

The application formulation and methanol extracts from wheat forage and straw were further analysed by HPLC to determine the enantiomer ratios of mefentrifluconazole. Chiral analysis was not conducted for wheat grain as mefentrifluconazole was not found in this matrix. The relative amounts of the two enantiomers were approximately 1:1 in the application formulations and in each matrix (Table 21).

Table 21 Enantiomer ratios from the treatment of wheat with chlorophenyl and triazole radiolabelled mefentrifluconazole.

Matrix	Enantiomer 1 (% ROI)	Enantiomer 2 (% ROI)
Chlorophenyl label		
Application Formulation	53.56	46.44
Forage	47.49	52.51
Straw	48.16	51.84
Triazole label		
Application Formulation	48.53	51.47
Forage	49.01	50.99
Straw	47.82	52.18

Notes:

ROI = region of interest (on the chromatogram).

All samples were extracted 42–182 days after sampling, and were then analysed 13–197 days after extraction. Total time between sampling to analysis was 196–281 days. The stability of the radioactive residues in wheat forage (stored at ≤-18 °C) and extracts of wheat forage (storage in fridge) was investigated using homogenates and extracts from both labels. Comparison of the chromatographic profiles between wheat forage extracted 84–85 days after sampling and re-extracted 623–624 days after sampling showed no significant changes to the composition and amounts of radioactivity in forage. Similarly, the chromatographic profiles between extracts analysed after 12–195 days of storage and re-analysed after 540–595 days of storage showed no significant changes to the composition and amounts of radioactivity in wheat forage extracts. Although storage stability was not assessed in wheat grain and straw matrices and extracts, all samples were extracted within 6 months of sampling. The extracts from wheat grain and straw were refrigerated for up to 197 days prior to analysis which is adequately covered by the storage period demonstrated in extracts from wheat forage. Furthermore extracts from grape leaves and soya bean seeds were also stable for up to 231 days and 374 days, respectively.

In summary, the metabolism of mefentrifluconazole is adequately understood in grapes, soya beans, and wheat, representing a fruit, a pulse crop, and a cereal crop. Unchanged parent was the predominant residue in grapes (berries, leaves, and stalks), wheat (forage and straw), and soya bean

(green pods, forage, and hulls). The parent was further conjugated by sugars resulting in numerous minor metabolites found in low levels compared to the parent. In contrast, in wheat grain and soya bean seed, the predominant component of the residue was triazole alanine formed by cleavage of the parent at the triazole-bridge. In wheat grain, triazole lactic acid was also a major component of the residue. Unchanged parent was absent in wheat grain and present in very low amounts in soya bean seed. Metabolites formed by conjugation with sugars were present in minor amounts (< 6 percent TRR and low relative to the parent).

Figure 1 shows a proposed metabolic pathway of mefentrifluconazole in crops.

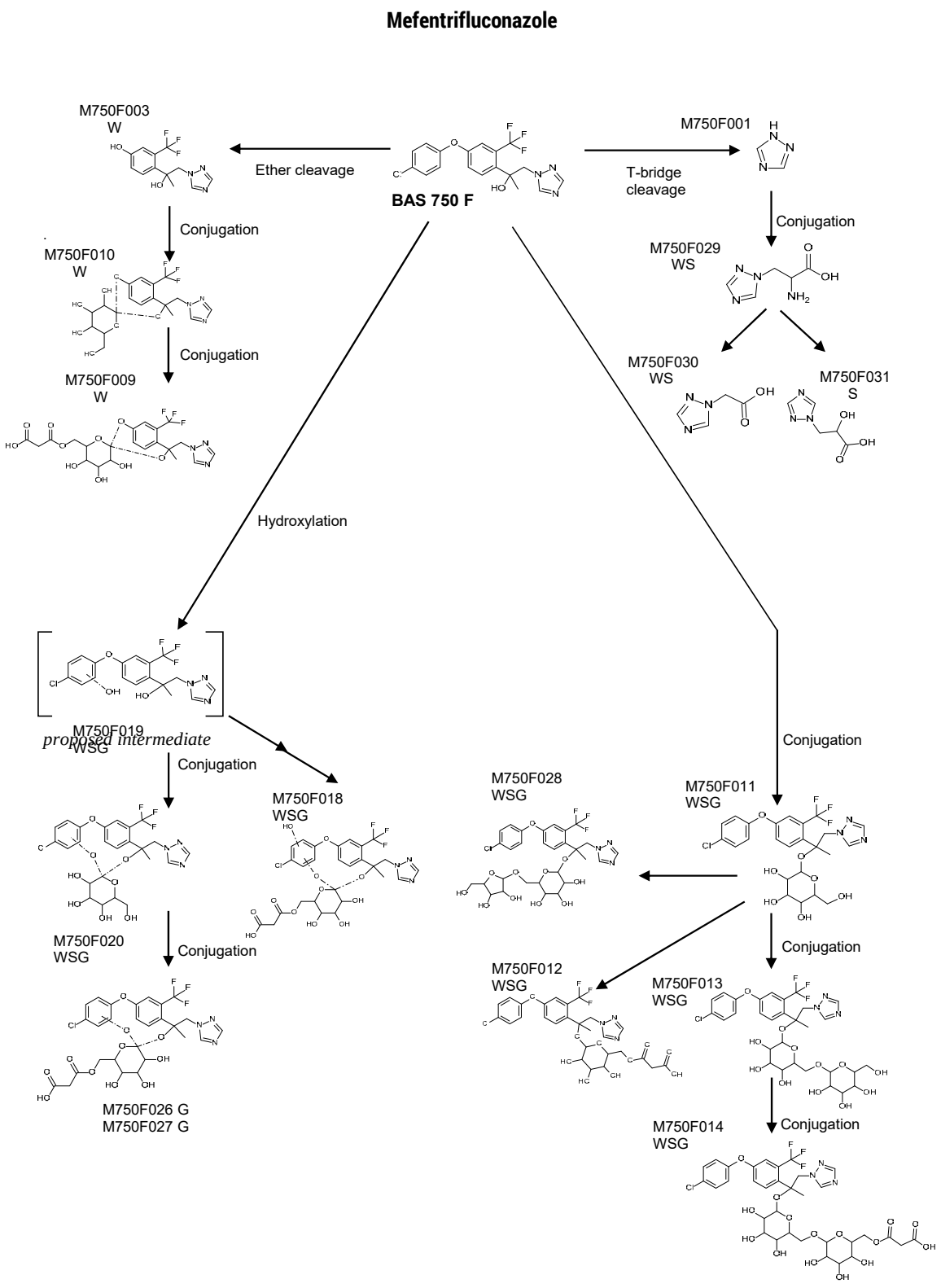


Figure 1 Proposed metabolic pathway of mefentrifluconazole in primary crops

Animal metabolism

Laboratory animal studies

Metabolism of mefentrifluconazole in rats was evaluated by the WHO Core Assessment Group of the 2020 JMPR.

Lactating goats

The metabolism of chlorophenyl-U-^{14}C-labelled (C-Label), triazole-3-(5)- ^{14}C-labelled (T-Label) or trifluoromethylphenyl-ring-U-^{14}C-labelled (TFMP-Label) mefentrifluconazole was investigated in lactating goats (British Saanen, 66–74 kg bw) (Thiaener et al., 2015, BASF DocID 2015_1078841). For the C and T labels, two goats each, and for the TFMP-label one goat, were dosed orally once daily for 14 consecutive days (C- and T-Label) or for 12 consecutive days (TFMP-Label). Feed consumption during the dosing period ranged from 0.72–1.78 kg/day, with animals offered 1 kg concentrate and 1 kg hay per day. The nominal daily doses were equivalent to 12 ppm in the diet (0.34 mg/kg bw). Milk production averaged 2.2 kg/day for the four goats. During the dosing period, urine and faeces were sampled once daily, while milk was collected twice daily, in the afternoon and in the morning and combined per 24 hour period. Following analysis of the individual whole milk samples the remaining bulk from the 6–7, 7–8, 8–9, 9–10, 10–11 and 11–12 day samples were combined and analysed. This whole milk pool was then divided into two equal portions, one was retained as whole milk and the second was centrifuged to separate the cream from the skimmed milk. Tissues, organs (liver, kidney, muscle and fat samples) and bile were collected after animal sacrifice, approximately 23 hours after administration of the last dose.

TRRs in liquid samples were determined by Liquid Scintillation Counting (LSC) while those in solid samples were determined by combustion analysis. Plateau levels of radioactive residues in milk were reached within 5–6 days after administration of the first dose (Table 22).

Table 22 TRRs in milk after administration of ^{14}C-Mefentrifluconazole to goats

Administration Day	C-label TRR (mg eq/kg)		T-label TRR (mg eq/kg)		TFMP-label TRR (mg eq/kg)
	goat 1	goat 2	goat 3	goat 4	goat 5
1	ND	ND	ND	ND	ND
2	0.014	0.015	0.076	0.045	0.021
3	0.027	0.031	0.156	0.099	0.049
4	0.029	0.030	0.220	0.262	0.058
5	0.033	0.037	0.261	0.372	0.074
6	0.031	0.041	0.273	0.347	0.075
7	0.029	0.046	0.285	0.317	0.074
8	0.028	0.042	0.311	0.310	0.080
9	0.028	0.040	0.289	0.284	0.074
10	0.028	0.038	0.281	0.259	0.071
11	0.028	0.035	0.285	0.275	0.062
12	0.025	0.039	0.279	0.254	0.061
13	0.027	0.038	0.271	0.228	0.056
14	0.028	0.053	0.263	0.224	-
15	0.030	0.059	0.253	0.224	-
Pooled sample	0.029		0.284		0.065

Most of the radioactivity was recovered in the excreta with urine containing 25.9–40.2 percent of the administered dose (AD) and faeces containing 34.5–49.6 percent of the AD. The radioactivity recovered in milk and tissues was low, each accounting for ≤ 2.2 percent of the AD.

Table 23 Material balance after administration of ^{14}C- Mefentrifluconazole to goats

Matrix	C-label Mean % AD	T-label Mean % AD	TFMP-label Mean % AD
Milk	0.25	2.16	0.35
Liver	0.40	0.25	0.52
Kidney	0.01	0.01	0.02

Matrix	C-label Mean % AD	T-label Mean % AD	TFMP-label Mean % AD
Muscle (flank)	0.02	0.12	0.07
Muscle (loin)	0.01	0.06	0.03
Fat (subcutaneous)	0.04	0.03	0.22
Fat (omental)	0.21	0.12	0.60
Fat (renal)	0.09	0.03	0.16
Urine	25.86	26.90	40.21
Feces	47.89	49.59	34.49
G.I. tract contents	3.35	2.63	3.76
G.I. tract	1.70	1.24	1.08
Bile	0.02	0.02	0.22
Whole blood	<0.01	Not analysed	<0.01
Cage wash	0.94	0.53	0.87
Total recovery	80.76	83.65	84.91

The calculated total radioactive residues (TRRs) in the pooled milk samples (144–288 h) of the C- and T-labels and the TFMP-label was 0.029 mg eq/kg, 0.273 mg eq/kg, and 0.062 mg eq/kg for whole milk and 0.016 mg eq/kg, 0.270 mg eq/kg and 0.036 mg eq/kg for skimmed milk, respectively. The TRRs in cream were 0.207 mg eq/kg, 0.289 mg eq/kg and 0.521 mg eq/kg for the C-label, the T-label and the TFMP-label, respectively. For tissues, TRRs were highest in liver (0.650–1.332 mg eq/kg), followed by kidney (0.352–0.422 mg eq/kg), composite fat sample (0.213–0.532 mg eq/kg) and composite muscle sample (0.047–0.223 mg eq/kg). In general, levels of radioactivity were lowest in milk and muscle (Table 24).

Table 24 TRRs in milk and tissues

Matrix	C-Label		T-Label		TFMP-Label	
	TRR measured (mg eq/kg)	TRR calculated (mg eq/kg)	TRR measured (mg eq/kg)	TRR calculated (mg eq/kg)	TRR measured (mg eq/kg)	TRR calculated (mg eq/kg)
Muscle (loin/flank)	0.044	0.047	0.222	0.223	0.099	0.098
Liver	1.122	1.085	0.655	0.650	1.468	1.332
Kidney	0.353	0.352	0.386	0.396	0.436	0.422
Fat (omental/ subcutaneous/renal)	0.307	0.309	0.215	0.213	0.515	0.532
Whole milk	0.029	0.029	0.284	0.273	0.065	0.062
Skim milk	0.016	0.016	0.286	0.270	0.031	0.036
Cream	0.204	0.207	0.266	0.289	0.491	0.521

Subsamples of liver, kidney and muscle (all labels) were sequentially extracted with methanol (3×) and water (2×). Subsamples of fat (all labels) were sequentially extracted with acetonitrile (2×) and isohexane (3×). After each extraction step, the samples were centrifuged and concentrated. The respective methanol and water extracts (liver, kidney, muscle) or acetonitrile and isohexane extracts (fat) were combined and analysed. The methanol extracts of kidney (C-label) and liver (C- and T-labels) were incubated with β-glucuronidase/arylsulfatase to investigate the presence of glucuronic acid conjugates M750F064 and M750F068.The unextracted residues were dried, homogenised and combusted for determination of the radioactive residues. In the case of liver, unextracted residues were subjected to protease hydrolysis.

Subsamples of the pooled whole milk and skimmed milk were extracted using acetonitrile and isohexane and centrifuged. Since after centrifugation, three phases were obtained, water was added to the supernatants to enable phase separation and the samples (whole milk and skimmed milk) were

centrifuged again. For skimmed milk, the centrifuged supernatant was filtered into a separating funnel. The acetonitrile phase and the isohexane phase of whole milk and skimmed milk were collected. For the C-label, the unextracted residue of whole milk and skimmed milk was subjected to additional extractions using methanol (3×) and water (2×; only for skimmed milk). The extracts were separated from the unextracted residues by centrifugation and filtered. The methanol and water extracts were combined while the unextracted residues were dried prior to analysis. Subsamples of the pooled cream were extracted once with acetonitrile and isohexane (2× for C-label). The acetonitrile and isohexane phases were collected and combined while the dried residues were homogenized prior to analysis.

Extraction of whole milk with acetonitrile released the majority of the radioactivity (86–96 percent TRR), with ≤5 percent TRR extracted using isohexane. Unextracted residues represented 2.0–7.5 percent TRR (0.001–0.020 mg eq/kg). In the acetonitrile extracts of whole milk, from the C- and TFMP-label, the parent compound was the major residue, accounting for 44.5–47.5 percent TRR (0.014–0.028 mg/kg) as was the metabolite M750F043, representing 14.2–25 percent TRR (0.004–0.016 mg eq/kg). Three additional minor metabolites were identified M750F022 (1.2–2.2 percent TRR; 0.001mg eq/kg), M750F041 (6.0–7.2 percent TRR; 0.002–0.004 mg eq/kg) and M750F072 (5.8–11.2 percent TRR; 0.002–0.004 mg eq/kg). In the acetonitrile extracts of whole milk, from the T-label, 1,2,4-triazole was the predominant metabolite detected, accounting for 78.4 percent TRR (0.214 mg eq/kg) with the parent compound representing 3 percent TRR (0.008 mg eq/kg). A similar metabolic profile was observed in skimmed milk and cream.

Although subjected to the same extraction procedure as milk, the distribution of radioactivity in fat was different. Moreover, isohexane extraction of the composite fat samples released the majority of the radioactivity (>91 percent TRR; >0.19 mg eq/kg) for all three labels. Acetonitrile-extracted and unextracted residues accounted for 1–5 percent TRR (0.003–0.011 mg eq/kg) and ≤4 percent TRR (0.008 mg eq/kg), respectively. In the isohexane extracts, parent compound was the main residue for all three labels (85–88 percent TRR; 0.18–0.47 mg/kg). Metabolite M750F022 was the only other metabolite detected in fat of the C- and TFMP-label studies (up to 5.8 percent TRR; 0.031 mg eq/kg) while 1,2,4-triazole was the only metabolite observed in the T-label study at 4.7 percent TRR (0.01 mg eq/kg).

Extraction of composite muscle samples with methanol released greater than 92 percent TRR, with ≤0.7 percent TRR extracted using water and <1.5 percent TRR remaining unextracted. In the methanol extract of muscle of the C- and TFMP-label, the parent compound was the main residue, accounting for 88–96 percent TRR (0.04–0.09 mg/kg). In the methanol extract of muscle of the T-label, the metabolite 1,2,4-triazole was the main residue (87 percent TRR; 0.19 mg eq/kg) with mefentrifluconazole accounting for 12 percent TRR (0.027 mg/kg). Metabolite M750F022 was only observed in muscle of the C-label and represented 6.7 percent TRR (0.003 mg eq/kg).

Similar to muscle, extraction of liver with methanol released greater than 88 percent TRR, with ≤2 percent TRR extracted using water. The remaining unextracted radioactivity (up to 9 percent TRR) was subjected to protease hydrolysis which released an additional 2–3 percent TRR (0.01–0.04 mg eq/kg), with 4–6 percent TRR (0.04–0.09 mg eq/kg) remaining as bound residues. In the methanol extracts, the parent compound represented one of the main residues for all three labels (26–50 percent TRR; 0.17–0.62 mg/kg) together with the metabolite M750F016 (10–15 percent TRR; 0.065–0.20 mg eq/kg). Metabolite M750F068, resulting from glucuronidation of the parent compound, was also observed in liver of all three labels but at lower levels (3–4 percent TRR; 0.03–0.06 mg eq/kg). In addition, the minor metabolite M750F022 and its glucuronide derivative M750F038, were found in the methanol extracts of the C- and TFMP-label (5–11 percent TRR; 0.05–0.15 mg eq/kg). For the T-label, 1,2,4-triazole and a likely conjugate of 1,2,4-triazole, which was converted entirely into 1,2,4-triazole after addition of hydrochloric acid, accounted together for 32 percent TRR (0.21 mg eq/kg).

Methanol extraction of kidney released greater than 96 percent TRR with ≤1 percent TRR being extracted with water and ≤3 percent TRR remaining unextracted. In the methanol extract of kidney, the parent compound was one of the main components for C-label (28 percent TRR; 0.100 mg/kg) and TFMP-label (46 percent TRR; 0.20 mg/kg). Conversely, for the T-label, triazole was the main component (68 percent TRR; 0.27 mg eq/kg) while the parent compound was only present at 10 percent TRR (0.04 mg/kg). In the C-label, the major metabolites M750F038 (14.8 percent TRR; 0.052 mg eq/kg) and M750F064 (11.8 percent TRR; 0.042 mg eq/kg) and M750F068 (18 percent TRR; 0.06 mg eq/kg) were identified. In addition, M750F022 was identified (6 percent TRR; 0.02 mg eq/kg). Incubation of the methanol extract with β-glucuronidase/arylsulfatase led to cleavage of glucuronic acid from M750F064, resulting in the formation of metabolite M750F022 and of M750F068 generating the parent compound. In the methanol extract of the TFMP-label, the metabolites M750F022 and M750F038 were also predominant, accounting for 14 percent TRR (0.06 mg eq/kg) and 11 percent TRR (0.05 mg eq/kg), respectively. The metabolites M750F003, M750F015, M750F016 and M750F072 were also observed but none represented greater than 4 percent TRR (0.02 mg eq/kg).

The results are shown in Tables 25 to 27. Urine, faeces and bile were also subjected to characterization/identification, where several other metabolites, in addition to those in milk and tissues, were observed.

Table 25 Characterisation and identification of C-Label residues in solvent extracts from goat milk, fat, muscle, liver and kidney

Fraction	Whole milk TRR = 0.029 mg eq/kg		Fat TRR = 0.307 mg eq/kg		Fraction	Muscle TRR = 0.044mg eq/kg		Liver TRR = 1.122 mg eq/kg		Kidney TRR = 0.353 mg eq/kg	
	% TRR	mg eq /kg	% TRR	mg eq /kg		% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg
Acetonitrile	85.6	0.025	1.1	0.003	Methanol	98.2	0.046	91.3	0.990	96.4	0.340
Mefentrifluconazole	*47.5*	*0.014*	*Not analysed*		*Mefentrifluconazole*	*87.9*	*0.042*	*49.9*	*0.541*	*28.3*	*0.100*
M750F016	-	-			*M750F016*	-	-	*11.8*	*0.128*	-	-
M750F022	*2.2*	*0.001*			*M750F022*	*6.7*	*0.003*	*4.8*	*0.052*	*5.8*	*0.021*
M750F038	-	-			*M750F038*	-	-	*6.5*	*0.070*	*14.8*	*0.052*
M750F041	*6.0*	*0.002*			*M750F041*	-	-	-	-	-	-
M750F043	*14.2*	*0.004*			*M750F043*	-	-	-	-	-	-
M750F068	-	-			*M750F068*	-	-	*3.0*	*0.033*	*17.8*	*0.063*
M750F072	*5.9*	*0.002*			*M750F072*	-	-	-	-	-	-
M750F064	-	-			*M750F064*	-	-	-	-	*11.8*	*0.042*
Unknowns	-	-			Unknowns	-	-	8.3	0.090	19.3	0.068
Isohexane	5.0	0.001	99.5	0.306	Water	0.3	<0.001	1.1	0.012	1.0	0.003
Mefentrifluconazole	*Not analysed*		*84.6*	*0.260*		*Not analysed*		*Not analysed*		*Not analysed*	
M750F022			*4.5*	*0.014*							
Methanol	5.8	0.002	-	-							
Total Extracted	96.4	0.028	100.6	0.309	Total Extracted	98.5	0.047	92.4	1.002	97.4	0.343
Total Identified	75.7	0.022	89.1	0.274	Total Identified	94.6	0.045	76.0	0.824	78.5	0.277
Total Characterized	17.4	0.005	1.1	0.003	Total Characterized	0.3	<0.001	9.5	0.103	20.3	0.071
Protease hydrolysate	-	-	-	-	Protease hydrolysate	-	-	3.4	0.037	-	-
Total Unextracted	3.5	0.001	-	-	Total Unextracted	1.5	0.001	4.5	0.049	2.6	0.009

Fraction	Whole milk TRR = 0.029 mg eq/kg		Fat TRR = 0.307 mg eq/kg		Fraction	Muscle TRR = 0.044mg eq/kg		Liver TRR = 1.122 mg eq/kg		Kidney TRR = 0.353 mg eq/kg	
	% TRR	mg eq /kg	% TRR	mg eq /kg		% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg
Accountability	100	0.029	101	0.309	Accountability	110	0.047	97	1.088	100	0.352

Table 26 Characterisation and identification of T-Label residues in solvent extracts from goat milk, fat, muscle, liver and kidney

Fraction	Whole milk TRR = 0.284 mg eq/kg		Fat TRR = 0.215 mg eq/kg		Fraction	Muscle TRR = 0.222 mg eq/kg		Liver TRR = 0.655 mg eq/kg		Kidney TRR = 0.386 mg eq/kg	
	% TRR	mg eq /kg	% TRR	mg eq /kg		% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg
Acetonitrile	92.4	0.252	5.0	0.011	Methanol	98.7	0.220	87.9	0.571	98.3	0.390
Mefentrifluconazole	*3.0*	*0.008*	*Not analysed*		*Mefentrifluconazole*	*11.9*	*0.027*	*26.2*	*0.170*	*10.3*	*0.041*
1,2,4-triazole	*78.4*	*0.214*			*1,2,4-triazole*	*87.3*	*0.194*	-	-	*68.1*	*0.270*
1,2,4-triazole derivative	-	-			*1,2,4-triazole derivative*[A]	-	-	*31.8*	*0.207*	-	-
M750F016	-	-			*M750F016*	-	-	*10.0*	*0.065*	-	-
M750F068	-	-			*M750F068*	-	-	*4.4*	*0.028*	-	-
Unknowns	*1.8*	*0.005*			*Unknowns*	-	-	*8.0*	*0.052*	*9.9*	*0.039*
Isohexane	0.1	<0.001	91.0	0.193	Water	0.7	0.002	2.0	0.013	0.4	0.002
Mefentrifluconazole	*Not analysed*		*84.9*	*0.180*		*Not analysed*		*Not analysed*		*Not analysed*	
M750F001			*4.7*	*0.010*							
Total Extracted	92.5	0.253	96.0	0.204	Total Extracted	99.4	0.222	89.9	0.584	98.7	0.392
Total Identified	81.4	0.222	89.6	0.190	Total Identified	99.2	0.221	72.4	0.470	78.4	0.311
Total Characterized	1.9	0.006	2.7	0.006	Total Characterized	0.7	0.002	10.0	0.065	10.3	0.041
Protease hydrolysate	-	-	-	-	Protease hydrolysate	-	-	2.2	0.014	-	-
Total Unextracted	7.5	0.020	4.0	0.008	Total Unextracted	0.7	0.001	6.7	0.044	1.2	0.005
Accountability	100	0.273	100	0.212	Accountability	101	0.223	97	0.628	100	0.397

Notes:

[A] The metabolite 1,2,4-triazole derivative was detected in a ratio of about 2:1 following HPLC analysis. The 1,2,4-triazole derivative was entirely converted to 1,2,4-triazole after addition of hydrochloric acid, evidence that the derivative was a conjugate of 1,2,4-triazole.

Table 27 Characterisation and identification of TFMP-Label residues in solvent extracts from goat milk, fat, muscle, liver and kidney

Fraction	Whole milk TRR = 0.065 mg eq/kg		Fat TRR = 0.515 mg eq/kg		Fraction	Muscle TRR = 0.099 mg eq/kg		Liver TRR = 1.468 mg eq/kg		Kidney TRR = 0.436 mg eq/kg	
	% TRR	mg eq /kg	% TRR	mg eq /kg		% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg
Acetonitrile	95.9	0.059	1.1	0.006	Methanol	98.8	0.097	91.5	1.219	97.9	0.420
Mefentrifluconazole	*44.5*	*0.028*	*Not analysed*		*Mefentrifluconazole*	*95.7*	*0.094*	*46.7*	*0.622*	*46.0*	*0.198*
M750F003	-	-			*M750F003*	-	-	-	-	*3.2*	*0.014*
M750F015	-	-			*M750F015*	-	-	-	-	*2.6*	*0.011*
M750F016	-	-			*M750F016*	-	-	*15.0*	*0.200*	*3.7*	*0.016*
M750F022	*1.2*	*0.001*			*M750F022*	-	-	*7.6*	*0.101*	*10.7*	*0.046*

Fraction	Whole milk TRR = 0.065 mg eq/kg		Fat TRR = 0.515 mg eq/kg		Fraction	Muscle TRR = 0.099 mg eq/kg		Liver TRR = 1.468 mg eq/kg		Kidney TRR = 0.436 mg eq/kg	
	% TRR	mg eq /kg	% TRR	mg eq /kg		% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg
M750F038	-	-			*M750F038*	-	-	*11.2*	*0.149*	*14.0*	*0.060*
M750F041	*7.2*	*0.004*			*M750F041*	-	-	-	-	-	-
M750F043	*25.0*	*0.016*			*M750F043*	-	-	-	-	-	-
M750F068	-	-			*M750F068*	-	-	*4.2*	*0.056*	-	-
M750F072	*5.8*	*0.004*			*M750F072*	-	-	-	-	*3.0*	*0.013*
Unknowns	*3.0*	*0.002*			*Unknowns*	-	-	*4.5*	*0.060*	*11.0*	*0.047*
Isohexane	2.3	0.001	98.4	0.524	Water	<0.1	<0.001	0.3	0.003	0.3	0.001
Mefentrifluconazole	*Not analysed*		*88.1*	*0.469*		*Not analysed*		*Not analysed*		*Not analysed*	
M750F022			*5.8*	*0.031*							
Total Extracted	98.1	0.061	99.5	0.53	Total Extracted	98.8	0.097	91.8	1.222	98.2	0.422
Total Identified	83.7	0.052	93.9	0.50	Total Identified	95.7	0.094	84.7	1.128	83.3	0.357
Total Characterized	5.3	0.003	4.5	0.024	Total Characterized	<0.1	<0.001	4.8	0.063	11.3	0.048
Protease hydrolysate	-	-	-	-	Protease hydrolysate	-	-	1.8	0.023	-	-
Total Unextracted	1.9	0.001	0.5	0.003	Total Unextracted	1.2	0.001	6.5	0.086	1.8	0.008
Accountability	100	0.062	100	0.533	Accountability	100	0.098	98	1.308	100	0.430

In order to analyse if one enantiomer of mefentrifluconazole was preferably metabolised in goat, enantiomer-specific analysis of the parent compound, isolated from selected matrices of the T-label and TFMP-label, was performed. While the ratio of both isomers was found to be approximately 50:50 in the doses administered to the animals and in the extracts of faeces, the relative amount of the (S)-isomer was lower compared to the (R)-isomer in the other investigated matrices (cream, liver and fat (T-label study) and kidney and muscle (TFMP-label study)). The relative amounts of (S)-isomer: (R)-isomer ranged from 20 percent:80 percent to 30 percent:70 percent. These findings demonstrated that matrix-specific differences were observed.

To demonstrate storage stability, analyses of stored extracts were performed for the samples of whole milk, skimmed milk (TFMP- and T- labels only), cream, liver, kidney, muscle and fat of all labels. Extracts were stored for up to 156 days prior to the first analysis with re-analysis occurring up to 428 days thereafter, for a minimum storage duration of 435 days. No significant changes were observed following comparison of the metabolic HPLC profiles of the various extracts.

The metabolic pathway of mefentrifluconazole in lactating goats appears to proceed via two main transformation reactions. The first is hydroxylation at the chlorophenyl-ring leading to M750F015, M750F016, M750F017, further glucuronidation generating M750F063 and M750F068. M750F041 and M750F091 are intermediates of the C-ring oxygenation reaction leading to C-ring hydroxyl metabolites (notably M750F016). Cleavage of the parent compound at the T-bridge generates 1,2,4-triazole as well as the two-ring metabolite M750F022, which itself is subject to oxidation (M750F038), followed by demethylation (M750F040), as well as to hydroxylation (M750F078), to sulfatation (M750F043), and to glucuronidation (M750F064).

The second transformation reaction, observed only to a minor extent, is cleavage of the parent molecule at the ether bridge, generating the two-ring metabolite M750F003. Notably, metabolites consisting of either the C-ring or the TFMP-ring were not observed in any of the samples of the C-label or the TFMP-label. Also seen only to a minor extent is hydroxylation at the methyl of the triazole bridge (M750F039, which is further sulfated to M750F072 or oxidized to M750F042).

The metabolic pathway of mefentrifluconazole in goats is ilusttrated in Figure 2.

Laying hen

The metabolism of chlorophenyl-U-^{14}C-labelled (C-Label), triazole-3-(5)- ^{14}C-labelled (T-Label) or trifluoromethylphenyl-ring-U-^{14}C-labelled (TFMP-Label) mefentrifluconazole was investigated in laying hens (Lohmann Brown, 1.5–2.0 kg) following repeated oral administration (Wenzel *et al*., 2015, BASF DocID 2015_1001001). The test item was administered once daily by gavage to laying hen (ten animals per label) for 14 consecutive days at a nominal dose of 12 ppm feed. The mean actual concentrations were 16.7 ppm feed (C-label), 15.9 ppm feed (TFMP-label) and 15.0 ppm feed (T-label), corresponding to daily means of 1.09, 1.07 and 1.08 mg/kg body weight, respectively. Feed consumption during the dosing period ranged from 96–142 g/day with birds offered 200 g of pellets/day. During the dosing period, excreta were collected once daily, while eggs were collected twice daily after which they were separated into egg whites and egg yolks. Tissues, organs (liver, kidney, muscle and fat samples) and bile were collected after animal sacrifice, 3–6 hours after administration of the last dose.

TRRs in liquid samples were determined by Liquid Scintillation Counting (LSC) while those in solid samples were determined by combustion analysis.

^{14}C-residues in egg yolk reached a plateau concentration within 7 days of dosing at 0.5 mg/kg, 0.6 mg/kg, and 0.3 mg/kg (C-, TFMP-, T-labels, respectively). In egg white, ^{14}C-residues reached a plateau within 3 days at 0.013 mg/kg for the C-label, within 5 days at 0.013 mg/kg for the TFMP-label and within 7 days at 0.39 mg/kg for the T-label (Table 28).

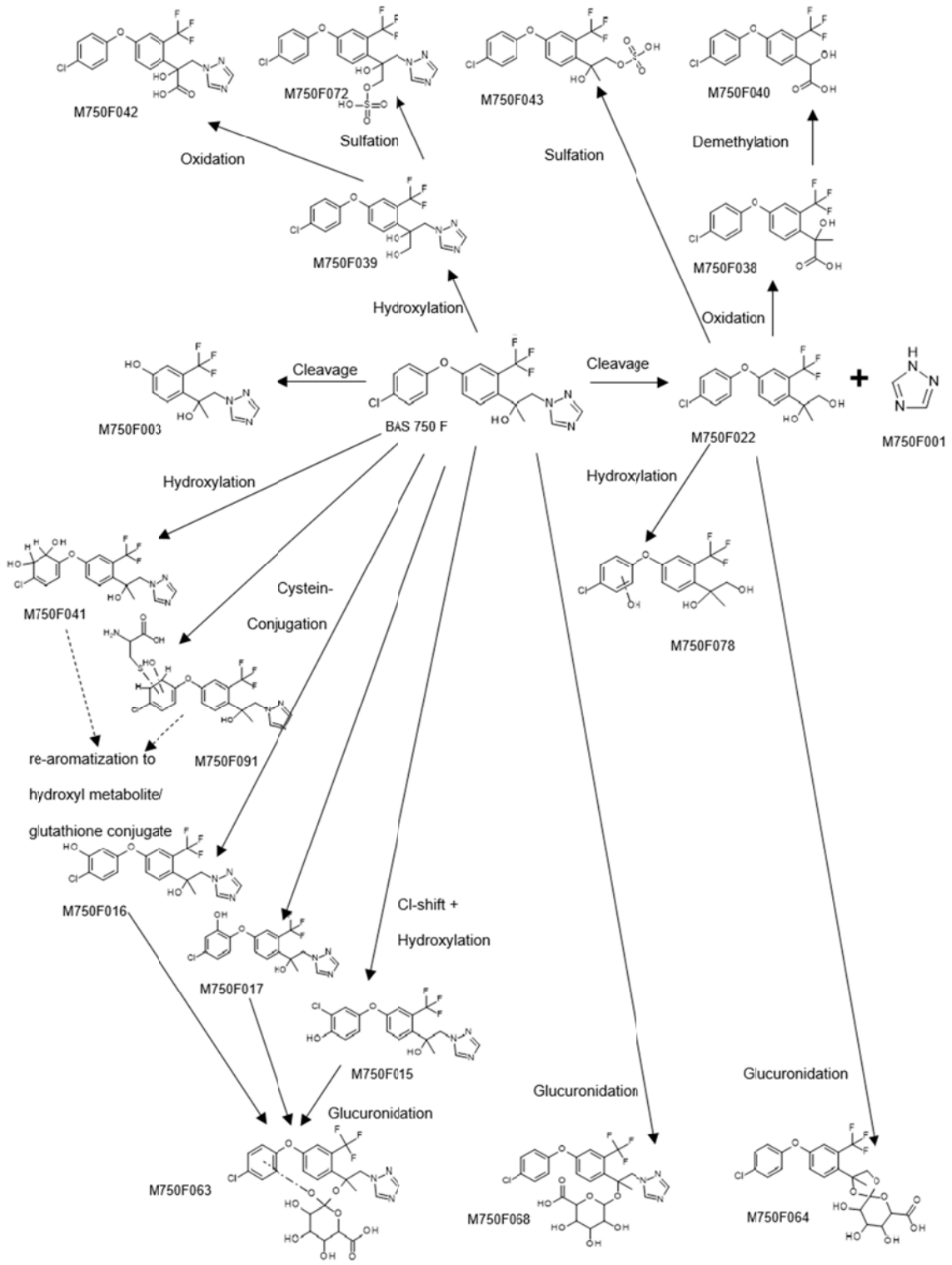


Figure 2 Proposed metabolic pathway of mefentrifluconazole in lactating goats

Table 28 TRR in egg white and yolk after administration of ^{14}C-Mefentrifluconazole to hen

Administration Day	C-label Measured TRR (mg eq/kg)		TFMP-label Measured TRRs (mg eq/kg)		T-label Measured TRR (mg eq/kg)	
	white	yolk	white	yolk	white	yolk
1	0.004	0.001	0.003	0.001	0.119	0.052

Administration Day	C-label Measured TRR (mg eq/kg)		TFMP-label Measured TRRs (mg eq/kg)		T-label Measured TRR (mg eq/kg)	
	white	yolk	white	yolk	white	yolk
2	0.009	0.043	0.008	0.039	0.260	0.138
3	0.013	0.121	0.009	0.138	0.300	0.178
4	0.012	0.244	0.009	0.227	0.323	0.215
5	0.012	0.334	0.013	0.384	0.314	0.234
6	0.012	0.472	0.007	0.460	0.359	0.277
7	0.011	0.571	0.011	0.617	0.387	0.301
8	0.009	0.595	0.014	0.622	0.363	0.301
9	0.006	0.556	0.009	0.666	0.415	0.322
10	0.009	0.424	0.008	0.658	0.384	0.308
11	0.008	0.471	0.010	0.639	0.366	0.302
12	0.007	0.454	0.010	0.665	0.390	0.311
13	0.008	0.448	0.010	0.648	0.344	0.292
Pooled sample	0.009	0.477	0.005	0.618	0.357	0.269

The radioactive residues in excreta amounted to 75–89 percent of the total administered dose (AD). For all labels, only low portions of the administered dose (≤ 0.3 percent) were retained in edible tissues or in egg (<1 percent of dose). The results are shown in Tables 29 and 30.

Table 29 Material balance after administration of ^{14}C-Mefentrifluconazole to hen

Matrix	C-label Mean % AD	TFMP-label Mean % AD	T-label Mean % AD
Egg white	0.01	0.02	0.55
Egg yolk	0.22	0.28	0.17
Partially formed eggs	0.08	0.14	0.09
Muscle (breast and thigh)	0.03	0.05	0.23
Liver	0.06	0.13	0.03
Kidney	0.01	0.01	0.01
Fat (omental, renal and subcutaneous)	0.13	0.10	0.01
Excreta	75.30	86.59	88.91
GI tract, contents	1.14	2.41	1.62
Bile	0.01	0.02	<0.00
Blood	<0.00	<0.00	<0.00
Cage wash	2.53	2.61	2.37
Total recovery	79.52	92.36	93.99

For the C- and TFMP-labels, ^{14}C-residues were highest in composite fat samples (0.72–1.1 mg eq/kg), followed by kidney (0.42–0.64 mg eq/kg), liver (0.31–0.58 mg eq/kg) and composite muscle samples (0.053–0.078 mg eq/kg). Following administration of the T-label, the trend was slightly different where TRRs were highest in kidney (0.57 mg eq/kg), followed by liver (0.50 mg eq/kg), muscle (0.36 mg eq/kg) and fat (0.21 mg eq/kg).

Table 30 TRRs in eggs and tissues

Matrix	C-label		TFMP-label		T-label	
	TRR measured (mg eq/kg)	TRR calculated (mg eq/kg)	TRR measured (mg eq/kg)	TRR calculated (mg eq/kg)	TRR measured (mg eq/kg)	TRR calculated (mg eq/kg)
Egg white	0.009	ND	0.005	ND	0.357	0.351
Egg yolk	0.477	0.469	0.618	0.613	0.269	0.300
Muscle (breast/thigh)	0.050	0.053	0.066	0.078	0.363	0.361

Matrix	C-label		TFMP-label		T-label	
	TRR measured (mg eq/kg)	TRR calculated (mg eq/kg)	TRR measured (mg eq/kg)	TRR calculated (mg eq/kg)	TRR measured (mg eq/kg)	TRR calculated (mg eq/kg)
Liver	0.320	0.311	0.582	0.580	0.480	0.498
Kidney	0.427	0.419	0.610	0.642	0.565	0.577
Fat (omental/subcutaneous/renal)	0.702	0.717	0.893	1.102	0.190	0.209

The homogenised samples of muscle, egg white (T-label), egg yolk, liver and kidney (TFMP- and T-labels) were extracted with methanol (3×) or methanol (3×) followed with water (2×). Kidney (C-label) was extracted sequentially 3× with methanol, 2× with dichloromethane and 1× with isohexane. Fat (C- and TFMP-labels) was extracted up to 3× with a mixture of acetonitrile/ isohexane (1/1, v/v) while fat from the T-label was extracted 3× with a mixture of methanol/isohexane (1/1, v/v). For all tested matrices, the solvent extracts were individually combined and the residue was dried.

After solvent extraction, the combined isohexane extract of fat (C-label) was partitioned 3× against acetonitrile. The methanol extract of muscle (T-label) was concentrated and partitioned with acetonitrile / isohexane (1/1, v/v) and water. The pooled methanol extracts of egg white (T-label) and egg yolk (T-label) were concentrated and partitioned up to 3× with acetonitrile / isohexane (1/1, v/v). The methanol extract of liver (C-label) was concentrated, supplemented with acetonitrile and partitioned against isohexane.

The post-extraction solids of egg yolk (C- and TFMP-labels), muscle (C-label), liver (all labels) and kidney (all labels) were subjected to protease treatment (suspension in 0.1 mol/L Tris (pH 7), incubation for 24–72 hours at 37 °C). The fatty acid conjugates in fat (TFMP-Lable) were cleaved enzymatically (suspension in 10 mL water (pH 7.7), incubation with 10 mg lipase over night under constant shaking at 37 °C) or via alkaline hydrolysis (incubated with 4 mL 2 mol/L NaOH for 1 hour in the rotary evaporator at room temperature).

Only egg whites from the T-label study contained sufficient radioactivity to be subjected to further analysis, where methanol and water extraction released almost all of the radioactivity (98.2 percent TRR; 0.350 mg eq/kg). M750F001 was the only identified metabolite in this matrix accounting for all of the radioactivity in the acetonitrile extract (83.2 percent TRR; 0.297 mg eq/kg). A loss of radioactive residues (13.4 percent TRR) was observed during the concentration and partition steps of the pooled methanol extract. However, a parallel workup of the extract revealed that approximately 7.0 percent TRR of this loss may be attributable to the metabolite 1,2,4-triazole.

Following sequential extractions of egg yolks with methanol and water, greater than 89 percent TRR was released, with 2.6 percent TRR remaining unextracted (T-label only). For the C- and TFMP-label studies where the PES accounted for 5–6 percent TRR, these were subjected to enzymatic hydrolysis which released up to 2.5 percent TRR (up to 0.015 mg eq/kg). For the C- and TFMP-label studies, M750F022 represented the major metabolite (39–47 percent TRR; 0.186–288 mg eq/kg). The parent compound and the fatty acid conjugates of the metabolite M750F022 (sum of M750F023, M750F024 and/or M750F025) were present in amounts of 6–12 percent TRR (0.031–0.071 mg eq/kg) and 14–15 percent TRR (0.066–0.091 mg/kg), respectively. In egg yolk of the T-label, 1,2,4-triazole and the parent were detected at similar levels (each accounting for approximately 42 percent TRR (0.12 mg eq/kg)).

Methanol and/or methanol/water extraction of muscle samples released greater than 85 percent TRR (>0.043 mg eq/kg) with 1.4–2.8 percent TRR remaining unextracted from the muscle samples collected from the TFMP- and T-label studies. The enzymatic hydrolysis of the muscle post-extraction solids (PES), from the C-label study, released 7.2 percent TRR (0.004 mg eq/kg) with 8.2 percent TRR

(0.004 mg eq/kg) remaining unextracted. A similar metabolic profile was observed for the C- and TFMP-labels with M750F022 representing the main component (50–77 percent TRR; 0.025–0.051 mg eq/kg). The parent, mefentrifluconazole and the fatty acid conjugates of M750F022 (sum of M750F023, M750F024 and/or M750F025) were present in low amounts of 5.6–7.4 percent TRR (0.003–0.005 mg eq/kg) and 9.8–19.5 percent TRR (0.007–0.010 mg eq/kg), respectively. In muscle from the T-label study, only 1,2,4-triazole was detected (91.4 percent TRR; 0.322 mg eq/kg).

For the C-, TFMP- and T-label studies, sequential extraction of liver with methanol/water released 83–100 percent TRR, with 3–14 percent TRR remaining bound. Enzyme hydrolysis of the unextracted radioactivity released an additional 2–7 percent TRR. Similar observations were made for the C-label and TFMP-labels regarding the metabolic profile, where M750F022 represented the major metabolite (29.3–36.7 percent TRR; 0.118–0.171 mg eq/kg). The parent mefentrifluconazole and the fatty acid conjugates of M750F022 (sum of M750F023, M750F024 and/or M750F025) were present in low amounts 5.8–7.2 percent TRR (0.03 mg/kg) and 6.9–11.6 percent TRR (0.021–0.068 mg/kg), respectively. Additionally, M750F034, a glutathione conjugate, was present at low levels in liver of the C-label (4.3 percent TRR; 0.014 mg eq/kg) and at considerably higher amounts in liver of the TFMP-label (20.1 percent TRR; 0.117 mg eq/kg). In liver of the T-label, 1,2,4-triazole was the main metabolite (85.2 percent TRR; 0.409 mg eq/kg) and the parent and M750F034 were detected at comparably low levels (≤6.7 percent TRR; 0.032 mg eq/kg).

Extraction of kidney with methanol/isohexane (C- and TFMP-label) and methanol/water (T-label) released 83.7–100 percent TRR (0.357–0.603 mg eq/kg) with ≤17 percent TRR remaining unextracted. Following enzymatic hydrolysis of the PES from the C- and TFMP-label studies, 4–10 percent TRR were solubilised. Similar to egg yolk and other tissues, comparable metabolic profiles were observed for the C-label and TFMP-label, with M750F022 representing the major fraction (20.1 percent TRR; 0.086–0.123 mg eq/kg). By contrast, the parent and the fatty acid conjugates of M750F022 (sum of M750F023, M750F024 and/or M750F025) were present in low amounts in kidney of the C-label, 4.0 percent TRR (0.017 mg/kg) and 3.9 percent TRR (0.017 mg eq/kg), respectively, while only mefentrifluconazole (3.7 percent TRR; 0.022 mg eq/kg) was detected in kidney of the TFMP-label. In kidney of the T-label study, only 1,2,4-triazole was identified (65.6 percent TRR; 0.371 mg eq/kg).

Extraction of fat with acetonitrile/isohexane (C- and TFMP- labels) and methanol/water (T-label) released >100 percent TRR with <1.4 percent TRR remaining in the PES. Similar metabolic profiles were observed between the C-label and TFMP-label where the fatty acid conjugates of M750F022 (sum of M750F023, M750F024 and M750F025) accounted for the main portion of radioactive residues (~42 percent TRR; 0.287–0.380 mg eq/kg) followed by metabolite M750F022 (25.4–41.1 percent TRR; 0.178–0.367 mg eq/kg). The parent was present at lower levels in fat of the C-label (5.4 percent TRR; 0.04 mg/kg) in comparison to the fat of the TFMP-label (11.7 percent TRR; 0.10 mg/kg). In fat of the T-label, 1,2,4-triazole was detected as the main metabolite (73.1 percent TRR; 0.139 mg eq/kg) with the parent present in lower amounts (20.1 percent TRR; 0.038 mg/kg).

The incubation of the pooled methanol extract of fat (TFMP-label) with lipase led to a cleavage of the fatty acid conjugates resulting in the metabolite M750F022. A second aliquot of the same extract) was subjected to alkaline treatment, where a complete cleavage of fatty acid conjugates was noted by HPLC analysis. Additionally, the pooled extract of fat after alkaline hydrolysis was partitioned against tetrahydrofuran (THF). The THF phase was analysed by HPLC, where only M750F022 was detected. Therefore, hydrolysis of the fatty acid conjugates appears to result in the formation of the metabolite M750F022 (Table 31).

Table 31 Characterisation and identification of C-label residues in solvent extracts from hen egg yolk, muscle, liver and kidney and fat

Fraction	Egg Yolk TRR = 0.477 mg eq/kg		Muscle TRR = 0.050 mg eq/kg		Liver TRR = 0.320 mg eq/kg		Kidney TRR = 0.427 mg eq/kg		Fraction	Fat TRR = 0.702 mg eq/kg	
	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg		% TRR	mg eq /kg
Methanol	88.3	0.421	85.0	0.043	79.0	0.253	82.1	0.350	Acetonitrile	82.6	0.580
Mefentrifluconazole	*6.5*	*0.031*	*5.6*	*0.003*	*7.2*	*0.03*	*4.0*	*0.017*	*Mefentrifluconazole*	*5.4*	*0.038*
M750F022	*39.0*	*0.186*	*49.9*	*0.025*	*36.7*	*0.118*	*20.1*	*0.086*	*M750F022*	*25.4*	*0.178*
M750F023	*2.6*	*0.012*	*8.0*	*0.004*	*2.0*	*0.006*	*1.7*	*0.007*	*M750F023*	*20.4*	*0.143*
M750F024	-	-	-	-	*1.1*	*0.003*	*0.8*	*0.004*	*M750F024*	-	-
M750F025	-	-	-	-	*3.8*	*0.012*	*1.4*	*0.006*	*M750F025*	-	-
M750F024 / M750F025	*11.3*	*0.054*	*11.5*	*0.006*	-	-	-	-	*M750F024 / M750F025*	*20.5*	*0.144*
M750F034	-	-	-	-	*4.3*	*0.014*	-	-	*M750F034*	-	-
Unknowns	*30.0*	*0.143*	*12.9*	*0.006*	*22.7*	*0.072*	*50.5*	*0.215*	*Unknowns*	*13.1*	*0.092*
Isohexane	-	-	-	-	-	-	0.4	0.002	Isohexane	18.2	0.128
Water	1.1	0.005	-	-	3.7	0.012	-	-	Acetonitrile	13.7	0.096
Unknowns	*1.1*	*0.005*	-	-	-	-	-	-	*M750F023*	*3.3*	*0.023*
Dichloromethane	-	-	-	-	-	-	1.2	0.005	*M750F024 / M750F025*	*7.0*	*0.049*
	Not analysed		*Not analysed*		*Not analysed*		*Not analysed*		*Unknowns*	*1.6*	*0.011*
									Isohexane	2.6	0.018
Total Extracted	89.4	0.426	85.0	0.043	82.7	0.265	83.7	0.357		100.8	0.707
Total Identified	59.4	0.283	75.0	0.038	55.0	0.176	28.0	0.120		81.9	0.575
Total Characterized	31.1	0.148	12.9	0.006	26.4	0.084	52.1	0.222		17.3	0.122
Post Extraction Solids (PES)	-	-	21.5	0.011	14.5	0.046	-	-		-	-
Protease hydrolysate	-	-	7.2	0.004	7.2	0.023	-	-		-	-
Total Unextracted	9.1[A]	0.043	8.2	0.004	7.3	0.023	14.5[B]	0.062		1.4	0.010
Accountability	98	0.469	106	0.054	97	0.311	98	0.419		102	0.717

Notes:

[A] The residue after solvent extraction was further investigated within another workup, where approximately 2.1 percent TRR (0.010 mg/kg) were extracted by protease solubilisation leading to a final residue of 4.0 percent TRR (0.019 mg/kg).

[B] The residue after solvent extraction was further investigated within another workup, where approximately 10.4 percent TRR (0.044 mg/kg) were extracted by protease solubilisation leading to a final residue of 2.2 percent TRR (0.010 mg/kg).

Table 32 Characterisation and identification of TFMP-label residues in solvent extracts from hen egg yolk, muscle, liver and kidney and fat

Fraction	Egg Yolk TRR = 0.618 mg eq/kg		Muscle TRR = 0.066 mg eq/kg		Liver TRR = 0.582 mg eq/kg		Kidney TRR = 0.610 mg eq/kg		Fraction	Fat TRR = 0.893 mg eq/kg	
	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg		% TRR	mg eq /kg
Methanol	93.7	0.578	109.2	0.072	92.3	0.537	97.4	0.594	Acetonitrile	112.1	1.001
Mefentrifluconazole	*11.5*	*0.071*	*7.4*	*0.005*	*5.8*	*0.034*	*3.7*	*0.022*	*Mefentrifluconazole*	*11.7*	*0.104*
M750F022	*46.7*	*0.288*	*77.1*	*0.051*	*29.3*	*0.171*	*20.1*	*0.123*	*M750F022*	*41.1*	*0.367*
M750F023	*5.3*	*0.032*	*5.8*	*0.004*	*3.6*	*0.021*	-	-	*M750F023*	*24.7*	*0.221*
M750F024	*9.0*	*0.056*	-	-	-	-	-	-	*M750F024*	*5.3*	*0.048*
M750F025	*0.6*	*0.003*	-	-	-	-	-	-	*M750F025*	*12.5*	*0.111*
M750F024 /	-	-	*4.0*	*0.003*	*8.0*	*0.047*	-	-	*M750F024 /*	-	-

Fraction	Egg Yolk TRR = 0.618 mg eq/kg		Muscle TRR = 0.066 mg eq/kg		Liver TRR = 0.582 mg eq/kg		Kidney TRR = 0.610 mg eq/kg		Fraction	Fat TRR = 0.893 mg eq/kg	
	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg		% TRR	mg eq /kg
M750F025									*M750F025*		
M750F034	-	-	-	-	*20.1*	*0.117*	-	-	*M750F034*	-	-
Unknowns	*10.2*	*0.062*	*4.6*	*0.003*	*17.4*	*0.102*	*62.2*	*0.380*	*Unknowns*	*12.2*	*0.109*
Isohexane	-	-	-	-	-	-	1.6	0.009	Isohexane	11.0	0.099
Water	0.6	0.004	1.2	<0.002	1.8	0.010	-	-	Acetonitrile	8.8	0.079
	Not analysed		*Not analysed*		*Not analysed*		*Not analysed*		*M750F023*	*2.7*	*0.025*
									M750F024	*0.8*	*0.007*
									M750F025	*3.0*	*0.027*
									Isohexane	1.1	0.010
Total Extracted	94.2	0.581	110.4	0.074	94.1	0.547	98.9	0.603		123.1	1.100
Total Identified	73.0	0.451	94.3	0.062	66.9	0.389	23.8	0.145		101.8	0.909
Total Characterized	10.8	0.065	5.8	0.005	19.2	0.112	63.8	0.389		13.3	0.119
Post Extraction Solids (PES)	5.1	0.032	-	-	5.6	0.033	6.3	0.038		-	-
Protease hydrolysate	2.5	0.015	-	-	2.8	0.016	4.2	0.026		-	-
Total Unextracted	2.2	0.013	2.8	0.002	2.4	0.014	1.7	0.011		0.3	0.002
Accountability	88	0.613	113	0.076	100	0.580	105	0.642		123	1.102

Table 33 Characterisation and identification of T-label residues in solvent extracts from hen egg white, egg yolk and muscle

Fraction	Egg White TRR = 0.357 mg eq/kg		Egg Yolk TRR = 0.269 mg eq/kg		Fraction	Muscle TRR = 0.353 mg eq/kg	
	% TRR	mg eq /kg	% TRR	mg eq /kg		% TRR	mg eq /kg
Methanol	97.0	0.346	107.9	0.290	Methanol	98.8	0.349
Acetonitrile	83.3	0.297	103.7	0.279	Acetonitrile/Isohexane	7.9	0.028
					Water	91.4	0.322
Mefentrifluconazole	-	-	*43.7*	*0.117*	*Mefentrifluconazole*	-	-
1,2,4-triazole	*83.2*	*0.297*	*41.4*	*0.111*	*1,2,4-triazole*	*91.4*	*0.322*
Unknowns	*13.4 (loss[A])*	*0.048 (loss[A])*	-	-	*Unknowns*	-	-
Isohexane	0.2	<0.001	5.3	0.014			
Water	1.2	0.004	1.0	0.003	Water	2.2	0.008
Total Extracted	98.2	0.350	108.9	0.293		101.00	0.357
Total Identified	83.2	0.297	85.1	0.228		91.4	0.322
Total Characterized	1.4	0.005	6.3	0.017		10.1	0.036
Total Unextracted	0.2	0.001	2.6	0.007		1.4	0.005
Accountability	98	0.351	111	0.300		102	0.362

Notes:

[A] Loss from the concentration and partition step of the pooled methanol extract.

Table 34 Characterisation and identification of T-label residues in solvent extracts from hen liver and kidney and fat

Fraction	Liver TRR = 0.480 mg eq/kg		Kidney TRR = 0.565 mg eq/kg		Fat TRR = 0.190 mg eq/kg	
	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg
Methanol	99.2	0.476	98.9	0.559	101.8	0.194
Mefentrifluconazole	*3.7*	*0.018*	-	-	*20.1*	*0.038*
1,2,4-triazole	*85.2*	*0.409*	*65.6*	*0.371*	*73.1*	*0.139*
M750F034	*6.7*	*0.032*	-	-	-	-
Unknowns	-	-	*26.5*	*0.150*	-	-
Isohexane	-	-	-	-	5.0	0.010
Water	1.3	0.006	1.1	0.006	-	-
Total Extracted	100.5	0.482	100.0	0.565	106.8	0.203
Total Identified	95.7	0.459	65.6	0.371	93.2	0.177
Total Characterized	1.3	0.006	27.6	0.156	5.0	0.010
Post Extraction Solids (PES)	3.3	0.016	2.0	0.011	-	-
Protease hydrolysate	1.8	0.009	1.3	0.008	-	-
Total Unextracted	1.0	0.005	0.8	0.005	3.2	0.006
Accountability	104	0.498	102	0.576	110	0.209

Chiral analysis of mefentrifluconazole residue in representative egg yolk and fat samples demonstrated that the ratio of enantiomers in both matrices was 43:57, comparable to the ratio in the administered dose which was determined to be 50:50

The stability of the radioactive residues in various frozen matrices (≤-18 °C) and extracts (storage in fridge) was investigated using homogenates and extracts from the C-label and T-label studies, representing all edible matrices. Comparison of the chromatographic profiles between sampling and the first analysis showed no significant changes to the composition and amounts of radioactivity for each matrix and extract for a storage duration of up to 274 days and 77 days, respectively. Similarly, between the first analysis and re-analysis, no significant changes to the composition and amounts of radioactivity for each matrix and extract were observed over the storage durations of up to 378 days and 354 days, respectively. No stability investigations were conducted for samples collected from the TFMP-label study, however, the stability demonstrated in the C-label and T-label studies can be extended to the samples from the TFMP-label study.

Overall, the results of the laying hen metabolism study show that the metabolic pathway appears to proceed via the cleavage of mefentrifluconazole at the azo-bridge of the propyl-triazole moiety resulting in the metabolites 1,2,4-triazole and M750F022. Metabolite M750F022 is further conjugated with fatty acids forming M750F023, M750F024 (both conjugated with an unsaturated fatty acid) and M750F025 (conjugated with a saturated fatty acid). Hydroxylation of mefentrifluconazole followed by epoxidation and conjugation with glutathione leads to the formation of the liver-specific metabolite M750F034. The incubation of the fat extracts (TFMP-Label) with lipase or sodium hydroxide led to a cleavage of the fatty acids conjugates yielding the metabolite M750F022.

Figure 3 shows a metabolic pathway of mefentrifluconazole in laying hen

In summary, the metabolism of mefentrifluconazole is comparable in both lactating goats and laying hens, although more extensive in laying hens. In addition to the parent, mefentrifluconazole, the major components identified in the tested goat matrices included the metabolites M750F043 (milk only), M750F016 and M750F038 (liver and kidney), M750F022 (kidney) as well as the T-label specific metabolite 1,2,4-triazole in milk and all tissues. In the hen matrices tested, the parent compound was present albeit

at low levels. The predominant metabolites observed in eggs and all tissues was M750F022 while the metabolites M750F023, M750F024 and/or M750F025, fatty acid conjugates of M750F022, were predominant in fat only. Similar to goats, the major T-label specific metabolite 1,2,4-triazole was present in significant levels in eggs and tissues.

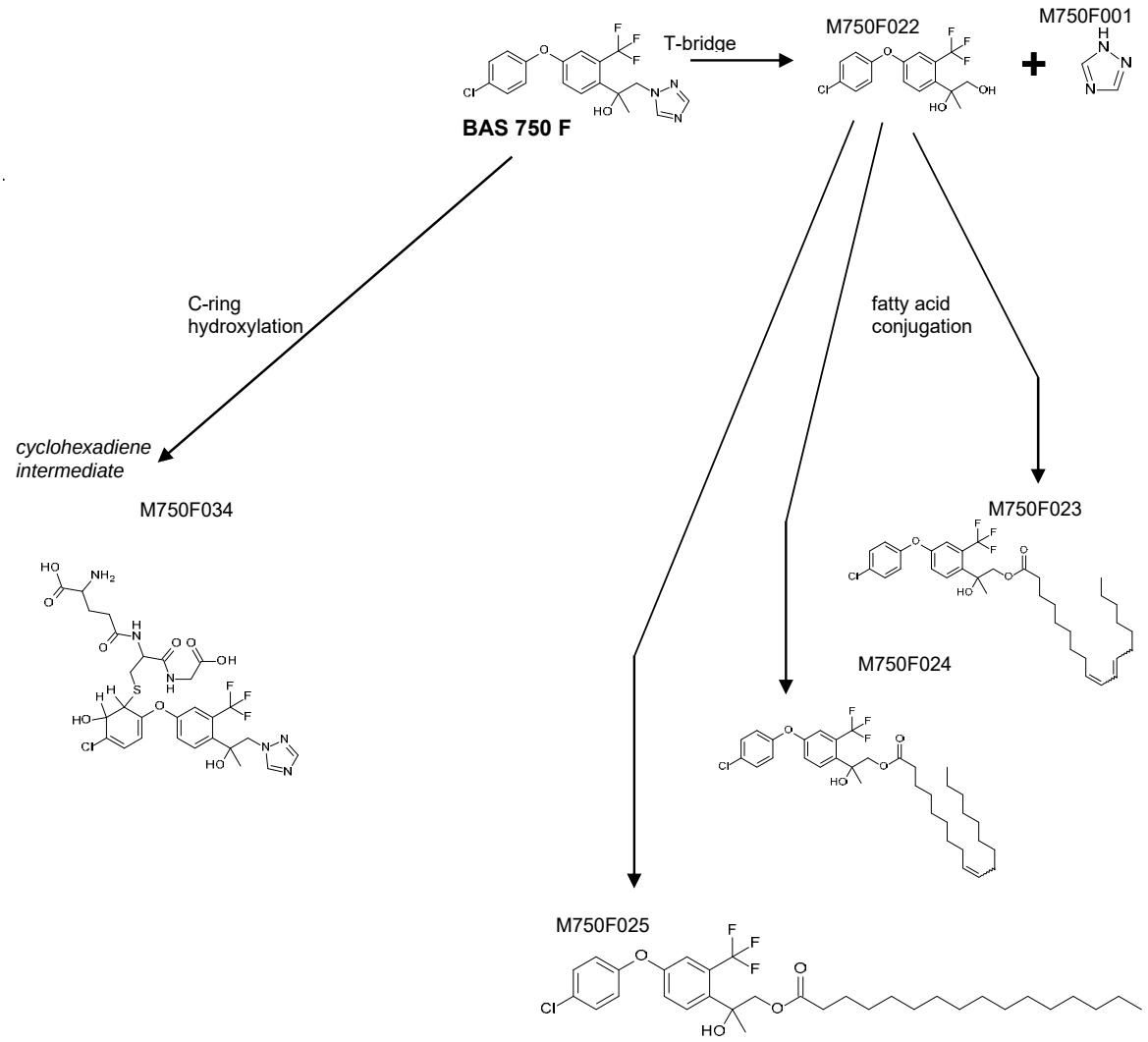


Figure 3 Proposed metabolic pathway of mefentrifluconazole in laying hen

ENVIRONMENTAL FATE

The Meeting received information on soil aerobic metabolism, hydrolysis and photolysis properties of mefentrifluconazole Studies were also received on the behaviour of [^{14}C]-mefentrifluconazole in confined rotational crops.

Route of degradation in soil

Aerobic soil metabolism

The aerobic soil metabolism of mefentrifluconazole was investigated in five agricultural soils

(Staudenmaier, 2015, BASF DocID 2014_1275177, 2014_1275178 and 2015_1003306), two from Europe (Germany; soil LUFA 5M and soil L10) and three from the United States (New Jersey and Indiana). Soil characteristics are summarized in Table 35, as per United States Department of Agriculture (USDA) soil texture classifications.

Table 35 Soil characteristics

Soil designation	Germany		United States		
	LUFA 5M	L10	New Jersey	New Jersey	Indiana
Textural class (USDA scheme)	Loamy sand	Loamy sand	Loam	Loam	Loam
Soil texture (%)					
Sand 0.050-2 mm	82.8	84.0	29	33	35
Silt 0.002-0.050	11.1	11.0	49	46	46
Clay <0.002 mm	6.1	5.0	22	21	19
Organic matter (%)	3.50	1.60	2.3	2.2	2.0
pH (H_2O)	7.9	6.6	6.9	6.8	6.3
pH ($CaCl_2$)	7.2	6.1	Not determined	6.4	5.8
Cation exchange capacity (cmol/kg)	11.4	3.7	9.1	8.5	10.3
Max water holding capacity (g/100 g dry soil)	25.2	26.9	37.0	33.3	33.3

The soils were treated separately with [chlorophenyl-U-^{14}C]mefentrifluconazole (C-label), [triazole-3(5)-^{14}C]mefentrifluconazole (T-label) or [trifluoromethylphenyl-ring-U-^{14}C] mefentrifuconazole (TFMP-label). The test materials were applied at a nominal rate of 150 g ai/ha. Soil was weighed into test vessels and placed into an incubation cabinet. The incubations were carried out in the dark in the laboratory under aerobic conditions at a soil moisture of 40 percent of the maximum water holding capacity and a temperature of 20 °C. A closed incubation system with continuous aeration (moistened air) was used with an attached trapping system for the determination of volatile compounds. During and at the end of the incubation, the microbial biomass was determined by the substrate induced respiration method, verifying that the soil was viable throughout the incubation period.

Samples from the C-, T-label and/or TFMP-label experiments were taken at 0, 3, 7, 14, 30, 58/62, 90/91 and 120/121 days after application (DAA). At each sampling time, two replicate soil samples were extracted twice with acetonitrile, twice with acetonitrile :water (80:20, v/v), and twice with acetonitrile :water (50:50, v/v). The individual extracts were analysed by liquid scintillation counting (LSC). The individual combined extracts were concentrated and analysed by radio-HPLC. The remaining soil after extraction was combusted in order to determine the amount of unextracted soil bound residues. These were further characterized by NaOH extraction and subsequent fractionation into fulvic acids, humic acids, and humins. A full material balance was provided for each sampling interval.

In the German soil samples treated with mefentrifluconazole radiolabelled in either the C-ring (LUFA 5M) or T-ring (LUFA 5M and L10), the amount of extracted radioactivity slightly decreased from an average of 99.0 percent and 99.1–99.5 percent TAR at day 0 to 82.4 percent and 81.9–86.0 percent TAR after 120/121 days of incubation for the respective labels. The material balance ranged from 98.8–100.5 percent TAR for both labels. The most prominent peak present in the extracts of the German soils consisted of the parent compound. During the course of the study, the amounts of parent compound in the total extracts slightly decreased from an average of 98.2 percent (C-label) and 98.9 percent (T-labels) TAR at day 0 to 80.8 percent and 81.2–83.5 percent TAR after 120/121 days of incubation. A number of metabolites were detected in low amounts, none of them exceeding 0.9 percent and 0.8 percent TAR at any sampling time for the C- and the T-labeled test materials, respectively. The metabolites 1,2,4-triazole and M750F003 were detected when the T-labeled test materials were applied, reaching maximum

amounts of about 0.5 percent TAR (121 DAAT; LUFA 5M soil) and 1.5 percent TAR (90 DAA; L10 soil) and 0.6 percent TAR (14 DAA; LUFA 5M soil) and 1.8 percent TAR (30 DAA; L10 soil).

In soil samples treated with the C-labeled material, unextracted soil residues increased from an average of 1.0 percent TAR on day 0 to a maximum of 12.7 percent TAR. In soil samples treated with the T-labeled material, unextracted soil residues increased from about 0.5–0.9 percent TAR at day 0 to about 12.6–17.9 percent TAR by the end of the study. About half of the unextracted residues could be attributed to the humin fraction. Mineralization was low and amounted to 4.7 percent (C-label) and 0.2–0.5 percent TAR (T-labels) at the end of the studies.

In the United States soil samples treated with mefentrifuconazole radiolabelled as either the C-label (New jersey), T-label (New Jersey and Indiana) or TFMP-label (New Jersey), the amount of extracted radioactivity decreased from an average of approximately 99 percent TAR at day 0 to 66–90 percent TAR at the end of the study, after 120/121 days of incubation. The material balance ranged from 95–103 percent TAR for all labels. The most prominent peak observed in the soil extracts was the parent compound, mefentrifluconazole declining from 98–99 percent TAR (day 0) to 63–87 percent TAR after 120/121 days. In soil extracts of samples treated with the T-labeled test material, the metabolite 1,2,4-(1H) triazole was detected at 0.2–0.8 percent TAR on day 0 and increased to a maximum of 5 percent TAR for the New Jersey soil and 1.2 percent TAR for the Indiana soil. Metabolite M750F003 was detected at a maximum level of 1.4 percent TAR (14 DAAT; New Jersey) and 0.6 percent TAR (58 DAT; Indiana). A number of other metabolites were also detected in very low amounts, none of them exceeding 1.4 percent (C-label) and 0.8–0.9 percent TAR (T-labels) at any sampling. For the TFMP-label, a number of metabolites were detected in low amounts, none of them exceeding 1.7 percent TAR at any sampling time. The metabolite M750F003 was the only metabolite identified reaching a maximum of 1.6 percent TAR at 30 DAA. The sum of metabolites in this soil sample never exceeded 2 percent TAR.

Compared to the German soil, unextracted residues in United States soil samples were formed in slightly higher amounts, increasing from an average of 0.8 percent (C-label) and 0.5 percent TAR (T-label and TFMP-label) on day 0 to a maximum of 19.5 percent TAR (C-label), 12.7–26.7 percent TAR (T-label) and 24 percent TAR (TFMP-label). About half of the unextracted residues could be attributed to the humin fraction. Mineralization was low to moderate and amounted to 9.7 percent (C-label), 0.3–0.5 percent TAR (T-labels) and 5.3 percent TAR (TFMP-label) at the end of the study.

Throughout the incubation period, R- and S-enantiomers of mefentrifluconazole (both labels) were almost equally present in the pooled acetonitrile as well as acetonitrile:water extracts of the German LUFA 5M and Indiana soils. In pooled extracts of New Jersey soil (both labels) and L10 soil (T-label), the ratio changed from an equal distribution of both enantiomers to a slightly higher ratio of the S-enantiomer of mefentrifluconazole at the end of the study (about 45:55 (R:S). 48:52 (R:S) and 46:54 (R:S)). Kinetic analysis and calculation of DT_{50} and DT_{90} values for mefentrifluconazole showed mefentrifluconazole to be persistent in soil (Table 36).

Table 36 DT_{50} and DT_{90} values in various soils in the United States

Soil	Label	Best-fit kinetic model	DT_{50}[A] (days)	DT_{90}[A] (days)
LUFA 5M	Chlorophenyl	DFOP	329	>1000
	Triazole	DFOP	356	>1000
L10	Triazole	DFOP	994	>1000
New Jersey	Chlorophenyl	DFOP	167	761
	Triazole	DFOP	>1000	>1000
	Trifluoromethylphenyl	DFOP	156	>1000
Indiana	Triazole	SFO	367	>1000

Notes:

DFOP: Double first-order in parallel.

SFO: Single first order.

[A] The predicted values were extrapolated well beyond the study duration and should be viewed with extreme caution.

A proposed degradation pathway of mefentrifluconazole in soil is shown in Figure 4.

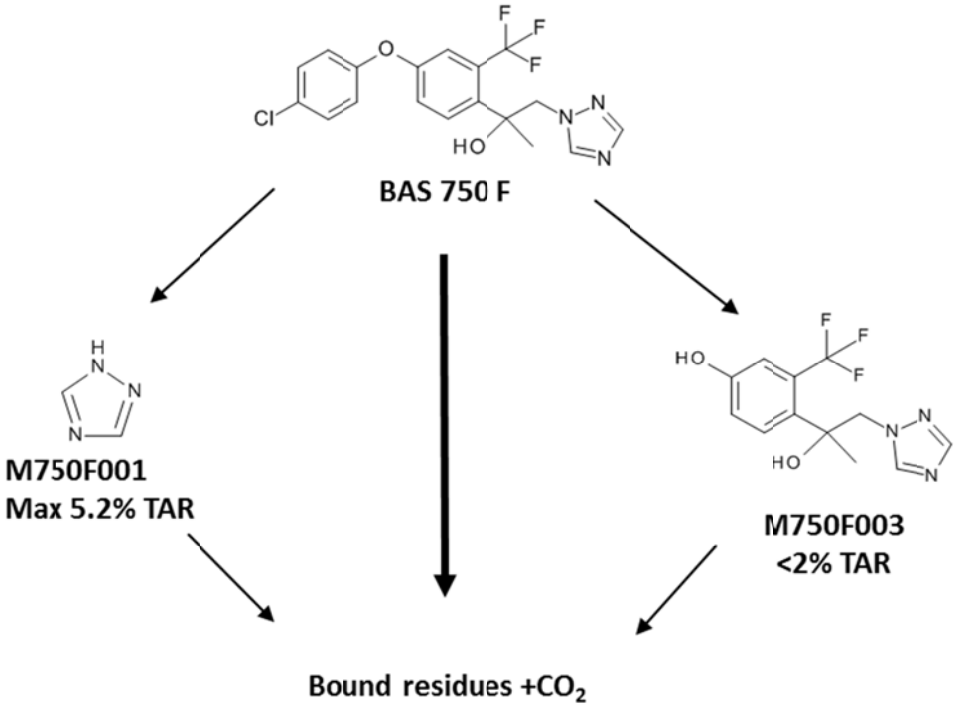


Figure 4 Degradation pathway of mefentrifluconazole in soil

Terrestrial field dissipation studies

United States

Terrestrial field dissipation studies were conducted in the United States at six different sites (Jacobson, 2016, BASFDocID 2015_7006396; Mitchell, 2017, BASF DocID 2016_7006418). Mefentrifluconazole formulated as an emulsifiable concentrate (EC) formulation (containing 100 g ai/L) was applied at the Illinois, North Dakota and Oklahoma sites, while at the New York, Washington, and California sites, the soluble concentrate (SC) formulation (containing 400 g ai/L) was applied. The target application rates, selected to represent a variety of proposed uses, were: 3 × 150 g ai/ha at 7-day intervals for the New York, North Dakota, Washington, and California sites; 3 × 200 g ai/ha at 14-day intervals for the Oklahoma site; and 2 × 150 g ai/ha at 7-day intervals at the Illinois site. Total application rates ranged from 300 g ai/ha for the Illinois site to 600 g ai/ha at the Oklahoma site while the rate at all other sites was 450 g ai/ha.

At each test site there were two test plots: one treated bare soil plot and one control bare soil plot. The treated plot at each test site was divided into three replicate subplots. The plots were kept in a bare soil condition throughout the study. The soil characteristics are shown in Table 37.

Table 37 Soil characteristics

	Soil segment							
	0-15 cm	15-30 cm	30-45 cm	45-60 cm	60-75 cm	75-90 cm	90-105 cm	105-120 cm
	New York soil							
Textural class (USDA scheme)	Silt loam	Silt loam	Silt loam	Silt loam	Silt loam	Silt loam	Silt loam	Silt loam
Soil texture (%)								
Sand	27	23	23	21	17	13	17	21
Silt	58	62	68	72	72	76	72	62
Clay	15	15	9	7	11	11	11	17
Organic matter (%)	4.3	2.7	1.09	0.31	0.31	0.22	0.17	0.22
pH	5.0	4.9	5.0	5.1	5.1	4.9	5.0	5.7
Cation exchange capacity (meq/100 g dry weight)	8.4	7.4	5.2	4.2	5.3	6.0	5.6	7.3
Moisture (gravimetric) at 1/3 bar (percent)	32.1	35.7	32.4	27.1	27.5	28.9	23.7	23.9
Moisture (gravimetric) at15 bar (percent)	9.8	8.7	5.5	3.7	5.5	6.2	5.4	6.2
	North Dakota soil							
Textural class (USDA scheme)	Clay	Clay	Clay	Clay	Sandy loam	Clay	Clay	Clay
Soil texture (%)								
Sand	30	34	42	40	56	38	32	44
Silt	29	19	17	13	9	15	17	9
Clay	41	47	41	47	35	47	51	47
Organic matter (%)	3.2	1.9	1.6	1.4	1.4	1.2	0.95	0.95
pH	7.5	7.6	7.7	7.8	7.8	8.0	7.9	8.0
Cation exchange capacity (meq/100 g dry weight)	33.9	36.5	34.9	33.3	33.0	31.1	30.2	34.1
Moisture (gravimetric) at 1/3 bar (%)	41.3	45.3	43.1	46.4	43.8	44.2	43.7	41.4
Moisture (gravimetric) at15 bar (%)	25.3	26.6	25.7	26.5	26.0	25.3	25.6	26.4
	Washington soil							
Textural class (USDA scheme)	Loamy sand	Sand	Sand	Loamy sand	Loamy sand	Sandy loam	Sandy loam	Sandy loam
Soil texture (%)								
Sand	86	88	90	84	76	70	64	68
Silt	11	11	9	15	21	29	33	29
Clay	3	1	1	1	3	1	3	3
Organic matter (%)	0.39	0.22	0.22	0.18	0.18	0.13	0.18	0.09
pH	8.3	8.2	8.2	8.2	8.1	8.1	8.4	8.4
Cation exchange capacity (meq/100 g dry weight)	7.6	8.0	8.1	8.9	9.6	10.7	13.7	14.6
Moisture (gravimetric) at 1/3 bar (%)	8.1	7.4	7.0	9.4	12.0	15.3	19.3	20.1
Moisture (gravimetric) at15 bar (%)	4.0	4.0	4.0	4.4	4.7	4.6	4.9	5.1
	California soil							
Textural class (USDA scheme)	Loamy sand	Sand	Sand	Loamy sand	Loamy sand	Sandy loam	Sandy loam	Sandy loam
Soil texture (%)								

	Soil segment							
	0-15 cm	15-30 cm	30-45 cm	45-60 cm	60-75 cm	75-90 cm	90-105 cm	105-120 cm
Sand	74	76	76	74	76	80	80	82
Silt	22	20	20	22	22	18	18	16
Clay	4	4	4	4	2	2	2	2
Organic matter (%)	0.70	0.48	0.31	0.13	0.13	0.09	0.26	0.31
pH	7.6	8.0	8.3	8.3	8.3	8.3	8.5	8.4
Cation exchange capacity (meq/100 g dry weight)	9.5	10.0	9.9	10.1	10.1	10.3	10.0	9.9
Moisture (gravimetric) at 1/3 bar (%)	11.2	10.6	11.7	11.9	11.1	11.3	9.4	10.0
Moisture (gravimetric) at 15 bar (%)	4.1	3.9	4.1	4.1	4.0	4.0	3.5	3.8
	Oklahoma soil							
Textural class (USDA scheme)	Sandy loam	Sandy loam	Sandy loam	Loam	Loam	Loam	Loam	Sandy clay loam
Soil texture (%)								
Sand	59	59	53	47	43	39	47	55
Silt	26	24	28	32	34	34	30	24
Clay	15	17	19	21	23	27	23	21
Organic matter (%)	0.67	0.97	0.76	0.71	0.63	0.63	0.50	0.42
pH	7.1	5.8	5.8	6.3	6.4	6.7	6.8	7.0
Cation exchange capacity (meq/100 g dry weight)	7.8	8.2	9.2	9.6	11.0	13.2	12.1	11.8
Moisture (gravimetric) at 1/3 bar (%)	10.8	12.8	14.7	17	20.4	23.1	20.0	18.9
Moisture (gravimetric) at15 bar (%)	5.0	6.1	7.0	7.4	9.0	10.9	10.1	9.6
	Illinois soil							
Textural class (USDA scheme)	Silty clay loam	Silty clay loam	Silty clay loam	Silty clay	Silty clay loam	Silty clay loam	Silty clay loam	Clay loam
Soil texture (%)								
Sand	15	9	9	11	15	7	13	23
Silt	52	52	56	46	48	54	52	46
Clay	33	39	35	43	37	39	35	31
Organic matter (%)	4.3	3.3	1.8	0.82	0.56	0.56	0.47	0.34
pH	6.0	6.0	.3	6.6	6.8	7.0	7.2	7.4
Cation exchange capacity (meq/100 g dry weight)	18.9	21.0	23.0	23.8	22.6	21.6	19.0	16.6
Moisture (gravimetric) at 1/3 bar (%)	33.9	34.9	36.8	39.8	37.9	35.9	33.5	29.6
Moisture (gravimetric) at15 bar (%)	14.0	15.8	19.0	22.0	18.1	17.3	15.7	12.8

Rainfall was supplemented with irrigation at all six of the test sites. At each sampling event, five cores to a depth of 120 cm were taken from one sampling block in each replicate area (subplot). This results in 15 cores being collected during a sampling event (except on days of application when a duplicate set of 0–8 cm cores were taken). Soil cores were sampled prior to and immediately after each test substance application and 2/3, 7, 15/18, 30/32, 58–61, 82–90, 162–183 (209 for Illinois site), 266–

294, 346–391, 579–592, 602–693 DALA. Soil cores were sectioned into segments of 0–15, 15–30, 30–45, 45–60, 60–75, 75–90, 90–105 and 105–120 cm.

The samples were analysed for mefentrifluconazole and the metabolites M750F003 and 1,2,4-triazole (not further discussed herein) using the LC-MS/MS method L0214/01. The validated LOQ and LOD were 0.002 mg/kg and 0.0004 mg/kg, respectively. Soil samples were stored for up to 132 days (4.3 months) prior to analysis. A freezer storage stability study using soil from each site to support the storage conditions of incurred samples is in progress. The results are shown in Table 38.

Table 38 Calculated [A] average residues of mefentrifluconazole (g ai/ha) in the 0–45 and 0–120 cm sampled soil profiles

DAFA	DALA	New York		DAFA	DALA	North Dakota	
		0-45 cm	0-120 cm			0-45 cm	0-120 cm
-1	-	0.00	0.00	-1	-	0.00	0.00
0	-	124	124	0	-	66	66
6	-	49	49	6	-	86	86
7	-	130	130	7	-	138	138
13	-	55	55	13	-	120	120
14	0	141	141	14	0	259	352
17	3	127	127	17	3	165	165
21	7	107	107	21	7	290	291
29	15	98	98	32	18	209	309
44	30	68	68	46	32	199	277
74	60	92	92	75	61	163	207
104	90	94	99	104	90	133	134
195	181	125	125	Not sampled			
280	266	429	429	308	294	141	141
405	391	57	57	403	389	86	86
524	510	0.00	0.00	Not sampled			
644	630	42	43	635	621	120	134
720	706	89	89	675	661	53	53
DAFA	DALA	Washington		DAFA	DALA	California	
		0-45 cm	0-120 cm			0-45 cm	0-120 cm
-5	-	0.00	0.00	-1	-	0.00	0.00
0	-	110	110	0	-	160	160
6	-	63	63	6	-	62	67
7	-	147	14	7	-	241	253
13	-	123	123	13	-	120	138
14	0	231	231	14	0	243	268
17	3	179	179	17	3	133	140
21	7	197	197	21	7	135	146
29	15	195	195	29	15	106	130
44	30	204	204	44	30	164	184
73	59	170	215	74	60	102	104
104	90	149	156	96	82	119	122
176	162	141	141	194	180	142	142
283	269	147	147	284	279	77	77
399	385	50	50	374	360	46	46
521	507	48	48	523	509	30	30
644	630	82	82	606	592	17	17
707	693	49	49	647	633	13	13
DAFA	DALA	Oklahoma		DAFA	DALA	Illinois	
		0-45 cm	0-120 cm			0-45 cm	0-120 cm
-1	-	0.00	0.00	-15	-22	0.00	0.00
0	-	121	121	0	-7	106	106

DAFA	DALA	New York		DAFA	DALA	North Dakota	
		0-45 cm	0-120 cm			0-45 cm	0-120 cm
13	-	99	135	6	-1	161	161
14	-	273	293	7	0	302	302
27	-	184	221	10	3	230	230
28	0	271	555	14	7	228	228
30	2	294	601	22	15	214	214
35	7	301	456	37	30	174	175
43	15	320	320	Not sampled			
58	30	257	257	68	61	252	252
86	58	287	287	93	86	68	68
118	90	332	332	Not sampled			
211	183	221	221	216	209	79	79
302	274	158	158	278	271	51	51
374	346	84	84	377	370	28	28
542	514	116	116	586	579	7	27
612	584	101	101	609	602	91	91
654	626	71	71	645	638	48	48

Notes:

DAFA = days after first application.

DALA = days after last application.

– Means not applicable.

[A] The mass per area values were calculated by multiplying the equivalent dry weight mass of soil in one hectare for each core segment by the analytical dry weight concentration of the analyte.

The total mass of mefentrifluconazole in the entire sampled soil profile (0–120 cm) as well as the total mass in the upper soil profile only (0–45 cm) were assessed. All kinetic analyses were conducted from the day of the last application onward. When assessing dissipation of the total mass of mefentrifluconazole in the entire 0–120 cm sample profile, the mefentrifluconazole DT_{50} values ranged from 33 to 315 days. DT_{50} values could not be determined at the New York test site due to high variability. Dissipation of the total mass of mefentrifluconazole in the upper 0–45 cm of the soil profile, which was proposed to better represent the actual dissipation behaviour of mefentrifluconazole, gave DT_{50} values of 81–386 days (Table 39). Given the low levels of M750F003 formed, kinetic assessment for the metabolite was not attempted.

Table 39 DT_{50}s of Mefentrifluconazole from field dissipation studies conducted in the United States

Field trial	Soil type (USDA)	Best-fit kinetic model	DT_{50} (days)
New York, bare plot	Data from the last 3 sampling events did not fit a data trend, therefore, it was not possible to obtain a reliable DT_{50} from this trial site.		
North Dakota, bare plot	Clay	SFO	386
Washington, bare plot	Loamy sand	SFO	317
California, bare plot	Loamy sand	SFO	230
Oklahoma, bare plot	Sandy loam	SFO	318
Illinois, bare plot	Silty clay loam	FOMC	81

In a separate study, mefentrifluconazole, formulated as a suspension concentrate (containing 400 g ai/L) was applied three times to bare-ground and turf-cropped plots at test sites located in California and Georgia (White, 2017, BASF DocID 2016_7006417). Mefentrifluconazole was applied at a target rate of 1 kg ai/ha for a total of 3 kg ai/ha. Re-treatment intervals varied between 14–18 days.

The study design consisted of a treated bare-soil plot, a treated turf-cropped plot, and an

untreated bare-soil control plot at each test site. The treated plots at each test site were divided into three replicate areas for soil sampling purposes. The soil characteristics are shown in Table 40.

Table 40 United States soil characteristics

	Soil segment							
	0-15 cm	15-30 cm	30-45 cm	45-60 cm	60-75 cm	75-90 cm	90-105 cm	105-120 cm
Soil designation	California – Bare soil plot							
Textural class (USDA scheme)	Sandy loam	Sandy loam	Loamy sand	Loamy sand	Sandy loam	Sandy loam	Sandy loam	Sandy loam
Soil texture (%)								
Sand	74	74	76	76	70	68	66	68
Silt	18	20	20	20	26	28	28	26
Clay	8	6	4	4	4	4	6	6
Organic matter (%)	0.62	0.1	0.13	0.09	0.13	0.09	0.13	0.18
pH	6.9	7.6	8.0	8.1	8.2	8.2	8.2	8.2
Cation exchange capacity (meq/100 g dry weight)	10.2	9.5	9.2	9.8	10.5	10.2	10.4	9.9
Moisture (gravimetric) at 1/3 bar (%)	14.2	11.5	10.4	11.3	12.8	13.8	16.3	16.1
Moisture (gravimetric) at15 bar (%)	5.4	4.6	4.3	4.4	4.6	4.9	5.5	4.7
Soil designation	California – Turf cropped soil							
	0-15 cm	15-30 cm	30-45 cm	45-60 cm	60-75 cm	75-90 cm	90-105 cm	105-120 cm
Textural class (USDA scheme)	Loamy sand	Sandy loam	Sandy loam	Sandy loam	Sandy loam	Sandy loam	Sandy loam	Sandy loam
Soil texture (%)								
Sand	76	70	70	72	66	60	68	66
Silt	18	26	26	24	30	34	28	28
Clay	6	4	4	4	4	6	4	6
Organic matter (%)	2.43	0.22	0.09	0.09	0.09	0.09	0.09	0.09
pH	8.3	8.4	8.7	8.5	8.9	8.9	9.0	9.0
Cation exchange capacity (meq/100 g dry weight)	8.0	8.1	8.4	9.3	10.3	10.3	10.2	8.7
Moisture (gravimetric) at 1/3 bar (%)	13.2	11.3	11.4	12.7	6.6	19.4	18.9	16.5
Moisture (gravimetric) at15 bar (%)	5.7	4.6	4.4	4.6	5.5	6.0	5.7	4.9
	Georgia – Bare soil plot							
Textural class (USDA scheme)	Sand	Sand	Sandy loam	Sandy loam	Sandy clay loam	Sandy clay loam	Sandy clay loam	Sandy clay loam
Soil texture (%)								
Sand	93	89	77	75	73	69	67	65
Silt	4	6	8	6	4	6	6	6
Clay	3	5	5	19	23	25	27	29
Organic matter (%)	0.57	0.57	0.35	0.31	0.09	0.00	0.00	0.00
pH	6.7	6.2	6.2	5.8	5.5	5.1	5.0	4.7
Cation exchange capacity (meq/100 g dry weight)	3.8	4.0	5.2	5.8	6.2	6.1	6.4	6.4
Moisture (gravimetric) at 1/3 bar (%)	5.0	5.4	10.0	13.1	15.1	16.4	16.9	16.8
Moisture (gravimetric) at15 bar (%)	2.1	2.8	6.3	9.3	10.9	13.1	12.9	12.4
	Georgia – Turf cropped soil							

	Soil segment							
	0-15 cm	15-30 cm	30-45 cm	45-60 cm	60-75 cm	75-90 cm	90-105 cm	105-120 cm
Textural class (USDA scheme)	Sand	Sand	Sandy loam	Sandy loam	Sandy clay loam	Sandy clay loam	Sandy clay loam	Sandy clay loam
Soil texture (%)								
Sand	91	91	81	75.	67	69	65	63
Silt	6	6	6	6	8	6	4	4
Clay	3	3	13	19	25	25	31	33
Organic matter (%)	0.92	0.75	0.35	0.00	0.70	0.13	0.18	0.18
pH	6.5	6.4	6.3	6.1	5.6	5.6	5.0	4.8
Cation exchange capacity (meq/100 g dry weight)	3.8	3.7	5.3	5.7	6.0	6.2	6.6	7.1
Moisture (gravimetric) at 1/3 bar (%)	5.0	4.8	9.2	13.9	5.5	17.4	18.7	20.4
Moisture (gravimetric) at15 bar (%)	2.2	2.2	6.1	10.3	10.5	12.	13.8	14.5

At each site overhead sprinkler irrigation was used to supplement natural precipitation. Soil core samples were taken from the soil surface to a depth of 120 cm at various times prior to and after each application from each replicate subplot and 0, 3, 7, 14/15, 30/32, 59, 83/87, 111, 180/182, 268/272, 386/392, 511/512, 632/634 and 667/678 days after the last application (DALA). Collected soil cores were sectioned into segments of 0–15, 15–30, 30–45, 45–60, 60–75, 75–90, 90–105 and 105–120 cm.

The samples were analysed for mefentrifluconazole and the metabolites M750F003 and 1,2,4-triazole (not further discussed herein) using the LC-MS/MS method D1503/01. The validated LOQ and LOD were 0.002 mg/kg and 0.0004 mg/kg, respectively.

Soil samples were stored for up to 24 months prior to analysis. Results from a soil storage stability study using fortified control soils from the California and Georgia test sites indicate that mefentriflconazole and M750F003 are stable for at least 21 months (Table 41).

Table 41 Calculated[A] average residues of mefentrifluconazole and M750F003 (g ai/ha) in the 0–45 and 0–120 cm sampled soil profiles

		Mefentrifluconazole		M750F003 (BASF equivalents)	
		0-45 cm	0-120 cm	0-45 cm	0-120 cm
DA1A	DALA	California – Bare soil			
-1	-	0.00	0.00	0.00	0.00
0	-	564	564	0.11	0.11
18	-	452	452	2.9	2.9
18	-	1396	1437	5.9	5.9
31	-	858	877	6.5	6.5
32	0	1657	1703	5.5	5.5
35	3	1083	1391	11	11
39	7	1118	1411	10	10
47	15	1102	1517	12	12
62	30	948	973	14	14
115	83	621	743	3.5	3.5
143	111	101	1091	4.2	4.2
212	180	425	425	3.8	3.8
300	268	1072	1076	5.9	5.9
418	386	398	414	4.1	4.1
544	512	280	287	2.3	2.3

		Mefentrifluconazole		M750F003 (BASF equivalents)	
		0-45 cm	0-120 cm	0-45 cm	0-120 cm
664	632	168	168	1.8	1.8
699	667	285	285	2.2	2.2
DA1A	DALA	California – Turf cropped			
-1	-	0.00	0.00	0.00	0.00
0	-	332	332	0.00	0.00
18	-	244	244	3.0	3.0
18	-	785	808	4.0	4.0
31	-	487	488	6.6	6.6
32	0	1078	1090	7.9	7.9
35	3	755	845	11	11
39	7	900	901	12	12
47	15	686	688	12	12
62	30	1198	1237	12	12
115	83	835	835	13	13
143	111	732	732	14	14
212	180	509	509	17	17
300	268	500	500	15	15
418	386	115	116	6.4	6.4
544	512	64	64	5.1	5.1
664	632	28	29	2.4	2.4
699	667	21	21	1.3	1.3
DA1A	DALA	Georgia – Bare soil			
-1	-	0.00	0.00	0.00	0.00
0	-	475	475	1.5	1.5
13	-	311	311	5.2	5.2
14	-	773	774	6.	6.1
27	-	580	580	11	11
28	0	1056	1056	15	15
31	3	748	748	13	13
35	7	606	606	16	16
42	14	497	497	14	14
60	32	526	526	13	13
87	59	362	363	13	13
115	87	321	321	9.9	10
210	182	314	314	11.1	11
300	272	382	382	11.2	11
420	392	224	224	4.5	4.5
539	511	173	173	4.4	4.4
662	634	174	174	3.7	3.7
706	678	151	151	2.9	2.9
DA1A	DALA	Georgia – Turf cropped			
-1	-	0.00	0.00	0.00	0.00
0	-	457	457	0.00	0.00
13	-	549	549	9.8	9.5
14	-	331	331	9.4	9.4
27	-	752	752	18	18
28	0	1678	1678	25	25
31	3	877	877	14	14
35	7	966	966	21	21
42	14	958	958	23	23
60	32	1522	1522	46	46
87	59	817	817	24	24
115	87	580	580	20	20
210	182	569	569	17	17

		Mefentrifluconazole		M750F003 (BASF equivalents)	
		0-45 cm	0-120 cm	0-45 cm	0-120 cm
300	272	344	344	20	20
420	392	60	60	4.3	4.3
539	511	21	21	1.6	1.6
662	634	13	13	0.81	0.81
706	678	9.5	9.5	0.78	0.78

Notes:

DA1A = days after first application.

DALA = days after last application.

– Means not applicable.

[A] The mass per area values were calculated by multiplying the equivalent dry weight mass of soil in one hectare for each core segment by the analytical dry weight concentration of the analyte in that.

The total mass of mefentrifluconazole in the entire sampled soil profile (0–120 cm) at each sampling event was used in the initial kinetic assessment. However, the distribution of mefentrifluconazole residue with depth and time did not appear to follow a typical pattern. Specifically, the residue concentration decreased quickly with depth, but then increased again in the lower half of the core, with highest subsoil concentrations typically observed in the deepest segment, 105–120 cm. Cross-contamination during processing of the 0–120 cm samples was likely the cause. Therefore, only the upper 0–45 cm of the soil profile was used to derive the kinetic dissipation kinetics for mefentrifluconazole.

Kinetic models were not fit to the metabolite M750F003 data due to the low and/or variable levels observed (Table 42).

Table 42 DT_{50}s of Mefentrifluconazole from Field Dissipation Studies Conducted in the United States

Field trial	Soil type (USDA)	0-45 cm		0-120 cm	
		Best-fit kinetic model	DT_{50} (days)	Best-fit kinetic model	DT_{50} (days)
California – bare soil	Sandy loam	SFO	269	FOMC	120
California – turf cropped soil	Sandy loam	SFO	192	SFO	187
Georgia – bare soil	Sand	DFOP	10	DFOP	10
California – turf cropped soil	Sand	DFOP	59	DFOP	59

Europe

Six field dissipation studies were conducted in representative growing regions of Northern and Southern Europe including Germany (2), Denmark, Northern France, Italy and Spain (Schaufele, 2015, BASF DocID 2015_1046920; Schaufele, 2015, BASF DocID 2015_1242234; Studenroth, 2015, BASF DocID 2015_1249176). An emulsifiable concentrate formulation containing 100 g/L of mefentrifluconazole was applied to bare soil at rates ranging from 152–166 g ai/ha. Immediately after application, plots were covered by a layer of sand of approximately 5 cm in thickness to protect the applied product from surface processes such as volatilization or photolysis, and to exclude any potential impact on the degradation of the test material caused by any of these processes.

Soil specimens were taken at intervals of up to 720 days after application and to a depth of 50 cm. Soil cores were cut into 10 cm sections.

Table 43 European soil characteristics

Soil designation	Bogense, Denmark		Lentzke, Germany		Goch-Nierswalde, Germany	
	0-30 cm	30-50 cm	0-35 cm	35-50 cam	0-30 cm	30-50 cm
Textural class (USDA scheme)	Sandy loam	Sandy loam	Loamy sand	Sandy loam	Silt loam	Loam
Soil texture (%)						
Sand	73.3	71.5	80.0	76.3	39.0	49.5
Silt	15.3	16.0	12.0	12.3	51.5	41.2
Clay	11.4	12.5	7.9	11.5	9.6	9.3
Organic matter (%)	1.8	0.9	1.2	0.4	2.8	0.5
pH (H_2O)	6.4	7.4	5.4	4.5	6.5	6.0
pH ($CaCl_2$)	6.9	7.9	5.9	5.4	7.1	6.7
Cation exchange capacity (meq/100 g dry weight)	7.2	7.0	3.8	2.6	10.2	3.8
Max water holding capacity (g/100 g dry soil)	32.8	28.8	22.6	19.7	39.0	26.2

Soil designation	Stotzheim, France (North)		Poggio, Renatico, Italy		Uterera, Spain		
	0-30 cm	30-50 cm	0-30 cm	30-50 cam	0-20 cm	20-40 cm	40-50 cm
Textural class (USDA scheme)	Silty clay loam	Silty clay loam	Silty clay loam	Silty clay loam	Loamy sand	Loamy sand	Sandy clay
Soil texture (%)							
Sand	13.6	13.2	15.6	14.5	87.9	83.1	58.5
Silt	57.6	51.5	49.8	49.2	3.8	4.9	5.0
Clay	29.2	35.2	34.6	36.2	8.3	12.1	36.5
Organic matter (%)	1.4	1.2	1.8	1.9	0.7	0.4	0.6
pH (H_2O)	7.4	7.6	7.6	7.6	7.4	7.0	6.7
pH ($CaCl_2$)	8.0	8.3	8.3	8.2	7.9	7.6	7.1
Cation exchange capacity (meq/100 g dry weight)	14.4	16.4	17.0	17.3	3.5	4.0	21.0
Max water holding capacity (g/100 g dry soil)	41.7	41.0	44.4	47.3	28.8	34.3	46.2

Rainfall was supplemented with irrigation, when necessary. Soil samples were stored for up to 20 months prior to analysis. Results from a soil storage stability study using fortified control soils indicate that mefentriflconazole and M750F003 are stable for at least 22 months.

Soil specimens were analysed for residues of mefentrifluconazole, M750F003 and 1,2,4-triazole (not further discussed herein) according to the LC-MS/MS method L0214/01. The results are shown in Table 44. The method involved extraction of the soil using acetonitrile:water (70:30). Field soil specimens from the treated plot were analysed to a depth until at least one soil layer was free of detectable residues (<LOD of 0.0004 mg/kg).

Table 44 Calculated[1] average residues of mefentrifluconazole (g ai/ha) in the individual European soil layers

	Soil depth (cm)							Soil depth (cm)					
DAA	0-10	10-20	20-30	30-40	40-50	Total	DAA	0-10	10-20	20-30	30-40	40-50	Total
	Lentzke, Germany							Goch-Nierswalde, Germany					
0	121	*	*	*	*	121	0	121	*	*	*	*	121
6	131	0	0	**	**	131	7	137	0	0	**	**	137
14	115	0	0	**	**	115	13	121	0	0	**	**	121
33	107	0	0	**	**	107	27	82	0	0	**	**	82
56	108	0	0	**	**	108	59	120	0	0	**	**	120
85	92	0	0	**	**	92	95	101	0	0	**	**	101

	Soil depth (cm)							Soil depth (cm)					
DAA	0-10	10-20	20-30	30-40	40-50	Total	DAA	0-10	10-20	20-30	30-40	40-50	Total
118	93	0	0	**	**	93	125	91	0	0	**	**	91
176	86	0	0	**	**	86	185	94	0	0	0	**	94
272	85	0	0	**	**	85	248	91	4.9	0	0	**	96
355	39	0	0	**	**	39	361	31	0	0	0	**	31
476	42	0	0	**	**	42	474	18	0	0	**	**	18
590	48	0	0	**	**	48	613	16	0	0	**	**	16
715	34	0	0	**	**	34	710	28	0	0	**	**	28
DAA	Stotzheim, France (North)						DAA	Poggio Renatico, Italy					
0	133	*	*	*	*	133	0	116	*	*	*	*	116
7	115	0	0	**	**	115	7	137	0	0	**	**	137
14	122	0	0	**	**	122	13	105	0	0	**	**	105
30	118	0	0	**	**	118	29	121	0	0	**	**	121
62	93	0	0	**	**	93	56	114	0	0	**	**	114
91	69	0	0	**	**	69	90	102	0	0	**	**	102
120	62	2.7	0	**	**	65	120	95	0	0	**	**	95
175	61	4.6	0	**	**	66	183	97	0	0	0	0	97
238	57	0	0	**	**	57	285	120	0	0	**	**	120
366	53	0	0	**	**	53	351	80	0	0	0	**	80
471	25	0	0	**	**	25	475	72	0	0	**	**	72
591	19	0	0	**	**	19	600	68	0	0	**	**	68
720	16	0	0	**	**	16	714	74	0	0	**	**	74
DAA	Utrera, Spain						DAA	Bogense, Denmark					
0	132	*	*	*	*	132	0	108	*	*	*	*	108
6	117	0	0	**	**	117	6	124	0	**	**	**	124
13	107	0	0	**	**	107	13	121	3.3	**	**	**	124
29	98	0	0	**	**	98	29	122	4.3	0	**	**	126
58	79	0	0	**	**	79	61	81	0	**	**	**	81
92	95	0	0	**	**	95	92	83	4.3	0	**	**	87
127	74	0	0	**	**	74	124	68	4.5	0	**	**	72
183	65	0	0	**	**	65	174	69	0	0	**	**	69
230	66	0	0	**	**	66	245	50	0	0	**	**	50
353	52	0	0	**	**	52	363	35	0	0	**	**	35
478	42	0	0	**	**	42	487	13	0	0	**	**	13
591	22	0	0	**	**	22	615	14	0	0	**	**	14
713	20	0	0	**	**	20	713	8	0	0	**	**	8

Notes:

[1] Residue values of mefentrifluconazole in mg/kg dry soil were converted to residue rates in g/ha taking into account the actual dry soil density of the individual field samples. These were summed up for all depths between 0 and 50 cm. Residue values <LOQ or <LOD were reported and treated as zero

DAA Days after application

* No sample taken

** Sample not analysed

Residues of mefentrifluconazole degraded at all six European field sites from 108–133 g ai/ha at day 0 to 8–74 g ai/ha within 2 years. Considering the distribution of mefentrifluconazole residues in the soil profiles, residues remained predominantly and exclusively in the top 0–10 cm layer of soils. No residues above the LOQ were detected below 20 cm. Residues of the metabolite M750F003 were not detected. The calculated DT_{50}s are shown in Table 45.

Table 45 DT_{50} of mefentrifluconazole from field dissipation studies conducted in Europe

Field trial	Soil type (USDA)	Best-fit kinetic model	DT_{50} (days)	Kinetic model	Normalized[A] DT_{50} (days)
Bogense, Denmark	Sandy loam	SFO	186	SFO	96
Lentzke, Germany	Loamy sand	SFO	351	SFO	184
Nierswalde, Germany	Silt loam	SFO	268	SFO	147
Stotzheim, France, North	Silty clay loam	DFOP	145	SFO	129
Poggio Renatico, Italy	Silty clay loam	SFO	847[B]	SFO	611
Utrera, Spain	Loamy sand	DFOP	200[C]	SFO	313

Notes:

[A] Normalised for soil moisture and soil temperature. The predicted DT_{50} value was extrapolated well beyond the study duration and should be viewed with extreme caution. Endpoint was derived with the initial concentration fixed to the mean of the measured values.

China

Terrestrial field dissipation studies were conducted at three sites in China representative of Northern, Central and Southern China (Chenchao, 2019, BASF DocID 2019_2047604; Shbaita, 2019, BASF DocID 2019_8000062). Mefentrifluconazole formulated as a suspension concentrate (SC) formulation (containing 400 g ai/L) was applied once to bare soil at rates of 414–484 g ai/ha.

At each test site there were two test plots: one treated bare soil plot and one control bare soil plot. The treated plot at each test site was divided into two subplots. The plots were kept in a bare soil condition throughout the study. Soil specimens were taken at intervals up to 185 days after application ad to maximum depth of 30 cm. Soil cores were cut into 10 cm sections.

Table 46 Chinese soil characteristics

Soil Designation	Hebei, Langfang	Jiling, Changchun	Guangxi, Nanning
Textural class (USDA scheme)	Loam	Clay loam	Clay
Soil texture (%)			
Sand	41	40	18
Silt	33	32	20
Clay	26	28	62
Organic matter (%)	1.69	3.45	4.31
pH	7.41	6.9	4.77
Cation exchange capacity (meq/100 g dry weight)	14.22	33.2	9.96

Soil samples were stored for up to 20 months prior to analysis. Results from a soil storage stability study using fortified control soils indicate that mefentriflconazole and M750F003 are stable for at least 22 months.

Soil specimens were analysed for residues of mefentrifluconazole according to the LC-MS/MS method L0214/01. The method involved extraction of the soil using acetonitrile:water (70:30). The validated LOQ was 0.015 mg/kg. The residues are shown in Table 47 and the DT_{50} in Table 48.

Table 47 Calculated[A] average residues of mefentrifluconazole (g ai/ha) in the individual soil layers

	Soil depth (cm)			
	0-10	10-20	20-30	Total
DAA	Hebei, Langfang			
0	281	-	-	281
6	234	0	0	234
16	180	0	0	180

	Soil depth (cm)			
	0-10	10-20	20-30	Total
30	195	0	0	195
61	86	0	0	86
88	85	0	0	85
11	109	0	0	109
153	88	0	0	88
179	77	0	0	77
DAA	Jilin, Changchun			
0	339	-	-	339
8	280	0	0	280
15	236	0	0	236
30	231	0	0	231
63	194	0	0	194
94	152	0	0	152
122	143	0	0	143
150	182	0	0	182
185	180	0	0	180
DAA	Nannimg, Guangxi			
0	246	-	-	281
7	231	0	0	234
14	162	0	0	180
30	178	0	0	195
61	142	0	0	86
93	92	0	0	85
121	86	0	0	109
149	76	0	0	88
181	56	0	0	77

Notes:

[A] Residue values of mefentrifluconazole in mg/kg dry soil were converted to residue rates in g/ha taking into account the actual dry soil density of the individual field samples. These were summed up for all depths between 0 and 50 cm. Residue values <0.015 mg/kg were reported and treated as zero.

DAA = Days after application.

Table 48 DT_{50} of mefentrifluconazole from field dissipation studies conducted in China

Field trial	Soil type (USDA)	Best-fit kinetic model	DT_{50} (days)	Kinetic model	Normalized[A] DT_{50} (days)
Hebei, Langfang	Sandy loam	SFO	76	SFO	118
Jilin, Changchun	Loamy sand	FOMC	110	SFO	320
Nannimg, Guangxi	Clay	SFO	106	SFO	225

Notes:

[A] Normalised for soil moisture and soil temperature

Summary of soil dissipation studies

The dissipation of mefentrifluconazole under field conditions has been studied in the field in eight studies in the United States (bare soil only), six studies in Europe and three studies in China. In summary, quantifiable residues of mefentrifluconazole were detected only in the first 20 cm of the soils. No residues above the LOQ were detected below 20 cm in any sample at any site. The summary is shown in Table 49. The overall geometric mean (non-normalised) DT_{50} was estimated to be 149 days.

Table 49 Summary of DT_{50} values for mefentrifluconazole

Field trial	Soil type (USDA)	Best-fit kinetic model	DT_{50} (days)
New York, bare plot	Data from the last 3 sampling events did not fit a data trend, therefore, it was not possible to obtain a reliable DT_{50} from this trial site.		
North Dakota, bare plot	Clay	SFO	386
Washington, bare plot	Loamy sand	SFO	317
California, bare plot	Loamy sand	SFO	230
Oklahoma, bare plot	Sandy loam	SFO	318
Illinois, bare plot	Silty clay loam	FOMC	81
California, bare soil	Sandy loam	FOMC	120
Georgia, bare soil	Sand	DFOP	10
Bogense, Denmark	Sandy loam	SFO	186
Lentzke, Germany	Loamy sand	SFO	351
Nierswalde, Germany	Silt loam	SFO	268
Stotzheim, France (North)	Silty clay loam	DFOP	145
Utrera, Spain	Loamy sand	DFOP	200
Hebei, Langfang	Sandy loam	SFO	76
Jilin, Changchun	Loamy sand	FOMC	110
Nannimg, Guangxi	Clay	SFO	106
		Maximum	386
		Geometric mean	149

Hydrolysis

The hydrolytic stability of [triazole-3(5)-^{14}C]-labelled mefentrifluconazole in buffered solutions at pH 4, 5, 7 and 9 for a study period of 30 days at 25 ºC was investigated (Hassink, 2015, BASF DocID 2015_1046919). No hydrolysis of the parent was observed as 98–101 percentTAR was still present in the test systems after the duration of incubation. No degradation products at greater than 2 percent TAR were identified, nor was there any evidence of a change in the isomer ratio of the test material. As mefentrifluconazole is stable in aqueous solutions at environmentally relevant pHs, half-lives could not be estimated.

Soil Photolysis

German soil (LUFA 5M; the same as the aerobic soil degradation study) was treated with [chlorophenyl-U-^{14}C] (C-label) and [triazole-3(5)-^{14}C]-labelled mefentrifluconazole (T-label) at an application rate of 150 g/ha (Hassink, 2014, BASF DocID 2014_1181666). Soil samples were placed under continuous irradiation. The incubation temperature was kept at 22 °C and the soil moisture was adjusted daily to approximately 60 percent of the maximum water holding capacity corresponding to pF 2.0–2.5. The samples for the dark control were kept in an incubation cabinet and treated in a similar manner.

Soil samples were collected at 0, 1, 3, 6/7, 10 and 15 days after application (DAA). The samples were extracted three times with acetonitrile and twice with acetonitrile/water (1:1, v/v). The extracts were analysed for total radioactivity and if the TAR was greater than 5 percent, the extracts were subjected to HPLC analysis. The soil remaining after extraction was extracted three times with NaOH and if the TAR was greater than 5 percent, the amount of alkali-soluble components was determined. The dried soil was then combusted, to determine the amount of unextracted residues.

The overall values for the material balance in the photolysis and the dark control were in the range of 98–105 percent TAR. Carbon dioxide was the only trapped volatile degradation product found in the trapping solutions. After 15 days of treatment the following amounts were detected: for the C-label, 1.1 percent TAR in the irradiated samples and 0.4 percent TAR in the dark control; T-label, 0.3 percent

TAR in the irradiated samples and 0.1 percent TAR in the dark control. The amount of extracted radioactive residues in the study with the C-label test material decreased from 99 percent TAR on day 0 to 92 percent TAR on day 15 in the photolysis test, and from ~100 percent TAR on day 1 to 96 percent TAR on day 15 in the dark control. Similarly, in the study with the T-label test material, extracted residues decreased from 99 percent TAR on day 0 to 97 percent TAR on day 15 in the photolysis test, and from 102 percent TAR to 99 percent TAR in the dark control.

At the end of the study about 5 percent TAR for the C-label and 7 percent TAR for the T-label remained unextracted in the irradiated soil samples. About 5 percent TAR was unextracted at the end of incubation of the dark control samples, for both radiolabels. The alkali-soluble radioactivity in the irradiated samples amounted to 2.3 percent TAR and 4.2 percent TAR, for the C-label and the T-label respectively, representing the fulvic acids and the humic acids. Similar values (2.0 percent TAR and 2.3 percent TAR, respectively) were obtained for the dark control samples.

Analysis of the organic extracts by radio-HPLC demonstrated that, after 15 days, the amount of mefentrifluconazole decreased to 87 percent TAR in the photolysis experiment and to 93 percent TAR in the dark control samples for the C-label. Similarly, for the T-label, the amount of mefentrifluconazole decreased within the same time (15 days) to 94 percent TAR in the photolysis experiment and to 96 percent TAR in the dark control samples. For both labels, several degradation products were detected in the extracts, but none of them appeared in amounts higher than 2 percent TAR.

Kinetic analysis and calculations of DT_{50} and DT_{90} values for mefentrifluconazole in soil were conducted by non-linear regression methods and demonstrated that, there was limited degradation of the parent compound observed for either label in both irradiated and dark control samples, hence photolysis was not an important route of dissipation. However, as the predicted DT_{50} values were extrapolated well beyond the study duration, the values listed in Table 50 should be viewed with caution.

Table 50 DT_{50}s of Mefentrifluconazole from soil photolysis studies

Label	Test system	DT_{50}	DT_{90}	Kinetic model
Chlorophenyl	Irradiated	93	310	SFO
	Dark control	173	574	
Triazole	Irradiated	170	565	
	Dark control	225	747	

Confined Rotational Crop Study

[Chlorophenyl-U-^{14}C] (C-label) and [triazole-3(5)-^{14}C]-labelled mefentrifluconazole (T-label), formulated as EC formulations, were applied to bare sandy loam soil, in plastic containers maintained in either a glass roofed vegetation hall, phytotron of in a glass house, at an application rate of 300 g ai/ha (Rabe, *et al*., 2015, BASF DocID, 2015_1001871). Spinach (variety *Corvette)*, white radish (variety *April Cross)* and spring wheat (*Thasos*) were sown 30/31, 120/122 and 364/365 days after the soil treatment. All crops were harvested at maturity and additional immature spinach samples as well as spring wheat forge samples (in part dried to hay) were collected 25–33 days and 49–55 days after planting (DAP), respectively. Homogenised plant samples were subjected to oxidative combustion to determine TRRs ("TRR combusted").

Table 51 Total radioactive residues in secondary crops following application to bare soil

Matrix	C-label			T-label		
	DAP [A]	TRR combusted (mg eq/kg)	TRR [B] calculated (mg eq/kg)	DAP	TRR combusted (mg eq/kg)	TRR [B] calculated (mg eq/kg)
	Plant-back interval 30/31 days					
Spinach (immature)	28	0.016	0.013	25	0.055	0.052
Spinach (mature)	41	0.014	0.009	44	0.063	0.057
Radish (top)	68	0.013	0.011	70	0.194	0.186
Radish (root)	68	0.010	0.009	70	0.281	0.267
Wheat (forage)	49	0.027	0.021	53	0.318	0.288
Wheat (hay)	49	0.085	0.076	53	0.761	0.681
Wheat (straw) [d]	105	0.240	0.239	105	1.058	1.039
Wheat (grain)	105	0.015	0.014	105	2.400	2.311
	Plant-back interval 120/122 days					
Spinach (immature)	33	0.011	0.009	32	0.114	0.116
Spinach (mature)	41	0.016	0.014	43	0.171	0.150
Radish (top)	57	0.006	0.006	59	0.209	0.197
Radish (root)	57	0.009	0.008	59	0.206	0.198
Wheat (forage)	50	0.030	0.024	52	0.417	0.387
Wheat (hay)	50	0.181	0.155	52	2.561	2.260
Wheat (straw) [D]	144	0.105	0.094	148	1.102	1.008
Wheat (grain)	144	0.039	0.039	148	3.389	3.252
	Plant-back interval 365/364 days					
Spinach (immature)	27	0.007	- [C]	33	0.096	0.094
Spinach (mature)	40	0.007	- [C]	46	0.108	0.097
Radish (top)	61	0.005	- [C]	61	0.100	0.100
Radish (root)	61	0.005	- [C]	61	0.093	0.098
Wheat (forage)	55	0.012	0.010	54	0.189	0.193
wheat (hay)	55	0.035	0.033	54	0.873	0.860
wheat (straw) [D]	137	0.078	0.076	138	0.947	0.916
wheat (grain)	137	0.032	0.033	138	2.258	2.221

Notes:

[A] DAT=days after soil treatment (soil aging interval), DAP=days after planting/sowing (cultivation interval),

[B] TRR=sum methanol and water extracts and the unextracted residues remaining after solvent extraction

[C] No extraction performed,

[D] Straw samples including chaff fraction

For samples which underwent extraction, comparable values were obtained for the "TRR calculated" (sum of extracted and unextracted). Significant uptake and translocation of radioactive residues from soil into the secondary crops was observed, particularly in the case of the T-label over all plant-back intervals (PBI) and matrices (particularly spring wheat grain) which is due to the uptake and translocation of high amounts of triazole-specific metabolites and/ or triazole. The highest levels of radioactive residues were found in spring wheat straw (30-day PBI) and hay (120-day PBI) in the case of the C-label and in the dry matrices spring wheat grain, hay and straw after the 122-day PBI in the case of the T-label. The TRRs in spinach and white radish matrices were generally lower compared to those in wheat matrices. Overall, residues remained similar or decreased at longer PBIs, except for wheat grain from the C-label study where residues peaked at the 120-day PBI followed by a slight decrease by the 365-day PBI, yet still higher than the TRRs at the 30-day PBI.

The homogenised plant samples were consecutively extracted with methanol (3×) and water (2×). Spinach and white radish extracts after the 365-day PBI (C-label) contained only low amounts of TRRs

(based on combustion analysis) and were therefore not subjected to any further analysis. Subsamples of the purified and/ or concentrated methanol extracts of representative crop matrices (C-label: mature spinach, 120-day PBI, white radish root, 30-day PBI, spring wheat forage, 365-day PBI and spring wheat hay, 365-day PBI; T-label: immature spinach, 31-day PBI) were fractionated using HPLC where the parent fraction was collected and concentrated, and the concentrated parent fraction was subjected to chiral analysis.

In the case of the C-label, the extractability of radioactive residues in all tested matrices, except wheat grain, ranged from 34-83 percent TRR, while those in wheat grain were significantly lower, ranging from 8–21 percent TRR. Extracted residues in the wheat matrices were lower at the longest PBI compared to the 30–day PBIs. In the case of the T-label, the extractability of TRRs was much higher than that of the C-label, ranging from 95–99 percent TRR for all spinach and white radish matrices and from 82–96 percent TRR for the spring wheat matrices. The main portions of the TRRs were generally extracted with methanol, except for spring wheat grain where comparable portions of the radioactive residues were extracted with methanol and with water (Table 52).

Table 52 Extractability of radioactive residues in rotational crops (C-Label)

Matrix	Distribution of radioactive residues								
	TRRs	Methanol Extract		Water Extract		Total Extracted		PES	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plant-back interval 30 days									
Spinach (immature)	0.013	80.5	0.010	2.2	0.0003	82.7	0.011	17.3	0.002
Spinach (mature)	0.009	78.0	0.007	1.8	0.0002	79.8	0.007	20.2	0.002
Radish (top)	0.011	67.3	0.007	12.0	0.0013	79.3	0.008	20.7	0.002
Radish (root)	0.009	53.2	0.005	2.4	0.0002	55.6	0.005	44.4	0.004
Wheat (forage)	0.021	68.7	0.015	2.1	0.0005	70.8	0.015	29.2	0.006
Wheat (hay)	0.076	70.0	0.053	5.0	0.0038	74.9	0.057	25.1	0.019
Wheat (straw)	0.239	53.7	0.128	11.6	0.0278	65.3	0.156	34.7	0.083
Wheat (grain)	0.014	0.0	0.000	7.8	0.0011	7.8	0.001	92.2	0.013
Plant-back interval 120 days									
Spinach (immature)	0.009	63.9	0.006	5.0	0.0005	68.9	0.006	31.1	0.003
Spinach (mature)	0.014	53.8	0.007	6.0	0.0008	59.8	0.008	40.2	0.005
Radish (top)	0.006	31.7	0.002	8.3	0.0005	39.9	0.002	60.1	0.004
Radish (root)	0.008	54.2	0.005	4.1	0.0003	58.2	0.005	41.8	0.004
Wheat (forage)	0.024	40.0	0.010	3.5	0.0009	43.5	0.011	56.5	0.014
Wheat (hay)	0.155	35.0	0.054	6.8	0.0106	41.8	0.065	58.2	0.090
Wheat (straw)	0.094	38.7	0.036	8.7	0.0082	47.4	0.045	52.6	0.050
Wheat (grain)	0.039	11.4	0.004	9.9	0.0039	21.2	0.008	78.8	0.031
Plant-back interval 365 days									
Wheat (forage)	0.010	34.8	0.004	4.6	0.0005	39.4	0.004	60.6	0.006
Wheat (hay)	0.033	44.5	0.014	6.1	0.0020	50.6	0.016	49.4	0.016
Wheat (straw)	0.076	25.5	0.019	8.3	0.0063	33.8	0.026	66.2	0.050
Wheat (grain)	0.033	7.3	0.002	7.8	0.0025	15.1	0.005	84.9	0.028

Table 53 Extractability of radioactive residues in rotational crops (T-Label)

Matrix	Distribution of radioactive residues								
	TRRs	Methanol Extract		Water Extract		Total Extracted		PES	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plant-back interval 31 days									
Spinach (immature)	0.052	93.4	0.049	2.4	0.0012	95.7	0.050	4.3	0.002
Spinach (mature)	0.057	95.2	0.054	1.3	0.0008	96.6	0.055	3.4	0.002
Radish (top)	0.186	87.3	0.162	7.7	0.0144	95.1	0.177	4.9	0.009

Matrix	Distribution of radioactive residues								
	TRRs	Methanol Extract		Water Extract		Total Extracted		PES	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Radish (root)	0.267	92.7	0.248	4.2	0.0112	96.9	0.259	3.1	0.008
Wheat (forage)	0.288	87.0	0.251	5.8	0.0167	92.8	0.267	7.2	0.021
Wheat (hay)	0.681	78.9	0.537	13.5	0.0921	92.4	0.630	7.6	0.052
Wheat (straw)	1.039	57.6	0.598	24.6	0.2559	82.2	0.854	17.8	0.185
Wheat (grain)	2.311	47.5	1.097	46.0	1.0634	93.5	2.161	6.5	0.150
Plant-back interval 120 days									
Spinach (immature)	0.116	93.1	0.108	2.3	0.0026	95.4	0.110	4.6	0.005
Spinach (mature)	0.150	92.2	0.139	2.7	0.0040	94.9	0.143	5.1	0.008
Radish (top)	0.197	81.4	0.160	14.1	0.0278	95.5	0.188	4.5	0.009
Radish (root)	0.198	91.8	0.182	4.3	0.0086	96.2	0.190	3.8	0.008
Wheat (forage)	0.387	84.9	0.328	6.4	0.0247	91.3	0.353	8.7	0.034
Wheat (hay)	2.260	56.7	1.282	30.0	0.6772	86.7	1.959	13.3	0.302
Wheat (straw)	1.008	68.2	0.687	20.0	0.2018	88.2	0.889	11.8	0.119
Wheat (grain)	3.252	49.3	1.603	46.4	1.5087	95.7	3.111	4.3	0.141
Plant-back interval 364 days									
Spinach (immature)	0.094	94.6	0.089	2.1	0.0020	96.7	0.091	3.3	0.003
Spinach (mature)	0.097	94.2	0.091	2.3	0.0022	96.4	0.093	3.6	0.003
Radish (top)	0.100	83.6	0.083	12.9	0.0128	96.5	0.096	3.5	0.004
Radish (root)	0.098	96.4	0.094	2.3	0.0023	98.7	0.097	1.3	0.001
Wheat (forage)	0.193	85.6	0.165	6.2	0.0120	91.8	0.177	8.2	0.016
Wheat (hay)	0.860	83.0	0.715	10.8	0.0928	93.8	0.807	6.2	0.053
Wheat (straw)	0.916	48.9	0.448	33.8	0.3095	82.7	0.757	17.3	0.159
Wheat (grain)	2.221	40.6	0.901	52.9	1.1742	93.4	2.075	6.6	0.146

The nature of the residue in extracts of all matrices, except spinach and white radish (365-day PBI; C-label) was investigated using two different HPLC methods (radiodetection). Assignment of chromatographic peaks and identification of metabolites was based on co-chromatography with ^{14}C-labelled reference compounds as well as by comparison of retention times and elution pattern (metabolic profile). Characterization of the PES was performed by sequential hydrolysis using aqueous ammonia, amylases/amyloglucosidase, macerozyme/cellulase, and tyrosinase/laccase.

Mefentrifluconazole was the main component detected in all tested samples from the C-label study (85.2 percent TRR and 91.2 percent TRR [0.008 and 0.012 mg/kg]) in immature and mature spinach respectively at 30-day PBI, 42.5–70.4 percent TRR [0.006–0.101 mg/kg] in the other tested matrices at 30-day PBI, 35.7–60.8 percent TRR [0.006–0.055 mg/kg] in spinach and wheat at 120-day PBI and 17.7–41.0 percent TRR [0.002–0.018 mg/kg] in wheat samples at 365-day PBI. No parent or metabolites were detected in radish roots or tops beyond the 30-day PBI. In spinach and wheat, minor metabolites accounting for 2.2–79.7 percent TRR (0.0003–0.018 mg eq/kg), 5.0–83.3 percent TRR (0.0004–0.032 mg eq/kg) and 21.5–65.4 percent TRR (0.004–0.022 mg eq/kg) at 30-, 120- and 365-day PBIs, respectively, were characterised by their chromatographic properties.

In the case of the T-label study, mefentrifluconazole was detected at the 31-day PBI in immature and mature spinach at 25.2 percent (0.013 mg/kg) and 13.9 percent TRR (0.08 mg/kg), in white radish top at 5.6 percent TRR (0.010 mg /kg) and in all wheat samples except grain at 4.1–5.0 percent TRR (0.015–0.043 mg/kg). At longer PBIs, the parent was only detected in wheat hay (122-day PBI: 1.3 percent TRR [0.030 mg/kg]) and straw (122-day PBI; 1.3 percent TRR [0.014 mg/kg] and 365-day PBI; 0.8 percent TRR [0.008 mg/kg]). In most cases, the main constituent in the crop matrices was triazole alanine (13.2–93.5 percent TRR; 0.022–0.982 mg eq/kg), followed by triazole lactic acid 8.8–38.6 percent TRR (0.005–0.807 mg eq/kg). The only exceptions were spring wheat hay (122 DAT) and spring wheat straw (all PBIs),

where, triazole lactic acid was the most abundant component among all the analytes identified. In spring wheat grain, triazole alanine (42.5–72.6 percent TRR; 0.98–2.36 mg eq/kg) was the main component followed by triazole acetic acid (20.0–24.3 percent TRR; 0.46–0.69 mg eq/kg). The sum of the triazole derivative metabolites and 1,2,4-triazole in all secondary crops ranged from 64.6–101.4 percent TRR (Tables 54 and 55).

Table 54 Summary of identified/characterized radioactive residues (C-label)

	Spinach (immature)		Spinach (mature)		Radish top		Radish root		Wheat forage		Wheat hay		Wheat straw		Wheat grain	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plant-back Interval 30 days																
Mefentrifluconazole	91.2	0.012	85.2	0.008	54.8	0.006	61.5	0.006	70.4	0.015	61.6	0.046	42.5	0.101	ND	ND
Total Characterized	2.2	0.0003	12.0	0.001	34.6	0.004	16.7	0.002	13.6	0.003	24.0	0.018	32.2	0.077	79.7	0.01
Plant-back Interval 120 days																
Mefentrifluconazole	60.8	0.006	51.7	0.007	ND	ND	ND	ND	43.7	0.011	35.7	0.055	35.7	0.034	ND	ND
Total Characterized	5.0	0.0004	11.2	0.002	39.9	0.002	58.2	0.005	41.5	0.010	41.1	0.064	28.2	0.026	83.3	0.032
Plant-back Interval 365 days																
Mefentrifluconazole	ND	ND	ND	ND	ND	ND	ND	ND	17.7	0.002	41.0	0.013	23.9	0.018	ND	ND
Total Characterized	ND	ND	ND	ND	ND	ND	ND	ND	43.3	0.004	21.5	0.007	22.6	0.017	65.4	0.022

Table 55 Summary of identified/characterized radioactive residues (T-label)

	Spinach (immature)		Spinach (mature)		Radish top		Radish root		Wheat forage		Wheat hay		Wheat straw		Wheat grain	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plant-back Interval 31 days																
Mefentrifluconazole	25.2	0.013	13.9	0.008	5.6	0.010	ND	ND	5.0	0.015	4.5	0.031	4.1	0.043	ND	ND
1,2,4-triazole	10.5	0.005	7.7	0.004	ND	ND	ND	ND	4.0	0.012	5.0	0.034	6.3	0.065	14.7	0.339
TA	42.9	0.022	56.0	0.032	45.4	0.084	61.8	0.165	43.1	0.124	45.6	0.311	13.2	0.137	42.5	0.982
TAA	2.5	0.001	5.5	0.003	4.2	0.008	ND	ND	10.9	0.031	20.8	0.142	15.0	0.156	20.0	0.462
TLA	8.8	0.005	18.3	0.010	22.4	0.042	30.9	0.083	33.7	0.097	23.7	0.162	35.3	0.366	13.8	0.319
Total Identified	89.9	0.047	101.5	0.058	77.6	0.144	92.8	0.248	96.7	0.279	99.7	0.679	73.8	0.767	91.0	2.103
Total Characterized	2.4	0.001	1.3	0.0007	10.5	0.020	0.7	0.002	4.3	0.012	6.1	0.042	12.9	0.134	5.4	0.125
Plant-back Interval 122 days																
Mefentrifluconazole	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.3	0.030	1.3	0.014	ND	ND
1,2,4-triazole	ND	ND	12.4	0.019	ND	ND	ND	ND	3.9	0.015	4.5	0.101	2.6	0.026	ND	ND
TA	60.1	0.070	52.9	0.080	93.5	0.184	62.6	0.124	44.3	0.171	31.7	0.717	33.1	0.333	72.6	2.361
TAA	3.2	0.004	ND	ND	ND	ND	ND	ND	7.9	0.030	10.0	0.226	18.0	0.182	21.2	0.689
TLA	25.0	0.029	34.2	0.051	ND	ND	23.0	0.046	36.4	0.141	36.6	0.827	34.2	0.344	ND	ND
Total Identified	88.4	0.102	99.5	0.150	93.5	0.184	85.6	0.169	92.4	0.357	84.1	1.901	89.2	0.899	93.8	3.050
Total Characterized	2.3	0.003	2.7	0.004	ND	ND	4.3	0.008	5.4	0.021	9.9	0.224	10.3	0.104	3.6	0.117
Plant-back Interval 364 days																
Mefentrifluconazole	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.8	0.008	ND	ND
1,2,4-triazole	ND	ND	7.9	0.008	2.6	0.003	ND	ND	ND	ND	4.2	0.036	4.0	0.036	1.1	0.023
TA	71.1	0.067	56.2	0.054	77.5	0.077	79.1	0.077	37.7	0.073	38.3	0.330	16.6	0.152	64.2	1.425

	Spinach (immature)		Spinach (mature)		Radish top		Radish root		Wheat forage		Wheat hay		Wheat straw		Wheat grain	
Component	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
TAA	ND	ND	3.7	0.004	4.5	0.004	ND	ND	8.7	0.017	11.7	0.101	18.9	0.173	24.3	0.539
TLA	25.0	0.023	33.6	0.032	10.7	0.011	17.5	0.017	37.5	0.072	37.5	0.323	38.6	0.354	ND	ND
Total Identified	96.1	0.090	101.4	0.098	95.2	0.095	96.6	0.095	83.8	0.162	91.7	0.789	78.9	0.722	89.5	1.987
Total Characterized	2.1	0.002	2.3	0.002	ND	ND	2.3	0.002	6.2	0.012	3.7	0.032	11.1	0.095	5.7	0.126

Notes:

TA: Triazole alanine; TAA: Triazole acetic acid; TLA: Triazole lactic acid.

The PES (post extracted solids) following methanol and water extraction of mature spinach, white radish top and root (all C-label, 30-day PBI) as well as all the wheat matrices were characterised by sequential solubilisation procedures applying treatment with aqueous ammonia solution and one or two incubations with enzyme mixtures. The ammonia residues of spring wheat grain were subsequently incubated with amylases and amyloglucosidase. The amylase/ amyloglucosidase residues of spring wheat grain as well as the ammonia residues of the other investigated matrices were incubated with macerozyme and cellulase. In the cases of spring wheat forage and hay (third plant back interval) and spring wheat straw, the PES were further incubated with tyrosinase and laccase. The supernatants resulting from the aqueous ammonia treatment and after incubation with enzymes were analysed by HPLC. The results are shown in Tables 56 to 58. Samples from the Plant-back Interval (PBI) of 365 days from the C-label study were not analysed.

Table 56 Characterization of post extracted solids (PES) of spinach and radish matrices (C-label)

	Spinach (immature)		Spinach (mature)		Radish top		Radish root	
Hydrolysis fraction[A]	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plant-back Interval 30 days								
PES	17.3	0.0022	20.2	0.0018	20.7	0.0022	44.4	0.0041
AM hydrolysate[B]	N/A	N/A	2.9	0.0003	4.0	0.0004	4.2	0.0004
AM residue	N/A	N/A	14.3	0.0013	15.5	0.0016	40.7	0.0038
A/G hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
A/G residue	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
M/C hydrolysate	N/A	N/A	7.3	0.0007	5.7	0.0006	10.1	0.0009
M/C residue	N/A	N/A	6.1	0.0005	10.3	0.0011	31.0	0.0029
T/L hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
T/L residue	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Hydrolysed TRRs	N/A	N/A	10.2	0.0009	9.7	0.0010	14.2	0.0013
Final Unextracted Residue	N/A	N/A	6.1	0.0005	10.3	0.0011	31.0	0.0029
Plant-back Interval 120 days								
PES	31.1	0.0029	40.2	0.0055	60.1	0.0037	41.8	0.0035
AM hydrolysate[B]	N/A	N/A	5.2	0.0007	N/A	N/A	N/A	N/A
AM residue	N/A	N/A	31.7	0.0043	N/A	N/A	N/A	N/A
A/G hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
A/G residue	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
M/C hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
M/C residue	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
T/L hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
T/L residue	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Hydrolysed TRRs	N/A	N/A	5.2	0.0007	N/A	N/A	N/A	N/A
Final Unextracted Residue	N/A	N/A	31.7	0.0043	N/A	N/A	N/A	N/A
Final Unextracted Residue	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Notes:

[A] AM, ammonia, A/G amylase/amyloglucosidase, M/C macerozyme/cellulase, T/L tyrosinase/laccase

[B] Only one hydrolysis step was performed

N/A: Not analysed

Table 57 Characterization of post extracted solids (PES) in wheat matrices (C-label)

	Forage		Hay		Straw		Grain	
Hydrolysis fraction[A]	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plant-back Interval 30 days								
PES	29.2	0.0062	25.1	0.0189	34.7	0.0828	92.2	0.0132
AM hydrolysate[B]	6.1	0.0013	8.5	0.0064	7.7	0.0183	23.2	0.0033
AM residue	21.6	0.0046	17.1	0.0129	25.9	0.0618	63.5	0.0091
A/G hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	28.4	0.0041
A/G residue	N/A	N/A	N/A	N/A	N/A	N/A	31.3	0.0045
M/C hydrolysate	5.4	0.0012	3.9	0.0030	2.2	0.0053	20.4	0.0029
M/C residue	13.3	0.0029	9.6	0.0072	22.8	0.0544	10.5	0.0015
T/L hydrolysate	N/A	N/A	N/A	N/A	0.9	0.0020	N/A	N/A
T/L residue	N/A	N/A	N/A	N/A	21.6	0.0516	N/A	N/A
Hydrolysed TRRs	11.5	0.0025	12.4	0.0094	10.8	0.0257	71.9	0.0103
Final Unextracted Residue	13.3	0.0029	9.6	0.0072	21.6	0.0516	10.5	0.0015
Plant-back Interval 122 days								
PES	56.5	0.0138	58.2	0.0901	52.6	0.0495	78.8	0.0309
AM hydrolysate[B]	20.0	0.0049	5.4	0.0084	8.5	0.0081	17.5	0.0069
AM residue	35.1	0.0086	48.8	0.0756	43.2	0.0407	59.5	0.0234
A/G hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	29.6	0.0116
A/G residue	N/A	N/A	N/A	N/A	N/A	N/A	26.5	0.0104
M/C hydrolysate	18.0	0.0044	28.9	0.0447	9.1	0.0085	14.9	0.0059
M/C residue	14.9	0.0036	21.9	0.0339	33.8	0.0318	8.5	0.0034
T/L hydrolysate	N/A	N/A	N/A	N/A	1.9	0.0018	N/A	N/A
T/L residue	N/A	N/A	N/A	N/A	29.3	0.0275	N/A	N/A
Hydrolysed TRRs	38.0	0.0093	34.3	0.0530	19.5	0.0184	62.0	0.0244
Final Unextracted Residue	14.9	0.0036	21.9	0.0339	29.3	0.0275	8.5	0.0034
Plant-back Interval 365-days								
PES	60.6	0.0063	49.4	0.0161	66.2	0.0500	84.9	0.0276
AM hydrolysate[B]	8.7	0.0009	8.0	0.0026	8.6	0.0065	14.7	0.0048
AM residue	44.0	0.0046	34.8	0.0113	54.9	0.0415	65.6	0.0213
A/G hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	18.3	0.0060
A/G residue	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
M/C hydrolysate	8.6	0.0009	5.8	0.0019	3.9	0.0029	17.2	0.0056
M/C residue	N/A	N/A	N/A	N/A	N/A	N/A	25.2	0.0082
T/L hydrolysate	3.0	0.0003	1.6	0.0005	1.7	0.0013	N/A	N/A
T/L residue	32.2	0.0033	32.2	0.0105	50.8	0.0384	N/A	N/A
Hydrolysed TRRs	20.4	0.0021	15.4	0.0050	14.3	0.0108	50.2	0.0163
Final Unextracted Residue	32.2	0.0033	32.2	0.0105	50.8	0.0384	25.2	0.0082

Notes:

[A] AM, ammonia, A/G amylase/amyloglucosidase, M/C macerozyme/cellulase, T/L tyrosinase/laccase.

[B] combined ammonia hydrolysate from two solubilisation steps in the cases of spring wheat hay, straw and grain; only one solubilisation step was performed in the case of spring wheat forage.

N/A: not analysed.

Table 58 Characterization of post extracted solids (PES) in wheat matrices (T-label)

	Forage		Hay		Straw		Grain	
Hydrolysis fraction[A]	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plant- back Interval 31 days								
PES	7.2	0.0208	7.6	0.0519	17.8	0.1851	6.5	0.1499
AM hydrolysate[B]	3.5	0.0100	3.7	0.0249	9.5	0.0990	1.9	0.0436
AM residue	2.7	0.0079	3.0	0.0208	6.6	0.0687	4.4	0.1020
A/G hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	3.1	0.0712
A/G residue	N/A	N/A	N/A	N/A	N/A	N/A	0.9	0.0216
M/C hydrolysate	N/A	N/A	0.7	0.0044	1.5	0.0155	0.5	0.0109
M/C residue	N/A	N/A	2.1	0.0144	4.9	0.0504	0.4	0.0097
T/L hydrolysate	N/A	N/A	N/A	N/A	0.5	0.0055	N/A	N/A
T/L residue	N/A	N/A	N/A	N/A	4.3	0.0450	N/A	N/A
Hydrolysed TRRs	3.5	0.0100	4.3	0.0294	11.6	0.1201	5.4	0.1257
Final Unextracted Residue	2.7	0.0079	2.1	0.0144	4.3	0.0450	0.4	0.0097
Plant-back Interval 122 days								
PES	8.7	0.0338	13.3	0.3017	11.8	0.1188	4.3	0.1410
AM hydrolysate[B]	3.0	0.0116	7.3	0.1652	4.3	0.0430	1.6	0.0505
AM residue	5.0	0.0193	5.4	0.1219	6.3	0.0637	2.6	0.0830
A/G hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	1.7	0.0558
A/G residue	N/A	N/A	N/A	N/A	N/A	N/A	0.7	0.0215
M/C hydrolysate	2.4	0.0094	2.6	0.0586	1.7	0.0167	0.3	0.0107
M/C residue	2.4	0.0091	2.6	0.0597	4.6	0.0465	0.2	0.0078
T/L hydrolysate	N/A	N/A	N/A	N/A	0.5	0.0049	N/A	N/A
T/L residue	N/A	N/A	N/A	N/A	3.9	0.0395	N/A	N/A
Hydrolysed TRRs	5.4	0.0210	9.9	0.2238	6.4	0.0646	3.6	0.1170
Final Unextracted Residue	2.4	0.0091	2.6	0.0597	3.9	0.0395	0.2	0.0078
Plant-back Interval 364 days								
PES	8.2	0.0159	6.2	0.0532	17.3	0.1587	6.6	0.1460
AM hydrolysate[B]	4.5	0.0088	3.1	0.0266	7.2	0.0657	4.1	0.0902
AM residue	3.4	0.0065	2.6	0.0227	8.6	0.0788	2.8	0.0616
A/G hydrolysate	N/A	N/A	N/A	N/A	N/A	N/A	1.3	0.0285
A/G residue	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
M/C hydrolysate	0.8	0.0015	0.5	0.0041	1.6	0.0145	0.3	0.0068
M/C residue	N/A	N/A	N/A	N/A	N/A	N/A	0.7	0.0165
T/L hydrolysate	0.2	0.0004	0.2	0.0014	0.7	0.0061	N/A	N/A
T/L residue	2.3	0.0045	1.9	0.0165	5.9	0.0540	N/A	N/A
Hydrolysed TRRs	5.5	0.0107	3.7	0.0320	9.4	0.0863	5.7	0.1256
Final Unextracted Residue	2.3	0.0045	1.9	0.0165	5.9	0.0540	0.7	0.0165

Notes:

[A] AM, ammonia, A/G amylase/amyloglucosidase, M/C macerozyme/cellulase, T/L tyrosinase/laccase.

[B] combined ammonia hydrolysate from two solubilisation steps in the cases of spring wheat hay, straw and grain; only one solubilisation step was performed in the case of spring wheat forage.

N/A: not analysed.

Hydrolysis of the PES of selected matrices demonstrated that both the parent mefentrifluconazole and the triazole derivative metabolites were associated with plant constituents, namely starch and cell wall polymers, neither of which was further characterised. The presence of radioactivity in the final unextracted residues, following the extensive hydrolysis steps, was evidence of incorporation into non-hydrolysable cell constituents.

Enantiomer-specific analysis of mefentrifluconazole in all representative crops and both labels showed that both S-enantiomer and R-enantiomer are present as a racemic mixture confirming that the

1:1 ratio of the test substance applied to bare soil remains unchanged. The unchanged enantiomer ratio indicates absence of preferential metabolism and /or translocation of one of the two enantiomers in rotational crops.

Directly after harvesting, samples were transferred in a freezer and stored at ≤ -20 °C until extraction. Extracts were stored in a refrigerator. Storage stability of the methanol extracts of the various matrices demonstrated that the metabolite profiles after 270–469 days of storage was comparable to the metabolite profiles obtained in the initial analysis of the extracts (14–127 days), thus confirming stability of the radioactive residues in extracts for at least 284 days.

In rotational crops cultivated on mefentrifluconazole-treated soil, the residues include mainly two components, the parent and triazole derivative metabolites, the latter being generated by cleavage of the parent molecule at the triazole bridge (Figure 5).

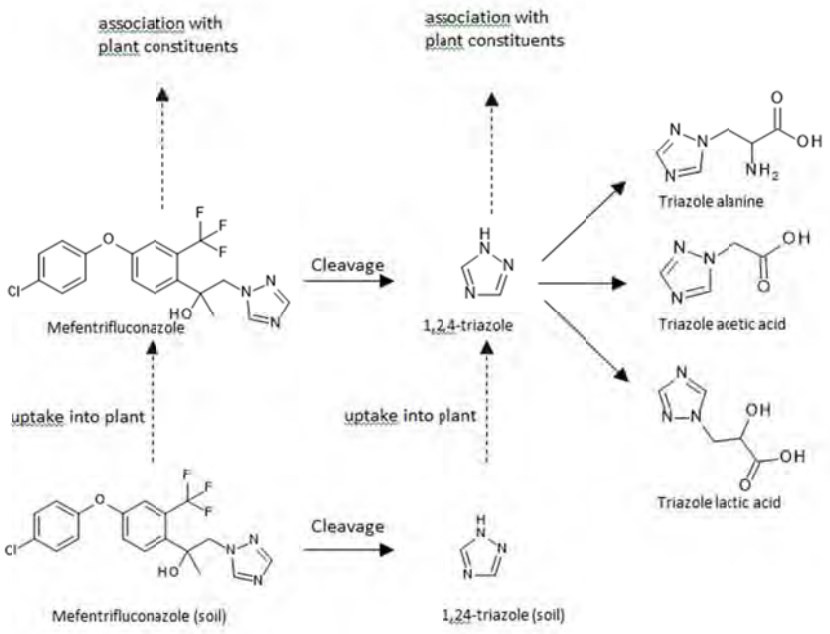


Figure 5 Proposed metabolic pathway of ^{14}C-Mefentrifluconazole in rotational crops

Field rotational crop study

United States

Field rotational crop studies were conducted in the American states of Georgia and Texas during the 2014 growing season (Schreier, 2016, BASF DocID, 2016_7006244). Treated plots received three broadcast soil directed spray applications, of an emulsifiable concentrate (EC) formulation, at rates of 0.196 to 0.207 kg ai/ha/application, with a 7-day retreatment interval, for a total rate of 0.595 to 0.614 kg ai/ha/season. The applications were made using 200 to 224 L/ha spray volumes with ground boom equipment. An adjuvant was added to the spray mixture for all applications at all trials. At intervals of 1, 3, 4 and 12 months following the last application to the bare soil, wheat (AGS 03108E-10E26, TAM 112 and Glenn), lettuce (romaine), and radish (Crunchy Royale and D'Avignon) were planted.

The residues of mefentrifluconazole in/on rotational crop RAC samples were quantitated using the LC-MS/MS Method D1511/01. The LOQ was 0.01 mg/kg. Procedural recoveries from control samples fortified with mefentrifluconazole at 0.01 to 10.0 mg/kg were 78 to 121 percent. Average procedural recoveries for individual crop matrices ranged from 88 percent to 106 percent. Samples were stored

frozen for up to 553 days (approximately 18 months) prior to extraction and analysis. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. The results are shown in Table 59.

Table 59 Mefentrifluconazole residues in rotational crops in the United States

Trial, Location, Year	Crop / Variety	Plant-back interval (months)	Application rate (kg ai/ha)	DALA	Commodity	Mefentrifluconazole, mg/kg [average]
R140430 Plains, GA 2014 - 2016	Wheat AGS 03108E10E26	1	0.596	109	Forage	<0.01, <0.01 [<0.01]
				182	Hay	<0.01, <0.01 [<0.01]
				213	Straw	<0.01, <0.01 [<0.01]
				213	Grain	<0.01, <0.01 [<0.01]
		3	0.595	170	Forage	<0.01, <0.01 [<0.01]
				243	Hay	<0.01, <0.01 [<0.01]
				274	Straw	<0.01, <0.01 [<0.01]
				274	Grain	<0.01, <0.01 [<0.01]
		4	0.595	201	Forage	<0.01, <0.01 [<0.01]
				274	Hay	<0.01, <0.01 [<0.01]
				279	Straw	0.02, 0.01 [0.02]
				305	Grain	<0.01, <0.01 [<0.01]
		13	0.597	479	Forage	<0.01, <0.01 [<0.01]
				538	Hay	<0.01, <0.01 [<0.01]
				587	Straw	<0.01, 0.01 [0.01]
				587	Grain	<0.01, <0.01 [<0.01]
R140432 Plains, GA 2014 - 2016	Lettuce Romaine	1	0.596	75	Leaves	<0.01, <0.01 [<0.01]
		3	0.596	136	Leaves	<0.01, <0.01 [<0.01]
		4	0.597	160	Leaves	<0.01, <0.01 [<0.01]
		12	0.596	440	Leaves	<0.01, <0.01 [<0.01]
R140434 Plains, GA 2014 - 2016	Radish Crunchy Royale	1	0.597	75	Tops	0.07, 0.07 [0.07]
				75	Roots	0.03, 0.02 [0.03]
		3	0.595	136	Tops	0.04, 0.03 [0.04]
				136	Roots	0.01, 0.02 [0.02]
		4	0.596	160	Tops	0.02, 0.02 [0.02]
				160	Roots	0.01, 0.02 [0.02]
		12	0.597	440	Tops	<0.01, <0.01 [<0.01]
				440	Roots	<0.01, <0.01 [<0.01]
R140431 Claude, TX 2014 - 2015	Wheat TAM112	1	0.601	223	Forage	2.38, 1.22 [1.80]
				264	Hay	0.53, 0.21 [0.37]
				301	Straw	0.01, 0.06 [0.04]
				301	Grain	<0.01, <0.01 [<0.01]
		3	0.603	285	Forage	0.57, 0.69 [0.63]
				326	Hay	0.87, 0.63 [0.75]
				363	Straw	NA
				363	Grain	<0.01, <0.01 [<0.01]
		4	0.602	315	Forage	1.13, 0.69 [0.91]
				356	Hay	2.65, 0.66 [1.66]
				363	Straw	NA
				393	Grain	<0.01, <0.01 [<0.01]
	Wheat Glenn	11	0.603	375	Forage	1.16, 1.57 [1.37]
				391	Hay	1.97, 0.75 [1.36]
				436	Straw	<0.01, <0.01 [<0.01]
				436	Grain	<0.01, <0.01 [<0.01]
R140433 Claude, TX 2014 - 2015	Lettuce Romaine	1	0.600	75	Leaves	<0.01, <0.01 [<0.01]
		3	0.614	136	Leaves	<0.01, <0.01 [<0.01]
		4	0.609	160	Leaves	<0.01, <0.01 [<0.01]

Trial, Location, Year	Crop / Variety	Plant-back interval (months)	Application rate (kg ai/ha)	DALA	Commodity	Mefentrifluconazole, mg/kg [average]
		12	0.602	440	Leaves	<0.01, <0.01 [<0.01]
R140435 Claude, TX 2014 - 2015	Radish D'Avignon	1	0.603	47	Tops	<0.01, <0.01 [<0.01]
				47	Roots	<0.01, <0.01 [<0.01]
		3	0.606	191	Tops	<0.01, <0.01 [<0.01]
				191	Roots	<0.01, <0.01 [<0.01]
		4	0.602	191	Tops	<0.01, <0.01 [<0.01]
				191	Roots	<0.01, <0.01 [<0.01]
		12	0.605	391	Tops	<0.01, <0.01 [<0.01]
				391	Roots	<0.01, <0.01 [<0.01]

With the exception of wheat matrices, mefentrifluconazole residues in lettuce and radish (roots and tops) collected from trials conducted in Georgia were equivalent to or higher than those from trials conducted in Texas. For wheat, residues of mefentrifluconazole were magnitudes greater in trials conducted in Texas compared to those in wheat matrices collected from trials conducted in Georgia, where mefentrifluconazole residues in almost all wheat RACs were <0.01 mg/kg.

Among the two locations, maximum individual mefentrifluconazole residues were 2.38, 0.69, 1.13, and 1.57 mg/kg in/on wheat forage samples collected following the 1, 3, 4 and 11 month PBIs, respectively, and were 0.53, 0.87, 2.65 and 1.97 mg/kg in/on wheat hay samples collected from the 1, 3, 4 and 12 month PBIs, respectively. Maximum individual mefentrifluconazole residues in/on wheat straw samples collected from the 1, 3, 4 and 12 month PBIs were 0.06, <0.01, 0.02 and 0.01 mg/kg, respectively. Residues of mefentrifluconazole in/on wheat grain and lettuce were <0.01 mg/kg (<LOQ) at all PBIs. Maximum mefentrifluconazole residues were 0.07, 0.04, 0.02 and <0.01 mg/kg in/on radish tops (leaves) collected from the 1, 3, 4 and 12 month PBIs, respectively, and were 0.03, 0.02, 0.02 and <0.01 mg/kg in/on radish root samples collected from the 1, 3, 4 and 12 month PBIs, respectively. Based on the findings of the study, limited uptake of mefentrifluconazole residues from the soil into the plants was observed and residues declined with longer PBIs.

Europe

During the 2014–2015 growing seasons, a total of four field trials were conducted using four rotational crops (wheat, carrot/radish, cauliflower/broccoli and lettuce/spinach) planted at 1, 4 and 12 months after the application to bare soil in different representative growing regions of Germany, Netherlands, Italy and Spain. Each field trial consisted of three treated plots.

An emulsifiable concentrate (EC) formulation was applied once to all plots at a rate equivalent to 271-327 g ai/ha. The spray volume used was 200 L/ha.

The residues of mefentrifluconazole in/on rotational crop RAC samples were quantitated using the LC-MS/MS method No. L0076/09 (535/1). The LOQ was 0.01 mg/kg. Average procedural recoveries from control samples fortified with mefentrifluconazole at 0.01, 0.1 and 1.0 ppm ranged from 77 to 92 percent.

Samples were stored frozen for up to 305 days (~10 months) prior to extraction and analysis. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. The results are shown in Table 60

Table 60 Mefentrifluconazole residues in rotational crops in Europe

Trial, location/Year	Crop / Variety	PBI (months)	Application rate (kg ai/ha)	DALA	Commodity or Matrix	Mefentrifluconazole Residues, mg
L140125 Palatinate, Germany 2015	Wheat / KWS Chamsin	1	0.313	69	Whole plant	<0.01
				89	Whole plant	<0.01
				135	Straw	<0.01
				135	Grain	<0.01
	Wheat / JB Asano	4	0.304	285	Whole plant	<0.01
				334	Whole plant	<0.01
				385	Straw	<0.01
				385	Grain	<0.01
	Wheat / KWS Chamsin	12	0.304	403	Whole plant	<0.01
				435	Whole plant	<0.01
				476	Straw	<0.01
				476	Grain	<0.01
	Carrot / Jeanette	1	0.313	134	Tops	<0.01
				134	Roots	<0.01
		4	0.304	216	Tops	<0.01
				216	Roots	<0.01
		12	0.304	463	Tops	<0.01
				463	Roots	<0.01
	Broccoli / Monteco	1	0.313	83	Inflorescences	<0.01
	Broccoli / Parthenon	4	0.304	184	Inflorescences	<0.01
	Broccoli / Monteco	12	0.304	426	Inflorescences	<0.01
	Lettuce / Babyleaf	1	0.313	58	Leaves	<0.01
				69	Leaves	<0.01
		4	0.304	148	Leaves	<0.01
				162	Leaves	<0.01
		12	0.304	393	Leaves	<0.01
				407	Leaves	<0.01
L140126 Limburg, Netherlands 2015	Wheat / Tybalt	1	0.292	89	Whole plant	<0.01
				111	Whole plant	<0.01
				145	Straw	<0.01
				145	Grain	<0.01
	Wheat / Winnetou	4	0.308	321	Whole plant	<0.01
				370	Whole plant	<0.01
				413	Straw	<0.01
				413	Grain	<0.01
	Wheat / KWS Chamsin	12	0.302	426	Whole plant	<0.01
				445	Whole plant	<0.01
				490	Straw	<0.01
				490	Grain	<0.01
	Carrot / Jubila	1	0.292	138	Tops	<0.01
				138	Roots	<0.01
		4	0.304	228	Tops	<0.01
				228	Roots	<0.01
		12	0.302	482	Tops	<0.01
				482	Roots	<0.01
	Cauliflower / Panther	1	0.292	118	Inflorescences	<0.01
	Cauliflower / Clarina	4	0.304	209	Inflorescences	<0.01

Mefentrifluconazole

Trial, location/Year	Crop / Variety	PBI (months)	Application rate (kg ai/ha)	DALA	Commodity or Matrix	Mefentrifluconazole Residues, mg
	Cauliflower / Freedom F1	12	0.298	427	Inflorescences	<0.01
	Spinach / Violin F1	1	0.292	74	Leaves	<0.01
				88	Leaves	<0.01
		4	0.313	158	Leaves	<0.01
				166	Leaves	<0.01
		12	0.298	406	Leaves	<0.01
				413	Leaves	<0.01
L140127 Bologna, Italy 2015	Wheat / Palesio	1	0.319	195	Whole plant	<0.01
				211	Whole plant	<0.01
				257	Straw	<0.01
				257	Grain	<0.01
		4	0.303	296	Whole plant	<0.01
				312	Whole plant	<0.01
				358	Straw	<0.01
				358	Grain	<0.01
		12	0.327	441	Whole plant	<0.01
				455	Whole plant	<0.01
				497	Straw	<0.01
				497	Grain	<0.01
	Radish / Candela di Ghiaccio	1	0.322	82	Tops	<0.01
				82	Roots	<0.01
		4	0.323	198	Tops	<0.01
				198	Roots	<0.01
		12	0.286	434	Tops	<0.01
				434	Roots	<0.01
	Cauliflower / Concept F1	1	0.298	106	Inflorescences	<0.01
		4	0.321	197	Inflorescences	<0.01
	Cauliflower / Chambord F1	12	0.285	470	Inflorescences	<0.01
	Spinach / America	1	0.271	71	Leaves	<0.01
				82	Leaves	<0.01
		4	0.327	180	Leaves	<0.01
				190	Leaves	<0.01
		12	0.274	426	Leaves	<0.01
				434	Leaves	<0.01
L140128 Sevilla, Spain 2015	Wheat / Arthur Nick	1	0.314	106	Whole plant	<0.01
				148	Whole plant	<0.01
				211	Straw	<0.01
				211	Grain	<0.01
	Wheat / JB Asano	4	0.323	196	Whole plant	<0.01
				238	Whole plant	<0.01
				301	Straw	<0.01
				301	Grain	<0.01
	Wheat / Arthur Nick	12	0.305	405	Whole plant	<0.01
				423	Whole plant	<0.01
				467	Straw	<0.01
				467	Grain	<0.01
	Radish / Candela di Ghiaccio	1	0.294	65	Tops	<0.01
				65	Roots	<0.01
		4	0.300	155	Tops	<0.01
				155	Roots	<0.01
	Radish / Largo Rojo	12	0.305	410	Tops	<0.01
				410	Roots	<0.01

Trial, location/Year	Crop / Variety	PBI (months)	Application rate (kg ai/ha)	DALA	Commodity or Matrix	Mefentrifluconazole Residues, mg
	Cauliflower / Sirente	1	0.294	135	Inflorescences	<0.01
		4	0.300	225	Inflorescences	<0.01
	Cauliflower / Caspe	12	0.305	432	Inflorescences	<0.01
	Spinach / Viroflay	1	0.294	64	Leaves	<0.01
				72	Leaves	<0.01
		4	0.300	154	Leaves	<0.01
				162	Leaves	<0.01
		12	0.305	405	Leaves	<0.01
				420	Leaves	<0.01

Residues of mefentrifluconazole in all tested crops, including a cereal grain (wheat), root crop (carrot and radish), leafy (lettuce and spinach) and Brassica (broccoli and cauliflower) vegetables were consistently below the LOQ of 0.01 mg/kg at all plant-back intervals of 1, 4 and 12 months in field rotational studies conducted in Germany, Netherlands, Italy and Spain.

In summary, the environmental fate data demonstrated that mefentrifluconazole is persistent in soil yet does not dissipate significantly, being almost exclusively detected in the top 15–20 cm. Notwithstanding its presence in the upper soil layer, the uptake of mefentrifluconazole residues was observed predominantly at the 30-day PBI before dissipating rapidly as a result of the cleavage of the triazole ring to generate the triazole derivative metabolites. Mefentrifluconazole is stable in aqueous solutions at environmentally relevant pHs and photolysis of mefentrifluconazole on the soil surface is not anticipated to be an important dissipation process.

The metabolism in rotational crops was assessed and determined to be similar to that in primary crops with no rotational crop specific metabolites. The magnitude of both mefentrifluconazole and triazole derivative metabolites was investigated under field conditions. Based on the results obtained in the field accumulation study, uptake of mefentrifluconazole residues is not expected

METHODS OF RESIDUE ANALYSIS

The Meeting received descriptions and validation data for several analytical methods for residues of mefentrifluconazole in diverse plant and animal matrices. Only the methods capable of analysing mefentrifluconazole will be reported herein.

Plants

Method L0076/09

Analytical method L0076/09 (de Paula José, 2015, BASF DocID 2015_3001681) involves extraction of soya bean (seed), dry beans (seed), tomato (whole fruit), citrus (whole fruit), wheat (grain), coffee (grain), and wheat (straw) with methanol:water: 2 mol/L HCl (70:25:5:v). For wheat straw, the extraction was conducted twice. The extract was transferred to a tube containing 0.2 mol/L NaOH solution and then partitioned with cyclohexane. The resulting organic phase was evaporated to dryness and reconstituted in methanol:water (50:50) prior to filtration and analysis.

The final extracts were quantified for residues of mefentrifluconazole using HPLC- and UPLC-MS/MS, comprising a C18 column that incorporated 0.1 percent formic acid in water and 0.1 percent formic acid in methanol mobile phases. Quantitation was by multiple reaction monitoring (MRM) in

positive mode with a mass transition of *m/z* 398 → 182 and m/z 398→ 133. The LOQ was 0.01 mg/kg for citrus fruit, coffee grains, dry bean seeds, soya bean seeds, tomato fruit, wheat grain and wheat straw.

Recovery and repeatability data for the determination of mefentrifluconazole residues in plant matrices are presented in Table 61. Average recovery values ranged from 73–100 percent for the three fortification levels tested while the percent RSD ranged from 1.1–14 percent. Additionally, two replicates of unfortified control samples were examined. All results from the control samples were below 0.002 mg/kg. (0.2× LOQ). No interfering peaks were detected in the samples analysed. The linearity of the detector response (r^2 > 0.9900), using non-matrix matched standards, over a concentration range of 0.04 to 2 ng/mL (n=6). For this study the matrix-matched standard solutions was not used for quantification because the sensitivity of the analyte was not influenced by matrix.

Analytical method L0076/09 (Ford, 2017, BASF DocID 2017_1192681) was independently validated for the determination of mefentrifluconazole residues in seven different plant matrices (orange fruit, coffee grains, dry bean seeds, soya bean grain, tomato fruit, wheat straw and grain) fortified at the LOQ of 0.01 mg/kg and 0.1 mg/kg. Average recoveries ranged from 76–100 percent with percentRSD ranging from 0.5–8.9 percent.

This method was also subjected to radiovalidation, where wheat (straw and forage), soya bean (green pod), and grape (berries) from the metabolism studies (Birk *et al*., 2015, BASF DocID 2014_1261057) were analysed using method L0076/09. When comparing the results from the residue analysis procedure against those of the metabolism extraction procedures, extraction efficiencies for mefentrifluconazole from wheat forage, wheat straw, soya bean green pods, and grape berries were 98 percent, 111 percent, 102 percent, and 93 percent, respectively, demonstrating the efficiency of the analytical method to extract incurred residues of mefentrifluconazole (Table 61). The analytical method L0076/09 was demonstrated to be suitable for the analysis of mefentrifluconazole in plant matrices.

Table 61 Validation of method L0076/09 for the analysis of mefentrifluconazole in plant matrices

Commodity	Analyte	Fortification levels (mg/kg)	No of samples	Range of recoveries (%)	Mean recovery (%)	RSD (%)	Reference
Citrus (fruit)	Mefentrifluconazole *m/z* 398→182	0.01	6	72-79	75	4.0	2015_3001681
		0.1	6	71-84	80	6.3	
		1.0	5	74-87	80	6.3	
	Mefentrifluconazole *m/z* 398→133	0.01	6	72-82	78	4.8	
		0.1	6	76-86	83	5.1	
		1.0	5	72-85	80	6.5	
	Mefentrifluconazole *m/z* 398→182	0.01	6	71-81	78	5.1	
		0.1	6	75-87	81	5.4	
		1.0	5	71-92	79	12	
	Mefentrifluconazole *m/z* 398→133	0.01	6	75-85	81	4.3	
		0.1	6	74-93	86	9.6	
		1.0	5	70-87	78	9.3	
	Mefentrifluconazole *m/z* 398→182	0.01	5	87-92	89	2.2	2017_1192681
		0.1	5	90-96	93	2.4	
	Mefentrifluconazole *m/z* 398→133	0.01	5	92-104	97	5.0	
		0.1	5	90-97	94	2.9	
Coffee (grain)	Mefentrifluconazole *m/z* 398→182	0.01	6	93-105	98	4.0	2015_3001681
		0.1	6	85-92	89	3.2	
		1.0	6	88-94	90	2.2	
	Mefentrifluconazole *m/z* 398→133	0.01	6	90-107	100	6.1	
		0.1	6	86-97	90	4.6	
		1.0	6	88-95	91	3.4	
	Mefentrifluconazole	0.01	6	78-89	83	4.4	

Commodity	Analyte	Fortification levels (mg/kg)	No of samples	Range of recoveries (%)	Mean recovery (%)	RSD (%)	Reference
	m/z 398→182	0.1	6	83-90	86	3.3	
		1.0	6	84-90	88	2.3	
	Mefentrifluconazole *m/z* 398→133	0.01	6	75-88	81	6.6	
		0.1	6	79-94	87	6.2	
		1.0	6	79-89	85	5.0	
	Mefentrifluconazole *m/z* 398→182	0.01	5	83-103	89	8.9	2017_1192681
		0.1	5	89-96	93	3.4	
	Mefentrifluconazole *m/z* 398→133	0.01	5	87-102	93	6.3	
		0.1	5	91-97	94	3.2	
Dry bean (seed)	Mefentrifluconazole *m/z* 398→182	0.01	6	82-89	85	2.8	2015_3001681
		0.1	6	77-85	82	3.4	
		1.0	6	75-83	78	4.6	
	Mefentrifluconazole *m/z* 398→133	0.01	6	77-88	83	6.5	
		0.1	6	70-93	83	9.2	
		1.0	6	76-85	81	4.1	
	Mefentrifluconazole *m/z* 398→182	0.01	6	84-97	91	5.7	
		0.1	6	80-99	87	7.9	
		1.0	6	80-89	84	4.8	
	Mefentrifluconazole *m/z* 398→133	0.01	6	82-103	91	10	
		0.1	6	75-94	84	9.2	
		1.0	6	75-92	83	8.2	
	Mefentrifluconazole *m/z* 398→182	0.01	5	90-91	90	0.5	2017_1192681
		0.1	5	87-89	88	1.0	
	Mefentrifluconazole *m/z* 398→133	0.01	5	97-111	105	6.1	
		0.1	5	89-92	90	1.3	
Soya bean (seed)	Mefentrifluconazole *m/z* 398→182	0.01	5	78-92	83	6.2	2015_3001681
		0.1	6	85-92	89	3.3	
		1.0	5	85-93	88	3.3	
	Mefentrifluconazole *m/z* 398→133	0.01	5	80-90	84	4.5	
		0.1	6	83-96	90	6.2	
		1.0	5	86-97	91	4.7	
	Mefentrifluconazole *m/z* 398→182	0.01	5	90-104	94	7.0	
		0.1	6	84-87	86	1.1	
		1.0	5	84-95	89	4.7	
	Mefentrifluconazole *m/z* 398→133	0.01	5	72-80	78	4.4	
		0.1	6	71-90	82	8.9	
		1.0	5	80-89	82	4.3	
	Mefentrifluconazole *m/z* 398→182	0.01	5	82-89	86	3.1	2017_1192681
		0.1	5	88-91	89	0.9	
	Mefentrifluconazole *m/z* 398→133	0.01	5	93-99	95	3.0	
		0.1	5	89-91	90	0.8	
Tomato (fruit)	Mefentrifluconazole *m/z* 398→182	0.01	5	78-96	87	8.4	2015_3001681
		0.1	6	73-85	79	6.2	
		1.0	5	70-79	73	4.85	
	Mefentrifluconazole *m/z* 398→133	0.01	5	81-91	86	5.1	
		0.1	6	73-83	79	4.8	
		1.0	5	71-80	75	4.8	
	Mefentrifluconazole *m/z* 398→182	0.01	5	82-97	89	6.9	
		0.1	6	73-92	82	8.1	
		1.0	5	70-82	77	7.4	
	Mefentrifluconazole *m/z* 398→133	0.01	5	72-85	78	6.3	
		0.1	6	72-88	79	9.2	
		1.0	5	72-87	79	8.0	
	Mefentrifluconazole	0.01	5	83-86	84	1.5	2017_1192681

Commodity	Analyte	Fortification levels (mg/kg)	No of samples	Range of recoveries (%)	Mean recovery (%)	RSD (%)	Reference
	m/z 398→182	0.1	5	85-88	87	1.3	
	Mefentrifluconazole	0.01	5	90-107	99	6.2	
	m/z 398→133	0.1	5	88-91	89	1.5	
Wheat (grain)	Mefentrifluconazole	0.01	5	78-87	84	9.2	2015_3001681
	m/z 398→182	0.1	6	83-88	85	2.8	
		1.0	6	79-105	87	10	
	Mefentrifluconazole	0.01	5	73-97	81	11	
	m/z 398→133	0.1	6	80-84	83	2.5	
		1.0	6	73-102	84	12	
	Mefentrifluconazole	0.01	6	72-84	78	6.8	
	m/z 398→182	0.1	6	70-93	82	9.4	
		1.0	5	86-111	92	11	
	Mefentrifluconazole	0.01	6	74-106	90	14	
	m/z 398→133	0.1	6	86-104	95	8.0	
		1.0	5	81-109	90	12	
	Mefentrifluconazole	0.01	5	89-93	90	1.9	2017_1192681
	m/z 398→182	0.1	5	88-92	91	1.9	
	Mefentrifluconazole	0.01	5	97-105	101	3.1	
	m/z 398→133	0.1	5	89-94	92	2.2	
Wheat (straw)	Mefentrifluconazole	0.01	6	73-92	86	8.6	2015_3001681
	m/z 398→182	0.1	6	75-87	80	6.3	
		1.0	6	74-86	80	6.5	
	Mefentrifluconazole	0.01	6	72-97	87	10	
	m/z 398→133	0.1	6	75-86	81	4.5	
		1.0	6	77-86	83	4.0	
	Mefentrifluconazole	0.01	5	70-77	73	4.1	
	m/z 398→182	0.1	6	71-83	78	5.8	
		1.0	6	71-96	85	11	
	Mefentrifluconazole	0.01	5	70-92	82	9.6	
	m/z 398→133	0.1	6	77-88	84	6.2	
		1.0	6	77-90	83	7.0	
	Mefentrifluconazole	0.01	5	79-85	82	3.8	2017_1192681
	m/z 398→182	0.1	5	82-87	84	2.9	
	Mefentrifluconazole	0.01	5	70-83	76	6.7	
	m/z 398→133	0.1	5	82-89	86	3.8	

Method L0295/01

Analytical method L0295/01 (Klimmek *et al*., 2015, BASF DocID 2015_1106708) is a multiresidue method (QuEChERs) that involves extraction of tomato (fruit), orange (whole fruit), dry bean seeds, wheat grain, and soya bean seeds with acetonitrile (water was added prior to extraction if needed). The extract was partitioned by adding a salt solution containing magnesium sulphate, sodium chloride, and sodium citrate followed by centrifugation. The acetonitrile phase was purified by freezing out at ≤ -18 °C (except for tomato fruit) and/or by dispersive SPE with primary/secondary amine (PSA) and Octadecyl silica (ODS) sorbent (QuEChERS modules C1 and C4, respectively). The extract was reconstituted into acetonitrile and water.

The final extracts were quantified for residues of mefentrifluconazole using LC-MS/MS, comprising a C18 column that incorporated 0.1 percent formic acid in acetonitrile and 0.1 percent formic acid in water mobile phases. MS/MS quantitation was by electrospray ionisation in positive mode with a mass transition of *m/z* 398 → 70, while confirmation analysis was conducted with a mass transition of

m/z 398→ 182. The LOQ was 0.01 mg/kg for tomato (fruit), orange (whole fruit), dry beans (seeds), wheat (grain) and dry soya beans (seeds).

Recovery and repeatability data for the determination of mefentrifluconazole residues in plant matrices are presented in Table 62. Average recovery values ranged from 70–97 percent for the two fortification levels tested while the percentRSD ranged from 1.1–20 percent. Additionally, two replicates of unfortified samples were examined and results were all below 0.3× LOQ. No interfering peaks were detected in the samples analysed. The linearity of the detector response (r^2 > 0.99), using solvent standards (tomato fruit, dry bean seeds, and dry soya bean seeds) and matrix-matched standards (wheat grain), over a concentration range of 0.15 to 7.5 ng/mL (n=7).

Analytical method L0295/01 (Richter *et al*., 2015, BASF DocID 2015_1240004) was independently validated for the determination of mefentrifluconazole residues in five different plant matrices (tomato fruit, wheat grain, dried broad beans, dried soya beans and whole orange) fortified at the LOQ of 0.01 mg/kg and 0.1 mg/kg. Average recoveries ranged from 85–110 percent with percentRSD ranging from 1.1–10 percent.

This method was also subjected to radiovalidation, where wheat (straw and forage), soya bean (green pod), and grape (berries) from the metabolism studies (Birk *et al*., 2015, BASF DocID 2014_1261057) were analysed using the QuEChERS multiresidue method. When comparing the results from the residue analysis procedure against those of the metabolism extraction procedures, extraction efficiencies for mefentrifluconazole from wheat forage, wheat straw, soya bean green pods, and grape berries were 80 percent, 59 percent, 99 percent, and 98 percent, respectively, which demonstrated the efficiency of the analytical method to extract incurred residues of mefentrifluconazole from most matrices. In the metabolism study, wheat straw samples were extracted three times with methanol and twice with water. However analytical method L0295/01 only extracted samples once with acetonitrile which may explain the low extraction efficiency for this method in wheat straw. The method L0295/01 was demonstrated to be suitable for the analysis of mefentrifluconazole in plant matrices (Table 62).

Table 62 Validation of method L0295/01 (QuEChERS) for the analysis of mefentrifluconazole in plant matrices

Commodity	Analyte	Fortification levels (mg/kg)	No of samples	Range of recoveries (%)	Mean mecovery (%)	RSD (%)	Reference
Tomato (fruit)	Mefentrifluconazole *m/z* 398→70	0.01	5	72-76	75	2.2	2015_1106708
		0.1	5	77-85	82	3.8	
	Mefentrifluconazole *m/z* 398→182	0.01	5	80-85	82	2.9	
		0.1	5	78-82	80	1.9	
	Mefentrifluconazole *m/z* 398→70	0.01	5	83-86	85	1.5	2015_1240004 ILV
		0.1	5	75-96	85	10	
	Mefentrifluconazole *m/z* 398→182	0.01	5	83-86	85	1.7	
		0.1	5	75-94	85	9.6	
Orange (whole fruit)	Mefentrifluconazole *m/z* 398→70	0.01	5	78-91	84	6.2	2015_1106708
		0.1	5	81-90	86	4.2	
	Mefentrifluconazole *m/z* 398→182	0.01	5	81-89	84	3.5	
		0.1	5	78-84	81	3.5	
	Mefentrifluconazole *m/z* 398→70	0.01	5	106-111	109	1.7	2015_1240004 ILV
		0.1	5	106-120	110	5.1	
	Mefentrifluconazole *m/z* 398→182	0.01	5	108-111	110	1.1	
		0.1	5	104-123	110	6.9	
Dry beans (seeds)	Mefentrifluconazole *m/z* 398→70	0.01	5	85-96	91	4.4	2015_1106708
		0.1	5	63-110	97	20	
	Mefentrifluconazole	0.01	5	88-96	92	3.7	

Commodity	Analyte	Fortification levels (mg/kg)	No of samples	Range of recoveries (%)	Mean mecovery (%)	RSD (%)	Reference
	m/z 398→182	0.1	5	62-102	90	18	
	Mefentrifluconazole	0.01	5	103-118	108	5.2	2015_1240004
	m/z 398→70	0.1	5	101-109	105	2.9	ILV
	Mefentrifluconazole	0.01	5	102-116	107	4.8	
	m/z 398→182	0.1	5	103-109	105	2.2	
Wheat (grain)	Mefentrifluconazole	0.01	5	73-89	84	7.5	2015_1106708
	m/z 398→70	0.1	5	88-96	92	4.3	
	Mefentrifluconazole	0.01	5	86-89	88	1.1	
	m/z 398→182	0.1	5	88-97	93	4.5	
	Mefentrifluconazole	0.01	5	105-117	110	4.2	2015_1240004
	m/z 398→70	0.1	5	108-113	110	1.9	ILV
	Mefentrifluconazole	0.01	5	101-118	107	6.1	
	m/z 398→182	0.1	5	107-113	110	2.1	
Dry soya beans (seeds)	Mefentrifluconazole	0.01	5	70-81	75	6.1	2015_1106708
	m/z 398→70	0.1	5	68-76	72	5.1	
	Mefentrifluconazole	0.01	5	68-75	72	3.9	
	m/z 398→182	0.1	5	64-78	70	8	
	Mefentrifluconazole	0.01	5 (4[1])	106-142[1]	110	4.7	2015_1240004
	m/z 398→70	0.1	5	108-115	110	2.9	ILV
	Mefentrifluconazole	0.01	5 (4[1])	104-145[1]	109	5.8	
	m/z 398→182	0.1	5	108-115	110	2.6	

Notes:

[1] Statistical outlier according to Dixons Q test (95 percent confidence interval) and was discarded for the calculation of the mean recovery and RSD.

Method 1511/01

Analytical method 1511/01 (Downs, 2016, BASF DocID 2015_7005822) was validated and considered acceptable as a method for data collection when analysing for residues of mefentrifluconazole in grape fruit, apple fruit, wheat grain, dried bean seed, and canola seed with an LOQ of 0.01 mg/kg in each matrix.

The method employed extraction by shaking with methanol:water (80:20, v/v). The extract is subsequently centrifuged, diluted with methanol:water (50:50, v/v), and filtered. The extracts were quantified for residues of mefentrifluconazole using LC-MS/MS, comprising a reversed phase column that incorporated 0.1 percent formic acid in water and 0.1 percent formic acid in acetonitrile mobile phases. MS/MS quantitation for mefentrifluconazole was by electrospray ionisation in positive mode with a mass transition of m/z 398 → 70, while confirmation analysis was conducted with a mass transition of m/z 400 → 70.

Recovery and repeatability data for the determination of mefentrifluconazole residues in plant matrices are presented in Table 63. Average recovery values ranged from 72–122 percent for the two fortification levels tested while the percentRSD ranged from 2–18 percent. Additionally, two replicates of unfortified samples were examined and results were all below 0.002 mg/kg (0.2× LOQ). No interfering peaks were detected in the samples analysed. The linearity of the detector response (r^2 > 0.9938), using solvent-based calibration standards, over a concentration range of 0.004 to 0.4 ng/mL (n=5).

This method was also subjected to radiovalidation, where wheat (straw and forage), soya bean (green pod), and grape (berries) from the metabolism studies (Rabe *et al*., 2017, BASF DocID 2016_1126366) were analysed using method 1511/11. When comparing the results from the residue

analysis procedure against those of the metabolism extraction procedures, extraction efficiencies for mefentrifluconazole from wheat forage, wheat straw, soya bean green pods, and grape berries were 98 percent, 100 percent, 114 percent, and 96 percent, respectively, which demonstrates the efficiency of the analytical method to extract incurred residues of mefentrifluconazole. The method 1511/01 was demonstrated to be suitable for the analysis of mefentrifluconazole in plant matrices (Table 63).

Table 63 Validation of method 1511/01 for the analysis of mefentrifluconazole in plant matrices

Commodity	Analyte	Fortification levels (mg/kg)	No of samples	Range of recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
Grape, fruit	Mefentrifluconazole *m/z* 398→70	0.01	5	99-105	103	2	2015_7005822
		1.0	5	112-128	121	7	
	Mefentrifluconazole *m/z* 398→182	0.01	5	97-104	99	3	
		1.0	5	113-131	122	6	
Apple, fruit	Mefentrifluconazole *m/z* 398→70	0.01	5	91-105	98	6	2015_7005822
		1.0	5	95-116	105	7	
	Mefentrifluconazole *m/z* 398→182	0.01	5	93-117	107	8	
		1.0	5	108-131	119	8	
Wheat, grain	Mefentrifluconazole *m/z* 398→70	0.01	5	70-75	72	3	2015_7005822
		1.0	5	99-136	114	14	
	Mefentrifluconazole *m/z* 398→182	0.01	5	67-79	72	6	
		1.0	5	88-136	108	18	
Bean, dried seed	Mefentrifluconazole *m/z* 398→70	0.01	5	102-109	105	3	2015_7005822
		1.0	5	101-114	106	5	
	Mefentrifluconazole *m/z* 398→182	0.01	5	103-118	109	5	
		1.0	5	105-125	112	8	
Canola, seed	Mefentrifluconazole *m/z* 398→70	0.01	5	96-116	103	8	2015_7005822
		1.0	5	94-100	97	3	
	Mefentrifluconazole *m/z* 398→182	0.01	5	95-116	103	8	
		1.0	5	95-103	99	3	

Animal matrices

Method L0272/01

Analytical method L0272/01 has been developed for the analysis of mefentrifluconazole in animal matrices, including bovine meat, milk, cream, fat, liver, kidney and hen eggs (Devine, 2015, BASF DocID 2015_1106707).

For matrices containing fat (milk, cream, fat), mefentrifluconazole is extracted using a mixture of acetonitrile and iso-hexane. An aliquot of the extract is centrifuged and partitioned twice using iso-hexane. For matrices containing proteins (muscle, kidney, liver and egg), mefentrifluconazole is extracted using a mixture of methanol/water/2N HCl (70/25/5, v/v/v). An aliquot of the extract is centrifuged and partitioned twice using cyclohexane. Final analysis is performed by LC-MS/MS and monitoring for two mass ion transitions (398 → 182 m/z for quantification and 398 → 133 m/z). The LOQ was 0.01 mg/kg.

Recovery and repeatability data for the determination of mefentrifluconazole residues in animal matrices are presented in Table 64. Average recovery values ranged from 73–110 percent for the two fortification levels tested while the percent RSD ranged from 0.4–11 percent. No interfering peaks were detected in the samples analysed. The linearity of the detector response (r^2 > 0.9997), using matrix matched and non-matrix matched standards, depending on the animal commodity, covered a range of concentrations of 0.04 to 10 ng/mL (n=8).

The LC-MS/MS method L0272/01 was successfully validated by an independent laboratory (Richter et al., 2015, BASF DocID 2015_1240005) for the determination of mefentrifluconazole residues in seven different animal matrices (milk, cream, fat, egg, meat, kidney and liver) fortified at the LOQ of 0.01 mg/kg and 0.10 mg/kg. Average recoveries ranged from 77–101 percent with percent RSD ranging from 2.2–19 percent. The method L0272/01 was demonstrated to be suitable for the analysis of mefentrifluconazole in all animal matrices based (Table 64)

Table 64 Recoveries of mefentrifluconazole in animal matrices by LC-MS/MS (L0272/01) Ion Mass Ion Mass Transition 398 → 133 m/z (confirmation)

	Fortification level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
Matrix		Transition 398 → 182 m/z (quantification)			Transition 398 → 133 m/z (confirmation)		
Bovine meat	0.01	80.4-89.4	85.0	4.4	85.3-97.8	93.0	5.8
	0.10	109-110	110	0.4	106-111	108	1.8
Bovine milk	0.01	71.9-89.1	82.0	8.8	68.3-82.7	76.5	7.0
	0.10	84.5-87.1	85.8	1.4	83.4-89.4	86.3	2.9
Bovine cream	0.01	69.4-75.3	72.6	3.2	69.0-75.2	72.1	3.8
	0.10	79.2-92.8	86.4	5.6	81.8-92.0	86.9	4.4
Bovine fat	0.01	70.1-94.1	80.2	11	71.4-89.0	80.8	8.6
	0.10	99.0-111	104	4.9	98.8-109	104	4.7
Bovine liver	0.01	83.2-94.0	87.5	5.2	84.6-92.0	88.3	3.9
	0.10	92.5-101	96.4	3.8	91.4-99.6	95.3	3.2
Bovine kidney	0.01	94.5-100	96.2	2.3	94.3-99.3	95.9	2.1
	0.10	94.2-106	101	4.6	89.1-104	100	6.7
Hen eggs	0.01	90.9-97.2	93.3	3.1	83.7-99.7	92.9	6.4
	0.10	102-107	105	2.0	110-111	110	0.5

Method D1704/01

Residues of mefentrifluconazole in livestock commodities are extracted with acidified acetonitrile by mechanical shaking; for "dry" matrices (liver, kidney, muscle, fat), the homogenized samples are soaked (hydrated) first by the addition of water (Downs, 2017, BASF DocID 2017_7008027). The residues in the extracts are cleaned-up and partitioned by shaking in the presence of a mixture of salts (magnesium sulfate and sodium acetate) into the organic layer, and centrifuged. The residues in an aliquot of the acetontrile layer are then further purified with the addition of a second salt mixture (containing $MgSO_4$ to remove residual water and primary secondary amine (PSA) sorbent to remove other interferences) and centrifuged. The residues in an aliquot of the resulting extract are diluted to final volume with 5 mM formic acid in acetonitrile:water (25:75, v/v). Final determination by LC-MS/MS was conducted by monitoring two MS/MS ion mass transitions (398→70 m/z for quantification and 400→70 m/z for confirmation). The LOQ was 0.01 mg/kg.

Matrix effects on the detection of mefentrifluconazole in extracts of bovine liver, kidney, muscle, fat, milk and poultry egg were found to be insignificant (< 20 percent). Therefore, solvent standards were used for quantification. All mean recoveries obtained at each fortification level were within the range of 78–100 percent with a RSD of 4–19 percent. Quantitation of residues in all samples was achieved using calibration curves calculated by linear regression (1/x weighting) of instrument responses. Linearity was demonstrated over a range of eight concentrations from 0.004 to 0.4 ng/mL (r=0.9981).

The LC-MS/MS method D1704/01 was successfully validated by an independent laboratory (Ivanov, 2017, BASF DocID 2017_1198105) for the determination of mefentrifluconazole residues in six different animal matrices (bovine liver, kidney, muscle, fat and hen eggs) fortified at the LOQ of 0.01 and 1.0 mg/kg. Average recoveries ranged from 86–110 percent with percent RSD of ≤ 15 percent,

demonstrating the reproducibility of the method. The results demonstrated the ability of the method to reliably determine residues of mefentrifluconazole in all animal matrices (Table 65).

Table 65 Recoveries of mefentrifluconazole in animal matrices by LC-MS/MS (Method D1704/01)

Matrix	Fortification level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
		Transition 398 → 70 m/z (quantification)			Transition 400 → 70 m/z (confirmation)		
Bovine liver	0.01	90-113	100	9	92-114	98	9
	1.0	88-100	94	5	81-100	90	8
Bovine kidney	0.01	82-105	94	11	82-114	99	15
	1.0	82-102	94	8	88-107	97	9
Bovine muscle	0.01	94-103	97	3	89-103	95	5
	1.0	91-100	94	4	86-98	93	4
Bovine fat	0.01	77-99	90	8	77-100	91	9
	1.0	67-88	78	9	70-88	78	7
Bovine milk	0.01	73-92	81	7	68-97	79	12
	1.0	82-109	92	13	78-114	93	18
Hen eggs	0.01	69-91	82	8	59-99	81	14
	1.0	82-103	89	8	76-111	89	15

Method L0309/01 – Metabolite M750F022

The analytical method L0309/01 was developed for the determination of the metabolite M750F022 in animal matrices including bovine meat, milk, fat, liver, kidney and hen eggs (Heger, 2015, BASF DocID 2015_1106706).

For fat containing matrices (milk and fat), samples are extracted twice with acetonitrile and iso-hexane. The resulting acetonitrile phase is partitioned twice against iso-hexane after which the acetonitrile phase is dried, dissolved in MeOH/H_2O (50/50) cleaned up on an SPE column prior to GC-MS analysis. For protein containing matrices (egg, muscle, liver and kidney) samples are extracted by macerating with MeOH/H_2O/2N HCl (70/25/5). The extract is partitioned twice against 0.2N NaOH and cyclohexane (muscle and liver) or dichloromethane (egg and kidney). An aliquot of the cyclohexane or dichloromethane phase is dried and dissolved in MeOH/H_20 (50/50) prior to clean up on an SPE column and analysis using GC-MS, monitoring one characteristic fragment ion for quantification (295 m/z) and two characteristic fragment ions for confirmation (297 m/z, 317 m/z). The LOQ was 0.01 mg/kg.

Recovery and repeatability data for the determination of residues of the metabolite M750F022 in animal matrices are presented in Table 66. Except for bovine fat, average recovery values ranged from 71–104 percent for the two fortification levels tested while the percent RSD ranged from 3.4–9.6 percent. In fat, average recoveries ranged from 108–124 percent with percentRSD ≤9.7 percent. No interfering peaks were detected in the samples analysed. The linearity of the detector response (r^2 > 0.9929), using matrix matched standards, covered a range of concentrations of 2.5 to 100 ng/mL (n=8).

During the validation of method L0309/01, the results demonstrated that the matrix-load in bovine fat and milk had a significant influence on the detection of the analyte. However, the validation was carried out using solvent standards. Therefore, the method was revalidated using matrix-matched standards in order to show the applicability of the method in these matrices (Heger, 2015, BASF DocID 2017/1002385). The mean recoveries resulting from these revalidation experiments were between 79 percent and 96 percent within the same fortification levels and fragment ions. The relative standard deviations for all fortification levels were ≤ 10 percent.

The GC-MS method L0309/01 was successfully validated by an independent laboratory (Bendig et al., 2015, BASF DocID 2015_1240006) for the determination of metabolite M750F022 residues in six

different animal matrices (milk, fat, egg, meat, kidney and liver) fortified at the LOQ of 0.01 mg/kg and 0.10 mg/kg. Average recoveries ranged from 72–101 percent with percentRSD ranging from 3.1–18 percent. Despite the minor modifications made during the ILV, none had an important impact on the analyte determination in animal matrices. Furthermore, based on acceptable recoveries and retention time, sensitivity and selectivity being comparable to those during method development, the method was shown to be reproducible.

The overall procedural validation of the GC-MS analytical method L0309/01 demonstrated the ability of the method to reliably determine residues of M750F022 in all animal matrices (Table 66).

Table 66 Recoveries of metabolite M750F022 in animal matrices by GC-MS

Matrix	Fortification level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
		Fragment 295 m/z (quantification)			Fragment 297 m/z			Fragment 317 m/z		
Bovine meat	0.01	75.1-89.7	82.4	7.7	74.6-84.4	79.2	5.5	72.5-87.4	79.9	6.9
	0.10	73.8-89.9	79.5	7.9	73.8-93.8	80.4	9.9	73.1-91.9	79.0	9.4
Bovine milk	0.01	74.1-88.9	79.5	8.2	75.8-88.8	80.2	6.9	74.9-88.9	79.7	6.7
	0.10	70.2-85.2	76.8	7.1	70.2-86.2	76.6	7.7	72.6-82.2	76.9	4.6
Bovine fat	0.01	118-129	124	3.4	112-127	121	5.1	104-122	114	6.6
	0.10	107-130	113	9.3	100-130	113	9.7	98-122	108	8.4
Bovine liver	0.01	78.1-87.8	83.9	5.8	79.6-87.8	82.8	3.9	78.7-85.5	82.1	3.4
	0.10	64.9-77.1	71.0	8.6	64.3-77.1	70.8	8.1	65.4-76.6	71.2	7.0
Bovine kidney	0.01	81.3-93.4	89.2	5.6	79.8-86.8	84.1	3.7	81.6-91.5	86.5	4.3
	0.10	70.8-80.0	75.6	4.6	70.3-75.9	73.4	3.5	71.9-82.3	77.3	5.0
Hen eggs	0.01	77.0-95.4	85.6	7.8	76.5-91.3	84.1	7.1	74.4-85.3	81.1	6.3
	0.10	94.8-117	102	8.7	96.1-115	102	7.5	95.1-119	104	9.6

Method L0309/02– M750F022 and Fatty Acid Conjugate M750F025 in poultry matrices

This method was developed to allow the determination of M750F022 and its fatty acid conjugates in hen matrices using GC-MS *(*Guedez Orozco, 2016, BASF DocID 2016_1001326). In order to determine the magnitude of residues of the fatty acid conjugates, the amount of M750F022 was determined in each sample twice, with and without hydrolysis. The fatty acid conjugates of M750F022 are hydrolysed to M750F022 using NaOH (10 M). In order to verify the functionality of the method, fortifications were done using the conjugate M750F025 as being representative of all the fatty acid conjugates, which are measured as M750F022. The fatty acid conjugates of M750F022 are extracted from fat containing matrices (fat, skin) with iso-hexane followed by two liquid-liquid partitions with acetonitrile. NaOH (10 M) together with tetrahydrofuran were added to the acetonitrile phase to initiate hydrolysis. The hydrolysate was dissolved in MeOH/water (50/50) and cleaned up using an SPE column. For protein containing matrices (muscle, liver and egg), the same procedure is followed, with the exception of the initial extraction where MeOH is used. Analysis is conducted using GC-MS monitoring one characteristic fragment ion for quantification (295 m/z) and two characteristic fragment ions for confirmation (297 m/z,

317 m/z). The LOQ was 0.01 mg/kg in each matrix for M750F022. Good linearity of the detector response was observed in the range of 2.5 to 100 ng/mL.

The analytical method L0309/02 is equivalent to the analytical method L0309/01 for the determination of M750F022 in animal matrices except for the extraction solvent used, which was changed to acetonitrile:iso-hexane (for fat containing matrices) or methanol (for protein containing matrices). This modification to the method was made because it was observed during method development that the use of HCl in the extraction solvent led to lower recoveries for the fatty acid conjugates. This does not have any impact on the extractability of M750F022, because the extraction solvent was the same as that used in the metabolism study.

The mean recovery values of the validation experiments were between 71 and 97 percent at both fortification levels of 0.01 and 0.10 mg/kg, respectively, except for hen liver and fat fortified at 0.10 mg/kg where recoveries were 62 percent (fragment 317 m/z) and 62–65 percent (all fragment ions), respectively (Table 67). The relative standard deviations for all fortification levels were ≤16 percent, demonstrating overall good repeatability.

Due to the consistently lower recoveries of the fatty acid conjugate M720F025 (reported as M750F022) in hen fat, method L0309/02 was revalidated (Heger, 2017, BASF DocID 2016_1002385). Following the revalidation, recoveries of M750F022 ranged from 79.1–82.1 (percent RSD ≤ 12.7), 73.0–84.1 percent (percent RSD≤14.9) and 78.2–85.9 percent (percent RSD≤14.3) when hen fat was fortified with the fatty acid conjugate at 0.01, 0.10 and 1.0 mg/kg, respectively.

The overall procedural validation of the GC-MS analytical method L0309/02 demonstrated the ability of the method to reliably determine residues of M750F025 as M750F022 in all hen matrices (Table 67)

Table 67 Recoveries (n=5) of M750F025 in hen matrices measured as M750F022 using GC-MS

Matrix	Fortification level (mg/kg)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
		Fragment 295 m/z (quantification)			Fragment 297 m/z			Fragment 317 m/z		
Hen egg	0.01	81.2-93.3	86.1	6.6	79.3-88.3	83.4	5.0	77.0-98.0	82.3	7.1
	0.10	79.6-89.8	84.4	5.3	79.3-90.1	84.2	5.8	79.1-90.6	84.1	5.4
Hen liver	0.01	63.3-89.0	75.4	16	66.8-88.7	75.3	12	70.7-78.1	73.7	4.3
	0.10	85.0-93.3	89.9	3.4	86.0-92.3	89.6	2.7	56.6-68.4	62.3	7.1
Hen muscle	0.01	89.5-107	96.9	7.3	85.1-105	94.5	8.4	78.0-103	86.8	13
	0.10	83.0-96.3	90.7	6.6	81.8-95.5	89.9	6.7	74.5-82.8	77.5	4.8
Hen fat	0.01	66.6-73.6	70.8	4.1	68.2-74.1	70.7	3.5	70.8-78.2	73.7	4.3
	0.10	58.8-71.1	65.3	7.7	58.8-70.3	65.0	7.6	56.6-68.5	62.3	7.1

The efficiency of the analytical methods L0272/01 and L0309/01 to adequately extract bio-incurred residues of mefentrifluconazole and M750F022 from samples collected from the lactating goat and laying hen metabolism studies was investigated (Thiaener *et al*., 2016, BASF DocID 2015_1161960). Cream, whole milk and kidney from lactating goats as well as fat, liver, muscle and egg yolk from laying hens were selected as edible matrices containing the target compounds and total residue concentrations above 0.010 mg/kg. The results of each extraction procedure were compared to the extraction performed within the respective metabolism studies.

For mefentrifluconazole, extraction efficiencies were 80 percent or higher for most matrices (milk, cream, muscle, kidney, fat, egg yolk), and lower for liver (46 percent) using method L0272/01. The extractability of mefentrifluconazole using the multi-residue method DFG S 19 was similar to the

extraction within the respective metabolism studies (95.1 to 112.1 percent) for the goat matrices and hen muscle and hen egg yolk, whereas the amounts of mefentrifluconazole extracted from hen fat (62.5 percent) and hen liver (74.3 percent) were lower. The extraction efficiency of the QuEChERS method was comparable to the metabolism study (80.1 to 99.1 percent) for most matrices; however, for hen egg yolk and fat, it was lower (73.0 and 6.0 percent, respectively) (Table 68).

For M750F022, extraction efficiencies were 90 percent or higher for most matrices (milk, cream, kidney, fat) and lower for egg yolk (66 percent), muscle (61 percent) and liver (46-50 percent) using method L0309/01. The concentrations of metabolite M750F022 extracted using method DFG S 19 were higher for egg yolk and cream (123.8 and 144.3 percent, respectively) or comparable for the other matrices (79.7 to 105.6 percent). The extractability of M750F022 using the QuEChERS method was similar to the extractability within the respective metabolism study for cream, kidney, liver and muscle (from 80.5 to 95.1 percent). Lower amounts of M750F022 were extracted from egg yolk (67.8 percent) and hen fat (19.0 percent) using QuEChERS (Table 69).

Table 68 Summary of extraction efficiency of methods L0272/01, DFG S19 and QuEChERS for mefentrifluconazole

Extraction Procedure	TRR (mg/kg)	mg/kg	% TRR	Extraction efficiency %[A]
Cream (goat)				
Goat metabolism study	0.207	0.156	75.6	100.0
L0272/01[B]	0.272	0.225	82.5	109.1
		0.207	75.8	100.2
Method DFG S 19		0.221	77.4	102.4
Method QuEChERS		0.201	73.7	97.5
Milk (goat)				
Goat metabolism study	0.062	0.028	44.5	100.0
L0272/01		0.025	40.1	90.1
Method DFG S 19		0.031	49.9	112.1
Method QuEChERS		0.027	44.1	99.1
Kidney (goat)				
Goat metabolism study	0.429	0.198	46.0	100.0
L0272/01		0.162	37.7	82.0
Method DFG S 19		0.194	45.2	98.1
Method QuEChERS		0.165	38.4	83.4
Fat (hen)				
Hen metabolism study	0.702	0.038	5.4	100.0
L0272/01		0.036	5.1	95.0
Method DFG S 19		0.023	3.3	62.5
Method QuEChERS		0.002	0.3	6.0
Liver (hen)				
Hen metabolism study	0.320	0.023	7.2	100.0
L0272/01[B]		0.011	3.3	46.4
		0.011	3.4	47.9
Method DFG S 19		0.017	5.3	74.3
Method QuEChERS		0.019	6.0	84.1

Extraction Procedure	TRR (mg/kg)	mg/kg	% TRR	Extraction efficiency %[A]
Muscle (hen)				
Hen metabolism study	0.050	0.003	5.6	100.0
L0272/01		0.003	5.2	94.1
Method DFG S 19		0.003	5.3	95.1
Method QuEChERS		0.002	4.4	80.1
Egg yolk				
Hen metabolism study	0.477	0.031	6.5	100.0
L0272/01		0.028	6.0	92.4
Method DFG S 19		0.030	6.3	97.7
Method QuEChERS		0.023	4.7	73.0

Notes:

[A]. extraction efficiency = amounts extracted using analytical method compared to amount extracted in metabolism study (normalizedto 100 percent). For cream (goat) extraction efficiency was calculated as the percent TRR in metabolism study divided by percent TRR extracted with analytical method.

[B]. In the case of cream (goat) and liver (hen), two different subsamples were extracted for confirmation.

Table 69 Summary of extraction efficiency of methods L0309/01, DFG S19 and QuEChERS for metabolite M750F022

Extraction Procedure	TRR (mg/kg)	mg/kg	% TRR	Extraction Efficiency (%) [A]
Cream (goat)				
Goat metabolism study	0.207	0.009	4.2	100.0
L0309/01[B]	0.272	0.012	4.5	106.9
		0.008	3.0	73.1
Method DFG S 19		0.016	6.0	144.3
Method QuEChERS		0.011	4.0	95.1
Milk (goat)				
Goat metabolism study	0.062	0.001	1.2	100.0
L0309/01		0.002	2.5	*(213.6)*[C]
Method DFG S 19		0.001	1.9	(162.2)[C]
Method QuEChERS		0.001	2.0	(172.3)[C]
Kidney (goat)				
Goat metabolism study	0.429	0.046	10.7	100.0
L0309/01		0.042	9.8	91.7
Method DFG S 19		0.049	11.3	105.6
Method QuEChERS		0.037	8.6	80.5
Fat (hen)				
Hen metabolism study	0.702	0.178	25.4	100.0
L0309/01		0.167	23.8	93.7
Method DFG S 19		0.142	20.3	79.7
Method QuEChERS		0.034	4.8	19.0
Liver (hen)				
Hen metabolism study	0.320	0.118	36.7	100.0
L0309/01[B]		0.058	18.3	49.7

Extraction Procedure	TRR (mg/kg)	mg/kg	% TRR	Extraction Efficiency (%) [A]
		0.057	17.7	48.2
		0.054	17.0	46.3
Method DFG S 19		0.103	32.3	88.0
Method QuEChERS		0.104	32.6	88.7
Muscle (hen)				
Hen metabolism study	0.050	0.025	49.9	100.0
L0309/01		0.015	30.4	60.9
Method DFG S 19		0.020	40.0	80.1
Method QuEChERS		0.022	43.4	87.0
Egg yolk				
Hen metabolism study	0.477	0.186	39.0	100.0
L0309/01 [B]		0.123	25.8	66.2
		0.102 [4]	21.3 [4)]	*(54.7)* [D]
Method DFG S 19		0.230	48.3	123.8
Method QuEChERS		0.126	26.4	67.8

Notes:

[A] extraction efficiency = amounts extracted using analytical method compared to amount extracted in metabolism study (normalized to 100 percent). For cream (goat) extraction efficiency was calculated as the percent TRR in metabolism study divided by percent TRR extracted using analytical method.

[B] As confirmation of the results obtained, for cream (goat) two different subsamples were extracted for confirmation. For liver (hen) three different subsamples were extracted. For egg yolk (hen), two different subsamples were extracted.

[C] The value calculated for milk appears to be an overestimation resulting from low analyte concentration.

[D] The value calculated for yolk is an underestimation since an additional 0.011 mg/kg (2.4 percent TRR) was recovered after concentration of the extract prior to HPLC analysis.

In summary, all analytical methods provided to the Meeting were determined to be acceptable for the analysis of mefentrifluconazole and Metabolite M750F022 in milk, eggs and bovine and poultry tissues and M750F025 (measured as M750F022) in hen matrices, based on average recoveries that were in the range of 70–120 percent and relative standard deviations of ≤20 percent. The detector responses were linear within the ranges tested. Radiovalidation information demonstrated the ability of the methods L0272/01 and L0309/01 to extract bioincurred residues of mefentrifluconazole and M750F022.

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

Plant matrices

The stability of mefentrifluconazole was investigated in wheat (whole plant, straw, grain), soya bean seed, rape seed, potato tuber, apple fruit, lemon fruit, dried bean seed, grape fruit, tomato fruit, and dried pea seed (Orozco *et al*., 2016, BASF DocID 2016_1112644). Samples were fortified at a concentration level of 0.10 mg/kg, stored frozen, and analysed after approximately 0, 30, 90, 180, 365, 545, and 730 days. Analytical method L0076/09 (de Paula José, 2015, BASF DocID 2015_3001681) was used to determine residues of mefentrifluconazole. The LOQ was 0.01 mg/kg in all matrices. The recoveries of mefentrifluconazole after frozen storage are summarized in Table 70.

Table 70 Stability of mefentrifluconazole residues in various commodities during frozen storage

Commodity	Storage period (days)	Spiking level (mg/kg)	Procedural Recoveries (%)	Stored Sample Residues (mg/kg)	Mean % remaining[A]

Commodity	Storage period (days)	Spiking level (mg/kg)	Procedural Recoveries (%)	Stored Sample Residues (mg/kg)	Mean % remaining[A]
Tomato, fruit	0	0.10	93.6, 87.5 [90.6]	0.090, 0.089 [0.090]	100
	31		98.0, 95.5 [96.8]	0.093, 0.094 [0.094]	104
	85		89.5, 93.5 [91.5]	0.086, 0.088 [0.087]	97
	177		83.0, 80.0 [81.5]	0.078 0.071 [0.075]	83
	358		93.0[2], 93.3[2] [93.2]	0079[2], 0.075[2] [0.077]	86
	546		92.0, 90.0 [91.0]	0.081, 0.074 [0.078]	87
	732		92.8, 90.3 [91.6]	0.082, 0.067 [0.075]	83
Apple, fruit	0	0.10	94.1, 91.0 [92.6]	0.087, 0.082 [0.085]	100
	30		96.5, 92.0 [94.3]	0.088, 0.090 [0.089]	105
	85		88.0, 77.5 [82.7]	0.093, 0.090 [0.092]	108
	182		98.5, 98.5 [98.5]	0.083, 0.081 [0.082]	96
	358		98.5, 93.0 [95.8]	0.085, 0.077 [0.081]	95
	547		97.0, 89.0 [93.0]	0.078, 0.079 [0.079]	93
	733		91.3, 94.3 [92.8]	0.082, 0.083 [0.083]	98
Grapes, fruit	0	0.10	94.1, 88.5 [91.3]	0.092, 0.086 [0.089]	105
	31		97.0, 98.5 [97.8]	0.092, 0.094 [0.093]	109
	85		90.5, 98.0 [94.3]	0.085, 0.085 [0.085]	100
	177		57.5, 83.5 [70.5]	0.088, 0.086 [0.087]	102
	358		94.3[2], 88.0[2] [91.1]	0.089[2], 0.084[2] [0.087]	102
	547		93.0, 98.0 [95.5]	0.087, 0.095 [0.091]	107
	733		94.3, 100 [97.2]	0.087, 0.090 [0.089]	105
Lemon, fruit	0	0.10	93.0, 89.0 [91.0]	0.090, 0.090 [0.090]	100
	29		97.5, 97.5 [97.5]	0.094, 0.094 [0.094]	104
	83		97.5, 97.0 [97.3]	0.094, 0.092 [0.093]	103
	182		102, 97.0 [99.5]	0.096, 0.095 [0.096]	107
	358		93.0[2], 92.3[2] [92.6]	0.094[B], 0.093[B] [0.094]	104
	547		103, 91.0 [97.0]	0.10, 0.096 [0.098]	109
	733		98.3, 95.3 [96.8]	0.093, 0.092 [0.093]	103
Wheat, grain	0	0.10	100, 95.1 [97.6]	0.094, 0.095 [0.095]	100
	30		105, 98.5 [102]	0.096, 0.093 [0.095]	100
	85		98.1, 87.0 [92.6]	0.096, 0.099 [0.098]	103
	182		106, 98.0 [102]	0.094, 0.089 [0.092]	97
	361		97.5, 105 [101]	0.098, 0.104 [0.101]	106
	547		106, 96 [101]	0.091, 0.096 [0.094]	99
	733		103, 98.3 [101]	0.091, 0.093 [0.092]	97
Bean, dried, seed	0	0.10	97.1, 91.5 [94.3]	0.093, 0.093 [0.093]	100
	30		101, 97.0 [98.8]	0.096, 0.092 [0.094]	101
	85		92.1, 88.5 [90.3]	0.094, 0.096 [0.095]	102
	182		106, 94.5 [100]	0.098, 0.097 [0.098]	105
	358		166, 98.0 [132]	0.090, 0.087 [0.089]	96
	547		104, 92.0 [97.8]	0.10, 0.104 [0.102]	110
	733		103, 95.3 [99.2]	0.098, 0.103 [0.101]	109
Peas, dried, seed	0	0.10	93.0, 90.5 [91.8]	0.091, 0.089 [0.090]	100
	29		99.0, 93.5 [96.3]	0.094, 0.096 [0.095]	106
	83		100, 96.0 [98.0]	0.089, 0.094 [0.092]	102
	182		108, 104 [106]	0.099, 0.10 [0.100]	111
	361		99.5, 91.0 [95.3]	0.096, 0.098 [0.097]	108
	550		95.0, 95.0 [95.0]	0.099, 0.098 [0.099]	110
	734		95.8, 97.3 [96.6]	0.089, 0.086 [0.088]	98
Soya bean, seed	0	0.10	101, 90.0 [95.3]	0.092, 0.088 [0.090]	100
	30		105, 90.5 [97.5]	0.089, 0.092 [0.091]	101
	85		82.0, 80.5 [81.3]	0.086, 0.086 [0.086]	96
	182		78.0, 98.0 [88.0]	0.089, 0.094 [0.092]	102
	361		93.5, 96.0 [94.8]	0.093, 0.088 [0.091]	101

Commodity	Storage period (days)	Spiking level (mg/kg)	Procedural Recoveries (%)	Stored Sample Residues (mg/kg)	Mean % remaining[A]
	550		101, 86.0 [93.5]	0.084, 0.093 [0.089]	99
	734		99.3, 87.8 [93.6]	0.082, 0.089 [0.086]	96
Rape, seed	0	0.10	77.5, 86.0 [81.8]	0.091, 0.090 [0.091]	100
	31		83.5, 87.0 [85.3]	0.089, 0.082 [0.086]	95
	85		93.5, 93.5 [93.5]	0.093, 0.090 [0.092]	101
	177		85.0, 100 [92.5]	0.081, 0.090 [0.086]	95
	369		87.0, 88.0 [87.5]	0.073, 0.080 [0.077]	85
	378[3]		88.0, 93.5 [90.8]	0.091, 0.096 [0.094]	103
	550		91.0, 95.5 [93.3]	0.094, 0.095 [0.095]	104
	735		92.3, 98.3 [95.3]	0.093, 0.091 [0.092]	101
Wheat Whole Plant No Roots	0	0.10	87.0, 90.5 [88.8]	0.092, 0.088 [0.090]	100
	31		91.5, 95.0 [93.3]	0.092, 0.094 [0.093]	103
	85		90.5, 90.0 [90.3]	0.087, 0.088 [0.088]	98
	177		97.5, 94.5 [96.0]	0.088, 0.096 [0.092]	102
	369		87.5, 94.5 [91.0]	0.084, 0.083 [0.084]	93
	550		99.0, 90.0 [94.5]	0.097, 0.094 [0.096]	107
	734		89.3, 96.8 [93.1]	0.090, 0.089 [0.090]	100
Wheat, straw	0	0.10	96.5, 85.5 [86.0]	0.085, 0.082 [0.084]	100
	29		91.0, 94.0 [92.5]	0.093, 0.091 [0.092]	110
	86		87.0. 84.0 [85.5]	0.090, 0.092 [0.091]	108
	184		89.0, 92.5 [90.8]	0.094, 0.091 [0.093]	111
	363		86.0, 85.5 [85.8]	0.090, 0.086 [0.088]	105
	551		91.0, 96.0 [93.5]	0.10, 0.097 [0.099]	118
	736		90.8, 84.3 [87.6]	0.094, 0.094 [0.094]	112
Potato, tuber	0	0.10	96.5, 88.0 [92.3]	0.089, 0.094 [0.092]	100
	29		97.5, 89.5 [93.5]	0.091, 0.079 [0.085]	92
	83		103, 97.0 [99.8]	0.092, 0.088 [0.090]	98
	184		89.0, 65.0 [77.0]	0.065, 0.057 [0.061]	66
	369		92.5, 93.0 [92.8]	0.080, 0.072 [0.076]	83
	378[C]		104, 97.0 [101]	0.092, 0.091 [0.092]	100
	551		93.0, 83.5 [88.3]	0.079, 0.074 [0.077]	84
	735		85.3, 91.8 [88.6]	0.082, 0.077 [0.080]	87

Notes:

[A] Normalised to Day 0 stored sample residues, not corrected for procedural recoveries.

[B] Samples were injected twice.

[C] Reserve samples (prepared in case of failing analyses, repetitions or additional samplings).

Residues of mefentrifluconazole were determined to be stable at ≤-18 ºC for up to 24 months in high water content commodities (tomato, apple, wheat whole plant), high oil content commodities (rape, seed), high protein content commodities (dried bean seed, dried pea seed, soya bean seed), high starch content commodities (potato tuber), high acid content commodities (grape, lemon), and wheat straw.

Animal matrices

In two freezer storage stability studies, samples of bovine liver, kidney, muscle, fat, milk and cream and hen eggs were fortified with the parent compound mefentrifluconazole (Heger, 2015, BASF DocID 2015_1106711) or metabolite M750F022 (Heger, 2015, BASF DocID 2015_1106710) at concentrations of 0.10 mg/kg/analyte (Tables 71 and 72). Samples were stored at ≤-18 ºC for a period of up to 180 days (6 months), which covered the sample storage interval in the feeding studies. Samples were analysed for the parent compound using the LC-MS/MS analytical method L0272/01 and for the metabolite using the GC-MS method L0309/01. The LOQ reported for each method was 0.010 mg/kg.

Residues of mefentrifluconazole and M750F022 were determined to be stable at ≤-18 ºC for up to 6 months in milk, cream, eggs and bovine and poultry tissues (Tables 71 and 72).

Table 71 Storage stability of the parent compound mefentrifluconazole in animal matrices

Matrix	Storage duration (days)	Procedural recoveries (%)	Residues (mg/kg)	Mean % remaining[A]
Bovine liver	0	96	0.11, 0.10 [0.105]	100
	28	99	0.09, 0.09 [0.09]	86
	-	-	-	-
	120	98	0.09,0.09 [0.09]	86
	177	94	0.10, 0.11 [0.105]	100
Bovine kidney	0	113	0.11, 0.10 [0.105]	100
	29	99	0.09, 0.09 [0.09]	86
	90	103	0.10, 0.10 [0.10]	95
	120	96	0.09, 0.09 [0.09]	86
	182	103	0.10, 0.10 [0.10]	95
Bovine muscle	0	105	0.09, 0.11 [0.10]	100
	30	98	0.09, 0.09 [0.09]	90
	89	106	0.10, 0.10 [0.10]	100
	120	101	0.09, 0.09 [0.09]	90
	182	112	0.09, 0.10 [0.095]	95
Bovine fat	0	93	0.09, 0.09 [0.09]	100
	32	87	0.08, 0.08 [0.08]	89
	85	92	0.09, 0.09 [0.09]	100
	117	89	0.09, 0.08 [0.085]	94
	180	86	0.08, 0.09 [0.085]	94
Bovine milk	0	95	0.10, 0.10 [0.10]	100
	29	90	0.08, 0.08 [0.08]	80
	84	97	0.10, 0.10 [0.10]	100
	116	90	0.08, 0.08 [0.08]	80
	178	101	0.09, 0.10 [0.095]	95
Bovine cream	0	98	0.10, 0.10 [0.10]	100
	32	94	0.09, 0.08 [0.085]	85
	83	98	0.10, 0.10 [0.10]	100
	118	96	0.09, 0.09 [0.09]	90
	177	101	0.09, 0.09 [0.09]	90
Hen egg	0	109	0.10, 0.11 [0.105]	100
	28	86	0.09, 0.09 [0.09]	86
	83	103	0.11, 0.11 [0.11]	105
	118	99	0.09, 0.09 [0.09]	86
	180	111	0.11, 0.10 [0.105]	100

Notes:

[A] Normalised to Day 0 stored sample residues, not corrected for procedural recoveries

Table 72 Storage stability of the metabolite M750F022 in animal matrices

Matrix	Storage duration (days)	Procedural recoveries (%)	Residues (mg/kg)	Mean % remaining[A]
Bovine liver	0	83	0.083, 0.081 [0.082]	100
	28	79	0.072, 0.068 [0.070]	85
	86	87	0.090, 0.081 [0.086]	105
	114	79	0.086, 0.080 [0.083]	101
	183	94	0.092, 0.077 [0.084]	102
Bovine kidney	0	74, 76 [75]	0.085, 0.082 [0.084]	100

Matrix	Storage duration (days)	Procedural recoveries (%)	Residues (mg/kg)	Mean % remaining[A]
	31	82	0.076, 0.077 [0.077]	92
	90	89	0.083, 0.077 [0.080]	95
	114	79	0.11, 0.096 [0.102]	121
	178	97	0.10, 0.084 [0.093]	111
Bovine muscle	0	71	0.070, 0.070 [0.070]	100
	29	80	0.093, 0.074 [0.084]	109
	91	76	0.074, 0.080 [0.077]	110
	115	65	0.093, 0.098 [0.095]	136
	181	70	0.10, 0.11 [0.106]	151
Bovine fat	0	107	0.10, 0.10 [0.101]	100
	28	100	0.088, 0.087 [0.087]	86
	87	108	0.093,0.10 [0.098]	97
	115	402	0.091, 0.094 [0.092]	91
	180	88	0.089, 0.089 [0.089]	88
Bovine milk	0	84	0.086, 0.083 [0.084]	100
	28	82	0.084, 0.080 [0.082]	98
	84	86	0.071, 0.084 [0.078]	93
	113	85	0.078, 0.079 [0.078]	93
	179	79	0.077, 0.083 [0.080]	95
Bovine cream	0	106	0.084, 0.086 [0.085]	100
	27	99	0.096, 0.097 [0.097]	114
	84	90	0.11, 0.095 [0.103]	121
	115	99	0.092, 0.096 [0.094]	110
	179	90	0.093, 0.093 [0.093]	109
Hen egg	0	74	0.092, 0.088 [0.090]	100
	28	84	0.082, 0.071 [0.077]	86
	85	94	0.087,0.094 [0.091]	101
	113	54	0.081, 0.076 [0.079]	88
	178	81	0.082, 0.078 [0.080]	89

Notes:

[A] Normalised to Day 0 stored sample residues, not corrected for procedural recoveries

USE PATTERNS

Mefentrifluconazole is used as SC or EC formulation for foliar, in-furrow or seed treatment. Table 73 summarizes the use pattern and Table 74 shows the crop rotation restrictions on United States labels for foliar-applied fungicides

Table 73 Registered uses of mefentrifluconazole for the crops for which residue trials were submitted

Crop	Country	Formulation	Application			Application rate per treatment			Cumulative rate per season (kg ai/ha)	PHI [days]
			Method	No.	Re-treatment interval [days]	kg ai/hL	Spray Volume [L/ha]	kg ai/ha		
Citrus fruits (Crop group 10-10)[A]	United States of America	SC or EC	Foliar	3	14	NS	-*	0.146	0.437	0
Pome fruits (Crop Group 11-10)[B]	United States of America	SC	Foliar	3	7	NS	-*	0.101	0.437	0
		SC		3	7	NS	-*	0.146		0

Crop	Country	Formulation	Application			Application rate per treatment			Cumulative rate per season (kg ai/ha)	PHI [days]
			Method	No.	Re-treatment interval [days]	kg ai/hL	Spray Volume [L/ha]	kg ai/ha		
Apples	Australia	SC	Foliar	3	7	0.006	NS	NS	0.018 kg ai/hL	7
Stone fruits (Crop Group 12)[C]	United States of America	SC	Foliar	3	7	NS	-*	0.123	0.437	0
		SC		3	7	NS	-*	0.146		0
Caneberries (Crop subgroup 13-07A)[D]	United States of America	SC	Foliar	3	7	NS	-*	0.146	0.437	0
Bushberries (Crop subgroup 13-07B)[E]	United States of America	SC	Foliar	3	7	NS	-*	0.146	0.437	0
Table grapes, raisins	Australia	SC	Foliar	3	7-21	0.006	NS	NS	0.018 kg ai/hL	7
Wine grapes		SC		3	7-21	0.006	NS	NS	0.018 kg ai/hL	7
Grape, table and raisin	United States of America	SC	Foliar	2	10 (same product) 21(another MFN product)	NS	-*	0.101	0.437	14
Grape, table and raisin	United States of America	SC	Foliar	2	10	NS	-*	0.112		14
Grapes, wine	United States of America	SC	Foliar	3	10	NS	-*	0.101	0.437	14
Grapes, wine	United States of America	SC	Foliar	3	10	NS	-*	0.146		14
Low growing berries (Crop group 13-07G)[F]	United States of America	SC	Foliar	3	7	NS	-*	0.146	0.437	0
Banana	Ecuador	SC	Foliar	4	14	NS	NS	0.140	NS	0
Avocado	El Salvador, Guatemala, Honduras	SC	Foliar	3	14	0.024	500	0.120	0.360	3
Avocado	Guatemala	SC	Foliar	1	NA	0.120	400	0.480	0.480	15
Papaya	El Salvador, Guatemala, Honduras	SC	Foliar	2	14	0.03	400	0.120	0.240	3
Mango	El Salvador, Guatemala, Honduras	SC	Foliar	3	14	0.015	800	0.120	0.360	3
Mango	Guatemala	SC	Foliar	3	14	0.120	800	0.960	2.88	1
Mango	China	SC	Foliar	3	10	0.016	NS	NS	0.048	14
Bulb vegetables (Crop group 3-07)[G]	United States of America	SC	Foliar	3	7	NS	NS	0.146	0.437	7

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Crop	Country	Formulation	Application			Application rate per treatment			Cumulative rate per season (kg ai/ha)	PHI [days]
			Method	No.	Re-treatment interval [days]	kg ai/hL	Spray Volume [L/ha]	kg ai/ha		
Onion, garlic	Guatemala, Honduras	SC	Foliar	3	7	NS	400	0.100	0.300	7
Leek	Guatemala, Honduras	SC	Foliar	1	NA	NS	400	0.100	0.100	NS
Onion, garlic, leek	El Salvador, Guatemala, Honduras	SC	Foliar	3	7	NS	400	0.120	0.360	7
Cucurbit vegetables (Crop group 9)[H]	United States of America	SC	Foliar	3	7	NS	NS	0.146	0.437	0
Watermelon, melon, pumpkin, cucumber	El Salvador, Guatemala, Honduras	SC	Foliar	3	7	NS	400	0.120	0.360	0
Fruiting vegetales (Crop group 8-10)[I]	United States of America	SC	Foliar	2	7	NS	NS	0.101	0.437	0
		SC	Foliar	3	7	NS	NS	0.146		0
Tomato, bell peppers, non-bell peppers	Columbia	SC	Foliar	1	NA	NS	600	0.140	NA	0
Tomato, non-bell peppers	El Salvador, Guatemala, Honduras	SC	Foliar	3	7	NS	400	0.120	0.360	0
Tomato, non-bell peppers	Guatemala, Honduras	SC	Foliar	3	7	NS	571	0.100	0.300	0
Tomato, non-bell peppers	El Salvador, Guatemala, Honduras	SC	Foliar	3	7	NS	400-669	0.120	0.360	0
Leafy vegetables (Crop group 4-16)[J]	United States of America	SC	Foliar	3	7	NS	NS	0.146	0.437	0
Legume vegetables (Crop group 6, except soybean, edamame)[K]	United States of America	SC	Foliar	2	7	NS	94	0.123	0.437	Bean forage, bean hay, pea vines, and pea hay may be fed no sooner than 21 DALA
		SC		2	7	NS	94	0.146		
		EC or SC		3	7	NS	94	0.146		21
Soybean	United States of America	EC or SC	Foliar	2	7	NS	94	0.146	0.292	21
		SC		2	14	NS	94	0.146	0.292	14 soybean forage 21 soybean seed and hay
Edamame	United States of America	EC or SC	Foliar	3	7	NS	94	0.146	0.437	21
		SC		2	14	NS	94	0.146		21

Crop	Country	Formulation	Application			Application rate per treatment			Cumulative rate per season (kg ai/ha)	PHI [days]
			Method	No.	Re-treatment interval [days]	kg ai/hL	Spray Volume [L/ha]	kg ai/ha		
Soybean	United States of America	Flowable	Seed treatment	1	NA	0.02 kg ai/100 kg seeds	NA	NA		NA
Non-grass forages (Crop group 18)[L]	United States of America	EC or SC	Foliar	3	7	NS	94	0.146	0.437	14
Root vegetables (Crop group 1B, except sugar beets)[M]	United States of America	SC	CloverFoliar	3	7	NS	-*	0.146	0.437	7
Root vegetables (Crop group 1B, except sugar beets)[M]	United States of America	SC	Foliar	3	7	NS	-*	0.101		7
Sugar beets	United States of America	EC or SC	Foliar	2	14	NS	94	0.146	0.292	7
Tuberous and corm vegetables (Crop subgroup 1C)[N]	United States of America	EC or SC	Foliar	3	7	NS	94	0.146	0.437	7
Potato	Ecuador	SC	Foliar	2	7	NS	400	0.08		7
Barley, spelt, triticale, wheat	France	EC	Foliar	1	NA	0.150	100-300	0.150		35
Winter wheat, spring wheat, durum wheat, spelt wheat, winter barley, spring barley, rye, triticale and oats	United Kingdom	EC	Foliar	2	NS	NA	100	0.146	0.292	When flowering anthesis complete (BBCH 69)
Barley, oats, rye, triticale, wheat	United States of America	SC	Foliar	2	14	NS	94	0.123	0.292	21
		EC or SC		2	14	NS	94	0.146		21
Sorghum (milo) and millet (pearl, proso)	United States of America	SC	Foliar	1	NA	NS	94	0.146	0.146	21
Sorghum (milo) and millet (pearl, proso)	United States of America	SC	Foliar	1	NA	NS	94	0.146	0.292	21
Sorghum (milo) and millet (pearl, proso)	United States of America	EC or SC	Foliar	2	14	NS	94	0.146	0.292	21
Barley, buckwheat, millet, oats, rye, sorghum, triticale, wheat	United States of America	Flowable	Seed treatment	1	NA	0.01 kg ai/100 kg seeds	NA	NA		NA

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Crop	Country	Formulation	Application			Application rate per treatment			Cumulative rate per season (kg ai/ha)	PHI [days]
			Method	No.	Re-treatment interval [days]	kg ai/hL	Spray Volume [L/ha]	kg ai/ha		
Oats	France	EC	Foliar	2	14	NS	100-300	0.150		35
Oats, winter rye	Lithuania	EC	Foliar	1	NA	NS	100-300	0.150		35
Winter wheat, spring wheat, winter triticale, summer triticale, winter barley, spring barley	Lithuania	EC	Foliar	1	NA	NS	100-300	0.100		35
		EC		2	14	NS	100-300	0.050		
Winter wheat, spring wheat, winter triticale, summer triticale, winter barley, spring barley	Lithuania	EC	Foliar	1	NA	NS	100-300	0.150		35
		EC		2	14	NS	100-300	0.075		
Rice	Brazil	SC	Foliar	2	15	0.14	100	0.14		35
Rice	China	SC	Foliar	2	5	0.024	500	0.12		21
Rice	Columbia	SC	Foliar	1	NA	NS	18-23 (aerial application)	0.18		21
Rice	Ecuador	SC	Foliar	2	15	NS	-*	0.14		21
Rice	Peru	SC	Foliar	2	14	NS	-*	0.12		21
Rice	Paraguay	SC	Foliar	1	NA	NS	-*	0.12		21
Rice	Philippines	SC	Foliar	2	10	0.03	400	0.12		21
Field corn, popcorn, silage, seed corn	United States of America	EC or SC	Foliar	2	14	NS	94	0.146	0.292	21
Sweet corn	United States of America	EC or SC	Foliar	2	7	NS	-*	0.146	0.437	
		EC or SC	Foliar	3	7	NS	94	0.146	0.437	21
Field corn, popcorn, sweet corn	United States of America	Flowable	Seed treatment	1	NA	0.01 kg ai/100 kg seeds	NA	NA	NA	NA
Grasses (Crop group 17)	United States of America	EC or SC	Foliar	3	7	NS	NA	0.146	0.437	0
Sugar cane	United States of America	SC	Foliar or In-furrow	2	14	NS	47 (Aerial) 23-94 (Ground)	0.146	0.292	14 DO NOT feed treated sugarcane commodities to livestock
Tree Nuts (Crop group 14)[0]	United States of America	SC	Foliar	3	7 (10 for pistachio)	NS	-*	0.123	0.437	14

Crop	Country	Formulation	Application			Application rate per treatment			Cumulative rate per season (kg ai/ha)	PHI [days]
			Method	No.	Re-treatment interval [days]	kg ai/hL	Spray Volume [L/ha]	kg ai/ha		
		SC		3	7 (10 for pistachio)	NS	-*	0.146		14
Rape seed (Crop subgroup 20A)[P]	United States of America	EC or SC	Foliar	2	14	NS	-*	0.146	0.292	21
Sunflower seed (Crop subgroup 20B)[Q]	United States of America	EC or SC	Foliar	2	14	NS	-*	0.146	0.292	21
Cottonseed (Crop subgroup 20C)	United States of America	SC	Foliar	2	7	NS	-*	0.146	0.292	30
		EC	In-furrow	3	7	NS	-*	0.146	0.437	30
Peanuts	United States of America	SC	Foliar	3	14	NS	-*	0.146	0.605	14 DO NOT graze or harvest for forage use.
		EC or SC		3	14	NS	-*	0.202		14 DO NOT graze or harvest for forage use.
Coffee beans	Columbia	SC	Foliar	1	NA	NS	300	0.107		45
		SC		1	NA	NS	300	0.160		45
Coffee beans	Ecuador	SC	Foliar	3	60	NS	400	0.160		45

Notes:

* United States spray volumes: 94 L/ha (ground) and 19 L/ha (aerial), unless otherwise specified.

NS: Not specified; NA: Not applicable.

PHI: Pre-harvest interval; on the United States labels, the PHI is the same as the grazing/feeding interval, unless otherwise specified.

[A] Citrus (subgroups 10-10a, 10-10b and 10-10c): inluding calamondin; citron; citrus hybrids; grapefruit (grapefruit, Japanese summer); kumquat; lemon; lime (lime, Australian desert, Australian finger, Australian round, Brown River finger, Mount White, New Guinea wild, Russell River, sweet, Tahiti); mandarin (Mediterranean, satsuma); orange (sour, sweet, tachibana, trifoliate); pummelo; tangelo; tangerine (mandarin); tangor; uniq fruit; cultivars, varieties, and/or hybrids of these.

[B] Pome fruits (crop group 11-10): including apple; azarole, crabapple; loquat; mayhaw; pear; Asian pear; quince; Chinese quince, Japanese quince, tejocote; cultivars, varieties, and/or hybrids of these.

[C] Stone fruits (crop group 12-12): including apricot; Japanese apricot; capulin; black cherry, Nanking cherry; sweet cherry, tart cherry; Chinese Jujube; nectarine; peach; plum; American plum, beach plum, Canada plum, cherry plum, Chickasaw plum, Damson plum, Japanese plum, Klamath plum, prune plum; plumcot; sloe; cultivars, varieties, and/or hybrids of these.

[D] Caneberries (crop subgroup 13-07A): including caneberry (blackberry; loganberry; raspberry, black and red; wild raspberry; cultivars, varieties, and/or hybrids of these).

[E] Bushberries (crop subgroup 13-07B): including aronia berry; blueberry, highbush; blueberry, lowbush; buffalo currant; Chilean guava; cranberry, highbush; currant, black; currant, red; elderberry; European barberry; gooseberry; honeysuckle, edible; huckleberry; jostaberry; juneberry (Saskatoon berry); lingonberry; native currant; salal; sea buckthorn; cultivars, varieties, and/or hybrids of these).

[F] Low growing berries (crop group 13-07G): including bearberry; bilberry; blueberry, lowbush; cloudberry; cranberry; lingonberry; muntries; partridgeberry; strawberry; cultivars, varieties, and/or hybrids of these.

[G] Bulb vegetables (crop group 3-07): including chive, fresh leaves; chive, Chinese, fresh leaves; daylily, bulb; elegans hosta; fritillaria, bulb; fritillaria, leaves; garlic, bulb; garlic, great-headed, bulb; garlic, serpent, bulb; kurrat; lady's leek; leek; leek, wild; lily, bulb; onion, Beltsville bunching; onion, bulb; onion, Chinese, bulb; onion, fresh; onion, green; onion, macrostem; onion, pearl; onion, potato, bulb; onion, tree, tops; onion, Welsh, tops; shallot, bulb; shallot, fresh leaves; cultivars, varieties, and/or

hybrids of these.

[H] Cucurbit vegetables (crop group 9): including chayote (fruit); Chinese waxgourd (Chinese preserving melon); citron melon; cucumber; gherkin; gourd, edible (includes hyotan, cucuzza, hechima, Chinese okra); Momordica spp (includes balsam apple, balsam pear, bittermelon, Chinese cucumber); muskmelon (includes true canteloupe, cantaloupe, casaba, crenshaw melon, golden pershaw melon, honeydew melon, honey balls, mango melon, Persian melon, pineapple melon, Santa Claus melon, and snake melon); pumpkin; squash, summer (includes crookneck squash, scallop squash, straightneck squash, vegetable marrow, zucchini); squash, winter (includes butternut squash, calabaza, hubbard squash, acorn squash, spaghetti squash); watermelon.

[I] Fruiting vegetables (crop group 8-10): including African eggplant; bush tomato; bell pepper; cocona; currant tomato; eggplant; garden huckleberry; goji berry; groundcherry; martynia; naranjilla; okra; pea eggplant; pepino; non-bell pepper; roselle; scarlet eggplant; sunberry; tomatillo; tomato; tree tomato; cultivars, varieties, and/or hybrids of these.

[J] Leafy vegetables (crop group 4-16): including amaranth, Chinese; amaranth, leafy; arugula; aster, Indian; blackjack; broccoli, Chinese; broccoli raab; cabbage, abyssinian; cabbage, Chinese, bok choy; cabbage, seakale; cat's whiskers; chamchwi; cham-na-mul; chervil, fresh leaves; chipilin; chrysanthemum, garland; cilantro, fresh leaves; collards; corn salad; cosmos; cress, garden; cress, upland; dandelion, leaves; dang-gwi, leaves; dillweed; dock; dol-nam-mul; ebolo; endive; escarole; fameflower; feather cockscomb; Good King Henry; hanover salad; huauzontle; jute, leaves; kale; lettuce, bitter; lettuce, head; lettuce, leaf; maca, leaves; mizuna; mustard greens; orach; parsley, fresh leaves; plantain, buckhorn; primrose, English; purslane, garden; purslane, winter; radicchio; radish, leaves; rape greens; rocket, wild; shepherd's purse; spinach; spinach, Malabar; spinach, New Zealand; spinach, tanier; Swiss chard; turnip greens; violet, Chinese, leaves; watercress; cultivars, varieties, and hybrids of these commodities.

[K] Legume Vegetables (crop group 6, except soybean and edamame); including broad bean (fava bean), chickpea (garbanzo bean), guar, jackbean, lablab bean, lentil, pigeon pea, sword bean. - Lupinus spp.: grain lupin, sweet lupin, white lupin, white sweet lupin. - Phaseolus spp.: field bean, kidney bean, lima bean, navy bean, pinto bean, runner bean, snap bean, tepary bean, wax bean. - Vigna spp.: adzuki bean, asparagus bean, blackeyed pea, catjang, Chinese longbean, cowpea, Crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean, yardlong bean. - Pisum spp.: dwarf pea, edible-pod pea, English pea, field pea, garden pea, green pea, snow pea, sugar snap pea.

[L] Non-grass Forages (crop group 18): including alfalfa; bean, velvet; clover (Trifolium spp., Melilotus spp.); kudzu; lespedeza; lupin; sainfoin; trefoil; vetch; vetch, crown; vetch, milk.

[M] Root vegetables (crop subgroup 1B): including Detailed Crop (Subgroup 1B) List - beet, garden; burdock, edible; carrot; celeriac; chervil, turnip-rooted; chicory; ginseng; horseradish; parsley, turnip-rooted; parsnip; radish; radish, oriental (daikon); rutabaga; salsify; salsify, black; salsify, Spanish; skirret; turnip.

[N] Tuberous and Corm Vegetables (crop subgroup 1C): arracacha; arrowroot; artichoke, Chinese; artichoke, Jerusalem; canna, edible; cassava, bitter and sweet; chayote (root); chufa; dasheen (taro); ginger; leren; potato; sweet potato; tanier; turmeric; yam bean; yam, true.

[O] Tree nuts (crop group 14-12): including African nut-tree; almond; beechnut; Brazil nut; Brazilian pine; bunya; bur oak; butternut; cajou nut; candlenut; cashew; chestnut; chinquapin; coconut; coquito nut; dika nut; ginkgo; Guiana chestnut; hazelnut (filbert); heartnut; hickory nut; Japanese horse-chestnut; macadamia nut; mongongo nut; monkeypot; monkey puzzle nut; okari nut; pachira nut; peach palm nut; pecan; pequi; pili nut; pine nut; pistachio; sapucaia nut; tropical almond; walnut (black, English); yellowhorn; cultivars, varieties, and/or hybrids of these.

[P] Rapeseed (crop subgroup 20A): including borage; crambe; cuphea; echium; flax seed; gold of pleasure; hare's ear mustard; lesquerella; lunaria; meadowfoam; milkweed; mustard seed; oil radish; poppy seed; rapeseed; sesame; sweet rocket; cultivars, varieties, and/or hybrids of these.

[Q] Sunflower seed (crop subgroup 20B): including calendula; castor oil plant; Chinese tallowtree; euphorbia; evening primrose; jojoba; niger seed; rose hip; safflower; stokes aster; sunflower; tallowwood; tea oil plant; vernonia; cultivars, varieties, and/or hybrids of these.

Table 74 Crop rotation restrictions on United States labels for foliar-applied fungicides

Rotational Crops	Planting Time From Last Applicaiton
Root and tuber vegetables (crop groups 1 and 2) Bulb vegetables (crop group 3-07) Leafy vegetabltes (crop group 4-16) Fresh herbs Brassicas (crop group 5-16) Legume vegetables, including soybeans (crop group 6) Foliage of legume vegetables (crop group 7)	0 days

Fruiting vegetables (crop group 8-10) Cucurbit vegetables (rop group 9) Fruit, small vine climbing, except fuzzy kiwi (subgroup 13-07F) Low-growing berries (crop group 13-07G) Cereals (crop groups 15 and 16) Grass and non-grass animal feeds (crop groups 17 and 18) Oilseeds (crop group 20) Peanut Stalk, stem and leaf petiole vegetables (crop group 22) Sugarcane Any other crop labeled for direct application of a product containing mefentrifluconazole	
Other food and feed crops, not listed above	May not be planted in rotation

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials for mefentrifluconazole on the following crops or crop groups:

Crops/Crop Groups	Table No.
Food commodities	
Citrus fruits	75
Pome fruits	76
Stone fruits	77
Caneberries	78
Blueberries	79
Grapes	80
Strawberry	81
Bananas	82
Avocado	83
Papaya	84
Mango	85
Bulb vegetables	86
Fruiting vegetables, cucurbits	87
Fruiting vegetables, other than cucurbits	88
Legume vegetables	89
Beans with pods	90
Peas with pods	91
Succulent beans without pods	92
Pulses	93
Dry beans	94
Dry peas	95
Soya beans	96
Lentils	97
Root vegetables	98
Potato	99
Wheat – North America	100

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Crops/Crop Groups	Table No.
Wheat - Europe	101
Barley – North America	102
Barley – Europe	103
Rice – United States	104
Rice – China	105
Rice – Brazil	106
Sorghum	107
Maize	108
Sweet corn	109
Sugar cane	110
Tree nuts	111
Rape seed	112
Sunflower seed – North America	113
Sunflower seed – Europe	114
Cottonseed	115
Peanuts	116
Coffee	117
Animal feeds	
Dry pea vines	118
Cowpea forage	119
Soya bean forage	120
Alfalfa forage	121
Clover forage	122
Dry pea hay	123
Cowpea hay	124
Soya bean hay	125
Alfalfa hay	126
Clover hay	127
Sugar beet tops	128
Wheat forage	129
Wheat whole plant, ears and rest of plant - Europe	130
Barley whole plant, ears and rest of plant - Europe	131
Sorghum forage	132
Maize forage	133
Sweet corn forage	134
Grass forage	135
Sorghum stover	136
Maize stover	137
Sweet corn stover	138
Wheat hay	139

Crops/Crop Groups	Table No.
Barley hay	140
Grass hay	141
Wheat straw - North America	142
Wheat straw - Europe	143
Barley straw - North America	144
Barley straw - Europe	145
Rice straw – United States	146
Rice husks and straw - China	147
Almond hulls	148
Peanut hay	149

Trials were generally well documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at residue levels like those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Unless stated otherwise, residue data are recorded unadjusted for recovery.

Residue values from the trials conducted in accordance with the critical GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Trial designs used non-replicated plots. Field reports provided data on the sprayers used, plot size, field sample size and sampling date.

Where duplicate field samples from an un-replicated plot were taken at each sampling time and were analysed separately, the mean of the two analytical results was taken as the best estimate of the residues in the plot and only the means are recorded in the tables. Similarly, where samples were collected from replicate plots the mean result is reported.

Citrus fruits

Table 75 Residues of mefentrifluconazole in whole citrus fruits from trials conducted in the United States following applications of an EC formulation using dilute and concentrated spray volumes (Bledsoe, 2017, BASF DocID 2017_7008898; Lucas, 2019; BASF DocID 2019_3000581)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
ORANGE							
Oviedo, FL; United States 2016 Valencia (R160246)[A]	3	150 154 156	- 14 14	697 699 702	83 83 83	0	0.146, 0.132 [0.139]
	3	152 155 153	- 14 14	1590 1587 1592	83 83 83	0	0.180, 0.159 [<u>0.170</u>]
Oviedo, FL; United States 2016 Navel	3	151 149 152	- 14 14	707 703 711	79 81 83	0	0.151, 0.193 [0.172]
	3	150	-	1587	73	0	0.210, 0.200 [<u>0.205</u>]

Mefentrifluconazole

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
(R160248)[A]		154 153	14 14	1602 1568	81 83		
Clemont, FL; United States 2016 Valencia (R160247)	3	152 152 155	- 14 14	702 692 703	73 83 83	0	0.166, 0.128 [0.147]
	3	152 156 154	- 14 14	1590 1593 1585	73 83 83	0	0.161, 0.189 [0.175]
Mims, FL; United States 2016 Navel (R160249)	3	152 151 151	- 13 14	711 710 698	79 81 83	0	0.188, 0.192 [0.190]
	3	151 150 154	- 13 14	1596 1590 1600	79 81 83	0	0.202, 0.198 [0.200]
Umatilla, FL; United States 2016 Valencia (R160250)	3	147 148 148	- 14 14	655 655 655	81 83 87	0	0.209, 0.174 [0.192]
	3	150 149 148	- 14 14	1871 1871 1871	81 83 87	0	0.162, 0.140 [0.151]
Apopka, FL; United States 2016 Hamlin (R160251)	3	147 149 148	- 14 14	514 514 514	83 85 89	0	0.447, 0.481 [0.464]
	3	149 149 150	- 14 14	3695 3695 3695	83 85 89	0	0.150, 0.176 [0.163]
Winter, Garden, FL; United States 2016 Valencia (R160252)	3	149 149 149	- 15 13	514 514 514	81 83 85	0	0.304, .0.362 [0.333]
	3	151 151 150	- 15 13	3695 3695 3695	81 83 85	0	0.166, 0.183 [0.175]
Groveland, FL; United States 2016 Hamlin (R160253)	3	150 148 150	- 14 14	514 514 514	81 85 89	0	0.300, 0.204 [0.252]
						7	0.354, 0.218 [0.286]
						14	0.432, 0.329 [0.381]
						21	0.338, 0.347 [0.343]
						28	0.297, 0.334 [0.316]
	3	151 152 151	- 14 14	3695 3695 3695	81 85 89	0	0.218,0.234 [0.226]
						7	0.191, 0.210 [0.201]
						14	0.189, 0.150 [0.170]
						21	0.143, 0.168 [0.156]
						28	0.134, 0.119 [0.127]
Raymondville, TX; United States 2016 Valencia (R160254)	3	152 153 152	- 14 13	706 707 702	79 79 81	0	0.165, 0.215 [0.190]
	3	150 153 152	- 14 13	2370 2369 2341	79 79 81	0	0.164, 0.136 [0.150]
Porterville, CA; United States 2016-2017 Atwood (R160255)	3	149 150 150	- 14 14	614 574 612	81 83 85	0	0.158, 0.126 [0.142]
	3	147 150	- 14	1655 1661	81 83	0	0.153, 0.148 [0.151]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
		150	14	1710	85		
Delano, CA; United States 2016 Atwood (R160256)	3	150 149 150	- 14 14	819 844 852	83 83 85	0	0.208, 0.279 [0.244]
	3	150 151 150	- 14 14	2571 2595 2554	83 83 85	0	0.131, 0.134 [0.133]
Sanger, CA; United States 2016 Werley (R160257)	3	151 151 150	- 14 14	795 795 795	83 85 87	0	0.210, 0.248 [0.229]
	3	150 152 151	- 14 14	1702 1702 1702	83 85 87	0	0.135, 0.180 [0.158]
Guasave, Sinaloa, Mexico 2017 Valencia (G175065)[B]	3	150 150 150	- 14 14	500 500 500	87 88 89	0	0.29
						7	0.66
						14	0.49
						21	0.67
Culiacan, Sinaloa, Mexico 2017 Valencia (G175066)[B]	3	150 150 150	- 14 14	500 500 500	85 86 88	0	0.47
						7	0.70
						14	0.56
						21	0.62
GRAPEFRUIT							
Oviedo, FL; United States 2016 Flame (R160258)	3	150 152 150	- 14 14	705 708 710	78 81 83	0	0.137, 0.236 [0.187]
	3	151 153 153	- 14 14	1591 1590 1609	78 81 83	0	0.240, 0.230 [0.235]
Mims, FL; United States 2016 Ray Red (R160259)	3	149 148 147	- 13 14	695 692 709	79 81 83	0	0.132, 0.133 [0.133]
	3	148 148 148	- 13 14	1592 1568 1574	79 81 83	0	0.121, 0.109 [0.115]
Umatilla, FL; United States 2016-2017 Ray Ruby (R160260)	3	150 151 150	- 14 14	889 889 889	77 79 87	0	0.197, 0.211 [0.204]
						7	0.195, 0.181 [0.188]
						14	0.132, 0.157 [0.145]
						21	0.122, 0.097 [0.110]
						28	0.130, 0.080 [0.105]
	3	150 151 150	- 14 14	3695 3695 3695	77 79 87	0	0.127, 0.146 [0.137]
						7	0.103, 0.095 [0.099]
						14	0.096, 0.096 [0.096]
						21	0.063, 0.065 [0.064]
						28	0.071, 0.057 [0.064]
Exeter, CA; United States 2016-2017 Melogold (R160261)	3	150 151 153	- 14 16	505 505 505	81 84 85	0	0.195, 0.127 [0.161]
	3	151 153 150	- 14 16	3695 3695 3695	81 84 85	0	0.068, 0.073 [0.071]
Raymondville, TX;	3	154	-	706	79	0	0.191, 0.178 [0.185]

Mefentrifluconazole

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
United States 2016 Rio Red (R160262)		157 155	14 13	707 702	81 83		
	3	153 157 153	- 14 13	2370 2369 2341	79 81 83	0	0.105, 0.115 [0.110]
Porterville, CA; United States 2016 Melogold (R160263)	3	154 157 155	- 14 13	706 707 702	79 81 83	0	0.120, 0.077 [0.099]
	3	153 157 153	- 14 13	2370 2369 2341	79 81 83	0	0.069, 0.074 [0.072]
LEMON							
Oviedo, FL; United States 2016 Meyer (R160264)	3	152 149 151	- 14 14	698 705 698	77 79 83	0	0.284, 0.252 [0.268]
	3	153 151 150	- 14 14	1597 1601 1588	77 79 83	0	0.377, 0.272 [0.325]
Winter Garden, FL; SA 2016 Bearss (R160265)	3	151 152 151	- 14 14	655 655 655	81 83 87	0	0.350, 0.317 [0.334]
	3	148 149 148	- 14 14	1871 1871 1871	81 83 87	0	0.323,0.242 [0.283]
San Luis Obispo, CA; United States 2016 Lisbon (R160266)	3	150 146 151	- 14 13	505 505 505	81 83 85	0	0.588, 0.619 [0.604]
	3	149 151 150	- 14 13	3695 3695 3695	81 83 85	0	0.193, 0.237 [0.215]
Woodlake, CA; United States 2016-2017 Lisbon (R160267)	3	151 149 150	- 14 16	505 505 505	81 84 85	0	0.429, 0.308 [0.369]
						7	0.288, 0.242 [0.265]
						14	0.245, 0.294 [0.270]
						21	0.284, 0.202 [0.243]
						28	0.236, 0.180 [0.208]
	3	150 148 150	- 14 16	3695 3695 3695	81 84 85	0	0.159, 0.175 [0.167]
						7	0.096, 0.135 [0.116]
						14	0.106, 0.095 [0.101]
						21	0.061, 0.071 [0.066]
						28	0.073, 0.070 [0.072]
Porterville, CA; United States 2016 Lisbon (R160268)	3	151 150 148	- 14 14	857 870 844	83 85 89	0	0.329, 0.262 [0.296]
	3	151	- 14 14	2630 2698 2684	83 85 89	0	0.230, 0.194 [0.212]
Richgrove, CA; United States 2016 Lisbon (R160269)	3	150 151 149	- 14 13	555 570 556	83 85 89	0	0.478, 0.394 [0.436]
	3	151 153 152	- 14 13	1502 1539 1538	83 85 89	0	0.317, 0.334 [0.326]
Sinaloa de Leyva,	3	150	-	500	81	0	0.98

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Sinaloa, Mexico 2017 Persa (G175067)		150 150	14 14	500 500	82 84	7	0.78
						14	0.58
						21	0.55

Notes:

[A] Applications were separated by a 5-month period, rendering the trials independent.

[B] Trial sites are separated by 164 km, rendering the trials independent.

Pome fruits

Table 76 Residues of mefentrifluconazole in pome fruits from trials conducted in the United States following applications of an SC formulation using dilute and concentrated spray volumes (Lucas, 2019, BASF DocID 2015_7005936)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
APPLE							
North Rose, NY; United States 2016 Cortland (R140484)[A, B]	3	150 151 151	- 7 7	886 891 891	81 81 85	0	0.49, 0.42 [0.46]
	3	149 149 149	- 7 7	1870 1865 1873	81 81 85	0	0.28, 0.27 [0.28]
North Rose, NY; United States 2016 Greening (R140485)[A]	3	149 150 149	- 7 8	883 889 882	81 81 85	0	0.30, 0.30 [0.30]
	3	152 151 151	- 7 8	1887 1882 1877	81 81 85	0	0.26, 0.28 [0.27]
North Rose, NY; United States 2016 Rome (R140486)[B]	3	152 152 150	- 7 7	899 888 887	77 77 81	0	0.38,0.46 [0.42]
						3	0.27, 0.30 [0.29]
						7	0.36, 0.28 [0.32]
						14	0.17, 0.16 [0.17]
						21	0.18, 0.19 [0.19]
	3	150 149 149	- 7 7	1869 1857 1854	77 77 81	0	0.33, 0.37 [0.35]
						3	0.19, 0.18 [0.19]
						7	0.19, 0.27 [0.23]
						14	0.18, 0.13 [0.16]
						21	0.15, 0.12 [0.14]
Cana, VA; United States 2016 Cana (R140487)	3	149 151 151	- 7 7	736 719 738	84 86 88	0	0.39, 0.47 [0.43]
	3	149 148 150	- 7 7	2829 2789 3011	84 86 88	0	0.33, 0.27 [0.30]
Buffalo, MN; United States 2016 Cortland (R140488)	3	153 153 153	- 7 7	502 502 501	81 85 85	0	<0.01, <0.01 [<0.01]
	3	152 153	- 7	1464 1468	81 85	0	<0.01, <0.01 [<0.01]

Mefentrifluconazole

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
		153	7	1471	85		
Dix, IL; United States 2016 Jonathon (R140489)	3	155 151 150	- 7 7	844 793 782	79 85 87	0	0.37, 0.36 [0.37]
	3	150 150 152	- 7 7	1906 1853 1868	79 85 87	0	0.30, 0.29 [0.30]
Branchton, ON; Canada 2016 Northern Spyres (R140490)[C]	3	142 147 149	- 7 7	678 705 715	81 85 85	0	0.16, 0.16 [0.16]
	3	147 149 149	- 7 7	1503 1533 1529	81 85 85	0	0.16, 0.13 [0.15]
Branchton, ON; Canada 2016 Ida Red (R140491)[C]	3	141 140 147	- 7 7	677 672 702	81 85 85	0	0.26, 0.26 [0.26]
	3	144 148 147	- 7 7	1477 1519 1512	81 85 85	0	0.18, 0.15 [0.17]
Cambridge, ON; Canada 2016 Gala (R140492)	3	147 152 147	- 7 7	702 728 705	81 85 85	0	0.48, 0.46 [0.47]
	3	144 145 141	- 7 7	1475 1484 1447	81 85 85	0	0.38, 0.55 [0.47]
Paradise, UT; United States 2016 Golden Delicious (R140493)	3	146 151 151	- 7 7	816 935 938	NS	0	0.24, 0.21 [0.23]
	3	146 155 149	- 7 7	1722 1930 1909	NS	0	0.17, 0.12 [0.15]
Porterville, CA; United States 2016 Granny Smith (R140494)	3	158 146 149	- 7 7	635 577 584	78 78 87	0	0.31, 0.19 [0.26]
	3	152 148 152	- 7 7	1799 1725 1805	78 78 87	0	0.32, 0.54 [0.43]
Ephrata, WA; United States 2016 Gala (R140495)[D]	3	149 149 151	- 7 7	467 466 472	83 85 87	0	0.60, 0.50 [0.55]
	3	150 151 150	- 7 7	1534 1540 1534	83 85 87	0	0.38, 0.32 [0.35]
Ephrata, WA; United States 2016 Braeburn (R140496)[D]	3	151 152 150	- 7 7	1100 1110 1091	NS	0	0.30, 0.30 [0.30]
						3	0.19, 0.29 [0.24]
						7	0.19, 0.28 [0.24]
						14	0.19, 0.25 [0.22]
						21	0.17, 0.11 [0.14]
	3	149 150 148	- 7 7	2021 2026 1991	NS	0	0.19, 0.25 [0.22]
						3	0.16, 0.17 [0.17]
						7	0.12, 0.14 [0.13]
						14	0.11, 0.11 [0.11]
						21	0.14, 0.12 [0.13]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Granger, WA; United States 2016 Grann Smith (R140497)	3	151 152 150	- 7 7	1100 1110 1091	NS	0	0.41, 0.37 [0.39]
	3	149 150 148	- 7 7	2021 2026 1991	NS	0	0.32, 0.29 [0.31]
Zilah, WA; United States 2016 Red Delicious (R140498)	3	152 149 148	- 7 7	799 784 780	NS	0	0.46, 0.44 [0.45]
	3	151 150 152	- 7 7	1458 1461 1465	NS	0	0.27, 0.35 [0.31]
PEARS							
Williamson, NY; United States 2016 Bartlett (R140475)	3	151 150 149	- 6 7	893 888 882	75 77 81	0	0.29, 0.30 [0.30]
	3	153 153 152	- 6 7	1885 1892 1872	75 77 81	0	0.27, 0.27 [0.27]
Buffalo, MN; United States 2016 Parker (R140476)	3	146 146 145	- 7 7	505 505 500	81 85 89	0	<0.01, <0.01 [<0.01]
	3	148 147 148	- 7 7	1485 1475 1486	81 85 89	0	0.01, <0.01 [<0.01]
Branchton, ON; Canada 2016 Bosc (R140477)[E]	3	149 146 144	- 6 6	696 683 672	81 85 85	0	0.72, 0.79 [0.76]
	3	146 145 144	- 6 8	1557 1539 1528	81 85 85	0	0.72, 1.12 [0.92]
Branchton, ON; Canada 2016 Bartlett (R140478)[E]	3	150 148 144	- 6 8	701 691 670	81 85 85	0	0.67, 0.79 [0.73]
	3	143 145 145	- 6 8	1525 1541 1543	81 85 85	0	0.48, 0.56 [0.52]
Lindsay, CA; United States 2016 Olympic (R140479)	3	151 151 148	- 7 6	694 695 673	87 87 89	0	0.28, 0.40 [0.34]
	3	150 149 151	- 7 7	2798 2863 2809	87 87 89	0	0.21, 0.24 [0.23]
Cottonwood, CA; United States 2016 20th Century (R140480)	3	149 149 149	- 7 7	704 704 704	81 85 89	0	0.10, 0.19 [0.15]
						3	0.14, 0.18 [0.16]
						7	0.31, 0.53 [0.42]
						14	0.39, 0.65 [0.52]
						21	0.30, 0.39 [0.35]
	3	148 148 148	- 7 7	1403 1403 1403	81 85 89	0	0.25, 0.50 [0.38]
						3	0.26, 0.54 [0.40]
						7	0.32, 0.37 [0.35]
						14	0.89, 0.46 [0.68]
						21	0.26, 0.24 [0.25]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Ephrata, WA; United States 2016 D'Anjou (R140481)	3	152 151 152	- 7 7	566 563 568	85 87 88	0	0.46, 0.34 [0.40]
	3	149 150 150	- 7 7	1867 1873 1872	85 87 88	0	0.24, 0.26 [0.25]
Zilah, WA; United States 2016 Bartlett (R140482)	3	152 153 153	- 7 7	975 998 962	NS	0	0.35, 0.33 [0.34]
	3	149 154 150	- 7 7	2132 2207 2155	NS	0	0.27, 0.23 [0.25]
Buena, WA; United States 2016 Bartlett (R140483)	3	153 150 149	- 7 7	826 813 803	NS	0	0.28, 0.36 [0.32]
	3	153 150 150	- 7 7	1808 1772 1780	NS	0	0.32, 0.27 [0.30]

Notes:

NS: Not specified.

[A] Applications were separated by a 2-week period, rendering the trials independent.

[B] Applications were made on the same day, rendering the trials dependent.

[C] Applications were made on the same day, rendering the trials dependent.

[D] Applications were separated by a 1-month period, rendering the trials independent.

[E] Applications were made on the same day, rendering the trials dependent.

Stone fruits

Table 77 Residues of mefentrifluconazole in whole stone fruits from trials conducted in the United States following application of an SC formulation using dilute and concentrate spray volumes (Hummel, 2016, BASF DocID, 2015_7005938)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
CHERRY							
Westley, CA; United States 2015 **Sweet cherry:** Royal Hazel (R140386)	3	149 148 148	- 7 7	704 703 703	79 81 89	0	0.33, 0.56 [0.45]
	3	148 148 148	- 7 7	1404 1404 1403	79 81 89	0	1.05, 1.10 [1.08]
Tulare, CA; United States 2015 **Sweet cherry:** Tulare (R140387)	3	150 150 151	- 6 7	838 689 803	85 87 89	0	0.93, 1.06 [1.00]
	3	150 150 151	- 6 7	3128 2932 2944	85 87 89	0	0.88, 0.96 [0.92]
Plainview, CA; United States 2015	3	150 151 150	- 7 7	740 840 820	85 87 87	0 3 7	0.65, 0.60 [0.63] 0.36, 0.57 [0.47] 0.49, 0.46 [0.48]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Sweet cherry: Rainer (R140388)						14	0.27, 0.33 [0.30]
						21	0.27, 0.20 [0.24]
	3	151	-	3326	85	0	1.10, 0.78 [0.94]
		151	7	3604	87	3	0.52, 0.86 [0.69]
		151	7	3451	87	7	0.42, 0.39 [0.41]
						14	0.59, 0.26 [0.43]
						21	0.39, 0.29 [0.33]
Ephrata, WA; United States 2014	3	152	-	568	81	0	0.93, 0.98 [0.96]
		151	7	565	85		
		153	7	569	87		
Sweet cherry: Skeena (R140389)[A]	3	152	-	1887	81	0	1.14, 0.96 [1.05]
		151	7	1885	85		
		151	7	1886	87		
Ephrata, WA; United States 2014	3	148	-	464	81	0	1.23, 1.66 [1.45]
		149	7	465	85		
		151	7	473	89		
Tart cherry: Balaton (R140390)[A]	3	151	-	1542	81	0	2.29, 1.80 [2.05]
		151	7	1542	85		
		151	7	1546	89		
Ridegeville, ON; Canada 2015	3	152	-	710	85	0	0.99, 0.91 [0.95]
		150	7	701	87		
		149	7	698	87		
Tart cherry: Montmorency (R140412)	3	152	-	1230	85	0	1.38, 1.39 [1.39]
		155	7	1252	87		
		148	7	1196	87		
Branchton, ON; Canada 2015	3	152	-	708	81	0	1.43, 1.66 [1.55]
		148	7	691	85		
		152	7	710	87		
Tart cherry: North Star (R140413)	3	154	-	1252	81	0	2.07, 2.43 [2.25]
		153	7	1243	85		
		154	7	1251	87		
Buffalo, MN; United States 2015	3	154	-	499	85	0	0.05, 0.03 [0.04]
		154	7	498	87		
		155	7	502	89		
Tart Cherry: Meteor (R140414)	3	153	-	971	85	0	0.03, 0.02 [0.03]
		153	7	972	87		
		153	7	972	89		
PEACH							
Alton, NY; United States 2014	3	152	-	898	79	0	0.43, 0.32 [0.38]
		150	8	890	81		
		149	7	885	81		
Virgil (R140391)	3	148	-	1852	79	0	0.49, 0.46 [0.48]
		151	8	1892	81		
		149	7	1867	81		
Chula, GA; United States 2015	3	156	-	544	77	0	0.37, 0.41 [0.39]
		151	7	522	81		
		151	7	512	87		
Hawthorne (R140889)	3	156	-	1260	77	0	0.49, 0.54 [0.52]
		151	7	1084	81		
		150	7	1076	87		
Morven, GA; United States 2015	3	151	-	491	76	0	0.39, 0.30 [0.35]
		149	7	511	81		
		151	7	493	87		
June Prince	3	152	-	1374	76	0	0.48, 0.45 [0.47]

Mefentrifluconazole

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
(R140890)		150 154	7 7	1312 1253	81 87		
Tifton, GA; United States 2015 June Prince (R140891)	3	152 150 151	- 7 7	483 508 500	76 77 81	0	0.32, 0.49 [0.41]
						3	0.19, 0.20 [0.20]
						7	0.12, 0.17 [0.15]
						14	0.07, 0.08 [0.08]
	3	150 153 150	- 7 7	1313 1305 1294	76 77 81	0	0.36, 0.31 [0.34]
						3	0.25, 0.20 [0.23]
						7	0.12, 0.14 [0.13]
						14	0.11, 0.07 [0.09]
Jordan Station, ON; Canada 2015 Glowing Star (R140428)[B]	3	151 151 155	- 7 7	707 706 725	79 81 NS	0	0.57, 0.53 [0.55]
	3	152 156 151	- 7 7	1233 1263 1224	79 81 NS	0	0.67, 0.73 [0.70]
Jordan Station, ON; Canada 2015 Brighton (R140429)[B]	3	149 147 156	- 7 7	695 686 728	85 87 87	0	0.27, 1.34 [0.81]
	3	150 269 147	- 7 7	1216 1257 1194	5 87 87	0	0.63, 0.36 [0.50]
Betrand, MO; United States 2014 Encor (R140395)[C]	3	154 152 150	- 8 7	571 564 556	76 81 87	0	0.33, 0.36 [0.35]
	3	151 151 150	- 8 7	1172 1170 1161	76 81 87	0	0.39, 0.37 [0.38]
Betrand, MO; United States 2014 Tyler (R140396)[C]	3	152 153 153	- 8 7	565 568 568	76 81 87	0	0.32, 0.32 [0.32]
	3	147 149 153	- 8 7	1140 1156 1183	76 81 87	0	0.40, 0.43 [0.42]
Harrah, OK; United States 2014 John Boy (R140397)	3	152 151 151	- 6 7	738 494 757	81 81 85	0	0.32, 0.26 [0.29]
	3	157 153 149	- 6 7	2188 2352 2476	81 85 87	0	0.63, 0.56 [0.60]
Corning, CA; United States 2014 Red Haven (R140398)	3	149 148 148	- 7 7	704 76 702	81 81 87	0	0.25, 0.19 [0.22]
	3	148 148 148	- 7 7	1403 1402 1403	81 81 87	0	0.87, 1.04 [0.96]
Kingsburg, CA; United States 2014 Late Ross (R140399)[D]	3	150 151 149	- 7 7	688 695 681	81 81 87	0	0.29, 0.28 [0.29]
	3	151 152 150	- 7 7	1426 1427 1417	81 81 87	0	0.64, 0.79 [0.72]
Kingsburg, CA; United States	3	152 151	- 7	721 703	81 81	0	0.39, 0.61 [0.50]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
2014		151	7	697	87		
Ross (R140400)[D]	3	151	-	1415	81	0	0.92, 1.00 [0.96]
		150	7	1409	81		
		149	7	1388	87		
Ephrata, WA; United States 2014	3	149	-	467	85	0	0.34, 0.19 [0.27]
		152	7	477	86		
		151	7	473	87		
Glowing Star (R140401)	3	151	-	1537	85	0	0.35, 0.36 [0.36]
		151	7	1545	86		
		151	7	1538	87		
PLUM							
Williamson, NY; United States 2014	3	150	-	889	79	0	0.33, 0.31 [0.32]
		151	9	891	81		
		149	7	882	85		
Shiro (R140402)	3	148	-	1848	79	0	0.28, 0.24 [0.26]
		148	9	1849	81		
		149	7	1867	85		
Buffalo, MN; United States 2015	3	146	-	503	85	0	<0.01, <0.01 [<0.01]
		147	7	507	87		
		144	7	497	89		
Black Ice (R140403)	3	146	-	1464	85	0	0.01, <0.01 [<0.01]
		145	7	1464	87		
		147	7	1464	89		
Branchton, ON; Canada 2015	3	151	-	705	81	0	0.84, 0.96 [0.90]
		151	6	705	85		
		146	8	683	85		
German (R140404)[E]	3	152	-	1235	81	0	1.05, 0.91 [0.98]
		147	6	1198	85		
		153	8	1243	85		
Branchton, ON; Canada 2015	3	151	-	706	81	0	0.66, 0.83 [0.75]
		150	6	698	85		
		145	8	678	85		
Italian (R140405)[E]	3	154	-	1250	81	0	0.95, 1.01 [0.98]
		150	6	1217	85		
		156	8	1269	85		
Orland, CA; United States 2014	3	179	-	705	79	0	0.03, 0.02 [0.03]
		148	7	700	NS		
		149	7	704	NS		
French (R140406)	3	148	-	1403	79	0	0.03, 0.03 [0.03]
		148	7	1402	NS		
		148	7	1403	NS		
Los Molinos, CA; United States 2014	3	149	-	704	79	0	0.23, 0.18 [0.21]
		148	7	701	81		
		148	7	700	85		
French (R140407)	3	148	-	1405	79	0	0.29, 0.31 [0.30]
		148	7	1404	81		
		148	7	1403	85		
Terra Bella, CA; United States 2015	3	153	-	784	85	0	0.07, 0.06 [0.07]
		155	7	801	87		
		152	7	780	89		
Yummy Beaut (R140892)	3	149	-	1936	85	0	0.14, 0.11 [0.13]
		156	7	1890	87		
		150	7	1844	89		
Lindsay, CA;	3	153	-	772	81	0	0.20, 0.20 [0.20]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
United States 2014 Angeleno (R140409)		151 150	7 7	741 749	85 89	3	0.23, 0.19 [0.21]
						7	0.17, 0.13 [0.15]
						14	0.11, 0.08 [0.10]
						21	0.12, 0.07 [0.10]
	3	151 150 150	- 7 7	1923 1807 1506	81 85 89	0	0.14, 0.14 [0.14]
						3	0.17, 0.15 [0.16]
						7	0.12, 0.12 [0.12]
						14	0.10, 0.12 [0.11]
						21	0.07, 0.10 [0.09]
Ephrata, WA; United States 2015 Early Italian (R140410)	3	149 148 150	- 7 7	467 464 468	81 85 87	0	0.25, 0.26 [0.26]
	3	150 151 151	- 7 7	1535 1538 1546	81 85 87	0	0.18, 0.20 [0.19]
Abbotsford, BC; Canada 2015 PR H1 (R140411)	3	154 151 152	- 9 7	614 602 610	82 83 83	0	0.30, 0.21 [0.26]
	3	149 144 152	- 9 7	983 962 1010	82 83 83	0	0.38, 0.35 [0.37]

Notes:

[A] Applications were separated by 1 day, rendering the trials dependent.

[B] Applications were made on the same day, rendering the trials dependent.

[C] Applications were made on the same day, rendering the trials dependent.

[D] Applications were separated by 11-day period, rendering the trials independent.

[E] Applications were made on the same day, rendering the trials dependent.

Table 78 Residues of mefentrifluconazole in caneberries (blackberries) from trials conducted in the United States following applications of an SC formulation (Shreier, 2018, BASF DocID 2018_7001820)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Weston, GA; 2016, Navaho (R160044)	3	151 150 149	- 7 7	537 556 558	81 83 87	0	0.30, 0.20 [0.25]
Lebanon, OK; 2016, Unknown (R160045)	3	154 152 147	- 7 7	588 613 596	85 87 89	0	0.79, 0.63 [0.71]
Oregon City, OR; 2016, Arden (R160046)	3	152 151 151	- 7 6	986 981 977	75-80 75-81 77-89	0	1.11, 1.32 [1.22]
Roseburg, OR; 2016, Kotata (R160047)	3	149 148 148	- 7 6	567 561 563	83 85 89	0	1.62, 1.03 [1.33]
Winston, OR; 2016, Chester (R160048)	3	150 150 151	- 7 8	571 571 576	81 85 89	0	1.11, 1.48 [1.30]
Tecumseh, MI; 2016, Triple Crown	3	150 150	- 7	538 536	70 76	0	0.46, 0.24 [0.35]
						1	0.38, 0.24 [0.31]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
(R160049)		150	7	534	81	3	0.34, 0.16 [0.25]
						7	0.08, 0.10 [0.09]
						10	0.13, 0.10 [0.12]

Table 79 Residues of mefentrifluconazole in blueberries from trials conducted in the United States following applications of an SC formulation (Shreier, 2018, BASF DocID 2018_7001820)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Pen Yan, NY;	3	157	-	587	80	0	0.69, 0.46 [0.58]
2016, Patriot		155	7	582	85		
(R160050)		152	8	571	89		
Dundee, NY;	3	149	-	572	81	0	0.78, 0.75 [0.77]
2016, Blue Ray		146	6	561	85		
(R160051)		142	8	548	89		
New Tripoli, PA;	3	146	-	920	5 % fruit coloured	0	0.72, 0.63 [0.68]
2016, Dixie		146	7	917	10-15 % fruit coloured		
(R160052)		147	8	926	60-80 % fruit coloured		
Alapaha, GA;	3	150	-	1031	79	0	0.61, 0.41 [0.51]
2016, Rebel		149	7	1099	85		
(R160053)		151	7	1039	89		
Plains, GA;	3	149	-	530	85	0	0.05, 0.06 [0.06]
2016, Ochlockonee		150	7	556	87		
Rabbiteye (R160054)		149	7	559	88		
Nevis, MN;	3	157	-	538	79-81	0	0.23, 0.12 [0.18]
2016, North Blue		152	7	523	85-87		
(R160055)		146	7	524	87-89		
Fenton, MI;	3	151	-	586	Full berry	0	0.68, 0.44 [0.56]
2016, Spaartan		150	7	592	Full berry		
(R160056)		156	7	614	Full berry		
Roseburg, OR;	3	149	-	569	83	0	3.07, 3.24 [3.16]
2016, Duke		147	7	558	85		
(R160057)		149	6	567	89		
Britton, MI;	3	151	-	586	Full berry	0	0.71, 0.76 [0.74]
2016, Draper		151	7	595	Full berry	1	0.52, 0.48 [0.50]
(R160058)		150	7	592	Full berry	3	0.29, 0.31 [0.30]
						7	0.14, 0.11 [0.13]
						10	0.06, 0.07 [0.07]

Grapes

Table 80 Residues of mefentrifluconazole in grapes from trials conducted in Canada and the United States following application of an SC formulation using dilute and concentrate spray volumes (Norris, 2016, BASF DocID 2016_7010091)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	DALA	Mefentrifluconazole (mg/kg)
WINE GRAPES						
Renton, ON;	3	150	-	504	14	0.63, 0.70 [0.67]
Canada		148	9	498	21	Not sampled

Mefentrifluconazole

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	DALA	Mefentrifluconazole (mg/kg)
2014 Concorde (R140826)		151	10	509		
	3	146	-	1963	14	0.45, 0.44 [0.45]
		147	9	1972	21	Not sampled
		148	10	2000		
Alton, NY; United States 2014 Cayuga White (R140824)	3	150	-	747	14	0.69, 0.63 [0.66]
		150	10	749	21	0.76, 0.73 [0.75]
		149	10	470		
	3	152	-	1888	14	0.83, 0.82 [0.83]
		152	10	1893	21	0.78, 0.75 [0.77]
		150	10	1873		
Breinigsville, PA; United States 2014 Corot noir (R140825)	3	150	-	432	0	0.28, 0.34 [0.31]
		151	8	436	3	0.27, 0.31 [0.29]
		148	10	428	7	0.25, 0.22 [0.24]
					14	0.20, 0.19 [0.20]
					21	0.17, 0.20 [0.19]
	3	152	-	2447	0	0.60, 0.54 [0.57]
		150	8	2419	3	0.74, 0.54 [0.64]
		151	10	2438	7	0.49, 0.46 [0.48]
					14	0.30, 0.20 [0.25]
					21	0.24, 0.21 [0.23]
Templeton, CA; United States 2014 Syrah noir (R140829)	3	154	-	457	14	0.38, 0.33 [0.36]
		153	10	442	21	0.32, 0.29 [0.31]
		150	10	485		
	3	152	-	2295	14	0.38, 0.38 [0.38]
		147	10	1992	21	0.32, 0.25 [0.29]
		151	10	2287		
Paso Robles, CA; United States 2014 Cabernet (R140830)	3	155	-	545	14	0.47, 0.56 [0.52]
		150	10	458	21	0.56, 0.69 [0.63]
		155	10	453		
	3	157	-	2375	14	1.04, 1.01 [1.03]
		154	10	2240	21	0.95, 0.84 [0.90]
		157	10	2048		
Dinuba, CA; United States 2014 Alicante (R140834)	3	150	-	459	0	0.29, 0.28 [0.29]
		149	11	480	3	0.20, 0.24 [0.22]
		149	9	462	7	0.19, 0.18 [0.19]
					14	0.19, 0.12 [0.16]
					21	0.13, 0.13 [0.13]
	3	151	-	2349	0	0.28, 0.40 [0.34]
		152	11	2206	3	0.30, 0.32 [0.31]
		151	9	2182	7	0.36, 0.36 [0.36]
					14	0.25, 0.40 [0.33]
					21	0.23, 0.26 [0.25]
Ephrata, WA; United States 2014 Chardonnay (R140835)	3	149	-	470	14	1.10, 1.03 [1.07]
		150	10	473	21	0.66, 0.86 [0.76]
		149	10	469		
	3	151	-	1877	14	0.81, 0.99 [0.90]
		151	10	1874	21	0.58, 0.86 [0.72]
		150	10	1873		
Oregon City, OR; United States 2014 Chardonnay (R140836)	3	150	-	436	14	0.23, 0.30 [0.27]
		154	10	440	21	0.33, 0.23 [0.28]
		148	10	424		
	3	147	-	1935	14	0.38, 0.43 [0.41]
		150	10	1981	21	0.22, 0.25 [0.24]
		149	10	1922		

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	DALA	Mefentrifluconazole (mg/kg)
TABLE GRAPES						
Kerman, CA; United States 2014 Thompson seedless (R140827)[A]	3	154	-	569	14	0.18, 0.28 [0.23]
		151	9	559	21	0.17, 0.12 [0.15]
		155	10	572		
	3	149	-	2333	14	0.84, 0.60 [0.72]
		150	9	2343	21	0.53, 0.36 [0.45]
		150	10	2347		
Kerman, CA; United States 2014 Thompson seedless (R140828)[A]	3	155	-	572	14	0.74, 0.63 [0.
		153	10	564	21	0.42, 0.35 [0.39]
		152	10	561		
	3	149	-	2338	14	0.31, 0.39 [0.35]
		149	10	2339	21	0.28, 0.53 [0.41]
		150	10	2346		
Porterville, CA; United States 2014 Crimson (R140831)	3	150	-	682	14	0.29, 0.33 [0.31]
		151	10	690	21	0.45, 0.37 [0.41]
		149	11	694		
	3	150	-	2060	14	0.22, 0.34 [0.28]
		150	10	2058	21	0.08, 0.14 [0.11]
		148	11	2032		
Kingsburg, CA; United States 2014 Crimson (R140832)	3	147	-	666	14	0.41, 0.55 [0.48]
		150	8	722	21	0.31, 0.31 [0.31]
		154	10	700		
	3	148	-	2028	14	0.41, 0.50 [0.46]
		147	8	2700	21	0.42, 0.33 [0.38]
		154	10	2106		
Terra Bella, CA; United States 2014 Crimson (R140833)	3	151	-	528	14	0.09, 0.13 [0.11]
		154	10	512	21	0.08, 0.06 [0.07]
		152	11	532		
	3	151	-	2240	14	0.32, 0.36 [0.34]
		148	10	2194	21	0.27, 0.30 [0.29]
		153	11	2270		

Notes:

[A] Applications were made on the same day, rendering the trials dependent.

Table 81 Residues of mefentrifluconazole in strawberries from trials conducted in the United States following applications of an SC formulation (Shreier, 2018, BASF DocID 2018_7001820)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Marion, NY; 2016, Lamora (R160059)	3	150	-	954	73	0	0.13, 0.16 [0.15]
		151	8	958	81-85		
		149	6	940	87		
Bradenton, FL; 2016, Radiant (R160060)	3	148	-	699	Mature	0	0.43, 0.45 [0.44]
		152	7	634	Fruiting berries		
		152	6	601	Ripe berries		
Aurora, SD; 2016, EVIE 2 (R160061)	3	150	-	568	65-81	0	0.44, 0.56 [0.50]
		147	7	556	65-81		
		150	8	570	81-85		
Paynesville, MN; 2016, Kent (R160062)	3	152	-	521	73-81	0	0.07, 0.09 [0.08]
		155	7	529	87-89		
		150	7	533	87-89		

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Fenton, MI; 2016, Apline (R160063)	3	152 152 149	- 7 7	589 587 577	87 88 89	0	<0.01, <0.01 [<0.01]
Yuba City, CA; 2016, San Andreas (R160064)	3	151 150 151	- 8 7	516 514 516	81 83 89	0	0.43, 0.43 [0.43]
Fresno, CA; 2016, Seascape (R160065)	3	150 149 148	- 7 7	658 652 648	85 87 89	0	0.59, 0.64 [0.62]
Dinuba, CA; 2016, Seascape (R160066)	3	149 147 149	- 7 7	656 645 652	85 87 89	0	1.01, 1.09 [1.05]
Grants Pass, OR; 2016, San Andreas (R160067)	3	151 152 151	- 7 7	516 518 516	79 83 89	0	0.30 ,0.28 [0.29]
Roseburg, OR; 2016, San Andreas (R160068)	3	158 155 156	- 7 6	525 516 520	81 86 89	0	0.22, 0.25 [0.24]
Britton, MI; 2016, Alpine (R160069)	3	150 151 152	- 7 7	580 584 589	87 88 89	0	<0.01, <0.01 [<0.01]
						1	<0.01, <0.01 [<0.01]
						3	<0.01, <0.01 [<0.01]
						7	<0.01, <0.01 [<0.01]
						10	<0.01, <0.01 [<0.01]

Bananas

Table 82 Residues of mefentrifluconazole in bagged and unbagged bananas from trials conducted in South America following applications of an SC formulation (Lucas, 2019, BASF DocID 2019_3000582)

Location; Year, Variety (Trial ID)	No.	Application			DALA	Portion analysed	Mefentrifluconazole (mg/kg)	
		Rate (g ai/ha)	RTI (days) (days)	Total rate (g ai/ha)			Bagged	Unbagged
Conchal; Brazil, 2017 Nanica (G175048)	5	140	14	700	0	Whole fruit	<0.01	0.37
					1		<0.01	0.65
					3		<0.01	0.26
					7		<0.01	0.44
					0	Peel	<0.01	0.24
					1		0.05	0.84
					3		0.45	0.16
					7		<0.01	1.62
					0	Pulp	<0.01	0.12
					1		<0.01	0.095
					3		0.035	0.012
					7		<0.01	0.21
Limera; Brazil, 2017 Prata (G175049)	5	140	14	700	0	Whole fruit	<0.01	0.74
					1		<0.01	0.44
					3		<0.01	0.59
					7		<0.01	0.55
					0	Peel	0.013, 0.013, 0.017 [0.014]	0.73, 0.71, 0.74, 0.38, 0.39 [0.59]
					1		0.059, 0.064,	0.95, 0.66, 0.83

Location; Year, Variety (Trial ID)	No.	Application			DALA	Portion analysed	Mefentrifluconazole (mg/kg)	
		Rate (g ai/ha)	RTI (days) (days)	Total rate (g ai/ha)			Bagged	Unbagged
							0.065 [0.063]	[0.81]
					3		0.11	1.6, 1.4 [1.5]
					7		0.026	0.65
					0	Pulp	<0.01	0.21
					1		<0.01	0.13
					3		<0.01	0.19
					7		<0.01	0.06
Campunas; Brazil 2017 Prata (G175050)	5	140	14	700	0	Whole fruit	0.015	0.47
					1		0.013	0.32
					3		<0.01	0.40
					7		0.010	0.54
					0	Peel	0.11	0.74
					1		0.012	0.65
					3		0.014	0.81
					7		<0.021	0.93
					0	Pulp	<0.01	0.034
					1		<0.01	0.028
					3		<0.01	0.052
					7		<0.01	0.037
Rio das Pedras; Bazil 2017 Prata (G175051)	5	140	14	700	0	Whole fruit	<0.01	0.47
					0/7		<0.01	0.28
					0	Peel	<0.01	0.55
					0/7		<0.01	0.15
					0	Pulp	<0.01	0.040
					0/7		<0.01	0.015
Tapirai; Brazil 2017 Prata (G175052)	5	140	14	700	0	Whole fruit	<0.01	0.026, 0.026, 0.025 [0.026]
					0/7		<0.01	0.040, 0.042, 0.043 [0.042]
					0	Peel	0.037	0.089
					0/7		0.024	0.081
					0	Pulp	<0.01	<0.01
					0/7		<0.01	<0.01
Manuel J.Calle/Cañar; Ecuador 2017 Valery (G175053)	5	140	14	700	0	Whole fruit	<0.01	0.19
					1		<0.01	0.34
					3		<0.01	0.57
					7		0.011	0.37
					0	Peel	0.024	0.43
					1		<0.01	0.49
					3		0.018	0.38
					7		0.016	0.78
					0	Pulp	<0.01	0.047
					1		<0.01	0.053
					3		<0.01	0.034
					7		<0.01	0.047
Simon Bolivar/ Guayas; Ecuador 2017 Williams (G175054)	5	140	14	700	0	Whole fruit	<0.01	0.35
					0/7		<0.01	0.19
					0	Peel	<0.01	0.50
					0/7		<0.01	0.61
					0	Pulp	<0.01	0.047
					0/7		<0.01	0.091
Municipio Zona	5	140	14	700	0	Whole fruit	0.18, 0.15, 0.14	0.14

Location; Year, Variety (Trial ID)	No.	Application			DALA	Portion analysed	Mefentrifluconazole (mg/kg)	
		Rate (g ai/ha)	RTI (days) (days)	Total rate (g ai/ha)			Bagged	Unbagged
Bananera / Departamento del Magdalena; Colombia 2017 Valery (G175055)							[0.16]	
					1		0.025	0.27, 0.22, 0.24 [0.24]
					3		0.021	0.16
					7		0.042	0.17
					0	Peel	0.15	0.52
					1		0.067	0.30
					3		<0.01	0.51
					7		0.017	0.47
					0	Pulp	0.030, 0.038, 0.032 [0.033]	0.11
					1		<0.01	0.057
					3		<0.01	0.085
					7		<0.01	0.14
Distrito de Santa Marta / Departamento del Magdalena; Colombia 2017 Williams (G175056)	5	140	14	700	0	Whole fruit	0.012	0.16
					0/7		<0.01	0.11
					0	Peel	<0.01	0.21
					0/7		<0.01	0.24
					0	Pulp	<0.01	0.039
					0/7		<0.01	0.057
El Retén / Departamento del Magdalena; Colombia 2017 Williams (G175057)	5	140	14	700	0	Whole fruit	0.043	0.12
					0/7		0.014	0.053
					0	Peel	0.66	0.065, 0.074, 0.078 [0.072]
					0/7		0.042	0.15, 0.16, 0.17 [0.16]
					0	Pulp	0.094	<0.01
					0/7		<0.01	0.011

Table 83 Residues of mefentrifluconazole in pitted whole avocadoes [expressed as whole fruit] from trials conducted in Brazil, Colombia and Mexico following applications of an SC formulation (Faria, 2021, BASF DocID 2021_2029386)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Mogi Mirim, São Paulo; Brazil 2021, Haas (G200080)	3	131	-	547	76	0	0.11
		122	14	508	77	1	0.20
		131	14	545	78	3	0.10
						7	0.065
Piraju, São Paulo; Brazil 2021, Haas (G200081)	3	129	-	539	75	0	0.33
		126	14	523	77	1	0.29
		132	14	548	78	3	0.36
						7	0.42
Medellin, Antioquia; Columbia Haas, 2021 (G200084)[A]	3	126	-	527	78	0	0.45
		137	14	574	78	1	0.31
		133	14	586	79	3	0.22
						7	0.20
Medellin, Antioquia; Columbia, 2021, Haas	3	142	-	592	77	0	0.33
		128	14	548	78		

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
(G200085)[A]		139	14	599	79		
Villa Donato Guerra, Mexico; 2021. Haas (G200082)	3	134	-	556	79-80	0	0.60
		133	14	554	80-81	1	0.52
		129	14	539	80-81	3	0.42
						7	0.50
Ocuituco, Morelos; Mexico 2021, Haas (G200083)	3	133	-	554	79	0	0.29
		133	14	552	79	1	0.28
		132	14	552	81	3	0.39
						7	0.39
						1	0.27
						3	0.32
						7	0.28

Notes:

[A] Applications were separated by 19 days, rendering the trials independent.

Table 84 Residues of mefentrifluconazole in whole papaya from trials conducted in Mexico and South America following applications of an SC formulation (Faria, 2021, BASF DocID 2021_2029387)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Piracicaba, São Paulo; Brazil 2021 Sunrise (G200086)	2	129	-	536	78	0	0.055
		129	14	536	81	1	0.044
						3	0.043
						7	0.038
	4	124	-	516	75	0	0.19
		125	14	520	77	1	0.17
		129	14	536	78	3	0.19
		122	14	509	81	7	0.071
Linares, ES; Brazil 2021 Sunrise (G200087)	2	125	-	522	77	0	0.067
		126	14	523	81	1	0.053
						3	0.066
						7	0.040
	4	126	-	525	75	0	0.068
		126	14	524	76	1	0.065
		126	14	525	77	3	0.058
		125	14	522	81	7	0.050
Araras, São Paulo; Brazil 2021 Papaya (G200173)	2	122	-	508	82	0	0.13
		123	13	511	84	1	0.058
						3	0.071
						7	0.064
	4	126	-	525	79	0	0.14
		124	14	516	81		
		122	14	508	82		
		123	13	512	84		
Armenia, Quindio; Columbia 2021 Tainung (G200091)	2	142	-	611	77	0	<0.01
		132	14	585	78	1	<0.01
						3	<0.01
						7	<0.01
	4	139	-	589	76	0	<0.01
		140	13	595	76	1	<0.01
		137	14	592	77	3	<0.01
		129	14	586	78	7	<0.01

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Municipio de Ignacio de la Llave,Veracruz; Mexico 2021 Marabol (G200089)	2	131 129	- 14	549 538	70-78 70-79	0	0.20
						1	0.17
						3	0.22
						7	0.14
	4	131 130 133 132	- 14 14 14	546 543 554 551	70-75 75-75 70-78 70-79	0	0.24
						1	0.19
						3	0.24
						7	0.20
Colima, Colima; Mexico 2021 Marivel (G200164)	2	132 131	- 14	548 543	70-79 70-81	0	0.22
						1	0.17
						3	0.19
						7	0.18
	4	133 137 130 133	- 14 14 14	554 569 541 554	70-79 70-79 70-79 70-81	0	0.37
						1	0.20
						3	0.34
						7	0.24
						1	0.17
						3	0.099
						7	0.093

Table 85 Residues of mefentrifluconazole in whole mango from trials conducted in China following applications of an SC formulation (Sun, 2019, BASF DocID 2021_2053597)

Location; Year, Variety (Trial ID)	No.	Nominal rate (kg ai/hL)	RTI (days)	Nominal spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Zhaoqing, Guangdong; 2018 Chokanan	3	0.016 0.016 0.016	- 8 10	675 675 675	72 73 73	0	0.64
						1	0.58
						7	0.41
						14	0.28, 0.28 [0.28]
						21	0.16, 0.16 [0.16]
Maoming, Guangdong; 2018, Mangifera indica L. cv. Zihua	3	0.016 0.016 0.016	- 7 7	675 675 675	72 73 74	14	0.16, 0.15 [0.16]
						21	0.07, 0.07 [0.07]
Nanning, Guangxi; 2018 No.82 Guire	3	0.016 0.016 0.016	- 7 7	675 675 675	73 74 75	14	0.20, 0.19 [0.20]
						21	0.11, 0.11 [0.11]
Yuxi, Yunnan; 2018 Unknown	3	0.016 0.016 0.016	- 7 7	675 675 675	71 72 74	0	0.38
						1	0.31
						7	0.20
						14	0.16, 0.16 [0.16]
						21	0.08, 0.07 [0.08]
Sanya, Hainan; 2018 Guifei	3	0.016 0.016 0.016	- 7 7	675 675 675	72 73 74	0	0.70
						1	0.43
						7	0.31
						14	0.12, 0.12 [0.12]
						21	0.05, 0.04 [0.05]
Zhangzhou, Fujian; 2018 Ai Wen	3	0.016 0.016 0.016	- 7 7	675 675 675	71 72 73	14	0.22, 0.21 [0.22]
						21	0.06, 0.06 [0.06]

Notes:

Residues in mango pulp sampled 14 and 21 DALA were all (n=12) <LOQ (0.01 ppm)

Bulb vegetables

Table 86 Residues of mefentrifluconazole in bulb vegetables from trials conducted in Canada and the United States following applications of an SC formulation (Brungardt, 2016, BASF DocID 2016_7010854)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH)	DALA	Mefentrifluconazole (mg/kg
BULB ONION							
Germansville, PA;	3	152	-	285	41-43	7	0.099, 0.081 [0.09]
2016, Stuttgarter		156	7	291	43-45		
(R161015)		157	6	293	47		
Stewardson, IL;	3	152	-	232	45	7	0.098, 0.11 [0.104]
2016, Ringmaster		155	8	217	47		
(R161016)		151	7	285	48		
Stilwell, KS;	3	154	-	284	45	6	0.028, 0.035 [0.032]
2016, Red Candy Apple		154	7	283	48		
(R161017)		153	7	280	49		
Jerseyville, ON; Canada	3	152	-	203	41	7	0.056, 0.038 [0.047]
2016, White Sweet Spanish		153	7	204	45		
(R161018)		145	7	194	48-49		
Elmira, ON; Canada	3	151	-	202	45-47	8	0.065, 0.11 [0.088]
2016, Safrane		156	7	208	47		
(R161019)		147	6	196	48		
Zearing, IA;	3	156	-	220	43	0	0.15, 0.13 [0.14]
2016		150	6	215	48	3	0.19, 0.073 [0.132]
Yellow		156	8	202	48	7	0.075, 0.14 [0.108]
(R161020)						10	0.067, 0.069 [0.068]
						15	0.060, 0.043 [0.052]
Hinton, OK; 2016	3	148	-	248	45	8	0.11, 0.078 [0.094]
Dixondale Candies		152	8	254	48		
(R161021)		149	7	248	48-49		
Wall, TX; 2016	3	151	-	269	45	7	0.013, 0.013 [0.013]
White Bermuda		149	7	274	47		
(R161022)		150	7	270	48		
King City, CA;	3	152	-	285	47	7	0.014, <0.01 [0.012]
2016, Marenge		153	7	287	48		
(R161023)		150	7	282	48		
Sanger, CA;	3	150	-	299	47	7	<0.01, <0.01 [<0.01]
2016, Candy Case		150	6	325	47		
(R161024)		152	8	330	47		
Nampa, ID;	3	148	-	187	45-47	8	0.029, 0.13 [0.080]
2016, Nunhems Vaquero		155	7	196	45-47		
(R161025)		144	7	182	48		
Payette, ID;	3	152	-	284	47	7	0.059, 0.045 [0.052]
2016, Sedona		154	7	288	47		
(R161026)		152	7	285	47		
Oregon City, OR;	3	151	-	234	42-44	7	0.040, 0.028 [0.034]
2016, Vaquero		156	7	245	43-45		
(R161027)		151	7	237	43-46		
GREEN ONIONS							
Deli, ON; Canada	3	154	-	207	41	7	0.29, 0.27 [0.28]
2016, Feast		154	7	208	43-45		
(R161014)		156	8	209	43-45		
Stewardson, IL;	3	155	-	241	19	7	0.40, 0.43 [0.42]
2016, Ringmaster		147	7	207	24		

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH)	DALA	Mefentrifluconazole (mg/kg
(R161028)		195	7	291	41		
Fresno, CA;	3	150	-	282	41	7	2.2, 1.9 [2.1]
2016, White Spear		148	7	279	43		
(R161029)[A]		150	7	282	45		
Fresno, CA;	3	151	-	292	41	0	1.7, 1.5 [1.6]
2016, Super Star Case		148	7	232	41	3	0.96, 1.3 [1.13]
(R161030)[A]		151	6	279	41	7	0.47, 0.31 [0.39]
						10	0.14, 0.23 [0.19]
						14	0.15, 0.14 [0.15]
Oregon City, OR;	3	148	-	230	14-16	7	0.12, 0.10 [0.11]
2016, Parade		150	7	235	14-16		
(R161031)		151	7	236	36-41		

Notes:

[A] Applications were separated by greater than 6 months, rendering the trials independent.

Fruiting vegetables, cucurbits

Table 87 Residues of mefentrifluconazole in fruiting vegetables, cucurbits, from trials conducted in the United States following applications of an SC formulation (Wyatt, 2021, BASF DocID 2021_2012679)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
CUCUMBER							
Elko, SC;	3	149	-	206	64-71	0	0.034, 0.023 [0.029]
2016, Space Master		149	7	206	66-73		
(R160070)		148	6	206	68-76		
Chula, GA;	3	149	-	225	71	0	0.036, 0.033 [0.035]
2016, SV4719CS F1		154	7	225	73		
(R160071)		150	7	225	85		
Oviedo, FL;	3	149	-	281	68	0	0.123, 0.069 [0.096]
2016, Park's Select Slicer		151	7	281	71		
(R160072)		150	7	281	72		
Delavan, WI;	3	149	-	196	83	0	0.027, 0.014 [0.021]
2016, Marketmore 76		149	7	206	85		
(R160073)		150	7	206	89		
Richland, IA;	3	150	-	234	67	0	0.015, <0.01 [0.013]
2016, Marketmore 76		151	7	234	71		
(R160074)		152	6	243	85		
Uvalde, TX; 2016	3	150	-	206	69	0	0.024, 0.025 [0.025]
Stonewall G4 F1		151	7	206	71		
(R160075)		152	7	262	82		
Porterville, CA; 2016	3	149	-	271	87	0	0.043, 0.042 [0.043]
Poinsett 76		150	7	271	88		
(R160076)		150	7	271	89		
Grants Pass, OR; 2016	3	155	-	290	84	0	0.023, 0.040 [0.032]
Kirby		157	7	290	88		
(R160077)		156	6	290	89		
Seven Springs, NC;	3	152	-	253	63	0	0.029, 0.032 [0.031]
2016, Lancer 152		150	5	309	63-66	3	0.021, 0.017 [0.019]
(R160078)		151	7	327	68-82	7	<0.01, <0.01 [<0.01]
						10	<0.01, <0.01 [<0.01]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
SUMMER SQUASH							
Alton, NY; 2016 Multipik F1 (R160079)	3	152 152 152	- 7 7	281 281 281	81-82 84-85 87-89	0	0.044, 0.061 [0.053]
Seven Springs, NC; 2016, Early Prolific Straightneck (R160080)	3	151 150 150	- 5 7	253 309 318	66 65-71 71-84	0	0.043, 0.048 [0.046]
Oviedo, FL; 2016 Summer Crookneck (R160081)	3	152 149 150	- 7 7	290 281 281	66 72 74	0	0.089, 0.080 [0.085]
Richland, IA; 2016 Yellow Crookneck OG (R160082)	3	150 151 150	- 7 7	253 234 234	51-52 56-58 85	0	0.052, 0.044 [0.048]
Carlyle, IL; 2016 Spineless Beauty (R160083)	3	151 149 151	- 8 6	271 243 215	69 87 89	0	0.086, 0.090 [0.088]
Porterville, CA; 2016 Black Beauty (R160084)	3	152 151 150	- 8 6	290 290 290	81 85 89	0	0.042, 0.039 [0.041]
Grants Pass, OR; 2016 Scallop, Early White Bush, Patty Pan (R160085)	3	151 155 154	- 7 6	281 290 281	84 88 89	0	0.012, <0.01 [0.011]
Delavan, WI; 2016 Early Prolific Straightneck (R160086)	3	150 149 150	- 7 7	196 206 206	81 84 87	0 3 7 10	0.013, 0.014 [0.014] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01]
MUSKMELON							
Chula, GA; 2016 Athena (R160087)	3	150 151 150	- 7 7	215 225 206	75 77 87	0	0.146, 0.173 [0.160]
Delavan, WI; 2016 Hale's Best (R160088)	3	150 149 149	- 7 7	215 206 215	83 86 89	0	0.129, 0.080 [0.105]
Richland, IA; 2016 Earlichamp F1 (R160089)	3	152 149 149	- 7 7	243 234 234	71 81 85	0	0.204, 0.230 [0.217]
Carlyle, IL; 2016 Athena (R160090)	3	150 151 152	- 7 7	253 234 299	79 82 87	0	0.124, 0.092 [0.108]
Uvalde, TX; 2016 Primo (R160091)	3	150 146 151	- 7 7	196 187 196	72 81 82	0	0.110, 0.113 [0.112]
Hughson, CA; 2016 Hale's Best Jumbo (R160092)	3	150 151 151	- 8 6	281 281 281	86 88 89	0	0.125, 0.161 [0.143]
King City, CA; 2016 Hale's Best Jumbo (R160093)	3	150 148 151	- 7 7	337 337 337	85-88 85-88 86-89	0	0.265, 0.152 [0.209]
Porterville, CA; 2016 Hale's Best Jumbo (R160094)	3	152 154 151	- 7 7	281 281 281	88 89 89	0 3 7 9	0.148, 0.181 [0.165] 0.172, 0.141 [0.157] 0.089, 0.071 [0.08] 0.127, 0.089 [0.108]

Fruiting vegetables, other than cucurbits

Table 88 Residues of mefentrifluconazole in fruiting vegetables, other than cucurbits, from trials conducted in the United States following applications of an EC formulation (Reeves, 2018, BASF DocID 2018_7005678; Brungardt, 2021, BASF DocID 2021_2029151)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
CHERRY TOMATO							
Gardner, ND; 2016	3	127	-	161	80	0	0.355, 0.365 [0.360]
Supersweet 100		142	7	179	81		
(R160148)		148	7	187	82		
Fitchburg, WI; 2016, Tami G	3	148	-	306	79-85	0	0.145, 0.105 [0.125]
(R160149)		148	6	304	79-86		
		150	8	274	79-86		
Madera, CA;	3	147	-	283	82	0	0.450, 0.360 [0.405]
2016, Naomi		147	6	283	84	3	0.350, 0.270 [0.310]
(R160150)		146	7	281	85	5	0.295, 0.360 [0.328]
						7	0.215, 0.305 [0.260]
						10	0.195, 0.205 [0.200]
TOMATO							
Alton, NY, 2016	3	148	-	282	81	0	0.180, 0.110 [0.145]
POLBIG F1		148	7	283	83		
(R160151)		148	7	282	86		
Jeffersonville, GA;	3	148	-	279	72	0	0.125, 0.160 [0.142]
2016, Red Bounty		150	7	281	72		
(R160152)		151	6	284	81		
Winter Garden, FL;	3	147	-	278	72	0	0.185, 0.105 [0.145]
2016, Celebrity		148	7	279	74		
(R160153)		148	7	280	81		
Greenville, FL;	3	148	-	210	71	0	0.270, 0.225 [0.248]
2016, Red Beauty		149	8	223	74		
(R160154)		149	6	248	82		
Northwood, ND;	3	152	-	281	81	0	0.021, 0.030 [0.026]
2016, Super Fantastic		153	7	284	82-83		
(R160155)		151	7	280	84-85		
Lime Springs, IA; 2016	3	150	-	278	75	0	0.070, 0.030 [0.050]
Mountain Spring		150	7	278	77		
(R160156)		154	7	285	79		
Richland, IA; 2016	3	154	-	239	81	0	0.213, 0.168 [0.190]
Celebrity		152	7	236	83		
(R160157)		151	7	235	85		
St. John, KS; 2016	3	180	-	226	81	0	0.100, 0.120 [0.110]
Brush Early Girl		150	7	232	83		
(R160158)		147	7	226	88		
Marysville, OH; 2016	3	153	-	330	83	0	0.100, 0.090 [0.095]
Roma		156	7	335	85		
(R160159)		153	7	329	86		
Delavan, WI; 2016	3	151	-	208	82	0	0.090, 0.060 [0.075]
BHN 594		150	7	207	86		
(R160160)		153	7	211	89		
York, NE; 2016	3	151	-	193	81	0	0.050, 0.044 [0.047]
Big Beef		151	7	198	82		
(R160161)		152	8	199	83		
Leonard, MO; 2016	3	151	-	265	81	0	0.044, 0.040 [0.042]
Celebrity F1		153	7	249	85		
(R160162)		156	7	310	85		

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Madera, CA; 2016	3	153	-	282	82	0	0.210, 0.132 [0.171]
Quality		153	6	282	84		
(R160163)		151	7	279	85		
King City, CA; 2016	3	152	-	339	85-88	0	0.105, 0.065 [0.085]
Ace		152	7	341	85-88		
(R160164)		152	7	340	87-89		
Fresno, CA; 2016	3	153	-	284	81	0	0.495, 0.245 [0.370]
SUN 6366		151	7	281	84		
(R160165)		151	7	280	87		
Yuba City, CA; 2016	3	153	-	280	83	0	0.242, 0.214 [0.228]
HM 3884		152	7	279	85	3	0.090, 0.065 [0.078]
(R160166)		155	7	285	89	5	0.072, 0.055 [0.064]
						7	0.060, 0.065 [0.062]
						10	0.042, 0.075 [0.058]
BELL PEPPER							
Jeffersonville, GA; 2016	3	152	-	280	71	0	0.204, 0.200 [0.202]
Green Pepper		153	7	283	72		
(R160167)		152	6	281	89		
Jennings, FL; 2016	3	152	-	238	79	0	0.047, 0.051 [0.049]
Alleglance		152	8	241	82		
(R160168)		151	7	240	87		
Dana, IA; 2016	3	153	-	282	79	0	0.068, 0.057 [0.062]
Double-up		150	7	278	86		
(R160169)		151	7	279	89		
Fitchburg, WI; 2016	3	150	-	266	67-75	0	0.052, 0.035 [0.044]
Excursion		153	7	257	67-76		
(R160170)		150	7	261	67-76		
Lime Springs, IA; 2016	3	153	-	283	69	0	0.230, 0.214 [0.222]
Revelution		150	8	277	71		
(R160171)		152	6	282	79		
Fulshear, TX; 2016	3	155	-	239	71	0	0.465, 0.390 [0.428]
California Wonder		154	6	237	73		
(R160172)		154	7	238	89		
Madera, CA; 2016	3	153	-	283	86	0	0.335, 0.259 [0.297]
Cypress		153	7	282	86		
(R160173)		153	7	283	89		
Yuba City, CA; 2016	3	152	-	279	81	0	0.842, 0.616 [0.729]
Lady Bell		152	7	278	82		
(R160174)		156	7	286	89		
Richland, IA; 2016	3	151	-	234	71	0	0.055, 0.074 [0.064]
California Wonder		151	7	240	74	3	0.086, 0.033 [0.059]
(R160175)		152	7	247	76	5	0.041, 0.052 [0.046]
						7	0.034, 0.038 [0.036]
						10	0.024, 0.028 [0.026]
NON-BELL PEPPER							
Larned, KS; 2016	3	152	-	234	71	0	0.260, 0.270 [0.265]
Mucho Macho		150	7	231	83		
(R160176)		153	7	222	88		
Fresno, CA; 2016	3	152	-	282	84	0	0.8994 0.305 [0.602]
Chingon		151	7	281	86		
(R160177)		151	7	281	88		
Wall, TX; 2016	3	150	-	261	84	0	0.150, 0.225 [0.188]
TAM		154	7	273	87	3	0.245, 0.230 [0.238]
(R160178)		150	7	265	89	5	0.190, 0.165 [0.178]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
						7	0.230, 0.175 [0.202]
						10	0.210, 0.145 [0.178]
Levelland, TX; 2020	3	149	-	275	82	0	0.440, 0.335 [0.388]
Jalepeno		148	6	275	86		
(R200070)		152	8	282	86		
Porterville, CA; 2020	3	150	-	290	84	0	0.369, 0.296 [0.333]
Mammoth		150	7	295	87		
(R200071)		151	7	295	89		

Leafy greens and Brassica leafy vegetables

Table 89 Residues of mefentrifluconazole in leafy greens and brassica leafy vegetables in Canada and the United Sates, following applications of an SC formulation (Wyatt, 2019, BASF DocID 2019_7002361)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Portion Analysed	Mefentrifluconazole (mg/kg)
HEAD LETTUCE								
Elmira, ON; Canada	3	155	-	206	41-43	0	With wrapper leaves	1.6, 2.5 [2.1]
2018, Butterhead		155	7	206	47-48			
(R180036)		157	8	206	49		Without wrapper leaves	1.5, 1.6 [1.6]
Richland, IA; United	3	147	-	196	43	0	With wrapper leaves	0.20, 0.44 [0.32]
States, 2018		152	7	206	46			
Deuce		150	6	262	49		Without wrapper leaves	<0.01, <0.01 [<0.01]
(R180037)								
Arroyo Grande, CA;	3	151	-	281	41	0	With wrapper leaves	1.3, 1.7 [1.5]
United States, 2018		152	7	281	47			
Regency		151	7	281	49		Without wrapper leaves	0.13, 0.050 [0.09]
(R180038)								
King City, CA; United	3	154	-	215	48	0	With wrapper leaves	0.77, 1.0 [0.89]
States, 2018		150	7	262	48-49			
Regency		154	6	271	49		Without wrapper leaves	0.055, 0.036 [0.046]
(R180039)								
Porterville, CA; United	3	150	-	281	47	0	With wrapper leaves	1.9, 2.4 [2.2]
States, 2018		150	7	271	48	3		1.8, 1.3 [1.6]
Regency		151	7	281	49	5		0.79, 0.95 [0.87]
(R180040)						7		1.1, 1.2 [1.2]
						10		1.4, 1.3 [1.4]
King City, CA; United	3	154	-	383	46	0	With wrapper leaves	1.6, 0.96 [1.3]
States, 2018,		151	7	365	48-49			
Vandenberg (R180041)		152	7	383	49			
Hickman, CA; United	3	149	-	281	48	0	With wrapper leaves	0.35, 0.19 [0.27]
States, 2018		149	7	281	49			
Vandenberg (R180042)		149	7	281	49			
Yuba City, CA; United	3	150	-	281	45	0	With wrapper leaves	0.09, 0.15 [0.12]
States, 2018		149	8	281	49			
Great Lakes 659		149	6	281	49			
(R180043)								
LEAF LETTUCE								
Elmira, ON; Canada	3	155	-	206	42-43	0	Leaves	3.1, 2.9 [3.0]
2018, Summer Star		159	7	215	48-49			
(R180044)		152	8	206	49			
Richland, IA; United	3	149	-	262	46-47	0	Leaves	8.3, 4.4 [6.4]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Portion Analysed	Mefentrifluconazole (mg/kg)
States, 2018, Batavian Bergam's Green (R180045)		152 150	6 8	281 262	47-48 49			
Porterville, CA; United States, 2018, Green Star (R180046)	3	152 152 149	- 7 7	281 281 281	48 48 49	0	Leaves	4.4, 4.4 [4.4]
Porterville, CA; United States, 2018 Starfighter R180047	3	151 151 149	- 7 7	299 309 299	46 48 49	0	Leaves	4.3, 4.1 [4.2]
King City, CA; United States 2018 Starfighter (R180048)	3	151 148 154	- 6 7	281 281 281	45-46 47-48 49	0	Leaves	2.8, 2.0 [2.4]
Hickman, CA; United States 2018 Romaine (R180049)	3	149 149 149	- 7 7	281 281 281	48 49 49	0	Leaves	2.1, 2.4 [2.3]
Yuba City, CA; United States, 2018 Red Salad Bowl (R180050)	3	150 149 150	- 7 7	281 281 281	48 48 49	0	Leaves	7.0, 7.4 [7.2]
Arroyo Grande, CA; United States, 2018 Big Star (R180051)	3	152 151 151	- 7 7	281 281 281	41 45 48	0	Leaves	2.8, 2.5 [2.7]
SPINACH								
Puslinch, ON; Canada 2018, SV3580 (R180053)	3	148 155 147	- 5 8	196 206 196	17-18 19 19	0	Leaves	5.3, 5.0 [5.2]
Richlands, NC; United States, 2018 Bloomsdale Long Standing (R180052)	3	152 154 148	- 7 7	253 224 224	14-16 17-21 48-49	0	Leaves	13, 11 [12]
Richland, IA; United States, 2018 Hybrid Savoyed Spinach Emperor F1 (R180054)	3	151 151 152	- 6 7	262 281 290	18-19 19 45-47	0	Leaves	18, 16 [17]
Raymondville, TX; United States, 2018 Bloomsdale Long Standing (R180055)	3	150 155 152	- 6 7	281 290 281	16-18 17-19 38-39	0	Leaves	12, 12 [12]
Porterville, CA; United States, 2018 Bloomsdale (R180056)	3	151 151 150	- 7 7	281 271 281	47 48 49	0 3 5 6 9	Leaves	12, 10 [11] 8.5, 7.2 [7.9] 9.2, 9.8 [9.5] 7.9, 7.6 [7.8] 8.0, 6.0 [7.0]
King City, CA; United States, 2018 Shasta (R180057)	3	156 154 149	- 6 8	383 383 383	14-17 41-47 49	0	Leaves	3.7, 3.8 [3.8]
Hickman, CA; United	3	150	-	281	45	0	Leaves	4.6, 4.6 [4.6]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Portion Analysed	Mefentrifluconazole (mg/kg)
States, 2018 Bloomsdale (R180058)		150 151	8 6	281 281	47 49			
Arroyo Grande, CA; United States, 2018 Racoon (R180059)	3	154 151 154	- 6 7	281 281 281	43 47 49	0	Leaves	6.2, 3.5 [4.9]
RADISH LEAVES								
Alton, NY; United States, 2016 Crunchy Royal (R160193)	3	150 151 150	- 7 7	282 282 282	12 13-14 47	7	Leaves	2.9, 3.6 [3.3]
Sneads, FL; United States, 2016, Crunchy Royal (R160194)	3	149 150 149	- 7 7	217 218 216	44 46 48	7	Leaves	8.5, 7.4 [8.0]
Bradenton, FL; United States, 2016, Early Scarlot Globe (R160195)	3	141 149 148	- 7 7	341 358 354	Vegetati ve -	7	Leaves	5.0, 4.8 [4.9]
Manilla, IN; United States, 2016, Sparkle White Tip (R160196)	3	152 155 150	- 8 6	210 207 207	V3 Mature -	8	Leaves	3.2, 3.2 [3.2]
Lime Springs, IA; United States, 2016 Cherry Bell (R160197)	3	147 152 154	- 8 7	281 281 281	10 12 16	7	Leaves	1.1, 1.1 [1.1]
Fresno, CA; United States, 2016 Rudolf OG (R160198)	3	148 150 149	- 7 7	279 283 280	45 46 49	7	Leaves	4.9, 5.3 [5.1]
Deerfield, MI; United States, 2016 Celesta (R160199)	3	150 151 154	- 7 7	196 198 195	47 48 49	3 5 7 10 14	Leaves	0.39, 0.38 [0.39] 0.30, 0.35 [0.33] 0.37, 0.37 [0.37] 0.36, 0.38 [0.37] 0.26, 0.34 [0.30]
MUSTARD GREENS								
Puslinch, ON; Canada 2018, Savanna (R180062)	3	143 149 145	- 8 7	187 196 196	14-15 17-18 17-18	0	Leaves	3.9, 4.3 [4.1]
Richlands, NC; United States, 2018, Southern Giant Curled (R180060)	3	149 156 150	- 7 7	243 224 234	14-16 17-19 48-49	0	Leaves	12, 12, [12]
Weston, GA; United States, 2018, Florida Broadleaf (R180061)	3	149 150 150	- 7 7	215 215 215	44 46 48	0	Leaves	5.3, 4.7 [5.0]
Richland, IA; United States, 2018, Florida Broadleaf (R180063)	3	151 150 150	- 7 7	271 215 281	16-17 35 48-49	0	Leaves	7.9, 8.6 [8.3]

Legume vegetables

Beans with pods

Table 90 Residues of mefentrifluconazole in beans with pods in the United States following application of an EC formulation. In each trial, an adjuvant was added (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
North Rose, NY; 2014 Carson (R140712)	3	151 152 149	- 7 7	283 285 280	55 60 69	21	<0.01, <0.01 [<0.01]
Chula, GA; 2014 Caprice (R140713)	3	145 153 151	- 7 7	373 204 393	26 29 65	21	<0.01, <0.01 [<0.01]
Hobe Sound, FL; 2014 Buffalo Bean (140714)	3	148 150 152	- 7 7	388 354 344	15 18 61	21	<0.01, <0.01 [<0.01]
Somers, IA; 2014 Bush Blue Lake (R140715)	3	150 152 153	- 7 7	235 238 240	61 65 71	21	<0.01, <0.01 [<0.01]
Gardner, ND; 2014 Carson Bush Wax (R140716)	3	153 159 154	- 6 6	188 197 191	63 70 75	0 7 14 21 28	0.02, 0.02 [0.02] <0.01, 0.04 [0.03] 0.02, 0.02 [0.02] <0.01, 0.02 [0.02] 0.03, 0.02 [0.03]
Parkdale, OR; 2015 Blue Lake 274 Bean (R140717)	3	150 152 154	- 7 7	185 187 187	51 59 65	21	<0.01, <0.01 [<0.01]

Peas with pods

Table 91 Residues of mefentrifluconazole in peas with pods in Canada and the United States following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Outlook, SK; Canada 2015, Homesteader Peas (R140742)	3	154 152 149	- 7 7	103 102 99	39-60 60-65 69-73	21	0.02, 0.02 [0.02]
North Rose, NY; United States, 2014 Knight (R140736)	3	152 151 150	- 7 7	286 283 282	35 39 50	21	<0.01, <0.01 [<0.01]
Dana, IA; United States 2015, Oregon Sugar Pod II (R140737)	3	149 152 152	- 7 7	234 239 239	61 65 71	21	<0.01, <0.01 [<0.01]
Gardner, ND; United States 2014, Alaska Garden Pea (R140738)	3	154 156 149	- 6 6	190 195 186	63 67 73	21	0.02, 0.03 [0.03]
Delavan, WI; United States, 2015, Wando pea (R140739)	3	149 145 150	- 8 7	165 163 162	18 19 65	21	<0.01, <0.01 [<0.01]
Campbell, MN; United States, 2014 Knight (R140741)	3	152 152 152	- 7 6	188 188 188	13 14-15 34	21	<0.01, <0.01 [<0.01]
Ephrata, WA; United States, 2015, Naches Pea (R140743)	3	151 150 149	- 7 7	141 140 140	34 38 61	21	<0.01, <0.01 [<0.01]
Abbotsford, BC; Canada 2015, Mr. Big (R140744)	3	155 146 152	- 7 7	206 195 203	64 67 71	21	0.02, 0.03 [0.03]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Dana, IA; United States	3	149	-	185	69	0	0.98, 1.29 [1.14]
2015, Super Sugar Snap		150	7	187	71	7	0.41, 0.18 [0.30]
(R140895)		148	7	185	73	14	0.23, 0.07 [0.15]
						21	0.10, 0.05 [0.08]
						28	0.04, 0.06 [0.05]

Succulent beans without pods

Table 92 Residues of mefentrifluconazole in succulent beans without pods in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Abbeville, GA; 2015	3	150	-	383	61	21	<0.01, <0.01 [<0.01]
Jackson Wonder Lima Bean		150	7	391	65		
(R140706)		155	7	401	71		
Chula, GA; 2015	3	150	-	209	64	21	<0.01, <0.01 [<0.01]
Jackson Wonder Lima Bean		147	7	197	66		
(R140707)[A]		152	7	204	68		
	3	149	-	211	71	0	<0.01, <0.01 [<0.01]
Chula, GA; 2015		151	7	211	75	7	<0.01, <0.01 [<0.01]
Jackson Wonder Lima Bean		150	7	214	79	14	<0.01, <0.01 [<0.01]
(R140708)[A]						21	<0.01, <0.01 [<0.01]
						28	<0.01, <0.01 [<0.01]
Gardner, ND; 2015	3	148	-	139	69	21	0.02, 0.01 [0.02]
Fordhook		149	7	186	75		
242 Bush Bean (R140709)		152	7	190	77		
Chico, CA; 2015	3	149	-	187	76	21	<0.01, <0.01 [<0.01]
Roma (R140710)		148	7	187	77		
		147	7	187	78		
Ephrata, WA; 2015	3	149	-	140	71	21	<0.01, <0.01 [<0.01]
Kingston Green Baby Lima		151	7	141	73		
(R140711)		151	7	141	75		

Notes:

[A] Applications were made 77 days apart, rendering the trials independent.

Succulent peas without pods

Table 93 Residues of mefentrifluconazole in succulent peas without pods in Canada and the United States following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial ID)	No.	Total cumulative rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH)	DALA	Mefentrifluconazole (mg/kg)
Abbotsford, BC; Canada	3	155	-	206	64	21	<0.01, <0.01 [<0.01]
2015,Mr. Big		146	7	195	67		
(R140744)		152	7	203	71		
Outlook, SK; Canada	3	154	-	103	39-60	21	<0.01, <0.01 [<0.01]
2015		152	7	102	60-65		

Location; Year, Variety (Trial ID)	No.	Total cumulative rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH)	DALA	Mefentrifluconazole (mg/kg)
Homesteader Peas (R140742)		149	7	99	69-73		
North Rose, NY; United States, 2014 Knight (R140736)	3	152 151 150	- 7 7	286 283 282	35 39 50	21	<0.01, <0.01 [<0.01]
Dana, IA; United States 2015, Oregon Sugar Pod II (R140737)	3	149 152 152	- 7 7	234 239 239	61 65 71	21	<0.01, <0.01 [<0.01]
Gardner, ND; United States 2014, Alaska Garden Pea (R140738)	3	154 156 149	- 6 6	190 195 186	63 67 73	21	<0.01, <0.01 [<0.01]
Delavan, WI; United States 2015, Wando pea (R140739)	3	149 145 150	- 8 7	165 163 162	18 19 65	21	<0.01, <0.01 [<0.01]
Campbell, MN; United States, 2014 Knight(R140741)	3	152 152 152	- 7 6	188 188 188	13 14-15 34	21	<0.01, <0.01 [<0.01]
Ephrata, WA; United States 2015, Naches Pea (R140743)	3	151 150 149	- 7 7	141 140 140	34 38 61	21	<0.01, <0.01 [<0.01]
Dana, IA; United States 2015, Super Sugar Snap (R140895)	3	149 150 148	- 7 7	185 187 185	69 71 73	0 7 14 21 28	0.04, 0.07 [0.06] <0.01, 0.02 [0.02] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01] <0.01

Pulses

Dry beans

Table 94 Residues of mefentrifluconazole in dry beans in Canada and the United States following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Outlook, SK; Canada 2015, Windbreaks (R140723)	3	150 152 149	- 7 7	100 101 99	79-80 79-81 83-84	21	0.03, 0.07 [0.05]
North Rose, NY; United States, 2014, CA Red Kidney (R140718)	3	150 151 151	- 7 7	214 216 215	76 77 78	21	<0.01, <0.01 [<0.01]
Dana, IA; United States 2015, Great Northern (R140719)	3	150 151 151	- 7 7	233 235 234	75 77 78-79	21	0.02, <0.01 [0.02]
Delavan, WI; United States, 2015, Pinto Field Bean (R140720)	3	152 151 150	- 7 7	168 168 193	77 79 81	21	<0.01, <0.01 [<0.01]
Stafford, KS; United States 2015, Pinto III (R140721)	3	147 146 143	- 7 7	165 164 161	81 83 86	0 7 14	0.03, 0.03 [0.03] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
						21	<0.01, <0.01 [<0.01]
						28	<0.01, <0.01 [<0.01]
Carrington, ND; United States, 2015, Buster Pinto Bean (R140722)	3	152 150 150	- 7 7	161 189 186	79 82 85	21	<0.01, <0.01 [<0.01]
Hinton, OK; United States 2014, Taylor Pinto Bean (R140724)	3	149 151 160	- 7 7	176 151 200	60 69 69-71	21	<0.01, 0.02 [0.02]
Chico, CA; United States 2014, Red Kidney (R140725)	3	148 148 148	- 7 7	187 187 187	81 83 85	21	<0.01, <0.01 [<0.01]
Parkdale, OR; United States, 2015, Blue Lake 274 Bean (R140726)	3	154 147 157	- 7 7	238 222 189	75 77 78	21	<0.01, <0.01 [<0.01]
Brandon, MB; United States, 2015, Windbreaker Pinto Bean (R140727)	3	148 151 150	- 7 7	198 202 200	55 80 68	21	<0.01, <0.01 [<0.01]

Dry peas

Table 95 Residues of mefentrifluconazole in dry peas following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Fort Saskatchewan, AB; Canada, 2015, Meadow Peas (R140734)	3	151 151 152	- 6 7	151 151 152	60 65 67	21	0.02, 0.02 [0.02]
Wakaw, SK; Canada 2015 Treasure Peas (R140735)	3	146 152 153	- 7 7	98 101 102	61-63 67-69 69-723	21	0.01, 0.01 [0.01]
Jamestown, ND; United States, 2014 4010 Forage Pea (R140728)	3	150 154 156	- 6 7	178 193 195	68 69 75	21	0.02, <0.01 [0.02]
Carrington, ND; United States, 2015, Oregon Trail Peas (R140729)	3	149 150 150	- 7 6	186 186 187	71 74 76	22	0.01, 0.01 [0.01]
Gran Island. NE; United States, 2015, Austrian Winter peas (R140730)	3	150 150 151	- 7 7	192 187 196	74 77 81	21	0.02, 0.02 [0.02]
American Falls, ID; United States, 2014 954-Genie (R140731)	3	151 151 150	- 7 7	188 187 186	77 79 79-81	21	0.08, 0.10 [0.09]
American Falls, ID; United States, 2015 Banner Peas (R140732)	3	150 152 153	- 7 7	187 190 191	71 74-75 76-77	0 7 14 21 28	0.02, 0.02 [0.02] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01]
Parkdale, OR; United States 2015, Columbia (R140733)	3	150 151 152	- 7 7	177 186 188	61 75 79	21	0.01, 0.01 [0.01]

Soya beans, dry

Table 96 Residues of mefentrifluconazole in dry soya bean seeds following application of an EC formulation in the United States. In each trial, an adjuvant was added (Crawford, 2016, BASF DocID 2015_7005933)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Chula, GA; 2014 HBK 7028 (R140686)	2	150 154	- 7	160 165	87 88	21	<0.01, 0.01 [0.01]
Chula, GA; 2015 Asgrow AG 7231 (R140687)	2	152 153	- 7	210 207	84 85	21	0.10, 0.02 [0.06]
Washington, LA; 2014 P95 Y70 (R140688)[A]	2	151 153	- 6	142 168	73 75	21	0.05, 0.04 [0.05]
Opelousas, LA; 2014 P95 Y70 (R140689) [A]	2	150 150	- 6	142 165	73 75	21	0.31, 0.30 [0.31]
Morrow, LA; 2014 Terral Rev 56R63 (R140690)	2	147 151	- 7	126 131	91 95	21	0.03, 0.03 [0.03]
Campbell, MN; 2014 Asgrow AG 0634 (R140691)	2	150 151	- 7	187 188	79 79	21	<0.01, <0.01 [<0.01]
Erie, ND; 2014 11 R08 RR2Y 91221152 (R140692)	2	154 155	- 8	193 194	80 83	21	<0.01, <0.01 [<0.01]
Perley, MN; 2014 POST 24R PC35 (R140693)	2	149 156	- 7	186 195	82 85	21	<0.01, <0.01 [<0.01]
Gardner, ND; 2014 S02 M9 (R140694)	2	154 156	- 7	146 140	84 87	0 7 14 21 28	0.20, 0.22 [0.21] <0.01, <0.01 [<0.01] 0.01, <0.01 [0.01] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01]
Northwood, ND; 2015 NT0090RR (R140695)	2	152 150	- 7	190 187	81 81	21	<0.01, <0.01 [<0.01]
Jefferson, IA; 2014 92Y75 Pioneer (R140696)[B]	2	152 151	- 7	236 235	85 85	0 7 14 21 28	<0.01, 0.02 [0.02] 0.01, <0.01 [0.01] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01]
Paton, IA; 2014 92Y75 Pioneer (R140697)[B]	2	152 151	- 7	236 235	85 85	21	<0.01, <0.01 [<0.01]
Dana, IA; 2014 92Y75 Pioneer (R140698)[B]	2	151 150	- 7	234 234	85 85	21	<0.01, <0.01 [<0.01]
Delavan, WI; 2015 A1024341 (R140699)	2	152 155	- 7	190 198	80 81	21	<0.01, <0.01 [<0.01]
Fisk, MO; 2015 48E3RR (R140700)	2	151 148	- 7	189 185	81 85	21	<0.01, <0.01 [<0.01]
Bloomfield, MO; 2015 NK 584-P4 (R140701)	2	150 148	- 7	188 185	79 84	21	<0.01, <0.01 [<0.01]
McClure, IL; 2015 5N479R2 (R140702)	2	148 151	- 7	185 189	79 83	21	<0.01, <0.01 [<0.01]
Enid, OK; 2014 HBK RY 4620 (R140703)	2	156 150	- 7	168 126	77-79 77-79	21	<0.01, <0.01 [<0.01]
Ringwood, OK; 2014 Unknown (R140704)	2	150 148	- 7	314 212	77-79 79-81	21	<0.01, <0.01 [<0.01]
Stafford, KS; 2015 P31T11R (R140705)	2	147 151	- 7	166 169	81 84	21	0.01, 0.01 [0.01]

Notes:

[A] Applications were made on the same day, rendering the trials dependent

[B] Applications were made on the same day, rendering the trials dependent

Dry lentils

Table 97 Residues of mefentrifluconazole in lentils following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932; Bledsoe, 2020, BASF DocID 2019_2075411)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Outlook, Saskatchewan; Canada 2015 CDC Invincible (R140746)	3	147	-	98	82-83	0	0.97, 1.22 [1.10]
		151	7	101	85-86	7	0.91, 0.93 [0.92]
		150	7	100	87-88	14	1.43, 0.85 [1.14]
						21	0.70, 0.65 [0.68]
						28	0.48, 0.61 [0.55]
Wakaw, Saskatchewan; Canada, 2015 CDC Invincible (R140747)	3	148	-	296	77-79	21	0.13, 0.14 [0.14]
		150	7	299	79-80		
		149	7	298	80-83		
Jamestown, ND; United States, 2015 Unknown (R140894)	3	155	-	106	75	20	0.06, 0.06 [0.06]
		156	7	106	78		
		142	8	97	80		
American Falls, ID; United States, 2019 Green (R190038)	3	151	-	168	75	22	0.26, 0.34 [0.30]
		148	6	168	77		
		148	7	168	79		
Salem, ID; United States 2019, Green (R190039)[1]	3	151	-	168	75	21	0.65, 0.44 [0.55]
		148	6	168	77		
		148	6	168	79		
Ephrata, WA; United States 2019, Brewer (R190040)	3	151	-	281	77	21	0.041, 0.048 [0.045]
		151	7	281	81		
		151	7	281	85		

Notes:

[1] Sample weights were 0.23 kg instead of the required 1 kg

Root and tuber vegetables

Root Vegetables

Table 98 Residues of mefentrifluconazole in root vegetables from trials conducted in the United States following applications of an EC formulation (Falk, 2016, BASF DocID 2016_7010183; Webber, 2018, BASF DocID 2016_7010852)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
CARROT ROOTS							
Alton, NY; United States, 2016 Caracas (R160183)	3	150	-	281	45	7	0.24, 0.19 [0.22]
		150	7	282	46-47		
		150	7	282	48		
Bradenton, FL; United States, 2016, Danvers 126 (R160184)	3	158	-	375	Vegetative Growth	7	0.13, 0.16 [0.15]
		154	7	345			
		154	7	359			

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Lenexa, KS; United States, 2016	3	149	-	191	46	6	0.17, 0.14 [0.16]
Nantindo (R160185)		150	7	196	47		
		156	7	203	48		
Blissfield, MI; United States,	3	150	-	188	45	7	0.12, 0.12 [0.12]
2016		152	7	203	46		
Caracas (R160186)		149	7	200	48		
Lime Springs, CA; United States,	3	151	-	281	45	7	0.11, 0.085 [0.098]
2016		150	7	281	77		
Imperator (R160187)		151	7	281	90		
Orland, CA; United States, 2017,	3	149	-	187	73	7	<0.01, <0.01 [<0.01]
CA 25		151	7	188	78		
(R160188)		151	7	189	81		
Marysville, CA; United States,	3	152	-	284	46	7	<0.01, <0.01 [<0.01]
2016, Red Cored Chantenay		151	8	283	47		
(R160189)		150	6	282	48		
Fresno, CA; United States, 2016	3	149	-	281	43	7	0.12, 0.10 [0.11]
Romance F1 (R160190)		147	7	277	45		
		155	7	290	47		
Porterville, CA; United States,	3	152	-	281	46	7	0.081, 0.037 [0.059]
2016		154	7	287	47		
Danvers (R160191)		150	7	282	49		
Payette, ID; United States, 2016	3	155	-	287	47	7	0.053, 0.050 [0.052]
Nelson (R160192)		150	7	279	48		
		154	6	286	48-49		
Lebanon, OK; United States,	3	152	-	304	44	3	0.22, 0.24 [0.23]
2016		155	7	299	46	5	0.21, 0.19 [0.20]
Scarlet Nantes/Red Cored		154	7	309	48	7	0.23, 0.20 [0.22]
Chantenay						10	0.22, 0.22 [0.22]
(R150200)						14	0.24, 0.23 [0.24]
RADISH ROOTS							
Alton, NY; United States, 2016	3	150	-	282	12	7	0.030, 0.038 [0.034]
Crunchy Royal (R160193)		151	7	282	13-14		
		150	7	282	47		
Sneads, FL; United States, 2016	3	149	-	217	44	7	0.051, <0.01 [0.031]
Crunchy Royal (R160194)		150	7	218	46		
		149	7	216	48		
Bradenton, FL; United States,	3	141	-	341	Vegetative	7	0.14, 0.12 [0.13]
2016, Early Scarlot Globe		149	7	358	Growth		
(R160195)		148	7	354	-		
Manilla, IN; United States, 2016,	3	152	-	210	V3	8	0.098, 0.12 [0.109]
Sparkle White Tip		155	8	207	Mature		
(R160196)		150	6	207	-		
Lime Springs, IA; United States,	3	147	-	281	10	7	0.071, 0.093 [0.082]
2016		152	8	281	12		
Cherry Bell (R160197)		154	7	281	16		
Fresno, CA; United States, 2016,	3	148	-	279	45	7	0.36, 0.40 [0.38]
Rudolf OG		150	7	283	46		
(R160198)		149	7	280	49		
Deerfield, MI; United States,	3	150	-	196	47	3	<0.01, <0.01 [<0.01]
2016		151	7	198	48	5	<0.01, <0.01 [<0.01]
Celesta (R160199)		154	7	195	49	7	<0.01, <0.01 [<0.01]
						10	<0.01, <0.01 [<0.01]
						14	<0.01, <0.01 [<0.01]
SUGAR BEET ROOTS[1]							

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Taber, AB; Canada 2015, 47RR75 (R140368)[A]	2	152 155	- 6	202 207	49 49	21	0.03, 0.05 [0.04]
Taber, AB; Canada 2015, 9103RR (R140369)[A]	2	153 153	- 7	153 153	39 39	21	0.15, 0.15 [0.15]
Boissevain, MB; Canada 2015, 9102RR R14A.0374)	2	147 152	- 7	295 305	39 39	21	0.11, 0.13 [0.12]
Carlyle, IL; 2014 Unknown (R140362)	2	146 154	- 7	98 104	31 33	14	0.57, 0.58 [0.58]
						21	0.40, 0.40 [0.40]
						28	0.14, 0.37 [0.26]
Highland, IL; 2014 Unknown (R140363)	2	161 157	- 7	147 157	38 39	14	0.06, 0.06 [0.06]
						21	0.05, 0.06 [0.06]
						28	0.03, 0.05 [0.04]
Wyoming, IL; 2014 Unknown (R140364)	2	148 151	- 7	138 144	38 38	21	0.03, 0.02 [0.03]
York, NE; United States, 2014, 48607 TT (R140365)	2	154 149	- 7	125 128	39 39	21	0.02, 0.01 [0.02]
Aurora, SD; United States, 2014, 48607 TT (R140366)	2	157 150	- 7	156 150	37 39	21	0.04, 0.05 [0.05]
St Lawrence, SD; United States, 2014, 48607 TT (R140367)	2	149 151	- 6	146 146	37 39	21	0.09, 0.09 [0.09]
Levelland, TX; United States, 2014, Phoenix (R140370)	2	151 148	- 7	190 185	49 49	21	0.08, 0.07 [0.08]
Nipoma, CA; United States, 2014, 48607 TT (R140371)	2	149 150	- 7	286 287	39 39	21	0.32 ,0.23 [0.28]
Blaine County, ID; United States, 2014 SX1521WRR (R140372)	2	148 150	- 7	121 118	39 39	21	0.05, 0.06 [0.06]
Minidoka County, ID; United States, 2014 Beta 2028 (R140373)	2	147 155	- 7	121 121	39 39	21	0.04, 0.04 [0.04]

Notes:

[1] In all sugar beet trials an adjuvant was added to the spray mixture.

[A] Applications were separated by 14 days rendering the trials independent.

Potato

Table 99 Residues of mefentrifluconazole in potato tubers following application of an EC formulation. In each trial, an adjuvant was added. (Schreier. 2016, BASF DocID 2016_7006671)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Outlook, SK; Canada 2015, Wisconsin Norland (R150265)	3	151 150 154	- 6 8	303 301 306	79-81 79-81 81	6	<0.01, <0.01 [<0.01]
Carberry, MB; Canada 2015, Russet Burbank (R150266)	3	149 151 149	- 7 7	198 202 198	69 43 47	7	<0.01, <0.01 [<0.01]
Germansville, PA; United States, 2015, Dark Red Norland (R150042)	3	152 155 157	- 8 6	286 291 295	67-69 41-45 46	7	<0.01, <0.01 [<0.01]
Lyons, NY; United States 2015, Reba	3	150 151	- 7	282 282	47 47	0	<0.01, <0.01 [<0.01]
						3	<0.01, <0.01 [<0.01]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
(R150043)		153	7	285	48-49	6	<0.01, <0.01 [<0.01]
						10	<0.01, <0.01 [<0.01]
						14	<0.01, <0.01 [<0.01]
Frenchtown, NJ; United States,	3	158	-	295	45	7	<0.01, <0.01 [<0.01]
2015, Waneta		156	7	291	45-46		
(R150044)		154	7	288	46-47		
Waterloo, NY; United States	3	150	-	279	46	7	<0.01, <0.01 [<0.01]
2015		150	7	280	47		
Red Norland (R150045)		150	7	280	48		
Weedsport, NY; United States	3	151	-	281	47	7	<0.01, <0.01 [<0.01]
2015		151	7	281	48		
Yukon Gold (R150046)		151	7	281	48		
Preston, GA; United States	3	149	-	168	90	7	<0.01, <0.01 [<0.01]
2015		150	7	168	92		
Red Pntiac (R150047)		149	7	171	96		
Hastings, FL; YSA	3	149	-	167	92	7	<0.01, <0.01 [<0.01]
2015		149	7	170	94		
Elkton (R150048)		149	7	167	94		
Paynesville, MN; United States,	3	150	-	189	47	7	<0.01, <0.01 [<0.01]
2015		150	8	188	49		
Red Pontiac (R150049)		149	7	188	49		
Perry, IA; United States	3	149	-	206	43	7	<0.01, <0.01 [<0.01]
2015		147	7	326	73		
Kennebeck (R150050)		146	7	328	91		
Farlin, IA; United States	3	149	-	175	41	0	0.02, 0.03 [0.03]
2015		151	7	182	45	3	0.02, 0.01 [0.02]
Kennebeck (R150051)		158	7	209	48	7	0.02, 0.01 [0.02]
						10	0.01, 0.03 [0.02]
						14	0.03, 0.05 [0.04]
Richland, IA; United States	3	149	-	189	46	7	<0.01, <0.01 [<0.01]
2015		151	7	199	47		
Atlantic (R150052)		151	7	211	47-48		
Muscatine, IA; United States	3	151	-	195	47	7	<0.01, <0.01 [<0.01]
2015		147	8	190	48		
Atlantic (R150053)		156	6	171	48		
Monte Vista, CO; United States,	3	151	-	311	85	7	<0.01, <0.01 [<0.01]
2015		150	7	240	87		
Centennial Russet (R150055)		156	7	312	89		
Fresno, CA; United States	3	149	-	187	42	7	<0.01, <0.01 [<0.01]
2015		150	7	188	45		
Red La Soda (R150056)		149	7	186	47		
Aberdeen, ID; United States	3	164	-	182	46	Lost due to the inability to collect protocol specified samples	
2015		149	8	186	47-48		
Russet Burbank (R150057)		164	6	204	48-49		
American Falls; ID; United States,	3	151	-	169	46	6	<0.01, <0.01 [<0.01]
2015		148	8	184	47-48		
Russet Burbank (R150058)		151	7	190	48-49		
Tulelake, CA; United States	3	152	-	284	86	6	<0.01, <0.01 [<0.01]
2015, Standard Russet Norkotah		150	7	280	87		
(R150059)		151	7	282	88		
Klamath Falls, OG; United States,	3	154	-	286	86	7	<0.01, <0.01 [<0.01]
2015		150	7	280	87		
Yukon Gold (R150060)		151	7	283	88		

Cereal grains

Wheat – North America

Table 100 Residues of mefentrifluconazole in wheat grain and aspirated grain fractions from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Taber, Alberta; Canada, 2015 AC Carberry (R140300)	2	150 145	- 14	200 193	71-75 73-75	21	0.09, 0.09 [0.09]
Elgin, Manitoba; Canada 2015, Cardale (R140307)	2	150 150	- 14	301 299	69-71 83	21	0.08, 0.10 [0.09]
Hague, Saskatchewan; Canada 2015, AC Vespar (R140308)	2	152 150	- 14	152 150	79-83 87-92	21	0.13, .0.08 [0.11]
Kipp, Alberta; Canada, 2015 AC Carberry (R140309)	2	152 151	- 14	101 100	75-77 81-85	21	0.11, 0.12 [0.12]
Fort Saskatchewan, Alberta; Canada, 2015, Harvest (R140310)	2	152 152	- 14	203 202	75 87	21	0.08, 0.09 [0.09]
Alvena, Saskatchewan; Canada 2015, Cardale (R140311)	2	151 148	- 14	180 177	75-76 83-85	21	0.07, 0.08 [0.08]
Brandon, Manitoba; Canada, 2015 Brandon (R140312)	2	156 149	- 14	104 99	77 87	21	0.09, 0.10 [0.10]
Delisle, Saskatchewan; Canada 2015, Marchwell (R140296)	2	154 149	- 14	154 149	71-73 83-85	21	0.10, 0.11 [0.11]
Athens, GA; United States, 2015 GA Gore (R140288)	2	151 151	- 14	281 293	65-69 77-83	21	0.08, 0.11 [0.10]
Stuttgart, AR; United States, 2014 TV8848 (R140289)	2	150 152	- 14	149 141	45-53 69-71	21	0.02, 0.05 [0.04]
Gardner, ND; United States, 2015 Elgin (R140290)	2	151 152	- 14	189 190	70 85	21	0.12, 0.16 [0.14]
St. Cloud, MN; United States, 2014 Faller (R140291)	2	150 150	- 14	188 187	57 77	21	0.05, 0.06 [0.06]
Paynesville, MN; United States 2014, Oklee (R140292)	2	150 150	- 14	191 191	35 87-89	21	<0.01, <0.01 [<0.01]
Fisk, MO; United States, 2015 Roane (R140293)	2	148 148	- 14	187 186	61 75	21	0.03, 0.03 [0.03]
East Bernard, TX; United States 2015, LA841 (R140294)	2	151 148	- 13	346 330	77 89	21	0.36, 0.17 [0.27]
Grand Island, NE; United States 2014, Prosper (R140295)	2	149 149	- 13	184 168	61 77	21	0.02, 0.03 [0.03]
Jamestown, ND; United States 2015, Prosper (R140297)[A]	2	147 148	- 14	184 185	69 75	20	0.07, 0.07 [0.07]
Jamestown, ND; United States 2015, Divide (R140298)[A]	2	156 147	- 14	146 137	69 76-77	21	0.09, 0.06 [0.08]
Hastings, NE; United States 2014 Prosper (R140299)	2	151 151	- 14	221 222	71 87	0	0.41, 0.30 [0.36]
						14	0.16, 0.13 [0.15]
						21	0.11, 0.13 [0.12]
						28	0.13, 0.11 [0.12]
						35	0.08, 0.07 [0.08]
Wall, TX; United States, 2015 TAM 113 (R140301)	2	151 148	- 14	181 181	73 85	20	0.04, 0.03 [0.04]
Groom, TX; United States, 2015 TAM 111 (R140302)	2	151 151	- 14	282 283	73 79	21	0.02, 0.02 [0.02]
Claude, TX; United States, 2015 TAM 112 (R140303)	2	153 147	- 14	285 276	73 75	21	0.02, 0.03 [0.03]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Lamed, KS; United States, 2015 LCS Wizard (R140304)	2	150 147	- 14	159 155	85 85	21	0.14, 0.10 [0.12]
Aberdeen, ID; United States, 2014 Alturas (R140305)	2	151 145	- 14	143 138	75 85	21	0.09, 0.17 [0.13]

Notes:

[A] Applications were separated by 4 days; rendering the trials dependent.

Wheat – Europe

Table 101 Residues of mefentrifluconazole in wheat grain from trials conducted in Europe following application of EC or SC formulations (Erdmann, 2015, BASF DocID, 2014_1010809; Ale, 2015, BASF DocID, 2015_1099704/2017_1141927)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
BadenWuerttemberg/ Kraichgau; Germany, 2014, Asano (L130166)	2	153 156	- 12	204 208	49 69	43	<0.01
Brandenburg; Germany, 2014 Smaragd (L130167)	2	146 153	- 14	195 203	49 69	49	<0.01
Stetten a. H. (Kraichgau); Germany 2014, Asano (L140168)	2	152 146	- 18	202 194	49 69	51	0.011
	2	152 150	- 18	202 200	49 69	51	<0.01
	2	147 153	- 18	196 204	49 69	51	0.024
Uedem, Germany, 2014 Elixier (L140169)	2	160 155	- 21	213 207	49 69	42 49	<0.01 <0.01
	2	156 156	- 21	208 208	49 69	42 49	<0.01 <0.01
	2	148 156	- 21	197 208	49 69	42 49	<0.01 <0.01
Limburg, Gennep; The Netherlands 2014, Premio (L130168)	2	161 158	- 20	215 211	49 69	49	<0.01
Ottersum, Netherlands, 2014 Tabsco (L140171)	2	154 150	- 21	205 200	49 69	42 49	0.014 <0.01
	2	148 150	- 21	197 213	49 69	42 49	<0.01 <0.01
	2	152 146	- 21	203 195	49 69	42 49	0.013 <0.01
Essex: United Kingdom, 2014 Solstice (L130169)	2	152 136	- 31	203 181	49 69	35 42 50	0.017 0.015 0.024
Rouzières de Toraine, Northern France, 2014 Atogi (L140170)	2	152 152	- 36	203 203	49 69	42 49	0.012 0.016
	2	148 155	- 36	197 207	49 69	42 49	0.012 0.014
	2	148 152	- 36	198 203	49 69	42 49	0.014 0.012
Midi-Pyrénées; Southern France, 2014, Tiepolo (L130170)	2	146 158	- 21	195 211	49 69	46	<0.01
St. Soulan; Southern France, 2014 Aprilio (L140174)	2	152 149	- 17	203 198	49 69	49	<0.01
	2	150	-	200	49	49	<0.01

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
		148	17	197	69		
	2	148 150	- 17	197 200	49 69	49	<0.01
Agios Georgios; Greece, 2014 Trofeo (L140175)	2	150 151	- 20	201 202	49 69	50	<0.01
	2	136 153	- 20	182 204	49 69	50	<0.01
	2	150 151	- 20	200 202	49 69	50	<0.01
Central Macedonia Pella; Greece, 2014, Trofeo (L130171)	2	149 150	- 21	199 200	49 69	54	<0.01
Emilia Romagna, Bologna; Italy 2014, Palassio (L130172)	2	151 151	- 14	201 202	49 69	48	<0.01
S. Martino Olearo; Italy, 2014 Avorio (L140176)	2	156 157	- 10	208 209	49 69	41 48	<0.01 <0.01
	2	157 154	- 10	209 205	49 69	41 48	<0.01 <0.01
	2	155 157	- 10	207 210	49 69	41 48	0.020 <0.01
Andalusia, Sevilla; Spain, 2014 Athur Nick (L130173)	2	154 152	- 14	205 203	49 69	43 49	0.017 0.018
Quintanar del Rey; Spain, 2014 Adagio (L140173)	2	154 156	- 20	205 208	49 69	51	0.025
	2	153 153	- 20	204 204	49 69	51	0.026
	2	155 152	- 20	206 203	49 69	51	0.056
La Gineta; Spain 2014 Califa (L140177)	2	149 156	- 19	199 208	49 69	49	<0.01
	2	150 148	- 19	200 198	49 69	49	<0.01
	2	150 148	- 19	200 198	49 69	49	<0.01

Barley –North America

Table 102 Residues of mefentrifluconazole in barley grain from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Minto, Manitoba; Canada 2015, Newdale (R140242)	2	150 152	- 14	160 162	52-59 83-85	21	0.51, 0.45 [0.48]
Hague, Saskatchewan; Canada 2015, CDC Austenson (R140243)	2	152 151	- 14	152 151	77 85-87	21	0.65, 0.77 [0.71]
Fort Saskatchewan, Alberta; Canada, 2015, Coalition (R140244)	2	153 151	- 14	204 201	73 77	21	0.37, 0.31 [0.34]
Hepburn, Saskatchewan; Canada 2015, CDC Austenson (R140245)	2	145 144	- 14	193 192	83 85	21	1.95, 1.39 [1.67]
Carberry, Manitoba; Canada 2015, Conlon (R140246)	2	154 155	- 14	103 103	85 89	21	0.48, 0.64 [0.56]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
North Rose, NY; United States 2014, AC Minoa (R140237)	2	150 152	- 14	187 190	52 73	21	0.19, 0.21 [0.20]
Paynesville, MN; United States 2014, Robust (R140238)	2	152 151	- 14	191 190	85-87 87-89	0	0.22, 0.19 [0.21]
						14	<0.01, <0.01 [<0.01]
						21	<0.01, <0.01 [<0.01]
						28	<0.01, <0.01 [<0.01]
						35	<0.01, <0.01 [<0.01]
Grand Island, NE; United States 2014, Haybet (R140239)	2	150 150	- 14	194 173	55 81	21	0.38, 0.35 [0.37]
Fresno, CA; United States 2015, Helena barley (R140240)	2	149 152	- 14	187 190	87 89	21	0.84, 0.75 [0.80]
Aberdeen, ID; United States 2014, Baroness (R140241)	2	155 152	- 14	147 145	75 85	21	0.27, 0.23 [0.25]

Barley – Europe

Table 103 Residues of mefentrifluconazole in barley grain from trials conducted in Europe following applications of EC or SC formulations. (Teresiak, 2014, BASF DocID, 2014_1010808; Ale, 2015, BASF DocID 2015_1099703/2017_1101701)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Rhineland Palatinate, Rheinhessen; Germany, 2013, Popino (L130174)	2	153 141	- 16	204 188	49 69	28	0.14
						35	0.15
						41	0.13
Brandenburg; Germany, 2013 Sandra (L130175)	2	146 146	- 14	195 195	49 69	53	0.014
Mauchenheim; Germany, 2014 Propino (L140158)	2	153 150	- 19	204 200	49 69	35	0.057
						43	0.048
	2	150 153	- 19	200 204	49 69	35	0.087
						43	0.061
	2	153 154	- 19	204 206	49 69	35	0.12
						43	0.083
Uedem; Germany, 2014 Meridian (L140159)	2	144 155	- 29	192 207	49 69	36	0.10
						41	0.077
	2	155 151	- 29	207 202	49 69	36	0.085
						41	0.071
	2	154 145	- 29	205 193	49 69	36	0.057
						41	0.039
Limburg, Gennep; The Netherlands 2013, Sequel (L130176)	2	149 163	- 27	199 217	49 69	34	0.13
						41	0.19
Ottersum; The Netherlands, 2014 Sequel (140160)	2	151 144	- 28	202 192	49 69	35	0.15
						41	0.10
	2	155 154	- 28	207 205	49 69	35	0.11
						41	0.10
	2	154 145	- 28	205 193	49 69	35	0.095
						41	0.087
Essex: United Kingdom, 2013 Cassata (L130177)	2	152 147	- 23	203 196	49 69	35	0.056
						41	0.071
Ugley Green; United Kingdom 2014 Flagon	2	151 151	- 24	202 201	49 69	35	0.22
						42	0.26
	2	150	-	200	49	35	0.28

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
(L140161)		158	24	211	69	42	0.25
	2	152	-	201	49	35	0.24
		148	24	198	69	42	0.21
Saint Pierre de Chevillé; Northern France 2014 Sandra (L140162)	2	142	-	190	49	41	0.050
		145	21	193	69		
	2	141	-	188	49	41	0.060
		140	21	187	69		
	2	142	-	190	49	41	0.034
		148	21	197	69		
Midi-Pyrénées; Southern France, 2013 Bamboo (L130178)	2	149	-	198	49	55	0.070
		140	23	187	69		
Tournecoupe; Southern France, 2014 Ketos (L140063)	2	162	-	217	49	42	0.10
		150	16	200	69		
	2	145	-	193	49		0.088
		152	16	203	69		
	2	145	-	193	49		0.11
		155	16	207	69		
Central Macedonia Pella; Greece, 2013 Moutso (L130179)	2	150	-	200	49	54	0.16
		150	11	201	69		
Prochoma; Greece, 2014 Chill (L140164)	2	151	-	201	49	43	0.030
		151	20	202	69		
	2	150	-	200	49		0.018
		152	20	202	69		
	2	151	-	201	49		0.026
		150	20	201	69		
Cuneo; Italy, 2013 Cometa (L130180)	2	147	-	196	49	48	0.10
		158	13	210	69		
Cassano D´Adda; Italy, 2014 Atomo (L140165)	2	153	-	204	49	34	0.10
		155	22	207	69	41	0.14
	2	148	-	198	49	34	0.12
		157	22	209	69	41	0.14
	2	146	-	195	49	34	0.19
		146	22	195	69	41	0.20
Andalusia, Sevilla; Spain, 2013 Prestige (1L130181)	2	150	-	200	49	29	0.42
		153	14	204	69	36	0.29
						42	0.41
Quintanar del Rey; Spain, 2014 Acapulco (L140166)	2	154	-	205	49	42	0.29
		150	20	200	69		
	2	146	-	195	49		0.059
		154	20	205	69		
	2	150	-	200	49		0.22
		152	20	203	69		
La Gineta; Spain, 2014 Hispanic (L140167)	2	142	-	190	49	48	0.029
		150	24	200	69		
	2	146	-	195	49		0.033
		151	24	201	69		
	2	146	-	195	49		0.020
		148	24	198	69		

Rice –United States

Table 104 Residues of mefentrifluconazole in paddy rice gain from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF

DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Stuttgart, AR; 2014 CL 152 (R140267)[A]_	2	148 146	- 14	153 151	58 85	21	1.63, 1.68 [1.66]
Stuttgart, AR; 2014 XL 745 Hybrid (R140268)[A]	2	150 145	- 14	154 150	58 86	23	1.13, 1.10 [1.12]
Cheneyville, LA; 2014 Cheniere (R140269)	2	148 159	- 14	226 242	86 87	21	1.86, 1.81 [1.83]
Glennonville, MO; 2014 CL 111 (R140270)	2	149 149	- 14	187 187	77 85	21	1.69, 1.64 [1.67]
Pollard, AR; 2014 XL 729 (R140271)	2	152 149	- 14	189 186	45 69	21	0.88, 0.81 [0.85]
Pocahontas, AR; CL XL745 (R140272)	2	150 149	- 14	188 186	43 65	21	0.38, 0.37 [0.38]
Fisk, MO; 2014 CL XL745 (R140273)[B]	2	150 149	- 14	187 187	45 65	21	0.63, 0.63 [0.63]
Fisk, MO; 2014 Francis (R140274)[B]	2	150 149	- 14	187 186	77 85	21	1.68, 1.80 [1.74]
East Bernard, TX; 2014 Presidio (R140275)	2	150 150	- 14	159 159	65 75	0	11.27. 11.33 [11.30]
						14	0.45, 0.48 [0.47]
						21	0.23, 0.31 [0.27]
						28	0.41, 0.44 [0.43]
						35	0.33, 0.27 [0.30]
Markham, TX; 2014 LX745 (R140276)	2	150 151	- 14	151 156	53 81	21	1.17, 1.14 [1.16]
Willows, CA; 2014 M205 (R140277)	2	148 148	- 14	187 187	61 85	21	<0.01, <0.01 [<0.01]
Maxwell, CA; 2014 M205 (R140278)	2	149 149	- 14	187 187	61 85	21	<0.01, <0.01 [<0.01]

Notes:

[A] Applications were made on the same day, rendering the trials dependent.

[B] Applications were separated by 37 days; rendering the trials independent.

Rice – China

Table 105 Residues of mefentrifluconazole in paddy rice and husked rice grain from trials conducted in China following application of an SC formulation. (Xiaohu, 2019, BASF DocID. 2020_2095671)

Location; Year, Variety	No.	Nominal Rate (g ai/ha)	Nominal RTI (days)	Nominal Spray Volume (L/ha)	BBCH	DALA	Portion analysed	Mefentrifluconazole (mg/kg)
Changchun City, Jilin Province; 2018 Jiudao 86	2	120 120	- 5	500	75	21	Paddy rice	1.3, 1.3 [1.3]
							Husked rice	0.80, 0.77 [0.79]
					77	28	Paddy rice	1.3, 0.48 [0.89]
							Husked rice	0.66, 0.13 [0.39]
Gaoyue Town, Huaibei City, Anhui Province; 2018 Xuyou 733	2	120 120	- 5	500	NS	7	Paddy rice	1.6, 1.3 [1.5]
							Husked rice	0.13, 0.29 [0.21]
					NS	14	Paddy rice	1.5, 1.2 [1.4]
							Husked rice	0.20, 0.14 [0.17]
					75	21	Paddy rice	1.0, 1.1 [1.1]
							Husked rice	0.069, 0.073 [0.071]
					77	28	Paddy rice	0.76, 0.76 [0.76]

Mefentrifluconazole

Location; Year, Variety	No.	Nominal Rate (g ai/ha)	Nominal RTI (days)	Nominal Spray Volume (L/ha)	BBCH	DALA	Portion analysed	Mefentrifluconazole (mg/kg)
							Husked rice	0.083, 0.086, [0.085]
					NS	35	Paddy rice	0.87, 0.73 [0.80]
							Husked rice	0.11, 0.11 [0.11]
Eshan Town, Fanchang County, Anhui Province; 2018, Suxiu 867	2	120 120	- 5	500	75	21	Paddy rice	1.0, 1.1 [1.0]
							Husked rice	0.071, 0.075 [0.073]
					77	28	Paddy rice	0.74, 0.72 [0.73]
							Husked rice	0.088, 0.088 [0.088]
Fotang Town, Yiwu City, Zhejiang; 2018, Liangyou 189	2	120 120	- 5	500	75	21	Paddy rice	1.4, 1.3 [1.4]
							Husked rice	0.086, 0.065[0.075]
					77	28	Paddy rice	0.98, 0.79 [0.89]
							Husked rice	0.078, 0.067 [0.073]
Huibu Town, Gao'an City, Jiangxi Province; 2018, Wanxiangyou 337	2	120 120	- 5	500	75	21	Paddy rice	1.4, 2.3 [1.8]
							Husked rice	0.18, 0.31 [0.24]
					77	28	Paddy rice	1.5, 1.5 [1.5]
							Husked rice	0.18, 0.15 [0.17]
Shizishan Street, Hongshan District, Wuhan, Hubei; 2018, Ejing 912	2	120 120	- 5	500	75	21	Paddy rice	2.4. 2.4 [2.4]
							Husked rice	0.47, 0.46 [0.47]
					77	28	Paddy rice	2.5. 2.4 [2.5]
							Husked rice	0.48, 0.46 [0.47]
Pingjiang Town, Yueyang City, Hunan; 2018 C Liangyou 4488	2	120 120	- 5	500	75	21	Paddy rice	2.0, 2.1 [2.1]
							Husked rice	0.32, 0.20 [0.26]
					77	28	Paddy rice	2.5, 2.4 [2.5]
							Husked rice	0.49, 0.50 [0.50]
Qidong County, Hengyang City, Hunan; 2018 C Liangyou 4488	2	120 120	- 5	500	NS	7	Paddy rice	2.5, 1.9 [2.2]
							Husked rice	0.36, 0.12 [0.24]
					NS	14	Paddy rice	1.9, 1.8 [1.9]
							Husked rice	0.21, 0.15 [0.18]
					75	21	Paddy rice	2.2, 2.3 [2.3]
							Husked rice	0.18. 0.19 [0.19]
					77	28	Paddy rice	1.2, 1.2 [1.2]
							Husked rice	0.15, 0.11 [0.13]
					NS	35	Paddy rice	1.2, 1.2 [1.2]
							Husked rice	0.093, 0.18 [0.14]
Changshun County, Guizhou Province; 2018 Yixiang 2115	2	120 120	- 5	500	NS	7	Paddy rice	0.89, 1.1 [1.0]
							Husked rice	0.14, 0.12 [0.13]
					NS	14	Paddy rice	0.82, 0.84 [0.83]
							Husked rice	0.16, 0.13 [0.15]
					75	21	Paddy rice	0.67, 0.63 [0.65]
							Husked rice	0.11, 0.088 [0.10]
					77	28	Paddy rice	0.34, 0.32 [0.33]
							Husked rice	0.061, 0.064 [0.063]
					NS	35	Paddy rice	0.51, 0.51 [0.51]
							Husked rice	0.074, 0.081 [0.078]
Xixiangtang District, Nanning City, Guangxi Zhuang; 2018, Y Liangyou No. 2	2	120 120	- 5	500	75	21	Paddy rice	0.65, 0.64 [0.65]
							Husked rice	0.11, 0.10 [0.10]
					77	28	Paddy rice	0.35, 0.32 [0.34]
							Husked rice	0.071, 0.072 [0.071]
Haitou Town, Xiashan District, Zhanjiang City, Guangdong; 2018 Jinzao 09	2	120 120	- 5	500	NS	7	Paddy rice	0.77, 0.76 [0.77]
							Husked rice	0.13, 0.14 [0.14]
					NS	14	Paddy rice	1.4, 0.73 [1.1]
							Husked rice	0.15, 0.11 [0.13]
					75	21	Paddy rice	0.75, 0.75 [0.75]

Location; Year, Variety	No.	Nominal Rate (g ai/ha)	Nominal RTI (days)	Nominal Spray Volume (L/ha)	BBCH	DALA	Portion analysed	Mefentrifluconazole (mg/kg)
							Husked rice	0.10, 0.13 [0.11]
					77	28	Paddy rice	0.63, 0.63 [0.63]
							Husked rice	0.087, 0.12 [0.10]
					NS	35	Paddy rice	0.48, 0.45 [0.47]
							Husked rice	0.098, 0.094 [0.096]
Yongfa Town, Chengmai County, Hainan Province; 2018, Boyou 125	2	120 120	- 5	500	75	21	Paddy rice	0.017, 0.041 [0.029]
							Husked rice	<0.01, <0.01 [<0.01]
					77	28	Paddy rice	0.015, 0.014 [0.015]
							Husked rice	<0.01, <0.01 [<0.01]

Rice – Brazil

Table 106 Residues of mefentrifluconazole in rice grain from trials conducted in Brazil following application of an SC formulation. In each trial, an adjuvant was added. (Castro, 2017, BASF DocID 2017_3001322)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Portion analysed	Mefentrifluconazole (mg/kg)
Ibiporã/Paraná; 2017 Paddy Rice - IRGA 428 (G150296)	2	153 153	- 15	100 100	45 57	35	Grain with hulls	<0.01
						45		<0.01
						55		<0.01
						65		<0.01
						35	Grain without hulls	<0.01
						45		<0.01
						55		<0.01
						65		<0.01
						35	Polished rice	<0.01
						45		<0.01
						55		<0.01
						65		<0.01
Bandeirantes/ Paraná; 2017 Upland Rice - IAC 58 (G150297)	2	153 153	- 15	100 100	71 75	35	Grain with hulls	0.034, 0.034 [0.034]
						45		0.052, 0.052 [0.052]
						55		<0.01
						65		<0.01
						35	Grain without hulls	<0.01
						45		<0.01
						55		<0.01
						65		<0.01
						35	Polished rice	<0.01
						45		<0.01
						55		<0.01
						65		<0.01
Londrina/Paraná; 2017 Upland Rice - IPR 117 (G150298)	2	153 153	- 15	100 100	61 65	35	Grain with hulls	0.041
						45		0.039
						55		<0.01
						65		<0.01
						35	Grain without hulls	<0.01
						45		<0.01
						55		<0.01
						65		<0.01

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Portion analysed	Mefentrifluconazole (mg/kg)
						35	Polished rice	<0.01
						45		<0.01
						55		<0.01
						65		<0.01
Santa Cruz do Sul/Rio Grande do Sul; 2017 Paddy Rice - Puitá INTA CL (G150299)	2	155 155	- 15	100 100	47 51	35	Grain with hulls	0.043, 0.44, 0.043 [0.043]
						45		0.084, 0.072, 0.074 [0.077]
						55		0.057, 0.053, 0.053 [0.054]
						65		0.039, 0.041, 0.033 [0.038]
						35	Grain without hulls	<0.01
						45		<0.01
						55		<0.01
						65		<0.01
						35	Polished rice	<0.01
						45		<0.01
						55		<0.01
						65		<0.01
Candelária/ Rio Grande do Sul; 2017 Paddy Rice - Puitá INTA CL (G150300)	2	154 153	- 15	100 100	47 49	35	Grain with hulls	0.055, 0.051, 0.050 [0.052]
						45		0.088, 0.082, 0.082 [0.084]
						55		0.054, 0.050, 0.051 [0.052]
						65		<0.01, <0.01, <0.01 [<0.01]
						35	Grain without hulls	<0.01
						45		<0.01
						55		<0.01
						65		<0.01
						35	Polished rice	<0.01
						45		<0.01
						55		<0.01
						65		<0.01

Sorghum

Table 107 Residues of mefentrifluconazole in sorghum grain from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Pollard, AR; 2014 53-67 (R140279)	2	149 149	- 14	186 187	85 87	22	0.49, 0.34 [0.42]
Paynesville, MN; 2014 L655 (R140280)	2	150 150	- 14	190 191	85 87	21	<0.01, <0.01 [<0.01]
Richland, IA; 2014 85Y40 (R140281)	2	152 151	- 14	130 228	85-87 87	21	0.46, 0.36 [0.41]
Fisk, MO; 2014 M 3838C (R140282)	2	149 151	- 14	186 188	83-85 87	21	0.27, 0.34 [0.31]
Hinton, OK; 2014 DKS29-28 (R140283)	2	151 151	- 14	179 204	75 85	21	0.76, 0.79 [0.78]
Raymondville, TX; 2014 DKS 51-01 (R140284)	2	155 156	- 14	191 193	55 75-80	21	0.22, 0.26 [0.24]
Grand Island, NE; 2014 A1005964 (R140285)	2	151 150	- 14	187 171	85 85	21	0.56, 0.48 [0.52]
Levelland, TX; 2014	2	151	-	189	56	21	0.18, .018 [0.18]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
DKS44-20 (R140286)		145	13	182	81		
Groom, TX; 2014	2	154	-	214	87	0	1.03, 1.00 [1.02]
H-390W (R140287)		148	14	209	89	14	1.16, 1.25 [1.21]
						21	1.03, 1.09 [1.06]
						28	1.22, 1.11 [1.17]
						35	1.18, 1.13 [1.16]

Maize

Table 108 Residues of mefentrifluconazole in maize grain from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Alton, NY; 2014	2	152	-	285	71	21	<0.01, <0.01 [<0.01]
232180 (R140247)		150	14	281	73		
Hawkinsville, Georgia; 2014	2	154	-	288	72-74	21	<0.01, <0.01 [<0.01]
Dekalb (R140248)		151	14	292	83-85		
Delavan, WI; 2014	2	150	-	158	85 R5	21	<0.01, <0.01 [<0.01]
DKC 49-94RIB (R140249)		147	14	157	86 R5		
Gardner, ND; 2014	2	149	-	139	76	21	<0.01, <0.01 [<0.01]
DKC33-53RIBAF2 (R140250)		148	15	139	78		
Erie, ND; 2014	2	152	-	142	85	21	<0.01, <0.01 [<0.01]
2Y188 (R140251)		150	14	140	87		
Oregon, WI; 2014	2	152	-	216	85	21	<0.01, <0.01 [<0.01]
DKC 49-29RIB (R140252)[A]		148	13	224	87		
Oregon, WI; 2014	2	148	-	210	85-87	21	<0.01, <0.01 [<0.01]
G96A69-3111 (R140253)[A]		147	13	222	87		
Stafford, KS; 2014	2	145	-	198	83	20	<0.01, <0.01 [<0.01]
Pioneer P1105AM (R140254)		153	14	171	84		
St. Cloud, MN; 2014	2	151	-	189	87	21	<0.01, <0.01 [<0.01]
DKC 38-03RIB (R140255)		153	14	191	87		
York, NE; 2014	2	150	-	190	69	21	<0.01, <0.01 [<0.01]
PO876CHR (R140256)[B]		150	14	190	89		
Paynesville, MN; 2014	2	151	-	188	69	0	<0.01, <0.01 [<0.01]
DK 1431		151	14	189	89	14	<0.01, <0.01 [<0.01]
(R140257)[C]						21	<0.01, <0.01 [<0.01]
						28	<0.01, <0.01 [<0.01]
						35	<0.01, <0.01 [<0.01]
Paynesville, MN; 2014	2	151	-	188	85	21	<0.01, <0.01 [<0.01]
DK 1431 (R140258)[C]		152	14	189	87		
Geneva, MN; 2014	2	149	-	159	83-85	21	<0.01, <0.01 [<0.01]
Pioneer 9834 (R140259)		151	14	174	85-87		
Richland, IA; 2014	2	150	-	325	83-85	21	<0.01, <0.01 [<0.01]
Pioneer P1498AM (R140260)		153	14	349	85-87		
Hendrick, IA; 2014	2	150	-	324	83-85	21	<0.01, <0.01 [<0.01]
Pioneer P1360HR (R140261)		152	14	169	85-87		
Kirkville, MO; 2014	2	149	-	182	83	21	<0.01, <0.01 [<0.01]
P1498AM (R140262)		150	14	184	85-87		
Fisk, MO; 2014	2	149	-	186	85	22	<0.01, <0.01 [<0.01]
RL8899YH B (R140263)		149	14	187	85		
Aquilla, MO; 2014	2	150	-	189	85	21	<0.01, <0.01 [<0.01]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
DeKalb DKC63-87 (R140264)		151	14	187	85		
York, NE; 2014	2	149	-	218	87	19	<0.01, <0.01 [<0.01]
DK 59-90 RIB (R140265)[B]		150	14	220	87		
East Bernard, TX; 2014	2	148	-	144	83	21	<0.01, <0.01 [<0.01]
P1395AM (R140266)		151	14	147	84		

Notes:

[A] Applications were made on the same day, rendering the trials dependent.

[B] Applications were separated by 2 days, rendering the trials dependent.

[C] Applications were separated by 1 day, rendering the trials dependent.

Sweet corn

Table 109 Residues of mefentrifluconazole in sweet corn kernels plus cob with husks removed from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID 2015_7005929)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Abbotsford, British Columbia;	3	164	-	431	69	21	<0.01, 0.02 [0.02]
Canada, 2014		146	6	385	71		
Honey and cream (R140577)		169	8	446	73		
Taber, Alberta; Canada	3	150	-	200	65	21	<0.01, <0.01 [<0.01]
2015		145	7	194	65		
148-4 (R140574)		155	6	207	65-67		
North Rose, NY; United States	3	154	-	308	38	21	<0.01, <0.01 [<0.01]
2014, BC 0805 (R140565)		155	6	305	61		
		150	8	300	65		
Alton, NY; United States	3	150	-	281	51	21	<0.01, <0.01 [<0.01]
2014		150	7	281	55		
Previous Gem (R140566)		150	7	280	63		
Chula, GA; United States	3	147	-	271	59	21	<0.01, <0.01 [<0.01]
2014		154	7	280	67		
Passion II (R140567)		147	7	275	73		
Newberry, FL; United States	3	153	-	195	59	21	<0.01, <0.01 [<0.01]
2014		154	7	188	63		
Passion II (R140568)		151	7	197	69		
Delavan, WI; United States	3	150	-	145	59	21	<0.01, <0.01 [<0.01]
2014		150	7	147	59		
NK 199 (R140569)		149	7	149	61		
Fitchburg, WI; United States	3	152	-	238	39-59	21	<0.01, <0.01 [<0.01]
2014		150	7	213	61		
Overland (R140570)		153	6	180	71		
St. Cloud, MN; United States	3	151	-	188	51	21	<0.01, <0.01 [<0.01]
2014		151	7	189	65		
Ambrosia (R140571)		150	7	188	71		
Paynesville, MN; United States	3	150	-	219	55-63	0	<0.01, <0.01 [<0.01]
2014		152	7	223	65-69	14	<0.01, <0.01 [<0.01]
Ambrosia (R140572)		150	7	225	69-73	21	<0.01, <0.01 [<0.01]
						28	<0.01, <0.01 [<0.01]
						35	<0.01, <0.01 [<0.01]
York, NE; United States	3	150	-	188	59	21	<0.01, 0.02 [0.02]
2014		151	7	188	65		
276A (R140573)		151	7	188	67		

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Fresno, CA; United States 2014 Silver Queen (R140575)	3	150 149 149	- 7 7	188 187 187	65 71 73	21	<0.01, 0.02 [0.02]
Aberdeen, ID; United States 2014 Ambrosia (R140576)	3	150 145 154	- 7 7	144 140 148	37 37 61	21	<0.01, 0.02 [0.02]

Sugar Cane

Table 110 Residues of mefentrifluconazole in sugarcane cane from trials conducted in the United States and Brazil following applications of an EC formulation (Bledsoe, 2019, BASF DocID 2019_7000661; Reis, 2016, BASF DocID 2017_3004001)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Jaboticabal, SP; Brazil 2016 RB 86-7515 (G150217)	3	149 158 155	- 30 30	250 265 260	39 42 45	15 30 45 60	0.019, 0.022 [0.021] 0.024, 0.026 [0.025] 0.019, 0.019 [0.019] 0.057, 0.051 [0.054]
Conchal, SP; Brazil 2014 SP 81-3250 (G150218)	3	143 146 153	- 30 30	600 610 640	39 39 39	15 30 45 60	0.019 0.016 0.022 0.027
Ribeirão Preto, SP; Brazil 2016 SP 89-1115 (G150219)	3	141 139 158	- 30 30	295 290 330	39 42 45	15 30 45 60	0.15, 0.13, 0.17 [0.15] 0.13, 0.14, 0.14 [0.14] 0.058, 0.052, 0.051 [0.054] <0.01
Engenheiro Coelho, SP; Brazil, 2016 RB 5156 G150220	3	141.0 141.0 148.1	- 30 31	0.590 0.590 0.620	39 39 39	15 30 45 60	0.060, 0.056 [0.058] 0.079, 0.074 [0.077] 0.037, 0.038 [0.038] 0.038, 0.039 [0.039]
Palmeira, PR; Brazil 2016 IAC SP 95-5000 G150221	3	150.2 149.1 155.8	- 30 30	0.262 0.312 0.326	39 43 47	15 30 45 60	0.014 0.010 <0.01 <0.01
Oviedo, FL; United States 2017, 2143 (R170038)	2	151 151	- 14	274 283	49 49	14	0.37, 0.38 [0.38]
Belle Glade, FL; United States, 2018 CPCL 00-4111 (R170039)	2	155 153	- 12	289 282	49 49	14	1.67, 0.27 [0.97]
Moore Haven, FL; United States, 2017 CP 961252 (R170040)	2	152 146	- 14	282 277	47 49	14	0.42, 0.41 [0.42]
Cheneyville, LA; United States, 2017 HoCP 96-540 (R170041)	2	157 161	- 13	200 203	14-15 nodes 14-15 nodes	14	0.33, 0.39 [0.36]
Morrow, LA; United States 2017, L01-299 (R170042)	2	157 168	- 16	194 197	8-9 nodes ~10-12 nodes	13	0.13, 0.82 [0.48]
Washington, LA; United States, 2017 540 (R170045)	2	155 151	- 14	276 280	Not recorded Not recorded	4 9 14	0.13, 1.05 [0.59] 0.25, 0.40 [0.33] 0.24, 0.37 [0.31]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
						19	0.059, 0.092 [0.076]
						24	0.10, 0.12 [0.11]
Raymondville, TX; United States, 2018 3388 (R170044)	2	153 148	- 14	281 281	38-39 38-39	14	0.25, 0.25 [0.25]
Waipahu, HI; United States 2018, 7052 (R170043)	2	149 153	- 13	235 238	85 85	14	0.054, 0.14 [0.097]

Tree Nuts

Table 111 Residues of mefentrifluconazole in nutmeats of tree nuts (pecans, pistachios, almonds) from trials conducted in the United States following application of an SC formulation. In each trial, an adjuvant was added. (Watt, 2016, BASF DocID 2015_7001273)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
PECANS							
Tifton, GA; 2014 Summer (R140578)	3	158 148 149	- 7 7	692 617 673	83 60 % shuck split 30 % nuts ripe for picking	13	<0.01, <0.01 [<0.01]
		151 149 151	- 7 7	1356 1225 1328	83 60 % shuck split 30 % nuts ripe for picking	13	<0.01, <0.01 [<0.01]
Chula; GA; 2014 Summer (R140579)	3	149 150 148	- 8 7	683 655 673	83 75 % shuck split 90 % shuck split	0	<0.01, <0.01 [<0.01]
						3	<0.01, <0.01 [<0.01]
						7	<0.01, <0.01 [<0.01]
						15	<0.01, <0.01 [<0.01]
						21	<0.01, <0.01 [<0.01]
		149 149 149	- 8 7	1328 1253 1309	83 75 % shuck split 90 % shuck split	0	<0.01, <0.01 [<0.01]
						3	<0.01, <0.01 [<0.01]
						7	<0.01, <0.01 [<0.01]
						15	<0.01, <0.01 [<0.01]
						21	<0.01, <0.01 [<0.01]
Port Barre, LA; 2014 Caddo (R140580)	3	152 152 151	- 7 7	926 1019 935	shuck split advanced shuck split 89	14	<0.01, <0.01 [<0.01]
		154 148 147	- 7 7	1459 1515 1525	shuck split advanced shuck split 89	14	<0.01, <0.01 [<0.01]
Stillwater, OK; 2014 Merramec (R140581)	3	145 150 151	- 7 7	599 711 692	81 85 85-89	13	<0.01, <0.01 [<0.01]
		148 149 150	- 7 7	1814 1861 1955	81 85 85-89	13	<0.01, <0.01 [<0.01]
Dill City, OK; 2014 Kanza R140582)	3	152 147 150	- 7 8	748 739 776	79-80 80-83 80-83	15	<0.01, <0.01 [<0.01]
	3	152 150 151	- 7 8	1487 1506 1525	79-80 80-83 80-83	15	<0.01, <0.01 [<0.01]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
PISTACHIOS							
Orland, CA; 2014 Kerman (R140583)	3	149 148 149	- 7 7	701 701 701	81 83 85	14	0.061, 0.026 [0.044]
	3	148 148 148	- 7 7	1394 1403 1394	81 83 85	14	<0.01, 0.011 [0.011]
Richgrove, CA; 2014 Pioneer (R140584)	3	151 150 151	- 7 7	655 655 645	79 79 81	0	<0.01, <0.01 [<0.01]
						3	0.057, <0.01 [0.029]
						7	<0.01, <0.01 [<0.01]
						14	<0.01, <0.01 [<0.01]
						21	0.012, 0.014 [0.013]
	3	151 151 151	- 7 7	1281 1281 1272	79 79 81	0	<0.01, 0.012 [0.011]
						3	0.041, 0.016 [0.029]
						7	<0.01, <0.01 [<0.01]
						14	<0.01, <0.01 [<0.01]
						21	0.012, 0.010 [0.011]
Terra Bella, CA; 2014 Kerman (R140585)	3	150 150 150	- 7 7	711 720 720	79 79 81	14	<0.01, <0.01 [<0.01]
	3	157 151 150	- 7 7	1412 1478 1403	79 79 81	14	<0.01, 0.012 [0.011]
ALMONDS							
Orland, CA; 2014 Nonpareil (R140586)	3	48 148 148	- 7 7	701 701 701	81 83 85	14	<0.01, <0.01 [<0.01]
	3	149 149 148	- 7 7	1403 1403 1403	81 83 85	14	<0.01, <0.01 [<0.01]
Strathmore, CA; 2014 Fritz (R140587)	3	153 148 157	- 7 7	505 486 533	81 81 81	0	<0.01, <0.01 [<0.01]
						3	<0.01, <0.01 [<0.01]
						7	<0.01, <0.01 [<0.01]
						14	<0.01, <0.01 [<0.01]
						21	<0.01, <0.01 [<0.01]
	3	154 151 157	- 7 7	1553 1525 1590	81 81 81	0	<0.01, <0.01 [<0.01]
						3	<0.01, <0.01 [<0.01]
						7	<0.01, <0.01 [<0.01]
						14	<0.01, <0.01 [<0.01]
						21	<0.01, <0.01 [<0.01]
Wasco, CA; 2014 Nonpareil (R140588)[A]	3	149 151 151	- 9 5	711 701 683	81 81 81	14	<0.01, <0.01 [<0.01]
	3	151 150 147	- 9 5	1216 1188 1150	81 81 81	14	<0.01, <0.01 [<0.01]
Wasco, CA; 2014 Fritz (R140589)[A]	3	152 151 151	- 6 8	524 514 514	85 85 89	14	<0.01, <0.01 [<0.01]
	3	150 150 151	- 6 8	2843 2825 2881	85 85 89	14	0.021, 0.021 [0.021]
Fresno, CA; 2014	3	149 150	- 6	683 683	81 81	15	<0.01, <0.01 [<0.01]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Monterey (R140590)		150	7	664	85-87		
	3	152	-	1590	81	15	<0.01, <0.01 [<0.01]
		150	6	1627	81		
		151	7	1721	85-87		

Notes:

[A] Applications were separated by 8-11 days; rendering the trials independent.

Oilseeds and oilfruits

Small seed oil seeds - Rapeseed

Table 112 Residues of mefentrifluconazole in rape (canola) seed from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Schreier, 2016, BASF DocID 2016_7006242)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Carberry, MB; Canada, 2015 Canterra 1970 (R140421)	2	152 152	- 14	203 202	65 75	21	0.12, 0.11 [0.12]
Neepawa, MB; Canada, 2015 L252 (R140422)	2	154 148	- 14	205 197	71 79	21	0.07, 0.05 [0.06]
Alvena; SK; Canada, 2015 Liberty Link L252 (R140423)	2	157 170	- 15	313 340	79-80 83-85	21	0.01, <0.01 [0.01]
Wakaw, SK; Canada, 2015 Roundup Ready 45H31 (R140424)	2	152 148	- 14	304 296	78-79 80-82	21	0.03, 0.05 [0.04]
Hepburn, SK; Canada, 2015 Roundup Ready 45H31 (R140425)	2	147 148	- 15	294 296	78-79 80-83	21	<0.01, <0.01 [<0.01]
Ft. Saskatchewan, AB; Canada, 2015, Roundup Ready D7454 (R140426)	2	150 150	- 14	200 200	74 78	21	0.32, 0.18 [0.25]
Andrew, AB; Canada, 2015 Roundup Ready D7454 (R140427)	2	151 154	- 14	200 205	77 80	21	0.13, 0.16 [0.15]
Plains, GA; United States 2015, Flint (R140415)	2	149 150	- 14	227 208	80 84	21	0.27, 0.23 [0.25]
Bagley, IA; United States 2014, 5440 (R140416)	2	156 149	- 14	214 247	80 87	7	0.21, 0.41 [0.31]
						10	0.21, 0.38 (0.30]
						14	0.39, 0.20 [0.30]
						21	0.78, 0.70 [0.74]
						28	0.47, 0.35 [0.41]
Northwood, ND; United States, 2015, 5440 (R140417)	2	150 150	- 14	280 281	67 72	21	0.04, 0.04 [0.04]
Jamestown, ND; United States, 2014, L252 (R140418)	2	152 152	- 14	190 218	75 85	21	0.01, 0.01 [0.01]
Blackfoot, ID; United States 2014, 09H7757 (R140419)	2	145 149	- 13	195 184	78 80	20	0.06, 0.05 [0.06]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
American Falls, ID; United States, 2014, V1037 (R140420)	2	152 157	- 14	190 195	65 77	22	0.05, 0.04 [0.05]

Sunflower seed – North America

Table 113 Residues of mefentrifluconazole in sunflower seeds from trials conducted in North America following applications of an EC formulation (Rosser, 2019, BASF DocID 2016_7010855)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
St-Marc-sur-Richelieu, QC; Canada, 2016, Sierra (R160216)	2	153 154	- 13	229 231	85 85	22	<0.01, <0.01 [<0.01]
Carberry, MB; Canada, 2016 Falcon (R160222)	2	150 154	- 15	110 113	71 83	20	0.038, 0.056 [0.047]
Brandon, MB; Canada, 2016 Falcon (R160223)	2	152 155	- 13	111 114	79 87	21	0.036, 0.046 [0.041]
Northwood, ND; United States 2016, Cobalt II (R160215)	2	149 147	- 14	185 137	79-80 84-86	20	0.054, 0.059 [0.057]
Cleveland, ND; United States 2016, RH1121 (R160217)	2	149 155	- 14	176 173	78 82	21	0.011, <0.01 [0.011]
Montpelier, ND; United States 2016, RH1121(R160218)	2	151 157	- 14	179 174	79 80	21	<0.01, <0.01 [<0.01]
Carrington, ND; United States 2016, Cobalt II (R160219)	2	150 150	- 14	139 140	79-80 84-86	21	<0.01, <0.01 [<0.01]
Grand Island, NE; United States 2016, 8N668S (R160220)	2	150 150	- 15	192 183	81 85	21	0.080, 0.043 [0.062]
Wall, TX; United States, 2016 8105N (R160221)	2	151 155	- 14	269 279	69-71 81	18	0.010, 0.013 [0.012]
Gardner, NY; United States, 2016 RH1121 (R160224)	2	146 149	- 14	174 171	77 85	14	<0.01, <0.01 [<0.01]
						21	<0.01, <0.01 [<0.01]
						28	<0.01, <0.01 [<0.01]
						35	<0.01, <0.01 [<0.01]

Sunflower seed – Europe

Table 114 Residues of mefentrifluconazole in sunflower seeds from trials conducted in Europe following applications of an SC formulation (Gálvez and Moreno, 2017, BASF DocID 2017_1018091; Gálvez, 2018, BASF DocID 2018_1013070)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Portion Analysed	Mefentrifluconazole (mg/kg)
Lentzke, BB; DEU 2016 NK Delfi (L160307)	2	113 108	- 13	301 289	59 69	0	Whole plant	1.0
						8	Rest of plant w/o roots	0.90
						8	Flower head	0.29
						70	Seed	<0.01
Donnelay, GES; FRA, 2016 Es Artic (L160308)	2	123 114	- 39	328 303	57-59 69	0	Whole plant	1.1
						14	Rest of plant w/o roots	1.6
						14	Flower head	0.27
						30	Seed	0.022
Refrancore, AT; ITA, 2016	2	110 110	- 14	300 300	59 69	0	Whole plant	2.4
						17	Rest of plant w/o roots	3.6

Mefentrifluconazole

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Portion Analysed	Mefentrifluconazole (mg/kg)
Olival (L160309)						17	Flower head	0.34
						37	Seed	<0.01
Lebrija, SE; ESP 2016 Kuzco (L160310)	2	114 112	- 25	304 297	59 69	0	Whole plant	3.7
						17	Rest of plant w/o roots	2.4
						17	Flower head	0.81
						26	Seed	0.015
Böhl, RP; DEU 2017 NK Delfi (L170117)	2	115 110	- 39	204 196	59 69	0	Whole plant	2.1
						7	Flower head	0.21
						7	Rest of plant w/o roots	2.6
						33	Seed	<0.01
Rohrau, NOE; AUT, 2017 DuPont Express Sun P64HE118 (L170118)	2	121 116	- 17	215 207	59 69	0	Whole plant	1.5
						18	Flower head	0.14
						18	Rest of plant w/o roots	0.87
						67	Seed	<0.01
Saint-Amand, WHT; BEL, 2017 ES Novamis CL (L170119)	2	109 115	- 23	194 205	59 69	0	Whole plant	1.6
						12	Flower head	0.21
						12	Rest of plant w/o roots	0.92
						57	Seed	<0.01
Ven-Zelderheide, LI; NLD, 2017 ES BiBA (L170120)	2	122 117	- 22	217 208	59 69	0	Whole plant	1.2
						14	Flower head	0.33
						14	Rest of plant w/o roots	0.55
						41	Seed	0.013
Donnelay, GES; FRA, 2017 ES IDYLLIC (L170121)	2	122 109	- 36	217 193	59 69	0	Whole plant	1.9
						14	Flower head	0.19
						14	Rest of plant w/o roots	0.92
						39	Seed	<0.01
Lentzke, BB; DEU 2017 NK Delfi (L170122)	2	112 113	- 16	199 201	59 69	0	Whole plant	0.72
						8	Flower head	0.16
						8	Rest of plant w/o roots	0.70
						74	Seed	<0.01
Castelnau D'Estrétefonds, OCC; FRA, 2017, Talento (L170123)	2	118 116	- 27	210 207	59 69	0	Whole plant	1.5
						22	Flower head	0.41
						22	Rest of plant w/o roots	2.1
						51	Seed	<0.01
Lalandusse, NAQ; FRA, 2017 ES SHAKIRA (L170124)	2	109 124	- 31	193 220	59 69	0	Whole plant	1.6
						19	Flower head	0.32
						19	Rest of plant w/o roots	2.0
						34	Seed	0.01
Kolchiko, GR-E; GRC 2017 P64LE25 (L170125)	2	113 113	- 14	200 201	59 69	0	Whole plant	2.4
						20	Flower head	0.19
						20	Rest of plant w/o roots	0.81
						48	Seed	<0.01
Quattordio, AL; ITA 2017 Club (L170126)	2	114 116	- 18	203 207	59 69	0	Whole plant	2.5
						11	Flower head	0.52
						11	Rest of plant w/o roots	3.8
						39	Seed	0.019
Lebrija, SE; ESP 2017 LG5461 (L170127)	2	121 116	- 9	215 207	59 69	0	Whole plant	2.2
						20	Flower head	0.55
						20	Rest of plant w/o roots	4.5
						41	Seed	0.017
Alcalá del Río, SE; ESP, 2017	2	116 115	- 14	205 204	59 69	0	Whole plant	4.4
						11	Flower head	0.72

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Portion Analysed	Mefentrifluconazole (mg/kg)
P64LL105 (L170128)						11	Rest of plant w/o roots	6.6
						32	Seed	0.044

Cottonseed

Table 115 Residues of mefentrifluconazole in cotton seeds from trials conducted in the United States following applications of an EC formulation (Phillips, 2018, BASF DocID 2018_7007470)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Portion Analysed	Mefentrifluconazole (mg/kg)
Jeffersonville, GA; 2017 ST 6182GLT (R170055)	3	146 149 150	- 6 8	275 279 282	77 80 80	33	Undelinted Cotton seed	0.024, 0.046 [0.035]
Proctor, AR; 2017 Stoneville 4949 GLT (R170056)	3	149 149 149	- 7 7	213 210 210	83 83-85 85-86	30	Undelinted Cotton seed	0.045, 0.061 [0.053]
Fisk, MO; 2017 CG3475B2XF (R170057)	3	147 149 148	- 8 6	185 187 187	81 82-83 83-84	29	Undelinted Cotton seed	0.029, 0.030 [0.030]
Washington, LA; 2017 DP 1639B2XF (R170058)	3	151 154 151	- 6 8	192 201 199	76-77 77-78 77-78	30	Undelinted Cotton seed	<0.01, 0.01 [0.01]
Uvalde, TX; 2017 DP 1044B2RF (R170059)	3	153 150 155	- 7 8	192 189 195	80 80 82	28	Undelinted Cotton seed	0.062, 0.043 [0.053]
Wolfforth, TX; 2017 Next Gen 4545 (R170060)	3	148 148 148	- 7 6	233 233 233	76 77 78	30	Undelinted Cotton seed	0.032, 0.031 [0.032]
Levelland, TX; United States 2017 DP1622 B2XF (R170061)	3	149 150 150	- 8 6	234 236 236	77 78 78	30	Undelinted Cotton seed	0.16, 0.075 [0.118]
							Gin by-products	1.47, 1.64 [1.56]
Littlefield, TX; United States 2017 Enlist WideStrike PHY300 W3F3 (R170062)	3	149 148 148	- 9 6	234 233 232	76 78 79-80	29	Undelinted Cotton seed	0.12, 0.077 [0.099]
							Gin by-products	3.80, 3.15 [3.48]
Wall, TX; United States 2017 FM 2007 GLT (R170063)	3	148 151 150	- 7 8	248 250 254	81 82 84	31	Undelinted Cotton seed	0.10, 0.099 [0.010]
							Gin by-products	4.14, 4.02 [4.08]
Yuba City, CA; United States, 2017 FM 1911 GLT (R170064)	3	150 149 150	- 7 7	189 188 189	78 81 83	31	Undelinted Cotton seed	0.065, 0.018 [0.042]
Paso Robles, CA; United States, 2017 DP358RF Pima (R170065)	3	150 145 148	- 6 7	283 273 279	79 81 81	29	Undelinted Cotton seed	0.035, 0.065 [0.050]
Sanger, CA; United States 2017, PHY 704 WRF Acala (R170066)	3	152 143 143	- 6 7	386 317 318	65 67-69 77-81	30	Undelinted Cotton seed	<0.01, <0.01 [<0.01]

Peanuts

Table 116 Residues of mefentrifluconazole in peanut nutmeat from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Andrews, 2016, BASF DocID 2016-7006298)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg) [
Elko, SC; United States	3	198	-	210	75	14	<0.01, <0.01 [<0.01]
2014, Bailey (R140337)		201	14	210	75		
		200	14	212	77		
Weston, GA; United States	3	200	-	197	74	14	<0.01, <0.01 [<0.01]
2014		200	14	211	78		
Georgia 06G (R140338)[A]		200	14	210	80		
Weston, GA; United States	3	200	-	197	72	14	<0.01, <0.01 [<0.01]
2014		200	14	212	77		
Georgia 06G (R140339)[A, B]		200	14	211	80		
Weston, GA; United States	3	200	-	212	77	13	<0.01, <0.01 [<0.01]
2014		199	14	210	80		
Georgia 06G (R140340)[B]		200	15	211	85		
Chula, GA; United States	3	200	-	196	75	14	<0.01, <0.01 [<0.01]
2014		199	14	195	79		
Georgia 09B (R140341)		200	15	206	85		
Abbeville, GA; United States	3	96	-	206	77	14	<0.01, <0.01 [<0.01]
2014		198	14	203	79		
Georgia 06G (R140342)		198	14	205	83		
Ellenton, GA; United States	3	197	-	190	75	14	<0.01, <0.01 [<0.01]
2014		201	14	197	77		
Georgia 09B (R140343)		200	14	205	83		
Winter Garden, FL; United States	3	198	-	235	79	14	<0.01, <0.01 [<0.01]
2014		202	14	239	86		
Georgia 06G (R140344)		197	14	233	88		
East Bernard, TX; United States	3	198	-	207	71	14	<0.01, <0.01 [<0.01]
2014		196	14	204	73		
Georgia 09B (R140345)		199	15	209	79		
Hinton, TX; United States	3	199	-	252	79	14	<0.01, <0.01 [<0.01]
2014		197	13	213	81-83		
Tamnut 0L06 (R140346)		197	16	229	81-83		
Edmonson, TX; United States 2014,	3	197	-	253	81	14	<0.01, <0.01 [<0.01]
ACI149		197	14	267	84		
(R140347)		194	14	262	86		
Danville, GA; United States	3	199	-	234	73	8	<0.01, 0.01 [0.01]
2014		200	14	235	75	10	<0.01, <0.01 [<0.01]
Georgia 06G (R140348)		198	14	233	77	14	<0.01, <0.01 [<0.01]
						17	<0.01, <0.01 [<0.01]
						22	<0.01, <0.01 [<0.01]

Notes:

[A] Applications were made on the same day, rendering the trials dependent.

[B] Applications were separated by 14-15 days, rendering the trials independent.

Coffee – South America

Table 117 Residues of mefentrifluconazole in coffee beans from trials conducted in South America following application of EC or SC formulations (Castro, 2016, BASF DocID 2016_3001981; Castro, 2017, BSF DocID 2017_3001321, Castro, 2018, BASF DocID 2018_3002901; Lucas, 2018, BASF DocID

2019_3000583)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Nominal spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Varginha, Minas Gerais; Brazil	3	134	-	400	71	15	0.011
2015		134	60	400	79	30	<0.01
Catucaí (G140309)		138	60	400	85	45	<0.01
						60	0.016
Ribeirão Preto, Sao Paulo; Brazil	3	142	-	400	75	15	0.014
2015		141	60	400	77	30	<0.01
Catucaí (G140310)		146	60	400	83	45	<0.01
						60	<0.01
Uberlândia, Minas Gerais; Brazil	3	137	-	400	75	15	0.13, 0.12 [0.13]
2015		137	60	400	81	30	0.097, 0.093 [0.095]
Mundo Novo (G140311)		136	60	400	89	45	0.069
						60	0.052
Engenheiro Coelho, Sao Paulo; Brazil	3	135	-	400	71	15	<0.01
2015		136	60	400	74	30	<0.01
Mundo Novo (G140312)		137	60	400	81	45	<0.01
						60	<0.01
Ibiporã, Paraná; Brazil	3	140	-	400	71	15	0.011
2015		144	60	400	76	30	<0.01
Catucaí (G140313)		146	60	400	82	45	<0.01
						60	<0.01
Ibiporã, Paraná; Brazil	3	171	-	400	71	15	<0.01, <0.01 [<0.01]
2016		166	60	400	75	30	<0.01, <0.01 [<0.01]
Catuai (G150270)		171	60	400	83	45	<0.01, <0.01 [<0.01]
						60	<0.01, <0.01 [<0.01]
Engenheiro Coelho, Sao Paulo; Brazil	3	171	-	400	71	15	<0.01, <0.01 [<0.01]
2016		163	60	400	77	30	<0.01, <0.01 [<0.01]
Oblata (G150271)		173	60	400	83	45	<0.01, <0.01 [<0.01]
						60	<0.01, <0.01 [<0.01]
Londrina, Paraná; Brazil	3	171	-	400	71	15	<0.01, <0.01 [<0.01]
2016		162	60	400	75	30	<0.01, <0.01 [<0.01]
Tupi (G150272)		167	60	400	83	45	<0.01, <0.01 [<0.01]
						60	<0.01, <0.01 [<0.01]
Ribeirão Preto, Sao Paulo; Brazil	3	161	-	400	77	15	<0.01, <0.01, <0.01 [<0.01]
2016		180	60	400	82	30	<0.01, <0.01, <0.01 [<0.01]
Catuai		178	60	400	87	45	<0.01, <0.01, <0.01 [<0.01]
(G150273)						60	<0.01, <0.01, <0.01 [<0.01]
Varginha, Minas Gerais; Brazil	3	167	-	400	77	15	<0.01, <0.01, <0.01 [<0.01]
2016		157	60	400	81	30	<0.01, <0.01, <0.01 [<0.01]
Catuai		163	60	400	85	45	<0.01, <0.01, <0.01 [<0.01]
(G150274)						60	<0.01, <0.01, <0.01 [<0.01]
Rio Claro, Sao Paulo; Brazil	3	163	-	400	70	15	<0.01, <0.01, <0.01 [<0.01]
2017		161	60	400	75	30	<0.01, <0.01, <0.01 [<0.01]
Obatã		160	60	400	81	45	0.014
(G160257)						60	0.013
Itirapina, Sao Paulo; Brazil	3	161	-	400	70	15	<0.01, <0.01, <0.01 [<0.01]
2017		158	60	400	80	30	0.028
Obatã		158	60	400	85	45	0.020
(G160258)						60	<0.01
Campinas, Sao Paulo; Brazil	3	166	-	400	70	15	0.010
2017		159	60	400	75	30	0.013
Tupi		160	60	400	80	45	0.012
(G160259)						60	0.021, 0.014, 0.016 [0.018]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Nominal spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Poços de Calda Minas Gerais; Brazil	3	164	-	400	70	15	0.018
2017		167	60	400	75	30	0.012
Cutuaí (G160260)		164	60	400	83	45	0.017, 0.014, 0.018 [0.016]
						60	0.017
Andradas Minas Gerais; Brazil	3	163	-	400	70	15	<0.01, <0.01, <0.01 [<0.01]
2017		167	60	400	75	30	<0.01
Cutuaí (G160261)		164	60	400	83	45	0.012, 0.014, 0.010 [0.012]
Parroquia Colonche, Santa Helena;	3	160	-	400	65	15	0.53
Ecuador, 2017		160	60	400	70	30	0.30
Robusta Tropical (G175044)[A]		160	60	400	81	45	0.18
						60	0.33
Parroquia Santa Helena, Santa	3	160	-	400	65	15	0.34
Helena; Ecuador		160	60	400	72	30	0.095, 0.11 [0.010]
2017		160	60	400	81	45	0.065, 0.079 [0.072]
Robusta Tropical (G175045)[A]						60	0.14
Santa Marta, Departamento Del	3	160	-	400	69	15	0.020
Magdalena; Colombia		160	60	400	72	30	0.010
2017		160	60	400	76	45	<0.01
(G175046)						60	<0.01
Aracataca;	3	160	-	400	70	15	0.016
Departamento Del Magdalena;		160	60	400	73	30	<0.01
Colombia		160	60	400	76	45	<0.01
2017 (G175047)						60	<0.01

Notes:

[A] Applications were made on the same day rendering the trials dependent.

Animal Feeds

Legume vegetables

Vines

Table 118 Residues of mefentrifluconazole in pea vines from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Jamestown, ND; United States	3	150	-	178	68	21	7.35, 7.55 [7.45]
2014		154	6	193	69		
4010 Forage Pea (R140728)		156	7	195	75		
Carrington, ND; United States	3	149	-	186	71	21	0.85, 0.79 [0.82]
2015		150	7	186	74		
Oregon Trail Peas (R140729)		150	6	187	76		
Gran Island. NE; United States	3	150	-	192	74	21	8.98, 10.03 [9.51]
2015		150	7	187	77		
Austrian Winter peas (R140730)		151	7	196	81		
American Falls, ID; United States	3	151	-	188	77	21	10.31, 10.04 [10.18]
2014		151	7	187	79		
954-Genie (R140731)		150	7	186	79-81		
American Falls, ID; United States	3	150	-	187	71	0	5.25, 5.63 [5.44]
2015		152	7	190	74-75	7	1.75, 1.96 [1.86]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Banner Peas (R140732)		153	7	191	76-77	14	1.53, 1.20 [1.36]
						21	1.28, 1.23 [1.26]
						28	0.62, 0.80 [0.71]
Parkdale, OR; United States	3	150	-	177	61	21	4.53, 4.03 [4.28]
2015		151	7	186	75		
Columbia (R140733)		152	7	188	79		
Fort Saskatchewan, AB; Canada	3	151	-	151	60	21	2.45, 2.01 [2.23]
2015, Meadow Peas (R140734)		151	6	151	65		
		152	7	152	67		
Wakaw, SK; Canada	3	146	-	98	61-63	21	2.68, 2.88 [2.78]
2015		152	7	101	67-69		
Treasure Peas (R140735)		153	7	102	69-723		

Cowpea – Forage

Table 119 Residues of mefentrifluconazole in cowpea forage from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Dana, IA; 2015	3	149	-	186	9-10	21	0.04, 0.05 [0.05]
Blackeye Cowpeas (R140866)		148	7	184	12-13		
		149	7	185	13-15		
Delavan, WI;	3	152	-	168	14	21	0.04, 0.03 [0.04]
2015		151	8	167	15		
Pinkeye Purple Hull (R140867)		152	6	168	17		
American Falls, ID;	3	155	-	194	10-12	21	0.08, 0.09 [0.09]
2015		167	8	180	13-19		
Blackeye Peas, Type 46 (R140868)		147	7	185	31-32		

Soya bean – Forage

Table 120 Residues of mefentrifluconazole in soya bean forage from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Chula, GA; 2014	2	150	-	216	19	21	1.71, 1.76 [1.74]
HBK 7028 (R140686)		152	7	223	60		
Chula, GA; 2015, Asgrow AG 7231	2	152	-	213	18	21	0.80, 0.77 [0.79]
(R140687)		153	7	206	19		
Washington, LA; 2014, P95 Y70	2	150	-	218	19	21	0.64, 0.65 [0.65]
(R140688)[A]		152	7	198	22		
Opelousas, LA; 2014, P95 Y70	2	156	-	226	22	21	1.83, 1.61 [1.73]
(R140689)[A]		148	7	193	22		
Morrow, LA; 2014, Terral Rev 56R63	2	146	-	127	49	21	2.10, 2.83 [2.47]
(R140690)		148	7	132	50		
Campbell, MN; 2014, Asgrow AG 0634	2	150	-	187	15	21	0.88, 1.44 [1.06]
(R140691)		150	7	187	17		
Erie, ND; 2014	2	152	-	142	60	21	2.20, 2.02 [2.11]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
11 R08 RR2Y 91221152 (R140692)		154	8	144	63		
Perley, MN; 2014 POST 24R PC35 (R140693)	2	150 156	- 7	140 146	60 63	21	2.72, 2.61 [2.67]
Gardner, ND; 2014 S02 M9 (R140694)	2	147 154	- 7	138 145	60 63	0	11.53, 20.51 [16.04]
						7	5.81, 5.35 [5.58]
						14	2.65, 3.18 [2.91]
						21	1.42, 1.45 [1.44]
						28	1.06, 1.03 [1.05]
Northwood, ND; 2015 NT0090RR (R140695)	2	150 153	- 7	187 190	12 13	21	0.20, 0.15 [0.18]
Jefferson, IA; 2014 92Y75 Pioneer (R140696)[B]	2	151 151	- 7	263 273	16 17	0	16.57, 14.72 [15.64]
						7	4.63, 4.98 [4.76]
						14	1.29, 1.16 [1.23]
						21	0.53, 0.47 [0.50]
						28	0.42, 0.27 [0.35]
Paton, IA; 2014 92Y75 Pioneer (R140697)[B]	2	150 151	- 7	262 273	15 16	21	1.22, 1.05 [1.14]
Dana, IA; 2014 92Y75 Pioneer (R140698)[B]	2	151 152	- 7	263 274	15 16	21	1.19, 1.10 [1.15]
Delavan, WI; 2015 A1024341 (R140699)	2	152 151	- 7	168 169	15 17	21	1.80, 1.97 [1.89]
Fisk, MO; 2015 48E3RR (R140700)	2	150 148	- 7	187 186	17 61	21	1.41, 1.62 [1.52]
Bloomfield, MO; 2015 NK 584-P4 (R140701)	2	148 149	- 7	184 186	16 18	21	0.29, 0.33 [0.31]
McClure, IL; 2015 5N479R2 (R140702)	2	150 153	- 7	187 191	16-17 61	21	1.08. 1.06 [1.07]
Enid, OK; 2014 HBK RY 4620 (R140703)	2	154 151	- 7	119 202	13-15 19	21	0.55, 0.57 [0.56]
Ringwood, OK; 2014 Unknown (R140704)	2	152 148	- 7	208 247	3-15 16-17	21	0.47, 0.60 [0.54]
Stafford, KS; 2015 P31T11R (R140705)	2	153 151	- 7	172 169	51 51	21	0.48, 0.53 [0.51]

Notes:

[A] Applications were made on the same day, rendering the trials dependent.

[B] Applications were made on the same day, rendering the trials dependent.

Alfalfa forage

Table 121 Residues of mefentrifluconazole in alfalfa forage from trials conducted in Canada and the United States following applications of an EC formulation (Csinos, 2019, BASF DocID 2019_7002384)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Minto, MB; Canada, 2018 Blend 4440 (R180025)	3	148 151 147	- 14 34	198 204 196	Mid Vegetative	14	0.83, 0.97 [0.90]
North Rose, NY; United States, 2018 NA (R180016)	3	148 151 151	- 14 28	166 170 170	19 22 36-38	11	3.89, 2.96 [3.42]
						14	6.24, 5.46 [5.85]
						43	0.06, 0.11 [0.09]
						73	<0.01, <0.01 [<0.01]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Shelbyville, IN; United States 2018 Beck's Caliber (R180017)	3	147 146 149	- 14 42	188 186 195	Not recorded	0 7 13 14 21 63	15.11, 18.72 [16.92] 4.90, 4.55 [4.73] 1.06, 1.26 [1.16] 3.28, 2.99 [3.13] 0.85, 0.98 [0.91] <0.01, <0.01 [<0.01]
Bagley, IA; United States 2018 Vernal (R180018)	3	151 146 154	- 15 32	153 157 169	14 51 36	13 14 49	2.61, 3.48 [3.05] 2.10, 2.29 [2.20] <0.01, <0.01 [<0.01]
Northwood, ND; United States, 2018 Multi – F-1 (R180019)	3	148 149 151	- 14 30	195 195 197	34-36 39-51 35-36	13 13 54	2.077, 1.639 [1.858] 2.436, 1.951 [2.194] <0.01, <0.01 [<0.01]
Gardner, ND; United States 2018 Multi – F-2 R180020	3	154 156 155	- 14 30	201 204 202	50 60 30	14 14 55	3.12, 3.32 [3.22] 0.95, 1.16 [1.06] 0.24, 0.22 [0.23]
Montpelier, ND; United States, 2018 Multi – F-2 (R180021)	3	150 152 154	- 14 26	196 199 200	45 55 30	14 14 56	3.77, 3.51 [3.64] 0.37, 0.31 [0.34] 2.09, 2.92 [2.50]
Monte Vista, CO; United States, 2018 Extend (R180022)	3	150 151 149	- 14 33	193 198 195	Mid Vegetative Early Bloom	14 14 37 63	1.99, 1.69 [1.84] 2.92, 3.83 [3.38] 0.10, 0.09 [0.10] 0.12, 0.17 [0.15]
Kerman, CA; United States 2018 Germains 825 GQ (R180023)	3	150 150 150	- 14 21	196 195 196	59 61 51	14 14 35 56	3.64, 3.42 [3.53] 2.45, 3.06 [2.76] 0.35, 0.33 [0.34] 0.13, 0.10 [0.11]
Idaho Falls, ID; United States, 2018 RR-WL372 (R180024)	3	155 155 147	- 14 41	204 214 195	Not recorded Mid Vegetative	13 14 14 64	2.02, 1.90 [1.96] 2.17, 1.50 [1.84] 4.19, 4.33 [4.26] 0.05, 0.05 [0.05]

Clover forage

Table 122 Residues of mefentrifluconazole in clover forage from trials conducted in the United States following applications of an EC formulation (Csinos, 2019, BASF DocID 2019_7002384)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
North Rose, NY; United States 2018 SS-030RCG (R180026)	3	152 151 151	- 14 42	169 167 171	35-36 38-39 26-28	14 14	1.59, 1.59 [1.59] 0.34, 0.42 [0.38]
TyTy, GA; United States 2018 Regal (R180027)	3	150 150 150	- 14 39	197 196 196	60 60 56	14 14	3.43, 3.75 [3.59] 1.67, 1.80 [1.74]
Washington, LA; United States 2018 White Clover (R180028)	3	154 155 152	- 14 15	212 216 196	65 65 65	14 14	5.38, 5.41 [5.40] 6.73, 4.78 [5.75]
Bagley, IA; United States 2018 Medium Red Clover (R180029)	3	145 151 150	- 15 32	146 161 166	13 51 35	0 7 13 15	16.46, 15.83 [16.2] 5.22, 3.71 [4.46] 4.28, 3.43 [3.86] 2.67, 3.28 [2.98]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
						20	1.78, 1.45 [1.62]
						28	1.19, 1.23 [1.21]
Aurora, SD; United States	3	154	-	190	39	14	1.28, 1.97 [1.63]
2018		152	14	178	49	15	1.14, 0.93 [1.04]
Arlington Red (R180030)		152	25	189	49		
Madill, OK; United States	3	157	-	197	14	14	3.02, 2.70 [2.86]
2018		148	14	165	18	14	6.00, 6.63 [6.32]
Alyce (R180031)		152	28	184	15		
Montpelier, ND; United States	3	154	-	200	40	14	6.51, 5.69 [6.10]
2018		151	14	198	50	14	1.45, 1.12 [1.28]
Medium Red Clover (R180032)		151	26	198	30		
Claude, TX; United States	3	148	-	192	Early	14	3.10, 2.29 [2.70]
2018		150	14	199	Vegetati	14	7.03, 6.53 [6.78]
Crimson (R180033)		149	48	195	ve		
Yuba City, CA; United States	3	151	-	198	13	14	0.15, 0.20 [0.18]
2018		151	13	198	24	14	0.14, 0.13 [0.14]
Crimson (R180034)		150	22	196	26		
Lamont, AB; CAN	2	151	-	196	14	14	2.76, 3.14 [2.95]
2018, Red Clover (R180035)[A]		150	14	196	34		

Notes:

[A] Third application was not made due to early freeze and snow.

Pea – Hay

Table 123 Residues of mefentrifluconazole in pea hay from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Fort Saskatchewan,	3	151	-	151	60	21	18	9.32, 8.69 [9.01]	11.36, 10.60
AB; Canada, 2015		151	6	151	65				[10.98]
Meadow Peas		152	7	152	67				
(R140734)									
Wakaw, SK; Canada	3	146	-	98	61-63	21	29	3.78, 3.40 [3.59]	5.32, 4.79
2015, Treasure Peas		152	7	101	67-69				[5.05]
(R140735)		153	7	102	69-723				
Jamestown, ND;	3	150	-	178	68	21	28	6.21, 9.08 [7.64]	8.62, 12.61
United States, 2014		154	6	193	69				[10.62]
4010 Forage Pea		156	7	195	75				
(R140728)									
Carrington, ND; United	3	149	-	186	71	21	19	8.23, 6.59 [7.41]	10.16, 8.13
States, 2015		150	7	186	74				[9.15]
Oregon Trail Peas		150	6	187	76				
(R140729)									
Gran Island. NE; United	3	150	-	192	74	21	31	7.83, 8.95 [8.39]	11.34, 12.97
States, 2015		150	7	187	77				[12.15]
Austrian Winter peas		151	7	196	81				
(R140730)									
American Falls, ID;	3	151	-	188	77	21	17	8.18, 8.98 [8.58]	9.86, 10.82
United States, 2014		151	7	187	79				[10.33]

Location; Year, Variety (Trial)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
954-Genie (R140731)		150	7	186	79-81				
American Falls, ID; United States 2015 Banner Peas (R140732)	3	150 152 153	- 7 7	187 190 191	71 74-75 76-77	0	23	14.46, 12.88 [13.67]	18.78, 16.73 [17.75]
						7		7.22, 8.80 [8.01]	9.38, 11.43 [10.40]
						14		3.17, 4.55 [3.86]	4.12, 5.91 [5.01]
						21		4.14, 4.70 [4.42]	5.38, 6.10 [5.74]
						28		3.08, 3.18 [3.13]	4.00, 4.13 [4.06]
Parkdale, OR; United States, 2015 Columbia (R140733)	3	150 151 152	- 7 7	177 186 188	61 75 79	21	17	5.89, 4.56 [5.22]	7.10, 5.49 [6.30]

Cowpea – Hay

Table 124 Residues of mefentrifluconazole in cowpea hay from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005932)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Dana, IA; 2015, Blackeye Cowpeas (R140866)	3	151 146 149	- 7 7	188 182 186	62-71 67-75 69-79	21	0.21, 0.67 [0.44]
Delavan, WI; 2015 Pinkeye Purple Hull (R140867)	3	152 149 146	- 7 7	169 163 161	17 19 19	21	0.78, 0.96 [0.87]
American Falls, ID; 2015, Blackeye Peas, Type 46 (R140868)	3	150 150 151	- 7 7	187 187 190	30-38 39-51 60-61	21	1.26, 1.96 [1.61]

Soya bean – Hay

Table 125 Residues of mefentrifluconazole in soya bean hay from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Crawford, 2016, BASF DocID 2015_7005933)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% Moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Chula, GA; 2014 HBK 7028 (R140686)	2	150 152	- 7	216 223	19 60	21	22	4.61, 4.26 [4.44]	5.91, 5.46 [5.68]
Chula, GA; 2015 Asgrow AG 7231 (R140687)	2	152 153	- 7	213 206	18 19	21	29	3.20, 3.08 [3.14]	3.95, 4.33 [4.14]
Washington, LA; 2014 P95 Y70 (R140688)[A]	2	150 152	- 7	218 198	19 22	21	49	1.87, 3.55 [2.71]	3.67, 6.96 [5.32]

Mefentrifluconazole

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% Moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Opelousas, LA; 2014 P95 Y70 (R140689)[A]	2	156 148	- 7	226 193	22 22	21	44	4.96, 1.76 [3.36]	8.86, 3.14 [6.00]
Morrow, LA; 2014 Terral Rev 56R63 (R140690)	2	146 148	- 7	127 132	49 50	21	35	6.80, 7.84 [7.32]	10.46, 12.06 [11.26]
Campbell, MN; 2014 Asgrow AG 0634 (R140691)	2	150 150	- 7	187 187	15 17	21	45	3.75, 2.90 [3.33]	6.82, 5.27 [6.05]
Erie, ND; 2014 11 R08 RR2Y 91221152 (R140692)	2	152 154	- 8	142 144	60 63	21	25	6.06, 7.18 [6.62]	8.08, 9.57 [8.83]
Perley, MN; 2014 POST 24R PC35 (R140693)	2	150 156	- 7	140 146	60 63	21	25	5.89, 7.67 [6.78]	7.85, 10.23 [9.04]
Gardner, ND; 2014 S02 M9 (R140694)	2	147 154	- 7	138 145	60 63	0	23	56.17, 63.48 [59.82]	72.95, 82.44 [77.70]
						7		14.65, 15.87 [15.26]	19.02, 20.61 [19.82]
						14		7.40, 8.67 [8.04]	9.61, 11.26 [10.43]
						21		3.67, 2.96 [3.31]	4.77, 3.84 [4.31]
						28		2.97, 2.85 [2.91]	3.86, 3.70 [3.78]
Northwood, ND; 2015 NT0090RR (R140695)	2	150 153	- 7	187 190	12 13	21	40	0.78,0.58 [0.68]	1.30, 0.97 [1.13]
Jefferson, IA; 2014 92Y75 Pioneer (R140696)[B]	2	151 151	- 7	263 273	16 17	0	50	25.39, 26.81 [26.10]	50.78, 53.62 [52.2]
						7		8.91, 8.40 [8.66]	17.82, 16.8 [17.31]
						14		1.98, 2.06 [2.04]	3.96, 4.12 [4.04]
						21		1.24, 2.23 [1.74]	2.48, 4.46 [3.48]
						28		0.48, 0.94 [0.71]	0.96, 1.88 [1.42]
Paton, IA; 2014 92Y75 Pioneer (R140697)[B]	2	150 151	- 7	262 273	15 16	21	39	2.87, 2.65 [2.76]	4.70, 4.34 [4.52]
Dana, IA; 2014 92Y75 Pioneer (R140698)[B]	2	151 152	- 7	263 274	15 16	21	37	3.06, 1.96 [3.01]	4.85, 3.11 [3.98]
Delavan, WI; 2015 A1024341 (R140699)	2	152 151	- 7	168 169	15 17	21	42	5.03, 4.50 [4.77]	8.67, 7.76 [8.21]
Fisk, MO; 2015 48E3RR (R140700)	2	150 148	- 7	187 186	17 61	21	14	6.15, 6.08 [6.12]	7.15, 7.07 [7.11]
Bloomfield, MO; 2015 NK 584-P4 (R140701)	2	148 149	- 7	184 186	16 18	21	28	1.09, 1.29 [1.19]	1.51, 1.79 [1.65]
McClure, IL; 2015 5N479R2 (R140702)	2	150 153	- 7	187 191	16-17 61	21	28	2.95, 2.80 [2.88]	4.10, 3.89 [3.99]
Enid, OK; 2014 HBK RY 4620 (R140703)	2	154 151	- 7	119 202	13-15 19	21	32	2.22, 1.84 [2.03]	3.26, 2.70 [2.98]
Ringwood, OK; 2014	2	152	-	208	3-15	21	43	1.86, 1.76	3.26, 3.09

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% Moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Unknown (R140704)		148	7	247	16-17			[1.81]	[3.18]
Stafford, KS; 2015 P31T11R (R140705)	2	153 151	- 7	172 169	51 51	21	30	1.57, 1.61 [1.59]	2.24, 2.30 [2.27]

Notes:

[A] Applications were made on the same day, rendering the trials dependent.

[B] Applications were made on the same day, rendering the trials dependent.

Alfalfa hay

Table 126 Residues of mefentrifluconazole in alfalfa hay from trials conducted in Canada and the United States following applications of an EC formulation (Csinos, 2019, BASF DocID 2019_7002384)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Minto, MB; Canada, 2018 Blend 4440 (R180025)	3	148 151 147	- 14 34	198 204 196	Mid Vegetative	14	4.62, 5.72 [5.17]
North Rose, NY; United States 2018 (R180016)	3	148 151 151	- 14 28	166 170 170	19 22 36-38	11 14 43 73	6.32, 7.88 [7.10] 8.28, 8.03 [8.15] 0.08, 0.13 [0.11] 0.16, 0.23 [0.20]
Shelbyville, IN; United States 2018 Beck's Caliber (R180017)	3	147 146 149	- 14 42	188 186 195	Not recorded	0 7 13 14 21 63	35.22, 44.60 [39.91] 12.52, 14.08 [13.30] 3.11, 2.30 [2.71] 7.77, 6.26 [7.02] 3.09, 3.34 [3.22] <0.01, <0.01 [<0.01]
Bagley, IA; United States, 2018 Vernal (R180018)	3	151 146 154	- 15 32	153 157 169	14 51 36	13 14 49	6.35, 5.73 [6.04] 5.05, 5.36 [5.20] <0.01, <0.01 [<0.01]
Northwood, ND; United States 2018, Multi – F-1 (R180019)	3	148 149 151	- 14 30	195 195 197	34-36 39-51 35-36	13 13 54	3.32, 3.73 [3.52] 4.27, 4.26 [4.26] <0.01, <0.01 [<0.01]
Gardner, ND; United States 2018, Multi – F-2 (R180020)	3	154 156 155	- 14 30	201 204 202	50 60 30	14 14 55	7.58, 8.37 [7.98] 3.97, 4.15 [4.06] 0.85, 0.49 [0.67]
Montpelier, ND; United States 2018, Multi – F-2 (R180021)	3	150 152 154	- 14 26	196 199 200	45 55 30	14 14 56	8.20, 8.94 [8.57] 1.43, 1.57 [1.50] 0.30, 0.52 [0.41]
Monte Vista, CO; United States 2018, Extend (R180022)	3	150 151 149	- 14 33	193 198 195	Mid Vegetative Early Bloom	14 14 37 63	2.68, 1.67 [2.18] 4.86, 4.06 [4.46] 0.35, 0.09 [0.22] 0.18, 0.15 [0.17]
Kerman, CA; United States 2018 Germains 825 GQ (R180023)	3	150 150 150	- 14 21	196 195 196	59 61 51	14 14 35 56	8.01, 5.97 [6.99] 6.34, 6.52 [6.43] 1.24, 0.82 [1.03] 0.32, 0.54 [0.43]
Idaho Falls, ID; United States 2018 RR-WL372	3	155 155 147	- 14 41	204 214 195	- Mid Vegetative	13 14 14	4.46, 3.83 [4.14] 5.44, 5.89 [5.67] 16.87, 16.64 [16.75]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
(R180024)						64	0.16, 0.20 [0.18]

Clover hay

Table 127 Residues of mefentrifluconazole in clover hay from trials conducted in Canada and the United States following applications of an EC formulation (Csinos, 2019, BASF DocID 2019_7002384)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Lamont, AB; Canada, 2018 Red Clover (R180035)[A]	2	151 150	- 14	196 196	14 34	14	6.00, 9.92 [7.96]
North Rose, NY; United States 2018 SS-030RCG (R180026)	3	152 151 151	- 14 42	169 167 171	35-36 38-39 26-28	14 14	3.83, 4.15 [3.99] 0.99, 1.26 [1.13]
TyTy, GA; United States 2018 Regal (R180027)	3	150 150 150	- 14 39	197 196 196	60 60 56	14 14	8.09, 6.28 [7.18] 5.40, 4.56 [4.98]
Washington, LA; United States 2018 White Clover (R180028)	3	154 155 152	- 14 15	212 216 196	65 65 65	14 14	7.94, 10.01 [8.97] 6.59, 9.98 [8.30]
Bagley, IA; United States 2018 Medium Red Clover (R180029)	3	145 151 150	- 15 32	146 161 166	13 51 35	0 7 13 15 20 28	46.40, 34.36 [40.38] 17.59, 14.20 [15.90] 9.57, 14.38 [11.98] 5.29, 7.16 [6.22] 3.58, 2.72 [3.15] 2.04, 2.03 [2.04]
Aurora, SD; United States 2018 Arlington Red (R180030)	3	154 152 152	- 14 25	190 178 189	39 49 49	14 15	4.94, 4.04 [4.49] 3.23, 4.19 [3.71]
Madill, OK; United States 2018 Alyce (R180031)	3	157 148 152	- 14 28	197 165 184	14 18 15	14 14	6.30, 9.56 [7.93] 15.71, 15.95 [15.83]
Montpelier, ND; United States 2018 Medium Red Clover (R180032)	3	154 151 151	- 14 26	200 198 198	40 50 30	14 14	17.57, 18.63 [18.1] 6.00, 5.64 [5.82]
Claude, TX; United States 2018 Crimson (R180033)	3	148 150 149	- 14 48	192 199 195	Early Mid Vegetative	14 14	4.01, 4.22 [4.11] 18.90, 14.92 [16.91]
Yuba City, CA; United States 2018, Crimson (R180034)	3	151 151 150	- 13 22	198 198 196	13 24 26	14 14	0.73, 1.28 [1.01] 0.71, 0.83 [0.77]

Notes:

[A] Third application was not made due to early freeze and snow.

Sugar Beet Tops

Table 128 Residues of mefentrifluconazole in sugar beet tops from trials conducted in North America following application an EC formulation. In each trial, an adjuvant was added (Falk, 2016, BASF DocID 2016_7010183)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	DALA	Mefentrifluconazole (mg/kg)
Taber, AB; Canada, 2015 47RR75 (R140368)[B]	2	152 155	- 6	202 207	21	0.46, 0.51 [0.49]
Taber, AB; Canada, 2015 9103RR (R140369)[B]	2	153 153	- 7	153 153	21	1.73, 1.94 [1.84]
Boissevain, MB; Canada, 2015 9102RR (R14A.0374)	2	147 152	- 7	295 305	21	3.50, 3.06 [3.28]
Carlyle, IL; United States 2014 (R140362)	2	146 154	- 7	98 104	14	7.95, 7.32 (7.64]
					21	6.77, 7.11 [6.94]
					28	3.94, 3.99 [3.97]
Highland, IL; United States 2014 (R140363)	2	161 157	- 7	147 157	14	1.68, 1.30 [1.49]
					21	1.27, 0.90 [1.09]
					28	0.62, 0.63 [0.63]
Wyoming, IL; United States, 2014 (R140364)	2	148 151	- 7	138 144	21	0.72, 0.73 [0.73]
York, NE; United States, 2014 48607 TT (R140365)	2	154 149	- 7	125 128	21	1.65, 2.56 [2.10]
Aurora, SD; United States, 2014 48607 TT (R140366)	2	157 150	- 7	156 150	21	1.78, 2.55 [2.16]
St Lawrence, SD; United States 2014, 48607 TT (R140367)	2	149 151	- 6	146 146	21	2.10[A], <0.01[A]
Levelland, TX; United States, 2014 Phoenix (R140370)	2	151 148	- 7	190 185	21	1.98, 1.86 [1.91]
Nipoma, CA; United States, 2014 48607 TT (R140371)	2	149 150	- 7	286 287	21	1.99, 1.58 [1.78]
Blaine County, ID; United States 2014, SX1521WRR (R140372)	2	148 150	- 7	121 118	21	1.08, 0.97 [1.03]
Minidoka County, ID; United States 2014, Beta 2028 (R140373)	2	147 155	- 7	121 121	21	1.53, 1.96 [1.77]

Notes:

[A] These top samples were analysed multiple times, first by a reinjection of the initial extracts and then by using aliquots from a fresh weighing of sample material. The mean result is shown. The results suggest that the untreated control sample, having a residue of 2.10 mg/kg, was switched with the treated sample. The result for the control sample is presumed to pertain to the treated sample.

[B] Applications were separated by 14 days, rendering the trials independent.

Subgroup of Cereal grains (including pseudocereals) feed products with high water (≥20 percent) content (forage and silage)

Wheat Forage

Table 129 Residues of mefentrifluconazole in wheat forage from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Minto, Manitoba, Canada, 2015, Carberry (R140306)	2	153 151	- 14	163 161	11-12 29-30	20	85	0.11, 0.08 [0.10]	0.73, 0.53 [0.63]
Elgin, Manitoba; Canada 2015, Cardale (R140307)	2	153 151	- 14	307 303	69-71 83	21	85	0.09, 0.08 [0.09]	0.60, 0.53 [0.57]
Hague, Saskatchewan; Canada,	2	150	-	150	79-83	21	77	0.07, 0.06	0.30, 0.26 [0.28]

Mefentrifluconazole

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
2015, AC Vespar (R140308)		150	14	150	87-92			[0.07]	
Kipp, Alberta; Canada 2015, AC Carberry (R140309)	2	150 156	- 14	100 104	75-77 81-85	21	83	0.03, 0.01 [0.02]	0.18, 0.06 [0.12]
Fort Saskatchewan, Alberta; Canada, 2015 Harvest (R140310)	2	155 149	- 14	207 199	75 87	21	71	0.67, 0.76 [0.72]	2.31, 2.62 [2.47]
Alvena, Saskatchewan; Canada, 2015 Cardale (R140311)	2	146 148	- 14	174 176	75-76 83-85	21	62	1.74, 1.46 [1.60]	4.58, 3.84 [4.21]
Brandon, Manitoba; Canada, 2015, Brandon (R140312)	2	151 148	- 14	101 99	77 87	21	70	0.92, 0.74 [0.83]	3.07, 2.47 [2.77]
Delisle, Saskatchewan; Canada, 2015, Marchwell (R140296)	2	152 154	- 14	152 154	71-73 83-85	21	77	0.03, 0.12 [0.08]	0.13, 0.52 [0.33]
Taber, Alberta; Canada 2015, AC Carberry (R140300)	2	151 151	- 14	201 202	71-75 73-75	21	75	0.16, 0.12 [0.14]	0.64, 0.48 [0.56]
Athens, GA; United States 2015, GA Gore (R140288)	2	152 154	- 14	291 290	65-69 77-83	21	71	1.03, 1.42 [1.23]	3.55, 4.90 [4.22]
Stuttgart, AR; United States, 2014, TV8848 (R140289)	2	152 154	- 14	152 153	45-53 69-71	21	67	1.10, 1.07 [1.09]	3.33, 3.24 [3.29]
Gardner, ND; United States, 2015, Elgin (R140290)	2	151 156	- 14	189 196	70 85	21	76	0.80, 0.95 [0.88]	3.33, 3.96 [3.65]
St. Cloud, MN; United States, 2014, Faller (R140291)	2	150 149	- 14	188 186	57 77	21	56	2.46, 2.38 [2.42]	5.59, 5.41 [5.50]
Paynesville, MN; United States, 2014, Oklee (R140292)	2	151 150	- 14	191 190	35 87-89	21	83	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
Fisk, MO; United States 2015, Roane (R140293)	2	149 149	- 14	186 187	61 75	21	77	0.56, 0.70 [0.63]	2.43, 3.04 [2.74]
East Bernard, TX; United States, 2015, LA841 (R140294)	2	150 149	- 13	335 333	77 89	21	71	1.88, 2.04 [1.96]	6.48, 7.03 [6.76]
Grand Island, NE; United States, 2014, Prosper (R140295)	2	148 148	- 13	176 175	61 77	21	76	0.40, 0.42 [0.41]	1.67, 1.75 [1.71]
Jamestown, ND; United States, 2015, Prosper (R140297)[A]	2	147 155	- 14	184 183	69 75	20	83	0.09, 0.06 [0.08]	0.53, 0.35 [0.44]
Jamestown, ND; United States, 2015, Divide (R140298)[A]	2	149 149	- 14	140 140	69 76-77	21	77	2.09, 1.51 [1.80]	9.09, 6.56 [7.83]
Hastings, NE; United States, 2014 Prosper (R140299)	2	150 150	- 14	220 221	71 87	0	76	7.62, 9.72 [8.67]	31.75, 40.50 [36.12]
						14		0.31, 0.52 [0.42]	1.29, 2.17 [1.73]
						21		0.23, 0.19 [0.21]	0.96, 0.79 [0.88]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
						28		<0.01, 0.20 [0.11]	<0.01, 0.83 [0.42]
						35		0.29, 0.23 [0.26]	1.21, 0.96 [1.08]
Wall, TX; United States 2015, TAM 113 (R140301)	2	147 148	- 14	175 181	73 85	20	80	0.75, 0.85 [0.80]	3.75, 4.25 [4.00]
Groom, TX; United States 2015, TAM 111 (R140302)	2	148 151	- 14	238 247	73 79	21	80	0.30, 0.18 [0.24]	1.50, 0.90 [1.20]
Claude, TX; United States 2015, TAM 112 (R140303)	2	151 152	- 14	242 250	73 75	21	67	2.08, 1.81 [1.95]	6.30, 5.48 [5.89]
Lamed, KS; United States 2015, LCS Wizard R140304)	2	150 151	- 14	169 169	85 85	21	83	0.80, 0.49 [0.65]	4.70, 2.88 [3.79]
Aberdeen, ID; United States, 2014, Alturas (R140305)	2	154 147	- 14	141 145	75 85	21	82	2.40, 1.06 [1.73]	13.33, 5.88 [9.61]

Notes:

[A] Applications were separated by 10 days; rendering the trials dependent.

Wheat Whole Plant, Ears, Rest of Plant – Europe

Table 130 Residues of mefentrifluconazole in wheat whole plant (no root), ears and rest of plant (without roots) from trials conducted in Europe following application of EC or SC formulations (Erdmann, 2015, BASF DocID, 2014_1010809; Ale, 2015, BASF DocID, 2015_1099704/2017_1141927)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	Portion analysed	DALA	Mefentrifluconazole Residues (mg/kg)
BadenWuerttemberg/ Kraichgau; Germany 2013, Asano (L130166)	2	153 156	- 12	204 208	49 69	Whole plant (no root)	0	3.5
						Ears	34	0.16
						Rest of plant (no roots)	34	2.1
Brandenburg; Germany 2013 Smaragd (L130167)	2	146 153	- 14	195 203	49 69	Whole plant (no root)	0	3.9
						Ears	34	0.38
							43	0.46
						Rest of plant (roots)	34	2.7
							43	4.8
Stetten a. H. (Kraichgau); Germany 2014 Asano (L140168)	2	152 146	- 18	202 194	49 69	Whole plant (no root)	0	2.6
						Ears	35	0.48
							42	0.41
						Rest of plant (no roots)	35	4.0
							42	4.2
	2	152 150	- 18	202 200	49 69	Whole plant (no root)	0	2.8
						Ears	35	0.46
							42	0.45
						Rest of plant (no roots)	35	2.8
							42	4.2
	2	147 153	- 18	196 204	49 69	Whole plant (no root)	0	3.1
						Ears	35	0.7
							42	0.40
						Rest of plant (no roots)	35	6.1
							42	5.2
Uedem, Germany 2014	2	160 155	- 21	213 207	49 69	Whole plant (no root)	0	2.5
						Ears	36	0.46

Mefentrifluconazole

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	Portion analysed	DALA	Mefentrifluconazole Residues (mg/kg)
Elixier (L140169)						Rest of plant (no roots)	36	0.78
	2	156 156	- 21	208 208	49 69	Whole plant (no root)	0	2.2
						Ears	36	0.38
						Rest of plant (no roots)	36	1.5
	2	148 156	- 21	197 208	49 69	Whole plant (no root)	0	2.2
						Ears	36	0.57
						Rest of plant (no roots)	36	1.1
Limburg, Gennep; The Netherlands 2013 Premio (L130168)	2	161 158	- 20	215 211	49 69	Whole plant (no root)	0	2.2
						Ears	34	1.6
							41	1.6
						Rest of plant (no roots)	34	0.42
							41	0.42
Ottersum, The Netherlands 2014 Tabsco (L140171)	2	154 150	- 21	205 200	49 69	Whole plant (no root)	0	2.4
						Ears	36	0.76
						Rest of plant (no roots)	36	3.5
	2	148 150	- 21	197 213	49 69	Whole plant (no root)	0	2.2
						Ears	36	0.70
						Rest of plant (no roots)	36	2.5
	2	152 146	- 21	203 195	49 69	Whole plant (no root)	0	2.0
						Ears	36	0.80
						Rest of plant (no roots)	36	3.1
Essex, United Kingdom 2013, Solstice (L130169)	2	152 136	- 31	203 181	49 69	Whole plant (no root)	0	2.8
Rouzières de Toraine, Northern France 2014 Atogi (L140170)	2	152 152	- 36	203 203	49 69	Whole plant (no root)	0	2.3
						Ears	35	0.60
						Rest of plant (no roots)	35	3.0
	2	148 155	- 36	197 207	49 69	Whole plant (no root)	0	2.6
						Ears	35	0.42
						Rest of plant (no roots)	35	2.6
	2	148 152	- 36	198 203	49 69	Whole plant (no root)	0	2.4
						Ears	35	0.39
						Rest of plant (no roots)	35	2.0
Midi-Pyrénées; Southern France, 2013 Tiepolo (L130170)	2	146 158	- 21	195 211	49 69	Whole plant (no root)	0	2.7
						Ears	35	0.10
							43	0.15
						Rest of plant (no roots)	35	0.46
							43	0.58
St. Soulan; Southern France 2014 Aprilio (L140174)	2	152 149	- 17	203 198	49 69	Whole plant (no root)	0	2.7
						Ears	34	0.16
							42	0.16
						Rest of plant (no roots)	34	0.91
							42	1.1
	2	150 148	- 17	200 197	49 69	Whole plant (no root)	0	3.2
						Ears	34	0.19
							42	0.16
						Rest of plant (no roots)	34	4.0
							42	1.3
	2	148 150	- 17	197 200	49 69	Whole plant (no root)	0	2.9
						Ears	34	0.30
							42	0.18
						Rest of plant (no roots)	34	1.7
							42	2.2
Agios Georgios; Greece	2	150	-	201	49	Whole plant (no root)	0	2.5

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	Portion analysed	DALA	Mefentrifluconazole Residues (mg/kg)
2014 Trofeo (L140175)		151	20	202	69	Ears	35	0.03
							43	0.061
						Rest of plant (no roots)	35	0.26
							43	0.60
	2	136 153	- 20	182 204	49 69	Whole plant (no root)	0	2.6
						Ears	35	0.063
							43	0.053
						Rest of plant (no roots)	35	0.29
							43	0.37
	2	150 151	- 20	200 202	49 69	Whole plant (no root)	0	3.8
						Ears	35	0.10
							43	0.091
						Rest of plant (no roots)	35	0.55
							43	0.50
Central Macedonia Pella; Greece 2013 Trofeo (L130171)	2	149 150	- 21	199 200	49 69	Whole plant (no root)	0	3.6
						Ears	35	0.25
							42	0.35
							49	0.34
						Rest of plant (no roots)	35	1.3
							42	2.2
							49	3.8
Emilia Romagna, Bologna; Italy 2013 Palassio (L130172)	2	151 151	- 14	201 202	49 69	Whole plant (no root)	0	2.4
						Ears	34	0.48
							42	0.71
						Rest of plant (no roots)	34	2.3
							42	2.1
S. Martino Olearo; Italy 2014 Avorio (L140176)	2	156 157	- 10	208 209	49 69	Whole plant (no root)	0	7.1
						Ears	34	0.40
						Rest of plant (no roots)	34	4.0
	2	157 154	- 10	209 205	49 69	Whole plant (no root)	0	6.6
						Ears	34	0.42
						Rest of plant (no roots)	34	3.2
	2	155 157	- 10	207 210	49 69	Whole plant (no root)	0	5.3
						Ears	34	0.6
						Rest of plant (no roots)	34	5.0
Andalusia, Sevilla; Spain 2013 Athur Nick (L130173)	2	154 152	- 14	205 203	49 69	Whole plant (no root)	0	6.3
						Ears	35	1.3
						Rest of plant (no roots)	35	12
Quintanar del Rey; Spain 2014 Adagio (L140173)	2	154 156	- 20	205 208	49 69	Whole plant (no root)	0	5.4
						Ears	35	3.5
							42	2.6
							49	0.93
						Rest of plant (no roots)	35	7.9
							42	8.8
							49	5.3
	2	153 153	- 20	204 204	49 69	Whole plant (no root)	0	4.8
						Ears	35	2.8
							42	2.3
							49	1.2
						Rest of plant (no roots)	35	10
							42	7.2
							49	6.6
	2	155	-	206	49	Whole plant (no root)	0	6.3

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	Portion analysed	DALA	Mefentrifluconazole Residues (mg/kg)
		152	20	203	69	Ears	35	3.7
							42	3.1
							49	2.1
						Rest of plant (no roots)	35	11
							42	13.2
							49	9.0
La Gineta; Spain 2014 Califa (L140177)	2	149 156	- 19	199 208	49 69	Whole plant (no root)	0	4.3
						Ears	35	0.22
							42	0.45
						Rest of plant (no roots)	35	1.7
							42	2.9
	2	150 148	- 19	200 198	49 69	Whole plant (no root)	0	3.9
						Ears	35	0.14
							42	0.19
						Rest of plant (no roots)	35	1.9
							42	2.6
	2	150 148	- 19	200 198	49 69	Whole plant (no root)	0	4.0
						Ears	35	0.091
							42	0.13
						Rest of plant (no roots)	35	1.2
							42	1.9

Barley Whole Plant, Ears, Rest of Plant – Europe

Table 131 Residues of mefentrifluconazole in barley whole plant, ears and rest of plant from trials conducted in Europe following application of EC or SC formulations (Teresiak, 2014, BASF DocID, 2014_1010808; Ale, 2015, BASF DocID 2015_1099703/2017_1101701)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	Portion analysed	DALA	Mefentrifluconazole (mg/kg)
RhinelandPalatinate, Rheinhessen; Germany, 2013, Popino (L130174)	2	153 141	- 16	204 188	49 69	Whole plant (no roots)	0	7.4
Brandenburg; Germany, 2013 Sandra (L130175)	2	146 146	- 14	195 195	49 69	Whole plant (no roots)	0	4.9
						Ears	27	0.082
							34	0.058
							42	0.065
						Rest of plant (no roots)	27	0.84
							34	0.52
							42	0.61
Mauchenheim; Germany 2014 Propino (L140158)	2	153 150	- 19	204 200	49 69	Whole plant (no roots)	0	3.6
						Ears	29	0.71
						Rest of plant (no roots)	29	3.6
	2	150 153	- 19	200 204	49 69	Whole plant (no roots)	0	3.6
						Ears	29	0.89
						Rest of plant (no roots)	29	3.0
	2	153	-	204	49	Whole plant	0	3.2

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	Portion analysed	DALA	Mefentrifluconazole (mg/kg)
		154	19	206	69	(no roots)		
						Ears	29	0.60
						Rest of plant (no roots)	29	3.1
Uedem; Germany 2014 Meridian (L140159)	2	144 155	- 29	192 207	49 69	Whole plant (no roots)	0	2.6
						Ears	27	0.42
						Rest of plant (no roots)	27	1.2
	2	155 151	- 29	207 202	49 69	Whole plant (no roots)	0	2.7
						Ears	27	0.33
						Rest of plant (no roots)	27	1.3
	2	154 145	- 29	205 193	49 69	Whole plant (no roots)	0	2.2
						Ears	27	0.15
						Rest of plant (no roots)	27	1.4
Limburg, Gennep; The Netherlands 2013 Squel (L130176)	2	149 163	- 27	199 217	49 69	Whole plant (no roots)	0	2.3
						Ears	28	1.2
						Rest of plant (no roots)	28	2.4
Ottersum; The Netherlands 2014 Sequel (L140160)	2	151 144	- 28	202 192	49 69	Whole plant (no roots)	0	2.9
						Ears	28	0.53
						Rest of plant (no roots)	28	1.6
	2	155 154	- 28	207 205	49 69	Whole plant (no roots)	0	2.7
						Ears	28	0.53
						Rest of plant (no roots)	28	2.1
	2	154 145	- 28	205 193	49 69	Whole plant (no roots)	0	2.8
						Ears	28	0.24
						Rest of plant (no roots)	28	1.3
Essex: United Kingdom 2013 Cassata (L130177)	2	152 147	- 23	203 196	49 69	Whole plant (no roots)	0	3.2
						Ears	28	0.71
						Rest of plant (no roots)	28	1.9
Ugley Green; United Kingdom 2014 Flagon (L140161)	2	151 151	- 24	202 201	49 69	Whole plant (no roots)	0	6.0
						Ears	29	0.93
						Rest of plant (no roots)	29	3.0
	2	150 158	- 24	200 211	49 69	Whole plant (no roots)	0	6.1
						Ears	29	1.3
						Rest of plant (no roots)	29	2.7

Mefentrifluconazole

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	Portion analysed	DALA	Mefentrifluconazole (mg/kg)
	2	152 148	- 24	201 198	49 69	Whole plant (no roots)	0	5.0
						Ears	29	1.1
						Rest of plant (no roots)	29	2.5
Saint Pierre de Chevillé; Northern France 2014 Sandra (L140162)	2	142 145	- 21	190 193	49 69	Whole plant (no roots)	0	2.5
						Ears	27	0.17
							34	0.19
						Rest of plant (no roots)	27	0.72
							34	0.86
	2	141 140	- 21	188 187	49 69	Whole plant (no roots)	0	2.0
						Ears	27	0.20
							34	0.26
						Rest of plant (no roots)	27	1.0
							34	1.5
	2	142 148	- 21	190 197	49 69	Whole plant (no roots)	0	2.4
						Ears	27	0.10
							34	0.13
						Rest of plant (no roots)	27	0.84
							34	1.2
Midi-Pyrénées; France 2013 Bamboo (L130178)	2	149 140	- 23	198 187	49 69	Whole plant (no roots)	0	3.4
						Ears	28	0.36
							34	0.13
							41	0.16
						Rest of plant (no roots)	28	0.49
							34	0.20
							41	0.18
Tournecoupe; Southern France 2014 Ketos (L140063)	2	162 150	- 16	217 200	49 69	Whole plant (no roots)	0	4.3
						Ears	28	0.46
							35	0.65
						Rest of plant (no roots)	28	2.4
							35	3.3
	2	145 152	- 16	193 203	49 69	Whole plant (no roots)	0	4.5
						Ears	28	0.43
							35	0.51
						Rest of plant (no roots)	28	2.0
							35	2.8
	2	145 155	- 16	193 207	49 69	Whole plant (no roots)	0	4.9
						Ears	28	0.41
							35	0.59
						Rest of plant (no roots)	28	2.0
							35	4.0
Central Macedonia Pella; Greece 2013 Moutso (L130179)	2	150 150	- 11	200 201	49 69	Whole plant (no roots)	0	7.2
						Ears	28	0.86
							35	1.0

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	Portion analysed	DALA	Mefentrifluconazole (mg/kg)
							42	1.7
						Rest of plant (no roots)	28	12
							35	12
							42	13
Prochoma; Greece 2014 Chill (L140164)	2	151 151	- 20	201 202	49 69	Whole plant (no roots)	0	5.6
						Ears	28	0.14
							36	0.31
						Rest of plant (no roots)	28	2.8
							36	2.8
	2	150 152	- 20	200 202	49 69	Whole plant (no roots)	0	5.3
						Ears	28	0.094
							36	0.26
						Rest of plant (no roots)	28	2.0
							36	2.8
	2	151 150	- 20	201 201	49 69	Whole plant (no roots)	0	5.5
						Ears	28	0.033
							36	0.18
						Rest of plant (no roots)	28	1.3
							36	1.4
Cuneo; Italy 2013 Cometa (L130180)	2	147 158	- 13	196 210	49 69	Whole plant (no roots)	0	3.9
						Ears	27	0.40
							34	0.31
							42	0.35
						Rest of plant (no roots)	27	1.8
							34	13
							42	1.9
Cassano D´Adda; Italy 2014 Atomo (L140165)	2	153 155	- 22	204 207	49 69	Whole plant (no roots)	0	4.4
						Ears	27	0.52
						Rest of plant (no roots)	27	2.3
	2	148 157	- 22	198 209	49 69	Whole plant (no roots)	0	4.9
						Ears	27	0.36
						Rest of plant (no roots)	27	2.2
	2	146 146	- 22	195 195	49 69	Whole plant (no roots)	0	3.4
						Ears	27	0.71
						Rest of plant (no roots)	27	2.3
Andalusia, Sevilla; Spain 2013, Prestige (L130181)	2	150 153	- 14	200 204	49 69	Whole plant (no roots)	0	5.7
Quintanar del Rey; Spain 2014 Acapulco (L140166)	2	154 150	- 20	205 200	49 69	Whole plant (no roots)	0	8.1
						Ears	28	3.9
							35	5.3
						Rest of plant (no roots)	28	20
							35	16

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	Portion analysed	DALA	Mefentrifluconazole (mg/kg)
	2	146 154	- 20	195 205	49 69	Whole plant (no roots)	0	9.2
						Ears	28	57
							35	6.0
						Rest of plant (no roots)	28	21
							35	21
	2	150 152	- 20	200 203	49 69	Whole plant (no roots)	0	6.7
						Ears	28	4.1
							35	3.7
						Rest of plant (no roots)	28	21
							35	20
La Gineta; Spain 2014 Hispanic (L140167)	2	142 150	- 24	190 200	49 69	Whole plant (no roots)	0	4.3
						Ears	28	0.29
							35	0.43
							41	0.33
						Rest of plant (no roots)	28	1.5
							35	1.6
							41	1.4
	2	146 151	- 24	195 201	49 69	Whole plant (no roots)	0	4.6
						Ears	28	0.23
							35	0.34
							41	0.40
						Rest of plant (no roots)	28	1.3
							35	2.0
							41	1.6
	2	146 148	- 24	195 198	49 69	Whole plant (no roots)	0	2.6
						Ears	28	0.17
							35	0.15
							41	0.13
						Rest of plant (no roots)	28	0.47
							35	0.56
							41	0.50

Sorghum Forage

Table 132 Residues of mefentrifluconazole in sorghum forage from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Pollard, AR; 2014, 53-67 (R140279)	2	151 151	- 14	189 189	85 87	22	74	0.19, 0.12 [0.16]	0.73, 0.46 [0.60]
Paynesville, MN; 2014 L655 (R140280)	2	151 151	- 14	191 180	85 87	21	71	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
Richland, IA; 2014 85Y40 (R140281)	2	149 151	- 14	229 235	85-87 87	21	79	0.43, 0.44 [0.44]	2.05, 2.10 [2.08]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Fisk, MO; 2014 M 3838C (R140282)	2	148 149	- 14	187 186	83-85 87	21	74	0.33, 0.33 [0.33]	1.27, 1.27 [1.27]
Hinton, OK; 2014 DKS29-28 (R140283)	2	147 150	- 14	117 130	75 85	21	76	0.79, 0.83 [0.81]	3.29, 3.46 [3.37]
Raymondville, TX; 2014 DKS 51-01 (R140284)	2	157 155	- 14	196 192	55 75-80	21	63	1.60, 1.66 [1.63]	4.32, 4.49 [4.41]
Grand Island, NE; 2014 A1005964 (R140285)	2	150 151	- 14	193 185	85 85	21	66	0.24, 0.28 [0.26]	0.70, 0.82 [0.76]
Levelland, TX; 2014 DKS44-20 (R140286)	2	152 150	- 13	190 187	56 81	21	68	1.64, 1.44 [1.54]	5.12, 4.50 [4.81]
Groom, TX; 2014 H-390W (R140287)	2	150 150	- 14	207 206	87 89	0	65	3.52, 3.72 [3.62]	10.06, 10.63 [10.34]
						14		1.11, 0.79 [0.95]	3.17, 2.26 [2.71]
						21		0.57, 0.63 [0.60]	1.63, 1.80 [1.72]
						28		0.41, 0.47 [0.44]	1.17, 1.34 [1.26]
						35		0.20, 0.32 [0.26]	0.57, 0.91 [0.74]

Maize Forage

Table 133 Residues of mefentrifluconazole in maize forage from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weigth
Alton, NY; 2014 232180 (R140247)	2	151 150	- 14	282 281	71 73	21	70	2.62, 1.67 [2.15]	8.73, 5.57 [7.15]
Hawkinsville, Georgia; 2014 Dekalb (R140248)	2	152 152	- 14	279 280	72-74 83-85	21	66	1.77, 1.04 [1.41]	5.20, 3.06 [4.13]
Delavan, WI; 2014 DKC 49-94RIB (R140249)	2	149 150	- 14	154 149	85 R5 86 R5	21	69	1.25, 0.69 [0.97]	4.03, 2.22 [3.13]
Gardner, ND; 2014 DKC33-53RIBAF2 (R140250)	2	147 147	- 15	138 138	76 78	21	69	1.45, 1.93 [1.69]	4.68, 6.22 [5.45]
Erie, ND; 2014 2Y188 (R140251)	2	155 153	- 14	145 143	85 87	21	65	1.28, 1.32 [1.30]	3.66, 3.71 [3.72]
Oregon, WI; 2014 DKC 49-29RIB (R140252)[A]	2	153 152	- 13	208 209	85 87	21	78	0.64, 0.63 [0.64]	2.91, 2.86 [2.89]
Oregon, WI; 2014 G96A69-3111 (R140253)[A]	2	153 152	- 13	209 208	85-87 87	21	78	0.89, 0.85 [0.87]	4.04, 3.86 [3.95]
Stafford, KS; 2014 Pioneer P1105AM (R140254)	2	147 152	- 14	165 170	83 84	20	70	0.55, 0.51 [0.53]	1.83, 1.70 [1.77]
St. Cloud, MN; 2014 DKC 38-03RIB (R140255)	2	149 150	- 14	186 188	87 87	21	70	1.40, 0.95 [1.18]	5.67, 3.17 [4.42]
York, NE; 2014 PO876CHR (R140256)[C]	2	149 150	- 14	188 181	69 89	21	75	0.43, 0.56 [0.50]	1.72, 2.24 [1.98]
Paynesville, MN; 2014 DK 1431 (R140257)[B]	2	150 151	- 14	187 189	69 89	0	84	0.24, 0.30 [0.27]	1.50, 1.88 [1.69]
						14		<0.01, <0.01	<0.01, <0.01

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weigth
								[<0.01]	[<0.01]
						21		<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
						28		<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
						35		<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
Paynesville, MN; 2014 DK 1431 (R140258)[B]	2	151 150	- 14	189 187	85 87	21	84	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
Geneva, MN; 2014 Pioneer 9834 (R140259)	2	150 149	- 14	155 135	83-85 85-87	21	74	0.54, 0.40 [0.47]	1.50, 1.11 [1.31]
Richland, IA; 2014 Pioneer P1498AM (R140260)	2	150 151	- 14	234 242	83-85 85-87	21	75	0.33, 0.30 [0.32]	1.32, 1.20 [1.26]
Hedrick, IA; 2014 Pioneer P1360HR (R140261)	2	151 151	- 14	146 242	83-85 85-87	21	74	0.39, 0.34 [0.37]	1.08, 0.94 [1.01]
Kirksville, MO; 2014 P1498AM (R140262)	2	151 151	- 14	208 135	83 85-87	21	73	0.66, 0.56 [0.61]	1.78, 1.51 [1.65]
Fisk, MO; 2014 RL8899YH B (R140263)	2	150 152	- 14	187 190	85 85	22	74	1.36, 1.18 [1.27]	3.78, 3.28 [3.53]
Aquilla, MO; 2014 DeKalb DKC63-87 (R140264)	2	152 150	- 14	190 187	85 85	21	78	0.71, 0.36 [0.54]	3.23, 1.64 [2.43]
York, NE; 2014 DK 59-90 RIB (R140265)[C]	2	151 150	- 14	222 220	87 87	19	73	0.75, 0.62 [0.69]	2.78, 2.30 [2.54]
East Bernard, TX; 2014 P1395AM (R140266)	2	151 147	- 14	154 143	83 84	21	58	2.16, 1.76 [1.96]	5.14, 4.19 [4.66]

Notes:

[A] Applications were made on the same day, rendering the trials dependent.

[B] Applications were separated by 2 days, rendering the trials dependent.

[C] Applications were separated by 1 day, rendering the trials dependent.

Sweet Corn Forage

Table 134 Residues of mefentrifluconazole in sweet corn forage from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005929)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Taber, Alberta; Canada, 2015 148-4 (R140574)	3	150 145 155	- 7 6	200 194 207	65 65 65-67	21	1.43, 1.92 [1.68]
Abbotsford, British Columbia; Canada, 2014 Honey and cream (R140577)	3	164 146 169	- 6 8	431 385 446	69 71 73	21	1.48, 2.69 [2.08]
North Rose, NY; United States, 2014 BC 0805 (R140565)	3	154 155 150	- 6 8	308 305 300	38 61 65	21	2.16, 2.28 [2.22]
Alton, NY; United States, 2014 Previous Gem (R140566)	3	150 150 150	- 7 7	281 281 280	51 55 63	21	1.41, 2.16 [1.78]
Chula, GA; United States, 2014 Passion II (R140567)	3	147 154	- 7	271 280	59 67	21	3.14, 3.24 [3.19]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
		147	7	275	73		
Newberry, FL; United States, 2014 Passion II (R140568)	3	153 154 151	- 7 7	195 188 197	59 63 69	21	1.58, 2.38 [1.98]
Delavan, WI; United States, 2014 NK 199 (R140569)	3	150 150 149	- 7 7	145 147 149	59 59 61	21	0.73, 0.76 [0.75]
Fitchburg, WI; United States, 2014 Overland (R140570)	3	152 150 153	- 7 6	238 213 180	39-59 61 71	21	3.05, 3.05 [3.05]
St. Cloud, MN; United States, 2014 Ambrosia (R140571)	3	151 151 150	- 7 7	188 189 188	51 65 71	21	1.97, 2.09 [2.03]
Paynesville, MN; United States, 2014 Ambrosia (R140572)	3	150 152 150	- 7 7	219 223 225	55-63 65-69 69-73	0 14 21 28 35	0.17, 0.29 [0.23] <0.01, 0.01 [0.01] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01] <0.01, <0.01 [<0.01]
York, NE; United States, 2014 276A (R140573)	3	150 151 151	- 7 7	188 188 188	59 65 67	21	2.69, 2.82 [2.76]
Fresno, CA; United States, 2014 Silver Queen (R140575)	3	150 149 149	- 7 7	188 187 187	65 71 73	21	2.84, 2.44 [2.64]
Aberdeen, ID; United States, 2014 Ambrosia (R140576)	3	150 145 154	- 7 7	144 140 148	37 37 61	21	1.26, 1.37 [1.32]

Grass forage

Table 135 Residues of mefentrifluconazole in grass animal feeds from trials conducted in Canada and the United States following applications of an EC formulation (Csinos, 2019, BASF DocID 2019_7002385)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
BERMUDA GRASS FORAGE							
Washington, LA; United States, 2018 Common (R180003)	3	152 151 152	- 14 14	187 196 206	16 16 37	0 14	14.25, 11.24 [12.75] 2.60, 2.88 [2.74]
Chula, GA; United States, 2018 Costal (R180004)	3	149 150 150	- 14 14	196 196 196	62 68 70	0 14	12.85, 9.37 [11.12] 4.41, 4.55 [4.48]
Madill, OK; United States, 2018 Costal (R180005)	3	151 154 156	- 14 14	187 168 196	32 36 51	0 14	32.44, 32.64 [32.54] 1.72, 1.27 [1.50]
Claude, TX; United States, 2018 Celebration (R180006)	3	151 148 151	- 14 14	196 196 196	Early Mid Late Vegetative	0 14	13.97, 16.42 [15.20] 5.88, 6.19 [6.04]
BLUE GRASS FORAGE							
North Rose, NY; United States, 2018 Kentucky (R180007)	3	155 149 152	- 13 14	178 168 168	Vegetative	0 7 14	9.41, 9.24 [9.33] 8.44, 8.12 [8.28] 2.88, 3.76 [3.32]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
						21	2.89, 3.86 [3.38]
						28	4.69, 3.55 [4.12]
Wolsey, SD; United States, 2018	3	149	-	196	15	0	21.43, 24.38 [22.91]
Kentucky		147	13	196	25	14	8.37, 5.93 [7.15]
(R180008)		148	15	196	27		
Grants Pass, OR; United States, 2019	3	149	-	196	33	0	6.67, 7.25 [6.96]
Kentucky		152	13	196	36	14	2.79, 2.70 [2.74]
(R180009)		152	14	196	41		
Idaho Falls, ID; 2018	3	152	-	196	32-35	0	17.29, 15.37 [16.33]
Sherman Big Blue		152	14	206	32-36	14	16.99, 13.80 [15.40]
(R180010)		150	14	206	36		17.48, 22.65 [20.07]
FESCUE FORAGE							
Gunton, MB; Canada, 2018	3	154	-	206	12	0	13.48, 17.74 [15.61]
Meadow		152	14	206	12		
(R180015)		151	14	206	31		
Shelbyville, IN; United States, 2018	3	148	-	187	Not recorded	0	11.56, 11.46 [11.51]
Pasture Fescue		149	14	187		14	3.54, 3.89 [3.71]
(R180011)		154	14	206			
Fresno, CA; United States,2018	3	149	-	196	Not recorded	0	9.02, 12.75 [10.88]
Tall		151	14	196		14	15.41, 10.91 [13.16]
(R180012)		150	14	196		14	5.45, 5.10 [5.28]
BROMEGRASS FORAGE							
Gardner, ND; United States, 2018	3	157	-	206	30	0	12.43, 13.79 [13.11]
Brome		151	14	196	50	14	5.87, 6.23 [6.05]
(R180013)		150	15	196	60		
Mitchell, NE; United States, 2018	3	147	-	178	35	0	29.70, 28.16 [28.93]
Smooth		150	14	178	37	14	11.77, 6.65 [9.21]
(R180014)		146	13	178	40		

Straw and hay of cereal grains (including pseudocereals)

Sorghum Stover

Table 136 Residues of mefentrifluconazole in sorghum stover from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Pollard, AR; 2014	2	149	-	186	85	22	76	0.80, 0.75 [0.78]	3.33, 3.12
53-67 (R140279)		149	14	187	87				[3.23]
Paynesville, MN; 2014	2	150	-	190	85	21	25	<0.01, <0.01	<0.01, <0.01
L655 (R140280)		150	14	191	87			[<0.01]	[<0.01]
Richland, IA; 2014	2	152	-	130	85-87	21	77	1.34, 1.56 [1.45]	5.83, 6.78
85Y40 (R140281)		151	14	228	87				[6.31]
Fisk, MO; 2014	2	149	-	186	83-85	21	73	0.34, 0.31 [0.33]	1.26, 1.15
M 3838C (R140282)		151	14	188	87				[1.21]
Hinton, OK; 2014	2	151	-	179	75	21	74	1.26, 1.16 [1.21]	4.85, 4.46
DKS29-28 (R140283)		151	14	204	85				[4.66]
Raymondville, TX; 2014	2	155	-	191	55	21	61	1.75, 1.43 [1.59]	4.49, 3.67
DKS 51-01 (R140284)		156	14	193	75-80				[4.08]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Grand Island, NE; 2014 A1005964 (R140285)	2	151 150	- 14	187 171	85 85	21	73	0.21, 0.47 [0.34]	0.78, 1.74 [1.26]
Levelland, TX; 2014 DKS44-20 (R140286)	2	151 145	- 13	189 182	56 81	21	65	1.30, 1.77 [1.54]	3.71, 5.06 [4.38]
Groom, TX; 2014 H-390W (R140287)	2	154 148	- 14	214 209	87 89	0	72	5.38, 7.35 [6.36]	19.21, 26.25 [22.73]
						14		2.47, 2.74 [2.61]	8.82, 9.78 [9.30]
						21		3.05, 2.06 [2.56]	10.89, 7.34 [9.12]
						28		2.51, 1.94 [2.22]	8.96, 6.93 [7.94]
						35		1.87, 2.82 [2.34]	6.68, 10.07 [8.38]

Maize Stover

Table 137 Residues of mefentrifluconazole in maize stover from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Alton, NY; 2014 232180 (R140247)	2	152 150	- 14	285 281	71 73	21	79	1.06, 1.14 [1.10]	5.05, 5.43 [5.24]
Hawkinsville, Georgia; 2014, Dekalb (R140248)	2	154 151	- 14	288 292	72-74 83-85	21	64	1.39, 1.26 [1.33]	3.86, 3.50 [3.68]
Delavan, WI; 2014 DKC 49-94RIB (R140249)	2	150 147	- 14	158 157	85 R5 86 R5	21	68	2.29, 1.77 [2.03]	7.16, 5.53 [6.34]
Gardner, ND; 2014 DKC33-53RIBAF2 (R140250)	2	149 148	- 15	139 139	76 78	21	74	1.70, 1.91 [1.81]	4.72, 5.31 [5.01]
Erie, ND; 2014 2Y188 (R140251)	2	152 150	- 14	142 140	85 87	21	65	2.92, 2.70 [2.81]	8.34, 7.71 [8.03]
Oregon, WI; 2014 DKC 49-29RIB (R140252)[A]	2	152 148	- 13	216 224	85 87	21	59	1.22, 1.24 [1.23]	2.98, 3.02 [3.00]
Oregon, WI; 2014 G96A69-3111 (R140253)[A]	2	148 147	- 13	210 222	85-87 87	21	41	1.73,1.73 [1.73]	2.93, 2.93 [2.93]
Stafford, KS: 2014 Pioneer P1105AM (R140254)	2	145 153	- 14	198 171	83 84	20	62	3.37, 4.01 [3.69]	8.87, 10.55 [9.71]
St. Cloud, MN; 2014 DKC 38-03RIB (R140255)	2	151 153	- 14	189 191	87 87	21	61	4.10, 4.37 [4.24]	10.51, 11.20 [10.86]
York, NE; 2014 PO876CHR (R140256)[C]	2	150 150	- 14	190 190	69 89	21	65	2.10, 1.53 [1.82]	6.00, 4.37 [5.19]
Paynesville, MN; 2014 DK 1431 (R140257)[B]	2	151 151	- 14	188 189	69 89	0	36	1.36, 1.67 [1.52]	2.12, 2.61 [2.37]
						14		0.05, 0.05 [0.05]	0.08, 0.08 [0.08]
						21		<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
						28		<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
						35		<0.01, <0.01	<0.01, <0.01

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
								[<0.01]	[<0.01]
Paynesville, MN; 2014 DK 1431 (R140258)[B]	2	151 152	- 14	188 189	85 87	21	21	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
Geneva, MN; 2014 Pioneer 9834 (R140259)	2	149 151	- 14	159 174	83-85 85-87	21	43	4.77, 6.55 [5.66]	8.37, 11.49 [9.93]
Richland, IA; 2014 Pioneer P1498AM (R140260)	2	150 153	- 14	325 349	83-85 85-87	21	75	2.28, 1.80 [2.04]	9.12, 7.20 [8.16]
Hendrick, IA; 2014 Pioneer P1360HR (R140261)	2	150 152	- 14	324 169	83-85 85-87	21	56	1.52, 1.30 [1.41]	3.45, 2.95 [3.20]
Kirkville, MO; 2014 P1498AM (R140262)	2	149 150	- 14	182 184	83 85-87	21	69	2.77, 2.25 [2.51]	8.94, 7.26 [8.10]
Fisk, MO; 2014 RL8899YH B (R140263)	2	149 149	- 14	186 187	85 85	22	49	5.41, 4.79 [5.10]	10.61, 9.39 [10.00]
Aquilla, MO; 2014 DeKalb DKC63-87 (R140264)	2	150 151	- 14	189 187	85 85	21	67	3.90, 4.28 [4.09]	11.81, 12.97 [12.39]
York, NE; 2014 DK 59-90 RIB (R140265)[C]	2	149 150	- 14	218 220	87 87	19	67	1.93, 2.23 [2.08]	5.85, 6.76 [6.30]
East Bernard, TX; 2014 P1395AM (R140266)	2	148 151	- 14	144 147	83 84	21	49	3.57, 4.92 [4.24]	7.00, 9.65 [8.33]

Notes:

[A] Applications were made on the same day, rendering the trials dependent.

[B] Applications were separated by 2 days, rendering the trials dependent.

[C] Applications were separated by 1 day, rendering the trials dependent.

Sweet Corn Stover

Table 138 Residues of mefentrifluconazole in sweet corn stover from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005929)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)	
							Fresh weight	Dry weight
Taber, Alberta; Canada, 2015 148-4 (R140574)	3	150 145 155	- 7 6	200 194 207	65 65 65-67	71	0.82, 0.57 [0.70]	0.99, 0.69 [0.84]
Abbotsford, British Columbia; Canada, 2014, Honey and cream (R140577)	3	164 146 169	- 6 8	431 385 446	69 71 73	55	1.48, 2.69 [2.08]	1.79, 3.26 [2.52]
North Rose, NY; United States, 2014 BC 0805 (R140565)	3	154 155 150	- 6 8	308 305 300	38 61 65	34	2.51, 2.58 [2.55]	3.04, 3.13 [3.09]
Alton, NY; United States, 2014 Previous Gem (R140566)	3	150 150 150	- 7 7	281 281 280	51 55 63	50	2.51, 2.58 [2.55]	3.04, 3.13 [3.09]
Chula, GA; United States, 2014 Passion II (R140567)	3	147 154 147	- 7 7	271 280 275	59 67 73	45	4.50, 2.42 [3.46]	5.45, 2.93 [4.19]
Newberry, FL; United States, 2014 Passion II (R140568)	3	153 154 151	- 7 7	195 188 197	59 63 69	35	0.89,1.15 [1.02]	1.08, 1.39 [1.24]
Delavan, WI; United States, 2014	3	150	-	145	59	51	0.36, 0.37	0.44, 0.45

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)	
							Fresh weight	Dry weight
NK 199 (R140569)		150 149	7 7	147 149	59 61		[0.37]	[0.45]
Fitchburg, WI; United States, 2014 Overland (R140570)	3	152 150 153	- 7 6	238 213 180	39-59 61 71	51	2.17, 3.27 [2.72]	2.63, 3.96 [3.30]
St. Cloud, MN; United States, 2014 Ambrosia (R140571)	3	151 151 150	- 7 7	188 189 188	51 65 71	64	2.54, 1.48 [2.01]	3.08, 1.79 [2.44]
Paynesville, MN; United States, 2014 Ambrosia (R140572)	3	150 152 150	- 7 7	219 223 225	55-63 65-69 69-73	52	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
						66	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
						73	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
						80	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
						87	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
York, NE; United States, 2014 276A (R140573)	3	150 151 151	- 7 7	188 188 188	59 65 67	41	1.30, 0.90 [1.10]	1.58, 1.09 [1.34]
Fresno, CA; United States, 2014 Silver Queen (R140575)	3	150 149 149	- 7 7	188 187 187	65 71 73	34	1.17, 1.95 [1.56]	1.42, 2.36 [1.89]
Aberdeen, ID; United States, 2014 Ambrosia (R140576)	3	150 145 154	- 7 7	144 140 148	37 37 61	42	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]

Wheat Hay

Table 139 Residues of mefentrifluconazole in wheat hay from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weigth
Minto, Manitoba; Canada 2015, Carberry (R140306)	2	151 153	- 14	163 161	11-12 29-30	20	24	1.32, 0.28 [0.80]	1.74, 0.37 [1.05]
Elgin, Manitoba; Canada 2015, Cardale (R140307)	2	153 151	- 14	307 303	69-71 83	21	21	0.21, 0.15 [0.18]	0.26, 0.19 [0.23]
Hague, Saskatchewan; Canada 2015, AC Vespar (R140308)	2	150 150	- 14	150 150	79-83 87-92	21	41	0.05, 0.59 [0.32]	0.08, 1.00 [0.54]
Kipp, Alberta; Canada, 2015 AC Carberry (R140309)	2	150 156	- 14	100 104	75-77 81-85	21	30	0.15, 0.14 [0.15]	0.21, 0.20 [0.21]
Fort Saskatchewan, Alberta; Canada, 2015, Harvest (R140310)	2	155 149	- 14	207 199	75 87	21	21	1.78, 1.58 [1.68]	2.25, 2.00 [2.12]
Alvena, Saskatchewan; Canada, 2015, Cardale (R140311)	2	146 148	- 14	174 176	75-76 83-85	21	26	3.12, 3.55 [3.34]	4.22, 4.80 [4.51]
Brandon, Manitoba; Canada 2015, Brandon (R140312)	2	151 148	- 14	101 99	77 87	21	20	2.09, 1.31 [1.70]	2.61, 1.64 [2.12]
Delisle, Saskatchewan; Canada, 2015, Marchwell (R140296)	2	152 154	- 14	152 154	71-73 83-85	21	36	0.09, 0.06 [0.08]	0.14, 0.09 [0.12]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weigth
Athens, GA; United States 2015, GA Gore (R140288)	2	152 154	- 14	291 290	65-69 77-83	21	39	2.08, 2.15 [2.12]	3.41, 3.52 [3.47]
Stuttgart, AR; United States, 2014, TV8848 (R140289)	2	152 154	- 14	152 153	45-53 69-71	21	39	2.72, 2.99 [2.86]	4.46, 4.90 [4.68]
Gardner, ND; United States 2015, Elgin (R140290)	2	151 156	- 14	189 196	70 85	21	31	2.44, 2.41 [2.43]	3.54, 3.49 [3.52]
St. Cloud, MN; United States 2014, Faller (R140291)	2	150 149	- 14	188 186	57 77	21	38	3.49, 3.59 [3.54]	5.63, 5.79 [5.71]
Paynesville, MN; United States 2014, Oklee (R140292)	2	151 150	- 14	191 190	35 87-89	21	63	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
Fisk, MO; United States 2015, Roane (R140293)	2	149 149	- 14	186 187	61 75	21	46	1.49, 1.40 [1.45]	2.76, 2.59 [2.68]
East Bernard, TX; United States, 2015, LA841 (R140294)	2	150 149	- 13	335 333	77 89	21	25	4.18, 4.69 [4.44]	5.57, 6.25 [5.91]
Grand Island, NE; United States, 2014, Prosper (R140295)	2	148 148	- 13	176 175	61 77	21	31	0.80, 0.97 [0.89]	1.16, 1.41 [1.29]
Jamestown, ND; United States 2015, Prosper (R140297)[A]	2	147 155	- 14	184 183	69 75	20	33	0.19, 0.03 [0.11]	0.28, 0.04 [0.16]
Jamestown, ND; United States 201, Divide (R140298)[A]	2	149 149	- 14	140 140	69 76-77	21	33	3.16, 4.32 [3.74]	4.72, 6.45 [5.58]
Hastings, NE; United States 2014, Prosper, (R140299)	2	150 150	- 14	220 221	71 87	0	41	21.58, 23.55 [22.56]	36.58, 39.92 [38.25]
						14		0.98, 1.46 [1.22]	1.66, 2.47 [2.07]
						21		0.92, 0.85 [0.89]	1.56, 1.44 [1.50]
						28		0.54, 0.62 [0.58]	0.92, 1.06 [0.99]
						35		0.43, 0.60 [0.52]	0.73, 1.02 [0.88]
Taber, Alberta; Canada, 2015, AC Carberry (R140300)	2	151 151	- 14	201 202	71-75 73-75	21	28	0.20, 0.25 [0.23]	0.28, 0.35 [0.32]
Wall, TX; United States, 2015 TAM 113 (R140301)	2	147 148	- 14	175 181	73 85	20	53	1.58, 1.85 [1.72]	3.36, 3.94 [3.65]
Groom, TX; United States 2015, TAM 111 (R140302)	2	148 151	- 14	238 247	73 79	21	52	0.33, 0.36 [0.35]	0.69, 0.75 [0.72]
Claude, TX; United States 2015, TAM 112 (R140303)	2	151 152	- 14	242 250	73 75	21	58	2.12, 2.37 [2.25]	5.05, 5.64 [5.35]
Lamed, KS; United States 2015, LCS Wizard (R140304)	2	150 151	- 14	169 169	85 85	21	61	2.07, 1.75 [1.91]	5.31, 4.49 [4.90]
Aberdeen, ID; United States 2014, Alturas (R140305)	2	154 147	- 14	141 145	75 85	21	24	4.02, 4.86 [4.44]	5.29, 6.39 [5.84]

Notes:

[A] Applications were separated by 4 days, rendering the trials dependent.

Barley Hay

Table 140 Residues of mefentrifluconazole in barley hay from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Minto, Manitoba; Canada, 2015, Newdale (R140242)	2	150 152	- 14	160 162	52-59 83-85	21	29	4.33, 4.57 [4.4]	5.35, 5.64 [5.50]
Hague, Saskatchewan; Canada, 2015, CDC Austenson (R140243)	2	152 150	- 14	152 150	77 85-87	21	30	0.47, 0.32 [0.40]	0.67, 0.46 [0.57]
Fort Saskatchewan, Alberta; Canada, 2015, Coalition (R140244)	2	149 151	- 14	199 201	73 77	21	20	5.40, 5.41 [5.41]	6.75, 6.76 [6.76]
Hepburn, Saskatchewan; Canada, 2015, CDC Austenson (R140245)	2	154 155	- 14	205 206	83 85	21	39	4.90, 3.87 [4.38]	8.03, 6.34 [7.19]
Carberry, Manitoba; Canada 2015, Conlon (R140246)	2	153 156	- 14	101 103	85 89	21	30	5.74, 5.66 [5.70]	8.20, 8.08 [8.14]
North Rose, NY; United States 2014, AC Minoa (R140237)	2	152 149	- 14	190 187	52 73	21	45	3.68, 6.30 [4.99]	6.69, 11.45 [9.07]
Grand Island, NE; United States 2014, Haybet (R140239)	2	151 150	- 14	131 194	55 81	21	21	4.06, 4.33 [4.20]	5.14, 5.48 [5.31]
Fresno, CA; United States, 2015, Helena barley (R140240)	2	150 150	- 14	188 187	87 89	21	57	4.85, 4.71 [4.78]	11.28, 10.95 [11.1]
Aberdeen, ID; United States 2014, Baroness (R140241)	2	156 153	- 14	153 146	75 85	21	19	6.21, 6.78 [6.50]	7.67, 8.37 [8.02]

Grass hay

Table 141 Residues of mefentrifluconazole in grass animal feeds from trials conducted in Canada and the United States following applications of an EC formulation (Csinos, 2019, BASF DocID 2019_7002385)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
BERMUDA GRASS HAY							
Washington, LA; United States, 2018, Common (R180003)	3	152 151 152	- 14 14	187 196 206	16 16 37	0 14	32.20, 32.13 [32.17] 6.96, 5.50 [6.23]
Chula, GA; United States, 2018 Costal (R180004)	3	149 150 150	- 14 14	196 196 196	62 68 70	0 14	18.46, 20.63 [19.55] 9.51, 10.87 [10.19]
Madill, OK; United States, 2018 Costal (R180005)	3	151 154 156	- 14 14	187 168 196	32 36 51	0 14	54.22, 51.00 [52.61] 2.37, 2.45 [2.41]
Claude, TX; United States, 2018 Celebration (R180006)	3	151 148 151	- 14 14	196 196 196	Early, Mid, Late Vegetative	0 14	32.49, 28.22 [30.36] 13.83, 14.42 [14.13]
BLUE GRASS HAY							
North Rose, NY; United States, 2018 Kentucky (R180007)	3	155 149 152	- 13 14	178 168 168	Vegetative Vegetative Vegetative	0 7 14 21 28	27.29, 26.39 [26.84] 21.35, 17.11 [19.23] 8.08, 10.63 [9.36] 4.94, 5.392[5.17] 4.92, 5.35 [5.13]
Wolsey, SD; United States, 2018, Kentucky (R180008)	3	149 147 148	- 13 15	196 196 196	15 25 27	0 14	64.05, 55.12 [59.59] 11.81, 13.51 [12.66]
Grants Pass, OR; United States 2019, Kentucky (R180009)	3	149 152 152	- 13 14	196 196 196	33 36 41	0 14	16.04, 18.71 [17.38] 7.26, 8.90 [8.08]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Idaho Falls, ID; United States	3	152	-	196	32-35	0	27.88, 38.78 [33.33]
2018, Sherman Big Blue		152	14	206	32-36	14	17.48, 22.65 [20.07]
(R180010)		150	14	206	36		27.29, 26.39 [26.84]
FESCUE HAY							
Gunton, MB; Canada, 2018	3	154	-	206	12	0	46.29, 46.61 [46.45]
Meadow		152	14	206	12		
(R180015)		151	14	206	31		
Shelbyville, IN; United States	3	148	-	187	Not recorded	0	20.79, 22.37 [21.58]
2018, Pasture Fescue		149	14	187		14	5.18, 5.98 [5.58]
(R180011)		154	14	206			
Fresno, CA; United States	3	149	-	196	Not recorded	0	24.41, 24.39 [24.40]
2018		151	14	196		14	17.17, 15.71 [16.44]
Tall (R180012)		150	14	196		14	12.65, 11.89 [12.27]
BROMEGRASS HAY							
Gardner, ND; United States	3	157	-	206	30	0	24.56, 25.64 [25.10]
2018		151	14	196	50	14	10.51, 12.17 [11.34]
Brome (R180013)		150	15	196	60		
Mitchell, NE; United States	3	147	-	178	35	0	37.02, 37.88 [37.45]
2018		150	14	178	37	14	35.68, 36.22 [35.95]
Smooth (R180014)		146	13	178	40		

Wheat straw – North America

Table 142 Residues of mefentrifluconazole in wheat straw from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID, 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Delisle, Saskatchewan;	2	154	-	154	71-73	21	30	7.80, 8.86	11.14, 12.66
Canada, 2015		149	14	149	83-85			[8.33]	[11.9]
Marchwell (R140296)									
Taber, Alberta; Canada	2	150	-	200	71-75	21	44	10.25, 8.77	18.30, 15.66
2015, AC Carberry		145	14	193	73-75			[9.51]	[16.98]
(R140300)									
Elgin, Manitoba; Canada	2	150	-	301	69-71	21	32	12.98, 11.62	19.09, 17.09
2015, Cardale (R140307)		150	14	299	83			[12.30]	[18.1]
Hague, Saskatchewan;	2	152	-	152	79-83	21	24	13.92, 14.01	18.32, 18.43
Canada, 2015, AC Vespar		150	14	150	87-92			[13.97]	[18.4]
(R140308)									
Kipp, Alberta; Canada	2	152	-	101	75-77	21	50	6.87, 7.42	13.74. 14.84
2015, AC Carberry		151	14	100	81-85			[7.14]	[14.29]
(R140309)									
Fort Saskatchewan, Alberta;	2	152	-	203	75	21	40	4.49, 4.07	7.48, 6.78
Canada, 2015		152	14	202	87			[4.28]	[7.13]
Harvest (R140310)									
Alvena, Saskatchewan;	2	151	-	180	75-76	21	22	8.55, 8.33	10.96, 10.68
Canada, 2015		148	14	177	83-85			[8.44]	[10.84]
Cardale (R140311)									
Brandon, Manitoba; Canada,	2	156	-	104	77	21	23	5.82, 7.64	7.56, 9.92
2015, Brandon (R140312)		149	14	99	87			[6.73]	[8.74]
Athens, GA; United States,	2	151	-	281	65-69	21	28	8.69, 8.77	12.07, 12.18

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
2015, GA Gore (R140288)		151	14	293	77-83			[8.73]	[12.13]
Stuttgart, AR; United States, 2014, TV8848 (R140289)	2	150 152	- 14	149 141	45-53 69-71	21	57	3.64, 5.45 [4.54]	8.46, 12.67 [10.56]
Gardner, ND; United States 2015, Elgin (R140290)	2	151 152	- 14	189 190	70 85	21	24	8.53, 11.06 [9.80]	11.22, 14.55 [13.4]
St. Cloud, MN; United States, 2014 , Faller (R140291)	2	150 150	- 14	188 187	57 77	21	25	20.11, 18.38 [19.25]	26.81, 24.51 [25.7]
Paynesville, MN; United States, 2014, Oklee (R140292)	2	150 150	- 14	191 191	35 87-89	21	23	<0.01, <0.01 [<.0.01]	<0.01, <0.01 [<.0.01]
Fisk, MO; United States 2015, Roane (R140293)	2	148 148	- 14	187 186	61 75	21	24	5.90, 4.44 [5.17]	7.76, 5.84 [6.80]
East Bernard, TX; United States, 2015, LA841 (R140294)	2	151 148	- 13	346 330	77 89	21	23	4.30, 3.91 [4.10]	5.58, 5.08 [5.33]
Grand Island, NE; United States, 2014 Prosper (R140295)	2	149 149	- 13	184 168	61 77	21	48	2.31, 2.33 [2.32]	4.44, 4.48 [4.46]
Jamestown, ND; United States, 2015, Prosper (R140297)[A]	2	147 148	- 14	184 185	69 75	20	18	11.84 (control) 14.88, 13.17 [14.02]	18.15, 16.06 [17.11]
Jamestown, ND; United States, 2015 Divide (R140298)[A]	2	156 147	- 14	146 137	69 76-77	21	26	12.22, 7.71 [9.96]	16.51, 10.42 [13.47]
Hastings, NE; United States 2014, Prosper (R140299)	2	151 151	- 14	221 222	71 87	0	28	7.96, 8.69 [8.32]	11.06, 12.07 [11.57]
						14		6.12, 6.07 [6.10]	8.50, 8.43 [8.47]
						21		4.55, 4.87 [4.71]	6.32, 6.76 [6.54]
						28		2.98, 3.51 [3.25]	4.14, 4.88 [4.51]
						35		3.14, 4.17 [3.66]	4.36, 5.79 [5.08]
Wall, TX; United States 2015, TAM 113 (R140301)	2	151 148	- 14	181 181	73 85	20	36	6.41, 5.69 [6.05]	10.02, 8.89 [9.46]
Groom, TX; United States, 2015, TAM 111 (R140302)	2	151 151	- 14	282 283	73 79	21	22	2.00, 2.90 [2.45]	2.56, 3.72 [3.14]
Claude, TX; United States, 2015, TAM 112 (R140303)	2	153 147	- 14	285 276	73 75	21	21	2.47, 1.87 [2.17]	3.13, 2.37 [2.75]
Lamed, KS; United States, 2015, LCS Wizard (R140304)	2	150 147	- 14	159 155	85 85	21	16	7.01, 10.29 [8.65]	8.34, 12.25 [10.30]
Aberdeen, ID; United States, 2014, Alturas (R140305)	2	151 145	- 14	143 138	75 85	21	28	2.36, 2.61 [2.49]	3.28, 3.62 [3.45]

Notes:

[A] Applications were separated by 10 days; rendering the trials dependent.

Wheat straw – Europe

Table 143 Residues of mefentrifluconazole in wheat straw (as received) from trials conducted in Europe following application of EC or SC formulations (Erdmann, 2015, BASF DocID, 2014_1010809; Ale, 2015,

BASF DocID, 2015_1099704/2017_1141927)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
BadenWuerttemberg/ Kraichgau; Germany, 2014, Asano (L130166)	2	153 156	- 12	204 208	49 69	43	3.9
Brandenburg; Germany, 2014 Smaragd (L130167)	2	146 153	- 14	195 203	49 69	49	5.5
Stetten a. H. (Kraichgau); Germany, 2014 Asano (L140168)	2	152 146	- 18	202 194	49 69	51	3.7, 3.4 [3.6]
	2	152 150	- 18	202 200	49 69	51	3.6, 3.6 [3.6]
	2	147 153	- 18	196 204	49 69	51	5.1, 4.7 [4.9]
Uedem, Germany 2014 Elixier (L140169)	2	160 155	- 21	213 207	49 69	42 49	1.3 1.6
	2	156 156	- 21	208 208	49 69	42 49	1.9 1.7
	2	148 156	- 21	197 208	49 69	42 49	1.3 1.9
Limburg, Gennep; The Netherlands, 2014 Premio (L130168)	2	161 158	- 20	215 211	49 69	49	2.3
Ottersum, The Netherlands 2014 Tabsco (L140171)	2	154 150	- 21	205 200	49 69	42 49	5.0 4.6
	2	148 150	- 21	197 213	49 69	42 49	4.3 4.4
	2	152 146	- 21	203 195	49 69	42 49	4.3 4.3
Essex: United Kingdom, 2014 Solstice (L130169)	2	152 136	- 31	203 181	49 69	35 42 50	10 8.6 6.2
Rouzières de Toraine, Northern France, 2014 Atogi (L140170)	2	152 152	- 36	203 203	49 69	42 49	3.4 3.1
	2	148 155	- 36	197 207	49 69	42 49	2.6 2.3
	2	148 152	- 36	198 203	49 69	42 49	2.2 2.1
Midi-Pyrénées; Southern France, 2014 Tiepolo (L130170)	2	146 158	- 21	195 211	49 69	46	0.50
St. Soulan; Southern France, 2014 Aprilio (L140174)	2	152 149	- 17	203 198	49 69	49	1.5, 1.5 [1.5]
	2	150 148	- 17	200 197	49 69	49	1.5, 1.6 [1.6]
	2	148 150	- 17	197 200	49 69	49	2.3, 2.4 [2.4]
Agios Georgios; Greece 2014 Trofeo (L140175)	2	150 151	- 20	201 202	49 69	50	0.46, 0.45 [0.46]
	2	136 153	- 20	182 204	49 69	50	0.53, 0.52 [0.52]
	2	150 151	- 20	200 202	49 69	50	0.81, 0.83 [0.82]
Central Macedonia Pella; Greece, 2014 Trofeo (L130171)	2	149 150	- 21	199 200	49 69	54	3.8
Emilia Romagna, Bologna; Italy, 2014 Palassio (L130172)	2	151 151	- 14	201 202	49 69	48	2.9
S. Martino Olearo; Italy, 2014	2	156	-	208	49	41	3.7

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray Volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Avorio (L140176)		157	10	209	69	48	4.6
	2	157 154	- 10	209 205	49 69	41 48	4.6 4.2
	2	155 157	- 10	207 210	49 69	41 48	4.1 3.9
Andalusia, Sevilla; Spain, 2014 Athur Nick (L130173)	2	154 152	- 14	205 203	49 69	43 49	9.9 18
Quintanar del Rey; Spain 2014 Adagio (L140173)	2	154 156	- 20	205 208	49 69	51	9.0, 8.6 [8.8]
	2	153 153	- 20	204 204	49 69	51	8.4, 8.7 [8.6]
	2	155 152	- 20	206 203	49 69	51	7.4, 6.6 [7.0]
La Gineta; Spain 2014 Califa (L140177)	2	149 156	- 19	199 208	49 69	49	3.1, 3.1 [3.1]
	2	150 148	- 19	200 198	49 69	49	2.5, 2.5 [2.5]
	2	150 148	- 19	200 198	49 69	49	1.4, 1.4 [1.4]

Barley Straw – North America

Table 144 Residues of mefentrifluconazole in barley straw from trials conducted in North America following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Minto, Manitoba; Canada 2015, Newdale (R140242)	2	150 152	- 14	160 162	52-59 83-85	21	41	8.94, 7.29 [8.12]	15.15, 12.36 [13.76]
Hague, Saskatchewan; Canada, 2015, CDC Austenson (R140243)	2	152 151	- 14	152 151	77 85-87	21	31	14.34, 14.50 [14.42]	20.78, 21.01 [20.90]
Fort Saskatchewan, Alberta; Canada, 2015 Coalition (R140244)	2	153 151	- 14	204 201	73 77	21	53	3.84, 2.99 [3.42]	8.17, 6.36 [7.27]
Hepburn, Saskatchewan; Canada, 2015 CDC Austenson (R140245)	2	145 144	- 14	193 192	83 85	21	32	14.72, 16.10 [15.41]	21.65, 23.68 [22.66]
Carberry, Manitoba; Canada 2015, Conlon (R140246)	2	154 155	- 14	103 103	85 89	21	19	6.96, 6.16 [6.56]	8.59, 7.60 [8.10]
North Rose, NY; United States 2014, AC Minoa (R140237)	2	150 152	- 14	187 190	52 73	21	33	3.38, 1.66 [2.52]	5.04, 2.48 [3.76]
Paynesville, MN; United States 2014 Robust (R140238)	2	152 151	- 14	191 190	85-87 87-89	0	30	1.28, 1.16 [1.22]	1.83, 1.66 [1.70]
						14		0.17, 0.25 [0.21]	0.24, 0.36 [0.30]
						21		<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
						28		<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
						35		<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
Grand Island, NE; United States, 2014, Haybet (R140239)	2	150 150	- 14	194 173	55 81	21	51	5.52, 5.60 [5.56]	11.26, 11.43 [11.30]
Fresno, CA; United States 2015, Helena barley (R140240)	2	149 152	- 14	187 190	87 89	21	17	13.06, 17.05 [15.06]	15.73, 20.54 [18.14]
Aberdeen, ID; United States 2014, Baroness (R140241)	2	155 152	- 14	147 145	75 85	21	19	1.54, 3.38 [2.46]	1.90, 4.17 [3.04]

Barley straw– Europe

Table 145 Residues of mefentrifluconazole in barley straw (as received) from trials conducted in Europe following application of EC or SC formulations (Teresiak, 2014, BASF DocID, 2014_1010808; Ale, 2015, BASF DocID 2015_1099703/2017_1101701)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Rhineland Palatinate, Rheinhessen; Germany 2013, Popino (L130174)	2	153 141	- 16	204 188	49 69	28	9.3
						35	15
						41	11
Brandenburg; Germany, 2013 Sandra (L130175)	2	146 146	- 14	195 195	49 69	53	1.0
Mauchenheim; Germany 2014 Propino (L140158)	2	153 150	- 19	204 200	49 69	35	5.9
						43	5.6
	2	150 153	- 19	200 204	49 69	35	6.8
						43	5.3
	2	153 154	- 19	204 206	49 69	35	5.0
						43	3.9
Uedem; Germany 2014 Meridian (L140159)	2	144 155	- 29	192 207	49 69	36	2.4
						41	2.5
	2	155 151	- 29	207 202	49 69	36	2.2
						41	3.1
	2	154 145	- 29	205 193	49 69	36	2.3
						41	2.1
Limburg, Gennep; The Netherlands, 2013, Sequel (L130176)	2	149 163	- 27	199 217	49 69	34	5.3
						41	5.6
Ottersum; The Netherlands 2014 Sequel (140160)	2	151 144	- 28	202 192	49 69	35	1.6
						41	4.3
	2	155 154	- 28	207 205	49 69	35	2.2
						41	3.6
	2	154 145	- 28	205 193	49 69	35	1.4
						41	2.2
Essex: United Kingdom, 2013 Cassata (L130177)	2	152 147	- 23	203 196	49 69	35	2.9
						41	3.9
Ugley Green; United Kingdom 2014 Flagon (L140161)	2	151 151	- 24	202 201	49 69	35	3.1
						42	2.7
	2	150 158	- 24	200 211	49 69	35	4.3
						42	3.7
	2	152 148	- 24	201 198	49 69	35	1.7
						42	1.7
Saint Pierre de Chevillé; Northern France 2014	2	142 145	- 21	190 193	49 69	41	0.99

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Sandra (L140162)	2	141 140	- 21	188 187	49 69	41	1.7
	2	142 148	- 21	190 197	49 69	41	1.2
Midi-Pyrénées; Southern France, 2013 Bamboo (L130178)	2	149 140	- 23	198 187	49 69	55	0.39
Tournecoupe; Southern France 2014 Ketos (L140063)	2	162 150	- 16	217 200	49 69	42	3.3
	2	145 152	- 16	193 203	49 69		2.4
	2	145 155	- 16	193 207	49 69		3.5
Central Macedonia Pella; Greece, 2013 Moutso (L130179)	2	150 150	- 11	200 201	49 69	54	6.4
Prochoma; Greece 2014 Chill (L140164)	2	151 151	- 20	201 202	49 69	43	1.9
	2	150 152	- 20	200 202	49 69		2.1
	2	151 150	- 20	201 201	49 69		1.2
Cuneo; Italy, 2013 Cometa (L130180)	2	147 158	- 13	196 210	49 69	48	4.2
Cassano D´Adda; Italy 2014 Atomo (L140165)	2	153 155	- 22	204 207	49 69	34 41	2.5 3.1
	2	148 157	- 22	198 209	49 69	34 41	2.8 4.6
	2	146 146	- 22	195 195	49 69	34 41	1.8 3.0
Andalusia, Sevilla; Spain 2013 Prestige (1L130181)	2	150 153	- 14	200 204	49 69	29 36 42	11 11 11
Quintanar del Rey; Spain 2014 Acapulco (L140166)	2	154 150	- 20	205 200	49 69	42	16
	2	146 154	- 20	195 205	49 69		18
	2	150 152	- 20	200 203	49 69		13
La Gineta; Spain, 2014 Hispanic (L140167)	2	142 150	- 24	190 200	49 69	48	1.9
	2	146 151	- 24	195 201	49 69		2.2
	2	146 148	- 24	195 198	49 69		0.62

Rice straw – United States

Table 146 Residues of mefentrifluconazole in rice straw from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Stuttgart, AR; 2014, CL 152 (R140267)[A]_	2	148 146	- 14	153 151	58 85	21	68	1.88, 1.74 [1.81]	5.88, 5.43 [5.66]
Stuttgart, AR; 2014, XL 745 Hybrid (R140268)[A]	2	150 145	- 14	154 150	58 86	23	61	2.48, 2.96 [2.72]	6.36, 7.59 [6.97]
Cheneyville, LA; 2014 Cheniere (R140269)	2	148 159	- 14	226 242	86 87	21	54	3.02, 2.94 [2.98]	6.56, 6.39 [6.48]
Glennonville, MO; 2014 CL 111 (R140270)	2	149 149	- 14	187 187	77 85	21	71	3.47, 3.21 [3.34]	8.90, 8.23 [8.56]
Pollard, AR; 2014 XL 729 (R140271)	2	152 149	- 14	189 186	45 69	21	77	1.81, 1.99 [1.90]	7.87, 8.65 [8.26]
Pocahontas, AR; 2014 CL XL745 (R140272)	2	150 149	- 14	188 186	43 65	21	74	2.25, 2.28 [2.27]	8.65, 8.77 [8.71]
Fisk, MO; 2014 CL XL745 (R140273)[B]	2	150 149	- 14	187 187	45 65	21	72	1.48, 1.82 [1.65]	5.28, 6.50 [5.89]
Fisk, MO; 2014 Francis (R140274)[B]	2	150 149	- 14	187 186	77 85	21	73	2.82, 2.18 [2.50]	10.44, 8.07 [9.26]
East Bernard, TX; 2014 Presidio (R140275)	2	150 150	- 14	159 159	65 75	0	75	2.19, 2.89 [2.54]	8.76, 11.56 [10.16]
						14		1.24, 1.34 [1.29]	4.96, 5.36 [5.16]
						21		0.96, 0.91 [0.94]	3.94, 3.64 [3.79]
						28		0.91, 1.01 [0.96]	3.64, 4.04 [3.84]
						35		1.09, 0.95 [1.02]	4.36, 3.80 [4.08]
Markham, TX; LX745 (R140276)	2	150 151	- 14	151 156	53 81	21	62	6.79, 6.55 [6.67]	17.87, 17.24 [17.55]
Willows, CA; 2014 M205 (R140277)	2	148 148	- 14	187 187	61 85	21	54	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]
Maxwell, CA; 2014 M205 (R140278)	2	149 149	- 14	187 187	61 85	21	61	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]

Notes:

[A] Applications were made on the same day, rendering the trials dependent.

[B] Applications were separated by 37 days; rendering the trials independent.

Rice husks and straw – China

Table 147 Residues of mefentrifluconazole in rice husks and straw from trials conducted in China following application of an SC formulation. (Xiaohu, 2019, BASF DocID. 2020_2095671)

Location; Year, Variety (Trial ID)	No.	Nominal Rate (g ai/ha)	Nominal RTI (days)	Nominal spray volume (L/ha)	BBCH	DALA	Portion analysed	Mefentrifluconazole (mg/kg)
Changchun City, Jilin Province; 2018 Jiudao 86	2	120 120	- 5	500	75	21	Rice husks	3.0, 3.1 [3.1]
							Straw	4.1, 3.0 [3.6]
					77	28	Rice husks	3.5, 1.7 [2.6]

Location; Year, Variety (Trial ID)	No.	Nominal Rate (g ai/ha)	Nominal RTI (days)	Nominal spray volume (L/ha)	BBCH	DALA	Portion analysed	Mefentrifluconazole (mg/kg)
							Straw	1.9, 3.2 [2.6]
Gaoyue Town, Huaibei City, Anhui Province; 2018 Xuyou 733	2	120 120	- 5	500	NS	7	Rice husks	6.7, 4.7 [5.7]
					NS	14	Rice husks	5.9, 5.0 [5.5]
					75	21	Rice husks	4.4, 4.6 [4.5]
							Straw	2.4. 1.7 [2.0]
					77	28	Rice husks	3.1, 3.1 [3.1]
							Straw	2.0, 2.5 [2.3]
					NS	35	Rice husks	3.5, 2.9 [3.2]
Eshan Town, Fanchang County, Anhui Province; 2018 Suxiu 867	2	120 120	- 5	500	75	21	Rice husks	4.3, 4.5 [4.4]
							Straw	2.8, 2.0 [2.4]
					77	28	Rice husks	3.0, 2.9 [3.0]
							Straw	2.6, 3.3 [2.9]
Fotang Town, Yiwu City, Zhejiang Province; 2018 Liangyou 189	2	120 120	- 5	500	75	21	Rice husks	6.0, 5.4 [5.7]
							Straw	6.9, 6.3 [6.6]
					77	28	Rice husks	4.1, 3.3 [3.7]
							Straw	3.2, 3.9 [3.5]
Huibu Town, Gao'an City, Jiangxi Province; 2018 Wanxiangyou 337	2	120 120	- 5	500	75	21	Rice husks	5.7, 9.1 [7.4]
							Straw	5.8, 8.5 [7.2]
					77	28	Rice husks	6.1, 6.3 [6.2]
							Straw	8.8, 9.3 [9.0]
Shizishan Street, Hongshan District, Wuhan, Hubei Province; 2018 Ejing 912	2	120 120	- 5	500	75	21	Rice husks	9.0, 9.0 [9.0]
							Straw	2.3, 2.4 [2.4]
					77	28	Rice husks	9.6, 9.2 [9.4]
							Straw	4.0, 4.3 [4.2]
Pingjiang Town, Yueyang City, Hunan Province; 2018 C Liangyou 4488	2	120 120	- 5	500	75	21	Rice husks	7.9, 8.8 [8.3]
							Straw	5.0, 4.0 [4.5]
					77	28	Rice husks	9.6, 9.2 [9.4]
							Straw	3.4, 5.6 [4.5]
Qidong County, Hengyang City, Hunan Province; 2018 C Liangyou 4488	2	120 120	- 5	500	NS	7	Rice husks	9.7, 8.1 [8.9]
					NS	14	Rice husks	7.8, 7.6 [7.7]
					75	21	Rice husks	9.2, 9.4 [9.3]
							Straw	3.2, 2.9 [3.1]
					77	28	Rice husks	5.0, 4.8 [4.9]
							Straw	1.8, 1.8 [1.8]
					NS	35	Rice husks	5.2, 4.7 [4.9]
Changshun County, Guizhou Province; 2018 Yixiang 2115	2	120 120	- 5	500	NS	7	Rice husks	3.5, 4.4 [4.0]
					NS	14	Rice husks	3.1, 3.3 [3.2]
					75	21	Rice husks	2.6, 2.5 [2.6]
							Straw	0.99, 0.96 [0.98]
					77	28	Rice husks	1.3, 1.2 [1.3]
							Straw	0.43, 0.55 [0.49]
					NS	35	Rice husks	2.0, 2.0 [2.0]
Xixiangtang District, Nanning City, Guangxi Zhuang Autonomous; 2018 Y Liangyou No. 2	2	120 120	- 5	500	75	21	Rice husks	2.5, 2.5 [2.5]
							Straw	1.5, 1.3 [1.4]
					77	28	Rice husks	1.3, 1.2 [1.3]
							Straw	0.63, 0.79 [0.71]
Haitou Town, Xiashan District, Zhanjiang City, Guangdong Province; 2018 Jinzao 09	2	120 120	- 5	500	NS	7	Rice husks	3.0, 2.9 [3.0]
					NS	14	Rice husks	5.6, 2.9 [4.3]
					75	21	Rice husks	3.0, 2.9 [3.0]
							Straw	0.12, 0.094 [0.11]
					77	28	Rice husks	2.5, 2.4 [2.5]
							Straw	0.12, 0.066 [0.09]
					NS	35	Rice husks	1.8, 1.7 [1.8]

Location; Year, Variety (Trial ID)	No.	Nominal Rate (g ai/ha)	Nominal RTI (days)	Nominal spray volume (L/ha)	BBCH	DALA	Portion analysed	Mefentrifluconazole (mg/kg)
Yongfa Town, Chengmai County, Hainan Province; 2018 Boyou 125	2	120 120	- 5	500	75	21	Rice husks	0.030, 0.14 [0.085]
							Straw	0.16, 0.11 [0.13]
					77	28	Rice husks	0.024, 0.016 [0.020]
							Straw	0.15, 0.082 [0.11]

Almond hulls

Table 148 Residues of mefentrifluconazole in almond hulls from trials conducted in the United States following application of an SC formulation. In each trial, an adjuvant was added. (Greenland, 2016, BASF DocID 2015_7005928)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	Mefentrifluconazole (mg/kg)
Orland, CA; 2014 Nonpareil (R140586)	3	148 148 148	- 7 7	701 701 701	81 83 85	14	0.28, 0.26 [0.27]
	3	149 149 148	- 7 7	1403 1403 1403	81 83 85	14	1.52, 1.80 [1.66]
Strathmore, CA; 2014 Fritz (R140587)	3	153 148 157	- 7 7	505 486 533	81 81 81	0	1.78, 2.10 [1.89]
						3	2.75, 2.83 [2.79]
						7	1.81, 1.68 [1.75]
						14	0.74, 0.75 [0.75]
						21	1.97, 1.67 [1.82]
	3	154 151 157	- 7 7	1553 1525 1590	81 81 81	0	2.03, 2.02 [2.03]
						3	2.24, 2.41 [2.33]
						7	1.12, 1.35 [1.24]
						14	1.33, 1.64 [1.49]
						21	0.95, 1.10 [1.03]
Wasco, CA; 2014 Nonpareil (R140588)[A]	3	149 151 151	- 9 5	711 701 683	81 81 81	14	0.91, 1.19 [1.05]
	3	151 150 147	- 9 5	1216 1188 1150	81 81 81	14	1.05, 1.16 [1.11]
Wasco, CA; 2014 Fritz (R140589)[A]	3	152 151 151	- 6 8	524 514 514	85 85 89	14	0.86, 1.25 [1.06]
	3	150 150 151	- 6 8	2843 2825 2881	85 85 89	14	1.74, 1.62 [1.68]
Fresno, CA; 2014 Monterey (R140590)	3	149 150 150	- 6 7	683 683 664	81 81 85-87	15	1.14, 1.12 [1.13]
	3	152 150 151	- 6 7	1590 1627 1721	81 81 85-87	15	0.71, 0.81 0.75]

Notes:

[A] Applications were separated by 8-11 days; rendering the trials independent.

Peanut hay

Table 149 Residues of mefentrifluconazole in peanut hay from trials conducted in the United States following application of an EC formulation. In each trial, an adjuvant was added. (Andrews, 2016, BASF DocID 2016_7006298)

Location; Year, Variety (Trial ID)	No.	Rate (g ai/ha)	RTI (days)	Spray volume (L/ha)	BBCH	DALA	% Moisture	Mefentrifluconazole (mg/kg)	
								Fresh weight	Dry weight
Elko, SC; 2014 Bailey (R140337)	3	198 201 200	- 14 14	210 210 212	75 75 77	14	25	6.69, 7.24 [6.96]	8.92, 9.65 [9.29]
Weston, GA; 2014 Georgia 06G (R140338)[A]	3	200 200 200	- 14 14	197 211 210	74 78 80	14	28	13.34, 7.26 [10.30]	18.53, 10.08 [14.30]
Weston, GA; 2014 Georgia 06G (R140339)[A, B]	3	200 200 200	- 14 14	197 212 211	72 77 80	14	26	4.35, 6.01 [5.18]	5.88, 8.12 [7.0]
Weston, GA; 2014 Georgia 06G (R140340)[B]	3	200 199 200	- 14 15	212 210 211	77 80 85	13	25	8.94, 4.41 [6.68]	11.92, 5.88 [8.90]
Chula, GA; 2014 Georgia 09B (R140341)	3	200 199 200	- 14 15	196 195 206	75 79 85	14	22	3.28, 3.65 [3.47]	4.20, 4.68 [4.44]
Abbeville, GA; 2014 Georgia 06G (R140342)	3	196 198 198	- 14 14	206 203 205	77 79 83	14	40	5.89, 6.86 [6.38]	9.82, 11.43 [10.63]
Ellenton, GA; 2014 Georgia 09B (R140343)	3	197 201 200	- 14 14	190 197 205	75 77 83	14	33	2.10, 2.09 [2.10]	3.13, 3.12 [3.13]
Winter Garden, FL; 2014 Georgia 06G (R140344)	3	198 202 197	- 14 14	235 239 233	79 86 88	14	30	4.78, 5.11 [4.95]	6.82, 7.30 [7.06]
East Bernard, TX; 2014 Georgia 09B (R140345)	3	198 196 199	- 14 15	207 204 209	71 73 79	14	49	14.16, 15.35 [14.76]	27.76, 30.10 [28.93]
Hinton, TX; 2014 Tamnut 0L06 (R140346)	3	199 197 197	- 13 16	252 213 229	79 81-83 81-83	14	28	2.43, 2.58 [2.51]	3.38, 3.58 [3.48]
Edmonson, TX; 2014 ACI149 (R140347)	3	197 197 194	- 14 14	253 267 262	81 84 86	14	27	10.98, 9.80 [10.39]	15.04, 13.42 [14.23]
Danville, GA; 2014 Georgia 06G (R140348)	3	199 200 198	- 14 14	234 235 233	73 75 77	8	20	8.40, 5.44 [6.92]	10.50, 6.80 [8.60]
						10		4.98, 7.75 [6.36]	6.22, 9.69 [7.95]
						14		5.52, 5.74 [5.63]	6.90, 7.18 [7.04]
						17		5.43, 3.84 [4.64]	6.79, 4.80 [5.80]
						22		3.46, 4.72 [4.09]	4.32, 5.90 [5.11]

Notes:

[A] Applications were made on the same day, rendering the trials dependent.

[B] Applications were separated by 14-15 days, rendering the trials dependent.

FATE OF RESIDUES IN STORAGE AND PROCESSING

Effects on the nature of the residue during processing

Radiolabelled [triazole-3(5)-^{14}C]-mefentrifluconazole and [chlorophenyl-U-^{14}C]-mefentrifluconazole were incubated in aqueous citrate-NaOH buffer solutions at concentrations of about 1 mg/L under three sets of conditions, each designed to simulate an appropriate process: 90 °C (pH 4, 20 minutes) to simulate pasteurisation, 100 °C (pH 5, 60 minutes), to simulate boiling, baking and brewing, and 120 °C (pH 6, 20 minutes in the dark) to simulate sterilisation (Hassink et al., 2014, BASF DocID 2014_1170665).

Total recovered radioactivity was measured for each test solution by LSC. Radioactive components were characterised by HPLC. Chiral analysis of mefentrifluconazole was also conducted using HPLC.

Under conditions representative of industrial and household processing procedures such as pasteurisation (pH 4, 90 °C, 20 min), baking, boiling, brewing (pH 5, 100 °C, 60 minutes) and sterilisation (pH 6, 120 °C, 20 minutes), mefentrifluconazole is shown to be stable. In addition, no degradation product exceeding 2 percent of total radioactivity was detected. Also, no change in the isomer ratio was observed. In conclusion, as mefentrifluconazole can be regarded as stable to hydrolysis, the nature of the residue is not affected by processing operations (Tables 150 and 151).

Table 150 Hydrolysis of mefentrifluconazole under simulated processing conditions

Compound	% total applied radioactivity			
	[triazole-3(5)-^{14}C]-mefentrifluconazole		[chlorophenyl-U-^{14}C]-mefentrifluconazole	
	Total	Mefentrifluconazole	Total	Mefentrifluconazole
pH 4, 90 °C, 20 mins				
Before test	100.0	Not analysed	100.0	Not analysed
After test	110.2	110.2	110.2	107.9
pH 5, 100 °C, 60 mins				
Before test	100.0	Not analysed	100.0	Not analysed
After test	110.2	110.2	108.3	107.1
pH 6, 120 °C, 20 mins				
Before test	100.0	Not analysed	100.0	Not analysed
After test	105.2	103.8	110.0	107.3

Table 151 Chiral analysis of mefentrifluconazole under simulated processing conditions

Compound	% total applied radioactivity							
	[triazole-3(5)-^{14}C]-mefentrifluconazole				[chlorophenyl-U-^{14}C]-mefentrifluconazole			
	Mefentrifluconazole		Other	Total	Mefentrifluconazole		Other	Total
	Isomer I	Isomer II			Isomer I	Isomer II		
pH 4, 90 °C, 20 mins								
Before test	51.2	48.8	-	100.00	49.1	50.1	0.8	100.0
After test	54.4	55.1	0.7	110.2	55.0	54.5	0.7	110.2
pH 5, 100 °C, 60 mins								
Before test	49.6	50.4	-	100.00	50.1	48.7	1.2	100.0
After test	55.7	54.5	-	110.2	52.9	55.4	-	108.3
pH 6, 120 °C, 20 mins								
Before test	50.1	49.9	-	100.0	48.4	50.2	1.5	100.0
After test	53.3	51.9	-	105.2	55.4	54.0	0.7	110.1

Residues after processing

Oranges

Three processing trials were carried out in the United States during the 2016 growing season (Bledsoe, 2017, BASF Reg. Doc. No. 2017_7008040) where oranges received three foliar airblast applications each at a rate of 736-754 g ai/ha, at 14-day intervals, of an EC formulation containing 100 g/L mefentrifluconazole. A nonionic surfactant (NIS) was added to the spray mixture for all applications. Mature oranges were harvested on the day of the last application (0 DALA) and transported to the processing facility under ambient conditions. At the processing facility, samples were processed within three days of receipt into juice, wet pomace, dried pomace, pulp, dried pulp, peel, peel after oil extraction, oil and marmalade according to commercial practices.

Washed oranges (6 kg) were hand peeled to generate the washed peel sample. A separate sample of washed oranges (190 kg) were then abraded for 45 seconds to scarify the flavedo for oil recovery. The scarified fruit was hand peeled to generate the peel after oil extraction sample. The remaining peeled, scarified fruit was used to make wet and dried pulp. To make wet and dried pulp, the peeled and scarified fruit (22 kg) was chopped using a food processor and fed through a pulper finisher.

The juice from the finisher was used to make marmalade. The pulp recovered from the finisher was pressed using a fruit press to form the wet pulp. A portion of the wet pulp was dried in an air dryer to < 10 percent moisture and the dried pulp was milled to a finished moisture content of 2.62–4.52 percent.

For the juice, scarified oranges (26 kg) were transferred to a juice extractor for juice removal. The collected juice was transferred to a pulper finisher and screened to remove the vesicular membranes, seeds, segment membranes, and peel fragments.

To make the oil, the collected oil-water emulsion from the scarification process was transferred to a sifter and screened to separate any flavedo fractions from the oil-water emulsion. The scarified flavedo fraction was used to make pomace. The oil-water emulsion was processed through a cream separator and centrifuged to separate the oil from the emulsion.

For pomace, peel from the juicing process was shredded using a food processor. The shredded peel was combined with the rag and seed from the juicing process and the scarified flavedo, lime (~95 percent CaO) was added and mixed, and then the limed pomace was pressed to generate the wet pomace sample. A portion of the wet pomace was dried in an air dryer to < 10 percent moisture and the dried pomace was milled to a finished moisture content of 2.62–4.36 percent.

To make the marmalade, orange rind was removed with a vegetable peeler and cooked for 20 minutes. An aliquot of juice from the pulp processing was cooked on a stove with 20 percent water added by weight. The cooked rind and juice were combined with lemon juice (3 tablespoons juice per kg fruit) and sugar (1.5 times the total weight of the fruit mixture) and boiled for 3 minutes. Pectin was added and the mixture was boiled for an additional 2 minutes to produce the finished marmalade which was packed into sterilized jars.

Residues of mefentrifluconazole in oranges and all processed commodities were determined using LC-MS/MS method D1511/01. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the oranges and processed commodities and analysis was 138 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 152.

Table 152 Mefentrifluconazole residues in orange and processed fractions with corresponding processing factors

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor[2]
RCN R160243 Florida, United States, 2016 Valencia 2246 g ai/ha DALA= 0	Whole fruit RAC[3]	0.56, 0.69 [0.63]	-
	Whole fruit RAC, preprocessing	0.48, 0.59 [0.54]	-
	Juice	<0.01, <0.01 [<0.01]	<0.02
	Wet pomace	0.69, 0.97 [0.83]	1.5
	Dried pomace	3.11, 3.55 [3.33]	6.2
	Pulp	0.01, 0.01 [0.01]	0.02
	Dried pulp	0.08, 0.07 [0.08]	0.15
	Peel	1.16, 1.66 [1.41]	2.6
	Peel after oil extraction	0.65, 0.86 [0.76]	1.4
	Oil	25.90, 15.34 [20.62]	38
	Marmalade	0.03[4], 0.07[4] [0.05]	0.09
RCN R160244, Florida, United States, 2016 Valencia 2225 g ai/ha DALA. = 0	Whole fruit RAC	0.68, 0.58 [0.63]	-
	Whole fruit RAC, preprocessing	0.70, 0.80 [0.75]	-
	Juice	<0.01, <0.01 [<0.01]	<0.01
	Wet pomace	1.65, 1.62 [1.64]	2.2
	Dried pomace	6.04, 6.00 [6.02]	8.0
	Pulp	0.03, <0.01 [<0.02]	<0.03
	Dried pulp	0.03, 0.03 [0.03]	0.04
	Peel	2.10, 2.77 [2.44]	3.3
	Peel after oil extraction	1.62, 1.94 [1.78]	2.4
	Oil	57.79, 48.76 [53.28]	71
	Marmalade	0.13[4], 0.04[4] [0.09]	0.12
RCN R160245 CA, United States, 2016 Werley 2251 g ai/ha DALA= 0	Whole fruit RAC	0.67, 0.87 [0.77]	-
	Whole fruit RAC, preprocessing	0.73, 0.57 [0.65]	-
	Juice	<0.01, 0.01 [<0.01]	<0.02
	Wet pomace	0.92, 1.34 [1.13]	1.7
	Dried pomace	4.03, 4.34 [4.19]	6.4
	Pulp	0.01, 0.01 [0.01]	0.02
	Dried pulp	0.07, 0.07 [0.07]	0.11
	Peel	1.80, 1.52 [1.66]	2.6
	Peel after oil extraction	0.96, 1.33 [1.15]	1.8
	Oil	26.64, 26.90 [26.77]	41
	Marmalade	0.30[4], 0.10[4] [0.20]	0.31

Notes:

[1] DALA = Days after last application.

[2] Average residue values of the preprocessed RAC (i.e. analysed just prior to processing) were used for the calculation of the processing factors.

[3] RAC = raw agricultural commodity.

[4] The result of multiple analyses of individual samples.

Apples

Three processing trials were carried out in the United States during the 2014 and 2015 growing seasons (Hummel, 2017, BASF DocID 2017_7000414) where apples received three foliar airblast applications each at a rate of 296-304 g ai/ha, at 6 to 8-day intervals, of an SC formulation containing 400 g/L mefentrifluconazole. An adjuvant was added to the spray mixture for all applications. Mature apples were harvested on the day of the last application (0 DALA) and transported to the processing facility under ambient conditions. At the processing facility, samples were processed within 4–9 days of receipt into

apple sauce, dried applies, canned apple, wet pomace, washed whole apples, apple juice, dried pomace, wash water, and fruit syrup according to commercial practices.

To produce washed whole apples and wash water, the fruit (25 kg) were first washed in a wire basket that was submerged multiple times in 19 L buckets of water. The wash water sample was collected directly from each water bucket.

For apple juice, wet and dry pomace, and fruit syrup, washed whole fruit samples were passed through a hammer mill to produce apple mash. The mash was separated into juice and wet pomace using a hydraulic press. A portion of the juice was heated in a kettle for 4 hours at 68–74 °C to produce fruit syrup. To pasteurize the juice, the juice sample was microwaves for 12 minutes at 100 percent power (equivalent to 85 °C on a 1200-watt microwave). To generate the dry pomace samples, a portion of the wet pomace was dried on a tray in a dehydrator for 18–20 hours at 57–63 ºC to a moisture content of approximately 10 percent.

To produce apple sauce, canned apple, and dried apple slices, washed whole apples were peeled, cut into quarter-inch slices, and cored. The quarter-inch slices were further cut in half. A portion of the apple slices were placed in a kettle with about 473 mL of water, heated to 71 °C for 20 minutes, and stirred to generate slices in syrup for canned fruit. Another portion of the apple slices was similarly placed in a kettle with about 473 mL of water, heated to 71 °C for 45 minutes, and mashed to generate apple sauce. The remaining apple slices were placed on dehydrator trays and dried at 66–71 °C for 20–24 hours to produce the dried apple slices with a moisture content of approximately 10 percent.

Residues of mefentrifluconazole in apples and all processed commodities were determined using LC-MS/MS method D1511/01. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the apples and processed commodities and extraction for analysis was 428 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 153.

Table 153 Mefentrifluconazole residues in apple and processed fractions with corresponding processing factors

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor[2]
RCN R140472 Wayne, NY, United States, 2014 Rome 899 g ai/ha DALA=0	Apple RAC[3]	0.73	-
	Processor RAC	0.48	-
	Washed whole apples	0.36	0.75
	Wash water	0.012	0.03
	Canned apples	0.026	0.05
	Fruit syrup	0.19	0.40
	Apple sauce	0.026	0.05
	Dried apples	0.15	0.31
	Apple juice	0.041	0.09
	Wet pomace	1.49	3.10
	Dried pomace	5.50	11.46
RCN R140473 Tehama, CA, United States, 2015 Summerfield 887 g ai/ha DALA=0	Apple RAC	0.06	-
	Processor RAC	0.08	-
	Washed whole apples	0.054	0.68
	Wash water	<0.01	<0.13
	Canned apples	<0.01	<0.13
	Fruit syrup	0.07	0.88
	Apple sauce	<0.01	<0.13
	Dried applies	0.02	0.25
	Apple juice	<0.01	<0.13

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor[2]
	Wet pomace	0.26	3.25
	Dried pomace	0.79	9.88
RCN R140474 Grant, WA, United States, 2014 Red Delicious 906 g ai/ha DALA=0	Apple RAC	0.78	-
	Processor RAC	0.73	-
	Washed whole apples	0.59	0.81
	Wash water	0.09	0.12
	Canned apples	0.18	0.25
	Fruit syrup	0.28	0.38
	Apple sauce	0.08	0.11
	Dried applies	0.24	0.33
	Apple juice	0.12	0.16
	Wet pomace	1.72	2.36
	Dried pomace	5.48	7.51

Notes:

[1] DALA = Days after last application.

[2] Mefentrifluconazole residue values of the Processed RAC (i.e. apples analysed just prior to processing) were used for the calculation of the processing factors.

[3] RAC = raw agricultural commodity.

Plums

Three processing trials were carried out in the United States during the 2014 and 2015 growing seasons (Hummel, 2017, BASF DocID 2017_7000415) where plums received three foliar airblast applications each at a rate of 298-307 g ai/ha, at 7-day intervals, of an SC formulation containing 400 g/L mefentrifluconazole. An adjuvant was added to the spray mixture for all applications. Mature plums were harvested on the day of the last application (0 DALA) and transported to the processing facility under ambient conditions. At the processing facility, samples were processed within 3 days of receipt into dried prunes, de-pitted plums, juice, puree, washed whole plums and wash water according to commercial practices.

To produce washed whole plums and wash water, the fruit (27 kg) were first washed in a wired basket that was submerged multiple times in 19L buckets of water. The wash water sample was collected directly from each water bucket. The remaining fruit was pitted to generate the de-pitted plum sample.

To make juice, puree, and prunes, portions of the washed pitted fruit (21 kg) was passed through an electric juice processor, passed through an electric food strainer/sauce maker, or dried on a tray in a dehydrator for 14-18 hours at 54-66 ºC to a moisture content of 15-18 percent, respectively.

Residues of mefentrifluconazole in plums and all processed commodities were determined using LC-MS/MS method D1511/01. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the plums and processed commodities and extraction for analysis was 603 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 154.

Table 154 Mefentrifluconazole residues in plum and processed fractions with corresponding processing factors

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole Residues (mg/kg)	Processing Factor[2]
RCN R140436 Wayne, NY,	Plum RAC[3]	0.570	-
	Processor RAC	0.413	-

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole Residues (mg/kg)	Processing Factor[2]
United States , 2014 Starking Delicious 903 g ai/ha DALA=0	Dried prune	1.061	2.57
	De-pitted plum	0.406	0.98
	Juice	0.033	0.08
	Puree	0.312	0.76
	Washed whole plum	0.446	1.08
	Wash water	0.017	0.04
RCN R140437 Tulare, CA, United States, 2014 French 913 g ai/ha DALA=0	Plum RAC	0.537	-
	Processor RAC	0.159	-
	Dried prune	0.678	4.26
	De-pitted plum	0.184	1.16
	Juice	0.032	0.20
	Puree	0.069	0.43
	Washed whole plum	0.185	1.16
	Wash water	<0.01	<0.06
RCN R140438 Grant, WA, United States, 2015 Early Italian 905 g ai/ha DALA=0	Plum RAC	0.275	-
	Processor RAC	0.209	-
	Dried prune	0.852	4.08
	De-pitted plum	0.235	1.12
	Juice	0.031	0.15
	Puree	0.116	0.56
	Washed whole plum	0.217	1.04
	Wash water	0.116	0.56

Notes:

[1] DALA = Days after last application.

[2] Mefentrifluconazole residue values of the Processed RAC (i.e. plums analysed just prior to processing) were used for the calculation of the processing factors.

[3] RAC = raw agricultural commodity.

Peach

Two processing trials were carried out in the United States during the 2017 growing season (Crawford, 2018, BASF DocID. No. 2018_7003950) where peaches received three foliar airblast applications each at a rate of 148–153 g ai/ha, at 7-day intervals, of an SC formulation containing 400 g/L mefentrifluconazole, applied either as a concentrated spray volume (591–764 L/ha) or dilute spray volume (1956–2304 L/ha). An adjuvant was added to the spray mixture for all applications. Mature peach (3.4 kg) were harvested on the day of the last application (0 DALA), frozen within 2.5 hours of collection, and shipped frozen by freezer truck to the processing facility ≤ 27 days later. At the processing facility, samples were processed by peeling whole peaches into peel and pulp sample.

Residues of mefentrifluconazole in peach fruit, peach pulp, and peach peel were determined using LC-MS/MS method D1511/01. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the peach and processed commodities and extraction was 250 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 155.

Table 154 Mefentrifluconazole residues in peach and processed fractions with corresponding processing factors

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor
R170049 Porterville, CA, United	Peach fruit (RAC)[2]	0.22, 0.12 [0.17]	-

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor
States, 2017) O'Henry 455 g ai/ha DALA=0	Peach peel	1.68, 1.26 [1.47]	8.65
	Peach pulp	0.048, 0.022 [0.035]	0.21
	Peach fruit (RAC)	0.25, 0.14 [0.20]	-
	Peach peel	1.98, 1.20 [1.59]	7.95
	Peach pulp	0.062, 0.046 [0.054]	0.27
R170104 Hinton, OK, United States, 2017 Red Haven 448 g ai/ha DALA=0	Peach fruit (RAC)	0.45, 0.42 [0.44]	-
	Peach peel	3.63, 3.56 [3.60]	8.18
	Peach pulp	0.021, 0.026 [0.024]	0.05
	Peach fruit (RAC)	0.30, 0.34 [0.32]	-
	Peach peel	2.19, 2.59 [2.39]	7.47
	Peach pulp	0.015, 0.013 [0.014]	0.04

Notes:

[1] DALA = Days after last application.

[2] RAC = raw agricultural commodity.

Grapes

Three processing trials were carried out in Germany during 2014 (Plier et al., 2016, BASF DocID 2014_1315284) where wine grapes received two foliar applications each at a rate of 430–460 g ai/ha, at 21–36-day intervals, of an EC formulation containing 100 g/L mefentrifluconazole. Mature grapes were harvested on the day of the last application (0 DALA, not discussed further) and 21 DALA and transported to the processing facility under ambient conditions. At the processing facility, grape samples were processed into must (naturally cloudy, deposit, separated), pomace, pasteurized juice, yeast deposit, wine, stalks, crush, and raisins according to commercial practices.

For grapes used to make rosé wine, juice was prepared by crushing unwashed grapes (51 kg) in a grape mill. The mash (stalks, flesh, skin, seed and juice) was pressed to extract the liquid. Samples of must naturally cloudy and pomace were taken. Subsequently, $K_2S_2O_5$ (SO_2) was added to the must as a preservative prior to clarification (natural sedimentation). After clarification, samples of must deposit and must were separated and collected. The raw juice was then pasteurized (83–87 °C for 2 minutes), bottled and sampled.

For grapes used to make red wine, juice was prepared by crushing red grape bunches (51 kg) in a stalk separator mill which removed the stalks from the rest of the mash. Stalks and crush were sampled. Subsequently, $K_2S_2O_5$ (SO_2) was added. Following crushing, the mash was heated up to 60 °C for two minutes and stirred. After cooling down, the mash was pressed to extract the liquid. Samples of must naturally cloudy and pomace were taken. After clarification must deposit and must were separated and sampled. The raw juice was then pasteurized (83–87 °C for 2 minutes), bottled and sampled.

For the vinification process (both rosé and red wine), raw juice was poured into glass vessels and pure culture yeast and nutrient salt was added to begin fermentation. After fermentation and clarification with $K_2S_2O_5$ (SO_2), the intermediate liquid was transferred to a new vessel and the yeast deposit was sampled. Bentonite was added to liquid to absorb proteins and $K_2S_2O_5$ (SO_2) was added. The resultant young wine was filtered after clarification and then bottled for maturation and stored at approximately 8 ºC. After maturation, the wine was sampled.

For the production of raisins, washed grape bunches were put in boiling water and then manually washed with cold tap water in a vessel. The bunches were dried in an oven (70 °C) for 23:20–26:00 hours until a moisture content of 10.08–10.90 percent was achieved. After drying, raisins were manually

removed from the stalks. Raisins and stalks were sampled.

Residues of mefentrifluconazole in grapes and all processed commodities were determined using LC-MS/MS method L0076/09. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the grapes and processed commodities and extraction was 518 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 156.

Table 156 Mefentrifluconazole residues in grape and processed fractions with corresponding processing factors

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole Residues (mg/kg)	Processing Factor[2]
L140390 Traustadt, Germany, 2014 Spät Burgunder (blauer) 870 g ai/ha DALA=21	Grapes	5.7	-
	Grapes RAC[3] before processing	4.8	-
	Rosé Wine Making	Rosé Wine Making	
	Must naturally cloudy	0.54	0.11
	Pomace	15	3.13
	Must deposit	2.1	0.44
	Must separated	0.32	0.07
	Pasteurized juice	0.21	0.04
	Yeast deposit	1.7	0.35
	Rosé wine	0.10	0.02
	Red Wine Making	Red Wine Making	
	Stalks	7.4	1.54
	Crush	7.8	1.63
	Must naturally cloudy	1.0	0.21
	Pomace	25	5.21
	Must deposit	1.8	0.38
	Must separated	0.76	0.16
	Pasteurized juice	0.56	0.12
	Yeast deposit	4.8	1.0
	Red wine	0.16	0.03
	Raisins	12	2.5
	Stalks (raisins)	19	3.96
L140391 Meißen, Germany, 2014 Domina 890 g ai/ha DALA=21	Grapes	8.3	-
	Grapes RAC before processing	6.1	-
	Rosé Wine Making	Rosé Wine Making	
	Must naturally cloudy	0.88	0.14
	Pomace	24	3.93
	Must deposit	4.6	0.75
	Must separated	0.35	0.06
	Pasteurized juice	0.31	0.05
	Yeast deposit	3.3	0.54
	Rosé wine	0.12	0.02
	Red Wine Making	Red Wine Making	
	Stalks	10	1.64
	Crush	9.4	1.54
	Must naturally cloudy	1.0	0.16
	Pomace	26	4.26
	Must deposit	1.2	0.20
	Must separated	0.84	0.14
	Pasteurized juice	0.79	0.13
	Yeast deposit	6.8	1.11
	Red wine	0.12	0.02

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole Residues (mg/kg)	Processing Factor[2]
	Raisins	24	3.93
	Stalks (raisins)	43	7.05
L140392 Kesten, Germany, 2014 Dornfelder 920 g ai/ha DALA=21	Grapes	0.9	-
	Grapes RAC before processing	1.1	-
	Rosé Wine Making	Rosé Wine Making	
	Must naturally cloudy	0.14	0.13
	Pomace	3.4	3.09
	Must deposit	0.98	0.89
	Must separated	0.079	0.07
	Pasteurized juice	0.059	0.05
	Yeast deposit	0.83	0.75
	Rosé wine	0.028	0.03
	Red Wine Making	Red Wine Making	
	Stalks	2.0	1.82
	Crush	1.1	1.00
	Must naturally cloudy	0.2	0.18
	Pomace	3.9	3.55
	Must deposit	0.2	0.18
	Must separated	0.15	0.14
	Pasteurized juice	0.14	0.13
	Yeast deposit	1.3	1.18
	Red wine	0.032	0.03
	Raisins	4.1	3.73
	Stalks (raisins)	6.9	6.27

Notes:

[1] DALA = days after last application.

[2] The processing factors were calculated by dividing the residue in the processed fraction by the residue for the RAC sample (processor RAC).

[3] RAC = raw agricultural commodity.

Strawberries

Three processing trials were carried out in Germany during the 2020 growing season (Plier *et al*., 2021, BASF DocID 2020/2108050) where strawberries received three foliar applications each at a rate of 315–342 g ai/ha, at 7 day intervals, of a suspension concentrate with 75 g/L mefentrifluconazole. Mature strawberries (BBCH 87) were harvested on the final day of application (0 DALA) and one day after the last application (1 DALA). Strawberry RAC samples were shipped at ambient temperature by car from the test facilities to the processing facility where they were stored under cooled conditions until processing. Samples were processed according to commercial practices into washed strawberries, wash water, canned strawberries, fruit syrup, jam before cooking, and jam after cooking.

Strawberry fruits without stems were manually washed in tap water for 3 minutes. Samples of wash water and washed strawberries were collected and placed into frozen storage until analysis.

For the production of jam, sugar (400 g) and glucose-syrup (800 g) were mixed with washed strawberries (900 g) and boiled for ~ 8–14 minutes until a dry substance of 63–65 percent was reached. Citric acid (8 g) and a 5 percent pectin solution (8 g pectin in 0.16 L water) were then added and the mixture was boiled until a dry substance of 60–62 percent was achieved. The jam was left to cool down for 120 minutes. The pH of the final product was 2.99–3.07. Samples of jam before cooking and jam after cooking (after cool down period) were taken and placed into frozen storage until analysis.

For the production of canned strawberries and fruit syrup, water (0.90 L), ascorbic acid (3.81 g), citric acid (2.25 g), and sugar (540–600 g) were mixed with washed strawberries (3000 g) and boiled for 30 seconds. The mixture was poured into jars and then filled with the water used for cooking until 2 cm below the rim. The jars were closed and placed into an autoclave at 91 °C for 2 minutes. Following pasteurization, the jars were allowed to cool for 120 minutes. The pH of the final product was 3.31–3.58. Samples of fruit syrup and canned strawberries were collected and placed into frozen storage until analysis.

Residues of mefentrifluconazole in strawberries and all processed commodities were determined using LC-MS/MS method L0076/09 (Plier *et al*., 2021, BASF DocID 2020/2108050). The limit of quantification (LOQ) was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the strawberries and processed commodities and extraction was 166 days. All samples were analysed within 7 days of extraction. Processing factors are shown in Table 157.

Table 157 Concentration of mefentrifluconazole residues in strawberry processed fractions and processing factors

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor
L200326 Gerichshain, Saxony, Germany, 2020 Elsanta 1007 g ai/ha	Strawberry, Field, 0 DALA	0.36	-
	Strawberry, Field, 1 DALA	0.19	-
	Strawberry, Processor RAC[3]	0.27	-
	Washed strawberries	0.32	1.19
	Wash water	0.17	0.63
	Canned strawberries	0.25	0.93
	Fruit syrup	0.054	0.20
	Jam before cooking	0.13	0.48
	Jam after cooking	0.13	0.48
L200327 Ebrach, Bavaria, Germany, 2020 Elsanta 983 g ai/ha	Strawberry, Field, 0 DALA	0.77	-
	Strawberry, Field, 1 DALA	0.77	-
	Strawberry Processor RAC[3]	0.75	-
	Washed strawberries	0.45	0.60
	Wash water	0.38	0.51
	Canned strawberries	0.58	0.77
	Fruit syrup	0.13	0.17
	Jam before cooking	0.16	0.21
	Jam after cooking	0.19	0.25
L200328 Motterwitz, Saxony, Germany, 2020 Elsanta 969 g ai/ha	Strawberry, Field, 0 DALA	0.83	-
	Strawberry, Field, 1 DALA	0.70	-
	Strawberry Processor RAC[3]	0.56	-
	Washed strawberries	0.39	0.70
	Wash water	0.37	0.66
	Canned strawberries	0.66	1.18
	Fruit syrup	0.17	0.30
	Jam before cooking	0.21	0.38
	Jam after cooking	0.24	0.43

Notes:

[1] DALA = Days after last application.

[2] Mefentrifluconazole residues values of the Processor RAC (i.e. strawberry samples analysed prior to processing) were used for the calculation of the processing factors.

[3] RAC = raw agricultural commodity, representative sub-samples were collected prior to processing.

Cucumbers

Three processing trials were carried out in Germany during the 2020 growing season (Plier et al., 2021, BASF DocID 2020/2108051) where cucumbers received three foliar applications each at a rate of 317–365 g ai/ha, at 6–7 day intervals, of a suspension concentrate with 75 g/L mefentrifluconazole. Fruits (BBCH 74–79) were harvested on the final day of application (0 DALA) and three days after the last application (3 DALA). Cucumber RAC samples were shipped at ambient temperature by car from the test facilities to the processing facility where they were processed immediately. Samples were processed according to commercial practices into canned gherkins, vegetable stock, wash water, washed gherkins, pickled gherkins, and brine.

Cucumber fruits without stems were manually washed in tap water for 3 minutes. Samples of wash water and washed gherkins were collected and placed into frozen storage until analysis.

For the production of pickled gherkins, a 5 percent (w/w) sodium chloride solution mixed with 1 percent (w/w) sugar was briefly boiled and then cooled down. This solution was filled into earthenware crocks containing washed cucumbers, which were weighted down. The crocks were closed and stored for 3 days at room temperature and subsequently 42–43 days at cooled conditions of 5–11 °C. Samples of brine and pickled gherkins were taken and placed into frozen storage until analysis.

For the production of canned gherkins and vegetable stock, water (2000 g), vinegar (800 g of 10 percent acid), salt (200 g), and sugar (400 g) were mixed and boiled for 30 seconds. The mixture was filled into jars containing washed cucumbers until 2 cm below the rim. The jars were closed and placed into an autoclave at 118–120 °C for 8–15 minutes. Following sterilization, the jars were allowed to cool for 60 minutes. Samples of vegetable stock and canned gherkins were collected and placed into frozen storage until analysis.

Residues of mefentrifluconazole in cucumbers and all processed commodities were determined using LC-MS/MS method L0076/09 (Plier et al., 2021, BASF DocID 2020/2108051). The limit of quantification (LOQ) was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the cucumbers and processed commodities and extraction was 140 days. All samples were analysed within 7 days of extraction. Processing factors are shown in Table 158.

Table 158 Concentration of mefentrifluconazole residues in cucumber processed fractions and processing factors

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor
L200346 Gerichshain, Saxony, Germany/ 2020 Conny F1 995 g ai/ha	Cucumber, Field, 0 DALA	0.11	-
	Cucumber, Field, 3 DALA	0.066	-
	Cucumber, Processor RAC[3]	0.056	-
	Canned gherkins	0.097	1.73
	Vegetable stock	< 0.010	< 0.18
	Wash water	0.026	0.46
	Washed gherkins	0.016	0.29
	Pickled gherkins	0.041	0.73
	Brine	< 0.010	< 0.18
L200347 Ebrach, Bavaria, Germany, 2020 Diamant F1 999 g ai/ha	Cucumber, Field, 0 DALA	0.36	-
	Cucumber, Field, 3 DALA	0.11	-
	Cucumber, Processor RAC[3]	0.21	-
	Canned gherkins	0.14	0.67
	Vegetable stock	0.013	0.06
	Wash water	0.058	0.28
	Washed gherkins	0.11	0.52

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor
	Pickled gherkins	0.054	0.26
	Brine	< 0.010	< 0.05
L200348 Motterwitz, Saxony, Germany, 2020 Conny F1 1009 g ai/ha	Cucumber, Field, 0 DALA	0.16	-
	Cucumber, Field, 3 DALA	0.13	-
	Cucumber, Processor RAC[3]	0.075	-
	Canned gherkins	0.066	0.88
	Vegetable stock	0.010	0.13
	Wash water	0.033	0.44
	Washed gherkins	0.039	0.52
	Pickled gherkins	0.063	0.84
	Brine	< 0.010	< 0.13

Notes:

[1] DALA = Days after last application.

[2] Mefentrifluconazole residues values of the Processor RAC (i.e. cucumber samples analysed prior to processing) were used for the calculation of the processing factors.

[3] RAC = raw agricultural commodity, representative sub-samples were collected prior to processing.

Tomatoes

Three processing trials were carried out in the United States during the 2016 growing season (Reeves, 2018, BASF DocID 2018/7005677) where tomatoes received three foliar applications each at a rate of 445–462 g ai/ha, at 6 to 8 day intervals, of an emulsifiable concentrate with 100 g/L mefentrifluconazole. An adjuvant (non-ionic surfactant) was added to the spray mixture for all applications. Tomato RAC samples and a single tomato fruit bulk sample were harvested on the final day of application (0 DALA) at the fruit ripening to full maturity growth stages (BBCH 82-89). Bulk tomato samples were shipped at ambient temperature within 24 hours of collection by overnight courier from the test facilities to the processing facility where they were stored under refrigerated conditions until processing. Samples were processed according to commercial practices into blanched tomatoes, blanching water, canned tomatoes, ketchup after pasteurization, paste, peeled tomatoes, puree, raw juice, sun-dried tomatoes, tomato peel, vegetable stock, wash water, washed tomatoes, and wet pomace.

Tomatoes were sorted by culling unsuitable fruits. Tomatoes to be processed were placed in water at temperatures of ~ 52–57 °C for 3–5 minutes and then rinsed with water. Defects and off-coloured areas were trimmed off prior to processing. Samples of wash water and washed tomatoes were collected and placed into frozen storage until analysis.

For the production of juice, paste, and puree, washed tomatoes were chopped in a Hobart food chopper and passed over a 6/64" screen. Material passing through the screen was pumped through a heat exchanger at ~ 90–97 °C and then passed through a 0.033" screen. Pulp material that did not pass through both screens was combined and collected as the representative sample of wet pomace. Tomato juice filtering through the 0.033" screen was checked for percent solids and acidity, and then adjusted accordingly to a pH of 4.5 or less with a 0.5 percent citric acid solution. Samples of raw juice were collected and placed into frozen storage until analysis. Tomato juice was used to produce puree and paste. Puree was generated by evaporating water from the tomato juice under heat and vacuum until a Brix range of 8–24 percent. Paste was produced using the same method but was concentrated to a Brix range of 24–30 percent. Both puree and paste were heated to ~ 85–91 °C and placed into metal cans and sealed. Cans were cooled in a 16–32 °C water bath for 28–32 minutes, and subsequently placed into frozen storage until analysis.

Tomato juice was also used in the production of ketchup. Tomato juice was evaporated to a solids content of 30–35 percent, and then vinegar (1.0–2.9 percent), sugar (4.5–5.5 pervent), and salt (0.9–1.2 percent) were added to the evaporated juice and stirred for 15 minutes. The mixture was screened through a sieve to remove large particles. Material passing through the screen was collected as ketchup, and placed into frozen storage until analysis.

Sun-dried tomatoes were produced by slicing cleaned tomatoes to ¼–3/8" thickness and put into a dehydrator, which dried the slices at ~ 49–54 °C for 24–26 hrs. The sun-dried tomatoes were placed into frozen storage until analysis.

For the production of canned tomatoes, washed tomatoes were blanched with 18–22 psi steam in a kettle for 1–3 minutes. Blanched tomatoes were placed into water to remove the skin. Blemishes were trimmed off following skin removal. The tomatoes were then put into cans with heated tomato juice, sealed, and cooked in a pressure cooker at 121–124 °C for 50–60 minutes. Canned tomatoes were cooled in a 16–32 °C water bath for 28–32 minutes, and subsequently placed into frozen storage until analysis.

Blanched tomatoes were produced by slicing an "X" on the bottom of each washed tomato and then placing into 98–101 °C water for 30–60 seconds. Tomatoes were removed from the boiling water and placed in cold water or a cold water bath. Tomato skins were removed and then the blanched tomatoes were cored. Samples of blanching water and blanched tomatoes were collected and placed into frozen storage until analysis.

Cleaned tomatoes were peeled by hand and representative samples of tomato peels and peeled tomatoes were collected and placed into frozen storage until analysis.

For the production of vegetable stock, cleaned tomatoes were cored and diced. The diced tomatoes were placed into 82–100 °C water for 45–60 minutes. The mixture was sieved after cooking to remove solids. Liquid passing through the sieve was collected as vegetable stock and placed into frozen storage until analysis.

Residues of mefentrifluconazole in tomatoes and all processed commodities were determined using LC-MS/MS method D1511/01 (Reeves, 2018, BASF DocID 2018/7005677). The limit of quantification (LOQ) was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the tomatoes and processed commodities and extraction was 675 days. All samples were analysed within 15 days of extraction. Processing factors are shown in Table 159.

Table 159 Concentration of mefentrifluconazole residues in tomato processed fractions and processing factors

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor
R160145 Greenville, FL, United States, 2016 Red Beauty 1346 g ai/ha DALA=0	Tomato, Field RAC	0.450	-
	Tomato, Processor RAC[3]	0.7500	-
	Blanched tomatoes	0.0487	0.06
	Blanching water	0.0540	0.07
	Canned tomatoes	0.0420	0.06
	Ketchup after pasteurization	0.2600	0.35
	Paste	0.7500	1.00
	Peeled tomatoes	0.0502	0.07
	Puree	0.2350	0.31
	Raw juice	0.0840	0.11
	Sun-dried tomatoes	5.0000	6.67
	Tomato peel	4.0000	5.33
	Vegetable stock	0.0740	0.10

Trial, location, year variety, total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor
	Wash water	0.0165	0.02
	Washed tomatoes	0.5000	0.67
	Wet pomace	2.1946	2.93
R160146 (Madera, CA, United States, 2016 Roma 1363 g ai/ha DALA=0	Tomato, Field RAC	0.625	-
	Tomato, Processor RAC[3]	0.6000	-
	Blanched tomatoes	0.0323	0.05
	Blanching water	0.0495	0.08
	Canned tomatoes	0.0485	0.08
	Ketchup after pasteurization	0.4050	0.68
	Paste	0.2950	0.49
	Peeled tomatoes	0.0371	0.06
	Puree	0.1650	0.28
	Raw juice	0.0455	0.08
	Sun-dried tomatoes	5.5000	9.17
	Tomato peel	2.5000	4.17
	Vegetable stock	0.1290	0.22
	Wash water	0.0380	0.06
	Washed tomatoes	0.4000	0.67
	Wet pomace	1.0500	1.75
R160147 Porterville, CA, United States, 2016 Dri 319 1352 g ai/ha DALA=0	Tomato, Field RAC	0.3275	-
	Tomato, Processor RAC[3]	0.4548	-
	Blanched tomatoes	0.0271	0.06
	Blanching water	0.0475	0.10
	Canned tomatoes	0.0216	0.05
	Ketchup after pasteurization	0.2550	0.56
	Paste	0.2100	0.46
	Peeled tomatoes	0.0146	0.03
	Puree	0.0900	0.20
	Raw juice	0.0380	0.08
	Sun-dried tomatoes	7.2613	15.97
	Tomato peel	1.1000	2.42
	Vegetable stock	0.0100	0.02
	Wash water	0.0511	0.11
	Washed tomatoes	0.4208	0.93
	Wet pomace	3.2476	7.14

Notes:

[1] DALA = Days after last application.

[2] Mefentrifluconazole residues values of the Processor RAC (i.e. tomato samples analysed just prior to processing) were used for the calculation of the processing factors.

[3] RAC = raw agricultural commodity, representative RAC sub-samples of the tomato bulk sample were collected prior to processing.

Soya beans

Three processing trials were carried out in the United States during the 2014 and 2015 growing seasons (Crawford, 2016, BASF DocID 2015_7005934) where soya beans received two foliar broadcast applications each at a rate of 148–155 g ai/ha, at 6 to 7 day intervals, of an SC formulation containing 100 g/L mefentrifluconazole. A second plot at each trial site was treated with two foliar broadcast applications each at a rate of 299–308 g ai/ha, at 14 to 15-day intervals, using the same formulation. An adjuvant (non-ionic spreader/sticker/surfactant) was added to the spray mixture for all applications. Mature soya bean seed was harvested 21–23 days after the last application (DALA) from the first plot and

77–91 DALA from the second plot. Samples were frozen and transported to the processing facility by freezer truck within 13 days of harvest or kept at ambient temperature and hand delivered to the processing facility within 1 day of harvest. At the processing facility, samples were processed into aspirated grain fraction, hulls, untoasted meal, toasted meal, crude oil, refined oil, defatted soy flour, soy milk, tofu, pollards, soya sauce, and miso according to commercial practices.

To generate aspirated grain fractions (AGF), whole soya bean seed samples (530 kg) that were harvested from the first treated plots were placed in a dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. Samples were moved into the system and aspirated for 75–120 minutes to remove light impurities (grain dust). Light impurities were classified using sieves. Impurities that went through the 2360 micron sieve were recombined to produce the AGF sample.

Whole soya bean seed samples (91 kg) harvested from the second treated plots with a moisture content > 13.5 percent were dried in an oven at 54–71 ºC until the moisture content was 10.0–13.5 percent. After drying, samples were cleaned by aspiration and screening. For production of hulls, untoasted meal, toasted meal, defatted soy flour, crude oil, and refined oil, whole soya beans were hulled using a roller mill and aspirated to separate the sample into hull and kernel. The kernel moisture content was adjusted to 13.5 percent, heated to 71–79 ºC in a mixer, and then flaked using a flaking roll. A portion of the flakes was removed for direct solvent extraction (no extrusion). The remaining flakes were extruded in a continuous processor where they were turned into collets by direct steam injection and compression. After extrusion, the collets were ground in a disc mill and dried in an oven at 66–82 ºC for 30–40 minutes. Ground collets and flakes were placed in stainless steel extractors and submerged in 49–60 ºC hexane for 30 minutes, drained, and repeated two more times for 15 minutes each. The miscella (crude oil and hexane) from each extraction were combined. The extracted flakes were desolventized with warm air and fractions of untoasted meal were collected. The remaining desolventized flakes were ground in a mill and sieved with a 62 mesh screen on a sifter. The resulting fraction passing through the screen was the defatted soy flour sample. Extracted collets were toasted by steam injection, heated to 104–116 ºC for 30–60 minutes, and then screened. The collected material passing through the screen was the toasted meal sample. The crude oil and hexane from the miscella were separated by vacuum evaporation. The crude oil was heated to 91–96 ºC, filtered, and fractions were then collected for analysis. Crude oil was neutralized by adding sodium hydroxide and mixing at a high rotations per minute (RPM) in a water bath for 90 minutes (at 20–24 ºC) and then mixed at a low RPM for 20 minutes (at 63–67 ºC). The neutralized oil was centrifuged, the refined oil was decanted and filtered, and the resultant fractions were alkali refined oil and soapstock (the latter was discarded). The alkali refined oil was heated and bleached by the addition of activated bleaching earth (1 percent by weight of oil). After vacuum filtration the resulting fractions were bleached oil and spent bleaching earth (the latter was discarded). Bleached oil was deodorized by steaming under vacuum for 28–32 minutes (at 220–230 ºC), followed by the addition of 0.5 percent citric acid solution (1 mL/100 g oil). The resulting fractions were deodorized oil and deodorizer distillates (the latter was discarded).

For soya milk and tofu production, whole cleaned soya beans (0.8 kg) were washed, soaked in water for at least 12 hours, ground and filtered. The filtered liquid (soya milk) was heated for 9–11 minutes at 91–96 ºC. A portion of the soya milk was then mixed and heated to 75–85 ºC. Calcium sulfate solution was added until the liquid (whey) became transparent and curd formed, upon which they were separated by centrifugation and the curds (tofu) were collected.

For the production of soya sauce and miso, cleaned whole soya beans (2 kg) were washed and soaked in reverse osmosis water for 10–14 hours and then pressure cooked at 220–230 ºC for 57–63 minutes. After cooling, the pressure-cooked soya bean kernel material was mixed slowly with ground roasted wheat, distilled water, and *Aspergillus oryzae* starter culture (0.1–0.2 percent by weight) for

approximately 5 minutes. The mixture was incubated in a forced air chamber at 29–36 ºC with a relative humidity (RH) of 55–75 percent for 24–72 hours and then lowered to 20–26 ºC at the same RH for another 24–72 hours to develop mature Koji. The mature Koji was mixed with 22–28 percent sea salt/distilled water brine solution and placed back into the chamber at an RH of 55–75 percent and incubated at temperatures of 15–20 ºC for the first 30 days, 25–30 ºC for the next 120 days, and 15–20 ºC for the last 30 days. After the fermentation process, raw soya bean sauce was separated from the spent Koji (miso) using a hydraulic press and filter paper. The raw soya sauce was filtered by cotton and vacuum and then pasteurized for 20–30 minutes at 70–80 ºC.

Pollard was produced by drying cleaned whole soya beans at 54-71 ºC until a moisture content <10 percent. The material was then milled in a mill and sieved with a 100 mesh screen twice. The material passing through the sieve was full-fat soy flour (which was discarded) while the material remaining in the sieve was collected as pollard.

Residues of mefentrifluconazole in soya bean and all processed commodities were determined using LC-MS/MS method D1511/01. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the soya beans and processed commodities and analysis was 539 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 160.

Table 160 Mefentrifluconazole residues in soya bean and processed fractions with corresponding processing factors

Trial, location, year, variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA[1]	Mefentrifluconazole (mg/kg)	Processing Factor[2]
R140748 Chula, GA, United States, 2014 Pioneer 95Y70	Seed (Field RAC)	297	21	<0.01	-
	Seed (Processor RAC)			0.013	-
	Aspirated Grain Fraction			1.21	93
	Seed (Field RAC)	599	91	<0.01	-
	Seed (Processor RAC)			<0.01	n/a
	Hulls			<0.01	n/a
	Meal (Toasted)			<0.01	n/a
	Crude Oil			<0.01	n/a
	Tofu			<0.01	n/a
	Soya Sauce			<0.01	n/a
	Pollards			<0.01	n/a
	Flour			<0.01	n/a
	Miso			<0.01	n/a
	Soya Milk			<0.01	n/a
	Refined Oil			<0.01	n/a
	Meal (Untoasted)			<0.01	n/a
R140749 Chenyville, LA, United States 2014 P54T94R	Seed (Field RAC)	306	23	<0.01	-
	Seed (Processor RAC)			<0.01	-
	Aspirated Grain Fraction			1.88	n/a[4]
	Seed (Field RAC)	611	77	<0.01	-
	Seed (Processor RAC)			0.012	-
	Hulls			<0.01	<0.83
	Meal (Toasted)			<0.01	<0.83
	Crude Oil			0.012	1.00
	Tofu			<0.01	<0.83
	Soya Sauce			<0.01	<0.83
	Pollards			<0.01	<0.83
	Flour			<0.01	<0.83
	Miso			<0.01	<0.83

Trial, location, year, variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA[1]	Mefentrifluconazole (mg/kg)	Processing Factor[2]
	Soya Milk			<0.01	<0.83
	Refined Oil			<0.01	<0.83
	Meal (Untoasted)			<0.01	<0.83
R120750 York, NE, United States 2015 322 RR2	Seed (Field RAC)	299	22	<0.01	-
	Seed (Processor RAC)			<0.01	-
	Aspirated Grain Fraction			2.51	n/a[4]
	Seed (Field RAC)	599	78	<0.01	-
	Seed (Processor RAC)			<0.01	-
	Hulls			<0.01	n/a
	Meal (Toasted)			<0.01	n/a
	Crude Oil			<0.01	n/a
	Tofu			<0.01	n/a
	Soya Sauce			<0.01	n/a
	Pollards			<0.01	n/a
	Flour			<0.01	n/a
	Miso			<0.01	n/a
	Soya Milk			<0.01	n/a
	Refined Oil			<0.01	n/a
	Meal (Untoasted)			<0.01	n/a

Notes:

[1] DALA = Days after last application.

[2] Mefentrifluconazole residue values of the Processor RAC (i.e. soya beans analysed just prior to processing) were used for the calculation of the processing factors. n/a = not applicable, because both RAC and processed commodity were below the LOQ.

[3] RAC = raw agricultural commodity.

[4] Since the RAC contained residues <LOQ, no reliable PF can be calculated. However, a concentration of the residue can be observed.

Sugar beets

Three processing trials were carried out in Germany during the 2015 growing season (Plier et al., 2016, BASF DocID 2015_1220032) where sugar beets received two foliar applications each at a rate of 740–800 g ai/ha, at 13 to 14-day intervals, of an EC formulation containing 100 g/L mefentrifluconazole. Tops and roots were harvested 0 and 20–21 days after the last application (DALA). Only 20–21 DALA root samples were sent for processing by car at ambient temperature and were processed 4–7 days after harvest. At the processing facility, samples were processed into washed beets, wash water, cossettes, pressed pulp, press water, raw juice, thin juice, thick juice, molasses, raw sugar, affinated syrup, refined sugar, dried pulp, and ensiled pulp according to commercial practices.

Sugar beet roots (289 kg) were washed in a cylindrical beet-washer and the wash water and washed beets were sampled. Washed beets were then sliced in a slicer set at a distance of 7 mm. The resulting cossettes were sampled. Cossettes were counter-current extracted with warm tap water in an extraction trough. After extraction the raw juice was sampled. The raw juice was purified by two-stage liming (cold pre-liming at 34.5–39.9 ºC with 250 CaO/L of lime milk up to a pH of 11.2 for 15-16 minutes followed by heating of the juice to 84.8–84.9 ºC for 15–48 minutes) and two-stage carbonatation (CO_2 was added until a pH of 11.0–11.2 was reached at a carbonatation temperature of 84.8–85.4 ºC and then filtered; for the second carbonatation, the filtrate was heated up to 94.8–95.0 ºC and CO_2 was added until the lime content in the juice reached a minimum pH of 9.0 to 9.2 and filtered). The resultant thin juice was sampled. The thin juice was concentrated in a single-stage evaporating plant at a temperature of 80–84

ºC (pressure of 0.45–0.55 bar) to a dry substance content of 60.6–66.5 percent. After evaporation the thick juice was sampled. The thick juice was concentrated at a pressure of 0.25 bar into a metastable supersaturation state and powdered sugar was injected as seed. Once crystallisation was finished, the massecuite was brought into the cooling crystalliser where the massecuite was cooled down to 19.6–36.4 ºC and then centrifuged (at approximately 3500 min^{-1}) to separate the molasses and raw sugar (air dried) which were both sampled. The raw sugar was mixed with distilled water for 25–45 minutes and then centrifuged (at approximately 3500 min^{-1}) to separate affinated syrup and pure sugar (air dried after centrifugation) which were both sampled. The wet pulp from the counter-current juice extraction was pressed and separated into press water and pressed pulp which were both sampled. An aliquot of the pressed pulp was dried in a drying chamber at 35 ºC until a moisture content of <10 percent was reached. The dried pulp was sampled. Another aliquot of the pressed pulp was fermented in a silage glass container under pressure at 20.0–22.2 ºC for 6 weeks to produce ensilage pulp that was sampled.

Residues of mefentrifluconazole in sugar beet roots and all processed commodities were determined using LC-MS/MS method L0076/09. TheLOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the sugar beet roots and processed commodities and analysis was 175 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 161.

Table 161 Mefentrifluconazole residues in sugar beet roots and processed fractions with corresponding processing factors

Trial, location, year, variety Total rate	Commodity or Matrix	Mefentrifluconazole Residues (mg/kg)	Processing Factor
L150310 Motterwitz, Saxony, Germany 2015 Julius 1510 g ai/ha DALA = 21	Roots without Tops (RAC)[2]	0.16	-
	Washed beets	0.044	0.28
	Wash water	0.083	0.52
	Cossettes	0.056	0.35
	Pressed pulp	0.12	0.75
	Press water	<0.01	<0.06
	Raw juice	0.019	0.12
	Thin juice	0.013	0.08
	Thick juice	0.045	0.28
	Molasses	0.14	0.88
	Raw sugar	<0.01	<0.06
	Affinated sugar	0.018	0.11
	Refined sugar	<0.01	<0.06
	Dried pulp	0.76	4.75
	Ensiled pulp	0.14	0.88
L150311 Gerichshain, Saxony, Germany 2015 Julius 1580 g ai/ha DALA = 20	Roots without Tops (RAC)	0.21	-
	Washed beets	0.069	0.33
	Wash water	0.070	0.33
	Cossettes	0.091	0.43
	Pressed pulp	0.18	0.86
	Press water	<0.01	<0.05
	Raw juice	0.026	0.12
	Thin juice	0.017	0.08
	Thick juice	0.073	0.35
	Molasses	0.23	1.10
	Raw sugar	<0.01	<0.05
	Affinated sugar	0.023	0.11
	Refined sugar	<0.01	<0.05

Trial, location, year, variety Total rate	Commodity or Matrix	Mefentrifluconazole Residues (mg/kg)	Processing Factor
	Dried pulp	1.1	5.24
	Ensiled pulp	0.24	1.14
L150312 Groß Santersleben, Saxony-Anhalt, Germany 2015 Artus 1520 g ai/ha	Roots without Tops (RAC)	0.34	-
	Washed beets	0.088	0.26
	Wash water	0.13	0.38
	Cossettes	0.34	1.00
	Pressed pulp	0.18	0.53
	Press water	<0.01	<0.03
	Raw juice	0.036	0.11
	Thin juice	0.021	0.06
	Thick juice	0.073	0.21
	Molasses	0.18	0.53
	Raw sugar	0.034	0.10
	Affinated sugar	0.062	0.18
	Refined sugar	0.033	0.10
	Dried pulp	1.1	3.24
	Ensiled pulp	0.23	0.68

Notes:

[1] DALA = Days after last application.

[2] RAC = raw agricultural commodity.

Potatoes

Three processing trials were carried out in the United States during the 2015 growing season (Schreier, 2016, BASF DocID 2016_7006672) where potatoes received three foliar broadcast applications each at a rate of 745–757 g ai/ha, at 6 to 7–day intervals, of an EC formulation containing 100 g/L mefentrifluconazole. Potato tubers were harvested 6–7 days after the last application (DALA). An adjuvant was added to the spray mixture for all applications. Potato tubers were shipped at ambient temperatures to the processing facility within 2 days of harvest. At the processing facility, samples were processed into peeled potato, wet peel, boiled potatoes, microwave/boiled potatoes, baked potato, fried potato, crisps (potato chips in the United States), chips (French fries), granules/flakes, process waste, ensiled potato, starch, dried pulp, and potato protein according to commercial practices.

Potatoes were washed in a tub for 5 minutes and culled by hand. Culled potatoes were sampled as the process waste fraction. Washed potatoes (36 kg) were batch steamed for 45–60 seconds at 100–120 psi and then batch scrubbed for 15–30 second with a peeler. The peels were collected. Peeled potatoes were further inspected to remove additional peel, rot, green or otherwise damaged potatoes. The trim waste was retained. A fraction of the steamed peeled potatoes were collected for anlaysis. The collected potato peels were hydraulically pressed and blended with the trim waste and the combined fraction was sampled as the wet peel. The remaining peeled potatoes were cut into slabs using a slicer, batch spray-washed in cold tap water, precooked at 70–77 ºC for 20 minutes in a kettle, and then cooled to <32 ºC for 20 minutes. The precooked potato slabs were then steam cooked at 94–100 ºC for 40–42 minutes in a steam cabinet. The cooked potato slabs were mashed in a modified meat grinder and mixed for 37 seconds in a mixer with an emulsion of food additives. The cooked mash was dried in a drum dryer into a thin sheet and broken into large flakes by hand. The flakes were then fed into a fruit press hammermill for uniform milling of the potato flakes which were sampled for analysis.

For the production of potato starch, washed potatoes (36 kg) were batch steamed for 45–60

seconds at 100–120 psi and then batch scrubbed for 15–30 second with a peeler. The water from the scrubber and peels were collected. The collected potato peels were hydraulically pressed and the collected starch water was combined with the water from the scrubber and filtered using a series of sieves. The filtered star water was centrifuged and the starch sample was collected for analysis.

For the production of potato crisps (chips) and fried potatoes, washed potatoes (14 kg) were batch peeled using a restaurant style Hobart peeler for 30 seconds. The peeled potatoes were cut into ~0.16 cm slices for crisps or ~0.5 cm slices for fried potatoes using a restaurant style slicer. The sliced potatoes were placed in a tub of hot water, drained over a screen, and fried in oil at 163–191 ºC for 90–145 seconds (crisps) or 3–3.5 minutes (fried potatoes). The fried potato crisps and fried potatoes were drained, salted (crisps), and sampled for analysis.

To generate boiled potatoes, washed potatoes (5 kg) were hand-peeled (stove-boiled potatoes only) and cut into quarters (stove-boiled and microwave-boiled potatoes). Unpeeled, quartered potatoes were placed in water and microwaved until an internal temperature of 88–92 ºC was attained. The microwaved-boiled potatoes were removed from the water and sampled for analysis. The peeled, quartered potatoes were boiled in water on a stove until an internal temperature of 88–92 ºC was attained. The stove boiled potatoes were removed from the water and sampled for analysis. For the production of baked potatoes, washed potatoes were baked in an oven at 210 ºC until an internal temperature of 88–92 ºC was reached. The baked potatoes were sampled for analysis. French fries were produced by slicing washed unpeeled potatoes into 0.5 cm strips using a fry cutter and then frying the strips in a fat fryer at 177–191 ºC got 2.5–3.0 minutes. After frying, the fresh French fries were drained and cooled and then sampled for analysis.

To generate potato pulp, protein, and ensiled potato, washed potatoes (40 kg) were chopped in a fruit press and hammermill and milled with a food processor. The collected pulp was hydraulically pressed in a fruit press and the pressed wet pulp was dehydrated in a tray dryer. The dried pulp was sampled for analysis. The collected potato water after pressing was centrifuged, filtered, and thermal processed at ~90 ºC while adjusting the pH to ~4.0 using H_2SO_4. The precipitated protein was recovered from the water using a filter. The potato protein was sampled for analysis. An aliquot of potato pulp (milled potatoes) was placed in a bag silo, sealed, and allowed to ferment for a minimum of 3 weeks at room temperature after which time the bag was opened, the pH of the ensiled potatoes was measured, and a sample was collected for analysis.

Residues of mefentrifluconazole in potatoes and all processed commodities were determined using LC-MS/MS method D1511/01. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the potatoes and processed commodities and analysis was 216 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 162.

Table 162 Mefentrifluconazole residues in potato and processed fractions with corresponding processing factors

Trial, location, year, variety Total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor
R150062 Lyons, NY, United States 2015 Reba 2254 g ai/ha DALA=6	Potato tuber Processor RAC[2]	<0.01, <0.01 [<0.01]	-
	Peeled potato	<0.01, <0.01 [<0.01]	n/a[3]
	Peel, wet	<0.01, <0.01 [<0.01]	n/a
	Boiled potatoes	<0.01, <0.01 [<0.01]	n/a
	Microwaved/boiled potatoes (unpeeled)	<0.01, <0.01 [<0.01]	n/a
	Baked potato	0.02, 0.02 [0.02]	n/a

Trial, location, year, variety Total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor
	Fried potato	<0.01, <0.01 [<0.01]	n/a
	Crisps	<0.01, <0.01 [<0.01]	n/a
	Chips	<0.01, <0.01 [<0.01]	n/a
	Granules/Flakes	<0.01, <0.01 [<0.01]	n/a
	Process waster	<0.01, <0.01 [<0.01]	n/a
	Ensiled	<0.01, <0.01 [<0.01]	n/a
	Starch	<0.01, <0.01 [<0.01]	n/a
	Dried pulp	0.01, 0.01 [0.01]	n/a
	Potato protein	0.01, 0.02 [0.02]	n/a
R150063 Weedsport, NY, United States 2015 Chieftain 2255 g ai/ha DALA=7	Potato tuber Processor RAC	0.02, 0.02 [0.02]	-
	Peeled potato	<0.01, <0.01 [<0.01]	<0.5
	Peel, wet	0.02, 0.03 [0.03]	1.50
	Boiled potatoes	<0.01, <0.01 [<0.01]	<0.5
	Microwaved/boiled potatoes (unpeeled)	<0.01, <0.01 [<0.01]	<0.5
	Baked potato	<0.01, 0.02 [<0.02]	<1.0
	Fried potato	<0.01, <0.01 [<0.01]	<0.5
	Crisps	<0.01, <0.01 [<0.01]	<0.5
	Chips	<0.01, <0.01 [<0.01]	<0.5
	Granules/Flakes	<0.01, <0.01 [<0.01]	<0.5
	Process waster	<0.01, <0.01 [<0.01]	<0.5
	Ensiled	<0.01, 0.02 [<0.02]	<1.0
	Starch	<0.01, 0.01 [<0.01]	<0.5
	Dried pulp	0.06, 0.07 [0.07]	3.50
	Potato protein	0.08, 0.05 [0.07]	3.50
R150064 Payette, ID, United States 2015 2257 g ai/ha DALA=7	Potato tuber Processor RAC	0.02, 0.03 [0.03]	-
	Peeled potato	<0.01, <0.01 [<0.01]	<0.33
	Peel, wet	0.06, 0.04 [0.05]	1.67
	Boiled potatoes	<0.01, <0.01 [<0.01]	<0.33
	Microwaved/boiled potatoes (unpeeled)	<0.01, <0.01 [<0.01]	<0.33
	Baked potato	<0.01, <0.01 [<0.01]	<0.33
	Fried potato	<0.01, <0.01 [<0.01]	<0.33
	Crisps	<0.01, <0.01 [<0.01]	<0.33
	Chips	<0.01, <0.01 [<0.01]	<0.33
	Granules/Flakes	<0.01, <0.01 [<0.01]	<0.33
	Process waster	<0.01, <0.01 [<0.01]	<0.33
	Ensiled	<0.01, 0.01 [<0.01]	<0.33
	Starch	<0.01, 0.01 [<0.01]	<0.33
	Dried pulp	0.04, 0.04 [0.04]	1.33
	Potato protein	0.03, 0.03 [0.03]	1.00

Notes:

[1] DALA = Days after last application.

[2] RAC = raw agricultural commodity.

[3] Since the RAC contained residues <LOQ, no reliable PF can be calculated. However, a concentration of the residue can be observed in baked potato, dried pulp, and potato protein from Trial ID R150062.

Wheat

Three processing trials were carried out in Germany during the 2014 growing season (Plier et al., 2015, BASF DocID 2014_1315283) where wheat received two foliar applications each at a rate of 420–490 g ai/ha, at 14 to 39-day intervals, of an EC formulation containing 100 g/L mefentrifluconazole. Wheat

whole plant no roots were harvested at 0, and 7–9 days after the last application (DALA). Wheat grain was harvested 45–60 DALA. Only samples collected at the 7–9 DALA and 45–60 DALA were sent for processing. Wheat samples for processing were shipped by car at ambient temperature from the field to the processing facility. Grain specimens were stored at room temperature until the start of processing (77–98 days after harvest). Whole plant no roots specimens were processed on the day of sampling. At the processing facility, samples were processed into wet silage, wilted silage, bran, flour, germ, middlings, shorts, gluten, gluten feed meal, starch, whole meal flour, whole grain bread, milled byproducts, and aspirated grain fraction, according to commercial practices.

For silage production, fresh harvested whole plants (5 kg) were dried in the field or in a dry oven at 35 ºC until a dry matter content of 35–55 percent was reached. Fresh wheat for wet silage production (dry matter content of 5–35 percent) and the dried wheat for wilted silage production were filled in special silage glass containers (under pressure) and stored closed at 20–25 ºC for about 6 weeks. Wet silage and wilted silage were sampled for analysis.

Wheat grain samples (61 kg) were cleaned and the aspirated grain fraction was sampled for analysis. Cleaned grain samples were moistened with tap water until a moisture content of 15–16 percent was reached (if necessary). Cleaned wheat grain (10 kg) was then milled in a closed system with different pairs of smooth rollers and sifter passages into straight flour, bran, and middlings. Samples of flour, bran and middlings were taken for analysis. Bran and middlings were further processed by mixing them together and then separating the mixture into shorts and low grade meal using a centrifuge/scouring machine. Samples of shorts were taken for analysis.

For the production of whole-meal flour and whole-meal bread, the same milling procedure used for the production of flour was used. After milling, the shorts were cracked into smaller pieces using an impact mill. All milling products of the process were used completely for the whole-meal and were mixed homogenously in a special flour mixer. Samples of whole-meal flour were taken for analysis. For baking whole meal bread, whole meal flour, yeast, salt, and water were mixed. The resulting dough was kneaded for 7 minutes, fermented for 20 minutes, reprocessed for 5 minutes, fermented a second time in a baking tin for 40 minutes, and then baked at 210–230 ºC for 50 minutes. A sample of whole-grain bread was taken for analysis.

To generate wheat germs, wheat grain (20 kg) was broken in a roller mill with 0.5 mm roller distance. The 400–1000 µm fraction was collected while the fraction > 1000 µm was broken again with a 0.3 mm roller distance. This procedure was repeated three times with a final roller distance of 0.2 mm. The fractions < 400 µm and the last fraction > 1000 µm were not processed further but were retained for the generation of milled byproducts. The 400–1000 µm fraction was fed through a special separator to obtain middlings/germ and bran. The bran was retained for the generation of milled byproducts. The middlings/germ mixture was milled into flour, bran, and small wheat germ discs in a mill with smooth rollers. The mixture was then sieved to separate the fractions. The flour was retained for the generation of milled byproducts. The bran/germ fraction was sieved again into fine bran/germ and coarse bran/germ fractions. From the separated germ discs, small parts of bran were removed manually and samples of germ were taken for analysis. The bran was mixed with the other fractions which were retained to generate milled byproducts. Milled byproducts were sampled for analysis.

For the production of starch and gluten, wheat grain (5 kg) was milled into straight flour, bran, and middlings. Straight flour and water were mixed to form hydrated dough which was then separated by centrifugation into wet starch, process water, and gluten (containing starch). The starch was washed with water and separated by centrifugation into starch, process water, and gluten twice. The gluten (containing starch) was also washed with water and centrifuged several times resulting in gluten after washing and process water fractions. Process water fractions were separated by centrifugation into starch and fibre.

Fibre was dried at 60 ºC. Wet starch fractions were dried at 60 ºC while wet gluten was dried by freeze drying. After the drying process, the dried products were milled. The milled starch fractions were combined. Starch and gluten samples were collected for analysis. The dried and milled fractions of fibre, starch, and gluten were then mixed to form gluten feed meal which was sampled for analysis.

Residues of mefentrifluconazole in wheat and all processed commodities were determined using LC-MS/MS method L0076/09. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the wheat and processed commodities and analysis was 305 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 163.

Table 163 Mefentrifluconazole residues in wheat and processed fractions with corresponding processing factors

Trial, location, year, variety Total rate	Commodity or Matrix	DALA[1]	Mefentrifluconazole (mg/kg)	Processing Factor[2]
FR 06/14/70, L140181 Motterwitz, Saxony, Germany 2014 Cubus 920 g ai/ha	Whole plant no roots, Field RAC[3]	7	3.1	-
	Whole plant no roots, Processing RAC		6.9	-
	Wet silage		7.6	1.10
	Wilted silage		8.0	1.16
	Grain, field RAC	56	0.14	-
	Grain, Processing RAC		0.13	-
	Bran		0.31	2.38
	Flour		<0.01	<0.08
	Germ		0.11	0.85
	Middlings		0.25	1.92
	Shorts		0.34	2.62
	Gluten		0.072	0.55
	Gluten feed meal		0.038	0.29
	Starch		<0.01	<0.08
	Whole meal flour		0.10	0.77
	Whole grain bread		0.070	0.54
	Milled byproducts		0.081	0.62
	Aspirated grain fractions		5.0	38.46
FR 06/14/25, L140182 Tützpatz, Mecklenburg-Western Pomerania, Germany 2014 Akteur 850 g ai/ha	Whole plant no roots, Field RAC	9	3.0	-
	Whole plant no roots, Processing RAC		2.5	-
	Wet silage		3.6	1.44
	Wilted silage		4.7	1.88
	Grain, field RAC	60	0.024	-
	Grain, Processing RAC		0.017	-
	Bran		0.063	3.71
	Flour		<0.01	<0.59
	Germ		0.031	1.82
	Middlings		0.066	3.88
	Shorts		0.077	4.53
	Gluten		<0.01	<0.59
	Gluten feed meal		<0.01	<0.59
	Starch		<0.01	<0.59
	Whole meal flour		0.017	1.00
	Whole grain bread		<0.01	<0.59
	Milled byproducts		0.019	1.12
	Aspirated grain fractions		0.37	21.76
FR 06/14/20, L140183 Blankenhagen,	Whole plant no roots, Field RAC	7	4.4	-
	Whole plant no roots, Processing RAC		3.2	-

Trial, location, year, variety Total rate	Commodity or Matrix	DALA[1]	Mefentrifluconazole (mg/kg)	Processing Factor[2]
Mecklenburg-Western Pomerania, Germany 2014 Ritmo 900 g ai/ha	Wet silage		3.8	1.19
	Wilted silage		6.5	2.03
	Grain, field RAC	45	0.028	-
	Grain, Processing RAC		0.034	-
	Bran		0.10	2.94
	Flour		<0.01	<0.29
	Germ		0.038	1.12
	Middlings		0.077	2.26
	Shorts		0.12	3.53
	Gluten		0.015	0.44
	Gluten feed meal		<0.01	<0.29
	Starch		<0.01	<0.29
	Whole meal flour		0.027	0.79
	Whole grain bread		0.019	0.56
	Milled byproducts		0.014	0.41
	Aspirated grain fractions		1.5	44.12

Notes:

[1] DALA = Days after last application.

[2] Mefentrifluconazole residue values of the Processing RAC (i.e. wheat samples analysed just prior to processing) were used for the calculation of the processing factors. n/a = not applicable.

[3] RAC = raw agricultural commodity.

Barley

Three processing trials were carried out in Germany during the 2014 growing season (Plier et al., 2015, BASF DocID 2014_1315282) where barley received two foliar applications each at a rate of 440–490 g ai/ha, at 15 to 24-day intervals, of an EC formulation containing 100 g/L mefentrifluconazole. Barley grain, harvested 43–56 days after the last application (DALA), were shipped by car at ambient temperature from the field to the processing facility. Grain specimens were stored at room temperature until the start of processing (116–140 days after harvest). At the processing facility, samples were processed into pearled (pot) barley, flour, bran, brewing malt, malt sprouts, beer, brewers grain (dried), and brewers yeast according to commercial practices.

Barley grain samples (49 kg) were cleaned and sieved through a 2.5 mm mesh and then combined wet and dry steeping was conducted to begin the malting process. After steeping, the malt sample underwent still germination (continuous turning for 166 hours at 14.5 °C, with a relative humidity of > 90 percent) and was kiln dried to a water content of 5.30–5.45 percent in a dry chamber. After kiln-drying, the germs were removed mechanically by a trimmer and brewing malt and malt sprouts were sampled for analysis.

To brew the malt, the malt sample (12 kg) was milled with a malt-mill, mashed in a heatable tun, and lautered in a refining vat for 2–3 hours to separate the wort from the insoluble malt components (brewer's grain). After separation, the brewers grain was dried at 50 °C until a dry matter content of <10 percent was attained, and then dried brewers grain was sampled for analysis. Hop pellets were added to the wort and the mixture was boiled for 1.5 hours at normal pressure. After boiling the flocs (hops draff) were separated in a whirlpool system for 20 minutes using intra-plant circulation. Fermentation was carried out in bottom fermentation containers at approximately 9 °C for 9–11 days. The yeast deposited on the tank bottom was sampled as brewers yeast prior to maturation. After fermentation, the beer was

matured by under warm conditions (room temperature) for 2 days, followed by cold maturation (under pressure at approximately 0.7–1.0 bar, 2 °C) for 21 days. The resultant rack beer was filtered and the final beer product was sampled for analysis.

For the production of pearled (pot) barley, the grain (5 kg) was cleaned and the moisture content was adjusted by drying or dampening to approximately 16 percent. The samples were then hulled until an abrasion of 20–25 percent was reached. The abrasion was sieved into bran and flour. Pot barley, bran, and flour were sampled for analysis.

Residues of mefentrifluconazole in barley and all processed commodities were determined using LC-MS/MS method L0076/09. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the barley grain and processed commodities and analysis was 254 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 164.

Table 164 Mefentrifluconazole residues in barley and processed fractions with corresponding processing factors

Trial, location, year, variety Total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor[2]
FR 05/14/70, L140178 Motterwitz, Saxony, Germany 2014 Quench 950 g ai/ha DALA=52	Grain, field RAC[3]	0.35	-
	Grain, Processing RAC	0.40	-
	Pearled/pot barley	0.065	0.16
	Flour	1.8	4.50
	Bran	1.7	4.25
	Brewing malt	0.20	0.50
	Malt sprouts	0.67	1.68
	Beer	<0.01	<0.03
	Brewers grain (dried)	0.95	2.38
	Brewers yeast	0.076	0.19
FR 05/14/25, L140179 Tützpatz, Mecklenburg-Western Pomerania, Germany 2014 Grace 890 g ai/ha DALA=56	Grain, field RAC	0.21	-
	Grain, Processing RAC	0.24	-
	Pearled/pot barley	0.029	0.12
	Flour	0.88	3.67
	Bran	1.2	5.00
	Brewing malt	0.12	0.50
	Malt sprouts	0.23	0.96
	Beer	<0.01	<0.04
	Brewers grain (dried)	0.58	2.42
	Brewers yeast	0.064	0.27
FR 06/14/20, L140180 Neubukow, Mecklenburg-Western Pomerania, Germany 2014 Quench 960 g ai/ha DALA=43	Grain, field RAC	0.23	-
	Grain, Processing RAC	0.22	-
	Pearled/pot barley	0.018	0.08
	Flour	0.70	3.18
	Bran	1.2	5.45
	Brewing malt	0.067	0.30
	Malt sprouts	0.24	1.09
	Beer	<0.01	<0.05
	Brewers grain (dried)	0.47	2.14
	Brewers yeast	0.042	0.19

Notes:

[1] DALA = Days after last application.

[2] Mefentrifluconazole residue values of the Processing RAC (i.e. barley grain samples analysed just prior to processing) were used for the calculation of the processing factors.

[3] RAC = raw agricultural commodity.

Rice

Three processing trials were carried out in the United States during the 2014 growing season (Reeves, 2019, BASF DocID 2015_7005931) where rice received two foliar applications each at a rate of 449–547 g ai/ha, at 14 day intervals, using an EC formulation containing 100 g/L mefentrifluconazole. An adjuvant was added to the spray mixture for all applications. Rice grain were harvested 21 days after the last application (DALA). Rice grain samples were stored ambient for durations less than 48 hours or stored frozen and shipped by freezer truck to the processing facility. At the processing facility, samples were stored frozen until processed into hulls, polished rice, and bran according to commercial practices.

Rice gain (60 kg) were cleaned by aspiration and screening using an aspirator to remove light impurities and then screened to separate foreign particles from the cleaned rough rice. The cleaned rough rice was then milled in a rice mill where the hull was removed in the hulling portion of the mill by rubber rollers. The hull material was separated from the brown rice by aspiration. After dehulling, the brown rice was milled in the milling chamber into white milled rice and bran by friction. Bran was separated from the white milled rice by air injection into the milling chamber. After exiting the milling chamber, bran was sieved to remove broken pieces of brown and white milled rice and small amounts of hull material from bran. Fractions of hull material, white milled rice (polished rice) and bran were collected for analysis.

Residues of mefentrifluconazole in rice and all processed commodities were determined using LC-MS/MS method D1511/01. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the rice and processed commodities and analysis was 723 days for the RAC and 437 days for the processed fractions. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 165.

Table 165 Mefentrifluconazole residues in rice and processed fractions with corresponding processing factors

Trial, location, year, variety Total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor[2]
R140858 Fisk, MO, United States, 2014 CL XL 745 898 g ai/ha DALA=21	Grain, Field RAC	7.22	-
	Grain, Processing RAC	2.01	-
	Hulls	8.37	4.16
	Polished rice	0.13	0.06
	Bran	1.16	0.58
R140859 Cheyneville, LA, United States, 2014 CL 111 1012 g ai/ha DALA=21	Grain, Field RAC	1.45	-
	Grain, Processing RAC	4.74	-
	Hulls	23.33	4.92
	Polished rice	0.11	0.02
	Bran	6.73	1.42
R140860 Biggs, CA, United States, 2014 M2 06 905 g ai/ha DALA=21	Grain, Field RAC	11.24	-
	Grain, Processing RAC	13.71	-
	Hulls	34.92	2.55
	Polished rice	0.08	0.01
	Bran	14.21	1.04

Notes:

* The mean values obtained in different weightings of the same sample.

DALA = Days after last application.

[2] In the United States study, mefentrifluconazole residue values of the Processing RAC (i.e. rice grain samples analysed just

prior to processing) were used for the calculation of the processing factors.

[3] RAC = raw agricultural commodity.

Maize

Three processing trials were carried out in the United States during the 2014 and 2015 growing seasons (Delinsky, 2016, BASF DocID 2016_7009425) where maize received two foliar applications each at a rate of 296–301 g ai/ha or 443–446 g ai/ha, at 14 to 22-day intervals, of an EC formulation containing 100 g/L mefentrifluconazole. An adjuvant was added to the spray mixture for all applications. Forage was harvested 13–17 days after the last application (DALA) from corn treated at the higher application rate. Maize grain was harvested 20–21 DALA from corn treated at the lower application rate and 52–69 DALA from corn treated at the higher application rate. Forage samples for processing were shipped under chilled conditions the day of harvest to the processing facility where they were stored at ambient temperature until processing. Grain samples for processing were either stored at ambient temperature for 2 days after harvest (R140861), shipped frozen on the day of harvest (R140862), or frozen for 4 days after harvest until being shipped frozen to the processing facility where all grain samples were stored frozen until processing. At the processing facility, samples were processed into silage, aspirated grain fractions, flour (wet milling), flour (dry milling), bran, gluten, gluten feed meal, starch (wet milling), germ, refined oil (wet milling), meal (dry milling), grits (dry milling), milled byproduct, and refined oil (dry milling) according to commercial practices.

Maize forage samples (55 kg) were fed into a chipper/shredder. The chopped material was placed in a container (micro-silo) and sealed for 21 days. Ensiled samples (silage) were collected for analysis.

Maize grain samples (300 kg) from the lower application were dried in an oven to a moisture content of 10.0–13.0 percent. Samples were then aspirated to remove light impurities (grain dust). Light impurities were classified using sieves of varying microns. The material passing through the 2360 micron sieve was recombined to produce one aspirated grain fraction (AGF).

Maize grain samples from the higher application (248 kg) were dried in an oven to a moisture content of 10.0–15.0 percent. Samples were then cleaned by aspiration and screening. Large and small screenings and light impurities were combined to produce milled byproducts.

In dry milling processes, cleaned whole maize grain (102 kg) was adjusted to a moisture content of 20.0–22.0 percent and then fed into a disc mill to crack the kernel. Corn stock from the mill was dried for 30 minutes at approximately 54–71 °C. Dried cornstock was screened with 0.31 cm screen to separate bran, germ, and large grits from grits, meal and flour. The latter three were separated using 14 mesh (~0.14 cm) and 62 mesh (~0.025 cm) sieves. The fraction on top of the 14 mesh sieve was grits; the fraction on top of the 62 mesh sieve was meal and the fraction through the 62 mesh sieve was flour. Bran, germ, and large grits were screened again to separate hull, germ, and grits using 0.51 cm and 0.48 cm screens. Germ fractions were dried to a final moisture content of 14.0–16.0 percent and processed fraction samples of grits, meal, flour, bran, and germ were collected for analysis.

Germ material (13 kg) was heated in a mixer for 10 minutes. Following heating, the material was flaked in a flaking roll with a gap setting of 0.018–0.025 cm. After flaking, some samples were sieved to remove any remaining endosperm from the germ flakes. Flaked material was placed in stainless steel batch extractors and after 30 minutes the miscella (crude oil and hexane) was drained and hexane was added to repeat the cycle two more times. Final two washings were at the same temperature range for 15– 30 minutes each. Following the final draining, the spent flakes were desolventized with ambient air to remove residual hexane. The resulting fractions were miscella and solvent extracted germ flakes. The

miscella was passed through a laboratory vacuum evaporator to separate the crude oil from the hexane. Crude oil was heated for hexane removal, filtered, and collected for refining. For alkali refining, the free fatty acid (FFA) of the crude oil was determined. Based on the FFA, weighed amounts of crude oil and 16 degree sodium hydroxide were placed in a water bath and mixed for 15 minutes on high RPM and then for 12 minutes on low RPM. Neutralized oil and soapstock were separated using centrifugation. Alkali refined oil samples were decanted and filtered prior to bleaching (by the addition of activated bleaching earth, 1.0 percent by weight of oil). The bleached oil was filtered. The resulting fractions were bleached oil and spent bleaching earth/filter aid. Bleached oil was steam bathed under vacuum. The oil was allowed to cool. During the cooling period a 0.5 percent citric acid solution was added to the oil. Resulting fractions were refined-bleached-deodorized oil (RBD oil) and deodorizer distillates. The RBD oil samples were collected for analysis.

In the wet milling processes, dried and cleaned maize grain (79 kg) were steeped in water containing 0.1–0.2 percent sulfurous acid for 22–48 hours. Steeped whole corn was passed through a disc mill and a majority of the germ and hull was removed using a hydroclone (water centrifuge). Germ and hull were dried to obtain a final moisture content between 5–10 percent. After drying, the germ and hull were separated using aspiration and screening. Cornstock (without germ and hull) ground in the disc mill was passed over a separator equipped with a 325 mesh (50 micron) screen. Process water passing through the screen was separated into starch and gluten using batch centrifugation. Starch was dried in an oven until the moisture content was less than 15.0 percent. The remainder of the gluten was dried utilizing a steam-heated drum dryer. The processed fraction samples of starch, gluten and gluten feed meal were collected for analysis.

Germ fractions (6 kg) were adjusted to a moisture content of 12 percent, heated in a mixer, flaked in the flaking roll and pressed in an expeller to liberate part of the crude oil. Resulting fractions are expelled crude oil and presscake with residual crude oil. The presscake was placed in stainless steel batch extractors and submerged for 30 minutes. The miscella was dried and fresh hexane added to repeat the cycle two more times. Following the final draining, the spent presscake was desolventized with ambient air to remove residual hexane. Resulting fractions from solvent extraction were miscella and solvent extracted presscake (germ cake). Miscella was passed through a laboratory vacuum evaporator to separate the crude oil from hexane. Crude oil was then heated for hexane removal. Crude oils from expelling and solvent extraction were filtered and combined for refining. Crude oil samples from the wet milling process were alkali refined, bleached, and deodorized utilizing the same methods used during the dry milling procedure.

For the production of wet milled flour, cleaned whole corn sample was boiled in solution of calcium hydroxide and water for 28–32 minutes and then steeped overnight. The liquid was drained and the corn was rinsed to remove fiber. The remaining corn was ground in a mill and the ground material was pressed into thin pieces and dried for 2–2.5 hours. The dried material was ground again in a pin mill and sieved with a 40 mesh screen. Material passing through the screen was wet milled flour, which was collected for analysis.

Residues of mefentrifluconazole in corn and all processed commodities were determined using LC-MS/MS method D1511/01. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the maize and processed commodities and analysis was 577 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 166.

Table 166 Mefentrifluconazole residues in maize and processed fractions with corresponding processing factors

Trial, location, year, variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA[1]	Mefentrifluconazole (mg/kg)	Processing Factor[2]
R140861 Fisk, MO, United States, 2014 RL8899YHB	Forage, Field RAC[3]	889	17	3.29, 2.85 [3.07]	-
	Forage, Processing RAC			1.36	-
	Silage			1.80	1.32
	Grain, Field RAC	298	21	<0.01, <0.01 [<0.01]	-
	Grain, Processing RAC			<0.01	-
	Aspirated grain fraction (AGF)			0.21	n/a
	Grain, Field RAC	889	59	<0.01, <0.01 [<0.01]	-
	Grain, Processing RAC			<0.01	-
	Flour – wet milling			<0.01	n/a
	Flour – dry milling			<0.01	n/a
	Bran			<0.01	n/a
	Gluten			<0.01	n/a
	Gluten feed meal			<0.01	n/a
	Starch			<0.01	n/a
	Germ			<0.01	n/a
	Meal			<0.01	n/a
	Grits			<0.01	n/a
	Milled byproducts			0.032	n/a
	Oil refined – dry milling			<0.01	n/a
	Oil refined – wet milling			<0.01	n/a
R140862 Richland, IA, United States/ 2014	Forage, Field RAC	892	14	2.60, 1.80 [2.20]	-
	Forage, Processing RAC			2.96	-
	Silage			2.55	0.86
	Grain, Field RAC	297	20	<0.01, <0.01 [<0.01]	-
	Grain, Processing RAC			<0.01	-
	Aspirated grain fraction (AGF)			0.24	n/a
	Grain, Field RAC	892	52	<0.01, 0.011 [<0.011]	-
	Grain, Processing RAC			<0.01	-
	Flour – wet milling			<0.01	n/a
	Flour – dry milling			<0.01	n/a
	Bran			0.017	n/a
	Gluten			<0.01	n/a
	Gluten feed meal			0.027	n/a
	Starch			<0.01	n/a
	Germ			<0.01	n/a
	Meal			<0.01	n/a
	Grits			<0.01	n/a
	Milled byproducts			0.101	n/a
	Oil refined – dry milling			<0.01	n/a
	Oil refined – wet milling			<0.01	n/a
R140863 York, NE, United States/ 2014	Forage, Field RAC	888	13	2.84, 3.00 [2.92]	-
	Forage, Processing RAC			2.88	-
	Silage			1.61	0.56
	Grain, Field RAC	296	21	<0.01, <0.01 [<0.01]	-
	Grain, Processing RAC			<0.01	-
	Aspirated grain fraction (AGF)			0.25	n/a
	Grain, Field RAC	888	69	<0.01, <0.01 [<0.01]	-
	Grain, Processing RAC			<0.01	-
	Flour – wet milling			<0.01	n/a
	Flour – dry milling			<0.01	n/a
	Bran			<0.01	n/a

Trial, location, year, variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA[1]	Mefentrifluconazole (mg/kg)	Processing Factor[2]
	Gluten			<0.01	n/a
	Gluten feed meal			<0.01	n/a
	Starch			<0.01	n/a
	Germ			<0.01	n/a
	Meal			<0.01	n/a
	Grits			<0.01	n/a
	Milled byproducts			0.088	n/a
	Oil refined – dry milling			<0.01	n/a
	Oil refined – wet milling			<0.01	n/a

Notes:

[1] DALA = Days after last application.

[2] Mefentrifluconazole residue values of the Processing RAC (i.e. corn samples analysed just prior to processing) were used for the calculation of the processing factors. n/a = not applicable. Since the corn grain processing RACs contained residues <LOQ, no processing factors could be calculated. However, a concentration of the residue was observed in several processed commodities (e.g. AGF, bran, gluten feed meal, milled byproducts).

[3] RAC = raw agricultural commodity.

Cotton

Three processing trials were carried out in the United States during the 2017 growing season (Moore and Phillips, 2019, BASF DocID 2018/7007471) where cotton received three foliar applications at a rate of 734–767 g a.i./ha, at 7 day intervals, of an emulsifiable concentrate containing 100g/L mefentrifluconazole. An adjuvant (non-ionic surfactant) was added to the spray mixture for all applications. Undelinted cottonseed and bulk seed cotton were collected from the untreated plot and treated approximately 30 days after last application (DALA). Undelinted cottonseed samples were placed into frozen storage within 3 hours of collection and transported to the processing facility by freezer truck. Bulk seed cotton samples were stored ambient for durations less than 55 hours, stored frozen and then shipped to the processing facility by freezer truck. At the processing facility, bulk seed cotton samples were processed into cottonseed hulls, cottonseed meal and refined oil according to commercial practices.

To produce undelinted cottonseed (RAC) fractions, seed cotton was cleaned with a stick extractor to remove the gin byproducts (gin trash) and ginned to separate ginned cottonseed (undelinted cottonseed) and lint in a Continental cotton gin. The resulting products were ginned cottonseed, lint, and gin trash. Undelinted cottonseed (RAC) fractions were collected and placed into frozen storage. The ginned cottonseed samples were mechanically delinted to produce delinted cottonseed.

To produce hull fractions, delinted cottonseed was cracked in a roller mill and the kernel and hull material were separated with a screen cleaner equipped with 10/64-inch and/or 12/64-inch screens. The hull material fractions were collected and the moisture content of the kernel was determined with an electronic moisture analyser. The moisture content of the kernel was adjusted to 13.5 percent by adding water and mixing. The samples were then allowed to equilibrate for a minimum of 2 hours.

For production of meal and refined oil, kernel material was heated in a steam heated mixer to 79–91 °C and held for 28–32 minutes. After heating, the material was flaked in a flaking roll with a gap setting of 0.008–0.013 inch. The flakes were extruded in a continuous processor (extruder) where they were turned into collets by direct steam injection. After extrusion, the material (collets) were ground and dried in an oven at 66–82 °C for 30–40 minutes. Ground collets were placed in stainless steel batch extractors and submerged in 49–60 °C hexane for 30 minutes, drained, and repeated two more times for 15 minutes each. The extracted solvent meal was toasted by steam injection, heated to 102–104 °C,

stopped at 104–116 °C and held there for 45 minutes. After cooling, the toasted meal samples were passed through a screen cleaner equipped with a 1/8-inch sieve. Toasted cottonseed meal was collected and placed into frozen storage. The crude oil and hexane from the miscella were separated by vacuum evaporation. The crude oil was heated to 91–96 °C, filtered and fractions were then collected for analysis. Crude oil was neutralized by adding sodium hydroxide and mixing at a high RPM in a water bath for 14–16 minutes (at 21–24 °C), followed by a low RPM for 11–13 minutes (at 63–67 °C). The neutralized oil was centrifuged, the refined oil was decanted and filtered and the resultant fractions were alkali refined oil and soapstock (the latter was discarded). The refined oil was heated to 40–50 °C and bleached by the addition of activated bleaching earth (1 percent by weight of oil). The temperature was increased to 85–100 °C and held for 10–15 minutes. After vacuum filtration, the resulting fractions were bleached oil and spent bleaching earth (the later was discarded). Bleach oil was deodorized by steaming under vacuum for 28–32 minutes (at 220–230 °C), followed by the addition of 0.5 percent citric acid solution (1 mL/100 g oil). The resulting fractions were alkali refined, bleached, and deodorized (RBD) oil. RBD oil was collected and placed in frozen storage.

Residues of mefentrifluconazole in cotton and all processed commodities were determined using LC-MS/MS method D1511/01. The limit of quantification (LOQ) was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the cotton and processed commodities and extraction for analysis was 364 days. All samples were analysed within 0 to 8 days of extraction. Processing factors are shown in Table 167.

Table 167 Concentration of mefentrifluconazole residues in cotton processed fractions and processing factors

Trial, location, year, variety Total rate	Commodity or Matrix	DALA[1]	Mefentrifluconazole Residues (mg/kg)	Processing Factor[2]
R170067 Jeffersonville, GA, United States, 2017 ST 6182GLT 2234 g ai/ha	Cottonseed RAC	33	0.47	-
	Processor RAC[3]	33-35	0.22	-
	Hulls		0.18	0.82
	Meal		0.013	0.06
	Refined oil		<0.01	<0.05
R170068 Uvalde, TX, United States, 2017 DP 1044B2RF 2257 g ai/ha	Cottonseed RAC	28	0.84	-
	Processor RAC		0.49	-
	Hulls		0.066	0.13
	Meal		<0.01	<0.02
	Refined oil		<0.01	<0.02
R170069 Edmonson, TX, United States, 2017 FiberMax 1911 2216 g ai/ha	Cottonseed RAC	30	2.91	-
	Processor RAC		5.45	-
	Hulls		0.41	0.08
	Meal		0.021	0.004
	Refined oil		0.022	0.004

Notes:

[1] DALA = Days after last application.

[2] The processing factor was calculated by dividing the residue in the processed fraction by the residue for the RAC sample (processor RAC).

[3] RAC = Raw agricultural commodity.

Coffee

Four processing trials were carried out in Brazil during the 2017 and 2018 growing seasons (José, 2019, BASF DocID 2019_2041531) where coffee received three foliar applications each at a rate of 783–

860 g ai/ha, at 60 day intervals, of an SC formulation containing 200 g/L each of mefentrifluconazole and pyraclostrobin. Pyraclostrobin residues were not investigated in this study. Coffee fruit were harvested 45 days after the last application (DALA). After sampling, coffee fruits were dried at ambient temperature. For the dried coffee samples, the dried fruits were sifted to remove leaves and branches. For the coffee bean samples, the dried fruits were peeled using a small processing machine to generate the dried coffee cherry samples. Samples were shipped frozen to the processing facility and remained frozen until processing. At the processing facility, samples were processed into concentrated liquor, instant coffee, and roasted ground coffee according to commercial practices.

Green coffee beans were roasted for 20 minutes at 200 °C to obtain a medium-strong roasted coffee. Distilled water was used to stop the roasting process. After roasting, the coffee beans were stored at room temperature for at least 18 hours to expel the CO_2 generated during roasting and to equilibrate its moisture content. Coffee beans were then ground in a cone mill.

To produce concentrated liquor, green coffee beans were roasted at 180 °C for 80–90 minutes and then stored at room temperature for at least 18 hours. The roasted coffee beans were then broken in a cone mill and sieved to remove the "fines". The coffee was then extracted using a coffee extraction pilot equipment containing seven columns of extraction. Filtered heated water (92 ± 4 °C) was pumped into the columns (water flow was 0.48 mL/minute) through the base and percolated the column in contact with the coffee. The extract was collected from the column. To generate instant coffee, coffee extract was left at room temperature for one hour and then dried in a spray dryer (air flow of 0.8 L/min, temperature of 175–180 °C).

Residues of mefentrifluconazole in coffee beans and processed commodities were determined using LC-MS/MS method L0076/09. The LOQ was 0.01 mg/kg for the RAC and all processed commodities. The maximum duration between sampling of the coffee and processed commodities and analysis was 253 days. Adequate storage stability data are available to support the storage conditions and durations for the samples in the present study. Processing factors are shown in Table 168.

Table 168 Mefentrifluconazole residues in coffee and processed fractions with corresponding processing factors

Trial, location, year, variety Total rate	Commodity or Matrix	Mefentrifluconazole (mg/kg)	Processing Factor
G160341 Indianópolis, MG, Brazil, 2017 Red Catuai 2415 g ai/ha DALA=45	Coffee beans RAC	0.25	-
	Dried coffee bean	3.4	13.6
	Concentrated liquor	0.016	0.06
	Instant coffee	0.046	0.18
	Roasted ground coffee	0.15	0.60
G160342 Araguari, MG, Brazil, 2017 Red Catuai 2394 g ai/ha DALA=45	Coffee beans RAC	0.204	-
	Dried coffee bean	2.6	12.7
	Concentrated liquor	0.020	0.10
	Instant coffee	0.071	0.01
	Roasted ground coffee	0.19	0.93
G160372 Leme, SP, Brazil, 2018 Obatã 2593 g ai/ha DALA=45	Coffee beans RAC	0.30	-
	Dried coffee bean	0.31	1.03
	Concentrated liquor	0.023	0.08
	Instant coffee	0.047	0.16
	Roasted ground coffee	0.19	0.63
G160373 Rio Claro, SP, Brazil, 2018 Obatã 2507 g ai/ha DALA=45	Coffee beans RAC	0.31	-
	Dried coffee bean	0.30	0.97
	Concentrated liquor	0.021	0.07
	Instant coffee	0.051	0.16
	Roasted ground coffee	0.18	0.58

Notes:

[1] DALA = Days after last application.

[2] RAC = raw agricultural commodity.

A summary of all processing factors estimated in various crops are shown in Table 169.

Table 169 Summary of mefentrifluconazole processed fractions with corresponding processing factors

Raw commodity	Processed commodity	Processing Factors	
		Individual	Best estimate
Orange	Juice	<0.02, <0.01, <0.02	0.02
	Wet pomace	1.5, 2.2, 1.7	1.7
	Dried pomace	6.2, 8.0, 6.4	6.4
	Pulp	0.02, <0.03, 0.02	0.02
	Dried pulp	0.15, 0.04, 0.11	0.11
	Peel	2.6, 3.3, 2.6	2.6
	Peel after oil extraction	1.4, 2.4, 1.8	1.8
	Oil	38, 71, 41	41
	Marmalade	0.09, 0.12, 0.31	0.12
Apple	Washed whole apples	0.75, 0.68, 0.81	0.75
	Canned apples	0.05, <0.13, 0.25	0.13
	Fruit syrup	0.40, 0.88, 0.38	0.40
	Apple sauce	0.05, <0.13, 0.11	0.11
	Dried apples	0.31, 0.25, 0.33	0.31
	Juice	0.09, <0.13, 0.16	0.13
	Wet pomace	3.10, 3.25, 2.36	3.10
	Dried pomace	11.5, 9.88, 7.51	9.88
Plum	Washed whole plum	1.08, 1.16, 1.04	1.1
	Dried prune	2.57, 4.26, 4.08	4.1
	De-pitted plum	0.98, 1.16, 1.12	1.1
	Juice	0.08, 0.20, 0.15	0.15
	Puree	0.76, 0.43, 0.56	0.56
Peach	Pulp	0.21, 0.27, 0.05, 0.04	0.13
Grape	Raisins	2.5, 3.93, 3.73	3.73
	Rosé Wine Process		
	Must naturally cloudy	0.11, 0.14, 0.13	0.13
	Pomace	3.13, 3.93, 3.09	3.13
	Must deposit	0.44, 0.75, 0.89	0.75
	Must separated	0.07, 0.06, 0.07	0.07
	Pasteurized juice	0.04, 0.05, 0.05	0.05
	Yeast deposit	0.35, 0.54, 0.75	0.54
	Rosé wine	0.02, 0.02, 0.03	0.02
	Red Wine Process		
	Stalks	1.54, 1.64, 1.82	1.64
	Crush	1.63, 1.54, 1.00	1.54
	Must naturally cloudy	0.21, 0.16, 0.18	0.18
	Pomace	5.21, 4.26, 3.55	4.26
	Must deposit	0.38, 0.20, 0.18	0.20
	Must separated	0.16, 0.14, 0.14	0.14
	Pasteurized juice	0.12, 0.13, 0.13	0.13
	Yeast deposit	1.0, 1.11, 1.18	1.11
	Red wine	0.03, 0.02, 0.03	0.03
Strawberry	Washed strawberries	1.19, 0.60, 0.70	0.70
	Canned strawberries	0.93, 0.77, 1.18	0.93
	Fruit syrup	0.20, 0.17, 0.30	0.20

Raw commodity	Processed commodity	Processing Factors	
		Individual	Best estimate
	Jam before cooking	0.48, 0.21, 0.38	0.38
	Jam after cooking	0.48, 0.25, 0.43	0.43
Cucumber	Washed gherkins	0.29, 0.52, 0.52	0.52
	Canned gherkins	1.73, 0.52, 0.88	0.88
	Pickled gherkins	0.73, 0.26, 0.84	0.73
	Brine	<0.18, <0.05, <0.13	0.13
	Vegetable stock	<0.18, 0.06, 0.13	0.13
Tomato	Blanched tomatoes	0.06, 0.05, 0.06	0.06
	Canned tomatoes	0.06, 0.08, 0.05	0.06
	Ketchup after pasteurization	0.35, 0.68, 0.56	0.56
	Paste	1.00, 0.49, 0.46	0.49
	Peeled tomatoes	0.07, 0.06, 0.03	0.06
	Puree	0.31, 0.28, 0.20	0.28
	Raw juice	0.11, 0.08, 0.08	0.08
	Sun-dried tomatoes	6.67, 9.17, 15.97	9.17
	Tomato peel	5.33, 4.17, 2.42	4.17
	Vegetable stock	0.10, 0.22, 0.02	0.10
	Washed tomatoes	0.67, 0.67, 0.93	0.67
	Wet pomace	2.93, 1.75, 7.14	2.93
Soya bean	Aspirated grain fraction	93	93
	Hulls	<0.83	0.83
	Meal (toasted)	<0.83	0.83
	Crude oil	1.0	1.0
	Tofu	<0.83	0.83
	Soya sauce	<0.83	0.83
	Pollards	<0.83	0.83
	Flour	<0.83	0.83
	Miso	<0.83	0.83
	Soy milk	<0.83	0.83
	Refined oil	<0.83	0.83
	Meal (untoasted)	<0.83	0.83
Sugar beet	Molasses	0.88, 1.1, 0.53	0.53
	Raw sugar	<0.06, <0.05, 0.10	0.06
	Affinated sugar	0.11, 0.11, 0.18	0.11
	Refined sugar	<0.06, <0.05, 0.10	0.06
	Dried pulp	4.75, 5.24, 3.24	4.75
	Ensiled pulp	0.88, 1.14, 0.68	0.88
Potato	Peeled tuber	<0.5, <0.33	0.33
	Wet peel	1.5, 1.67	1.6
	Boiled	<0.5, <0.33	0.33
	Microwaved/boiled	<0.5, <0.33	0.33
	Baked	<1.0, <0.33	0.33
	Fried	<0.5, <0.33	0.33
	Crisps	<0.5, <0.33	0.33
	Chips	<0.5, <0.33	0.33
	Granules/flakes	<0.5, <0.33	0.33
	Starch	<0.5, <0.33	0.33
	Dried pulp	3.5, 1.33	2.4
	Potato protein	3.5, 1.0	2.3
Wheat forage	Wet silage	1.10, 1.44, 1.19	1.2
	Wilted silage	8.0, 4.7, 6.5	6.5
Wheat grain	Bran	2.38, 3.71, 2.94	2.94
	Germ	0.85, 1.82, 1.12	1.12
	Middlings	1.92, 3.88, 2.26	2.26

Raw commodity	Processed commodity	Processing Factors	
		Individual	Best estimate
	Shorts	2.62, 4.53, 3.53	3.53
	Gluten	0.55, <0.59, 0.44	0.55
	Gluten feed meal	0.29, <0.59, <0.29	0.29
	Starch	<0.08, <0.59, <0.29	0.29
	Whole meal flour	0.77, 1.00, 0.79	0.79
	Whole grain bread	0.54, <0.59, 0.56	0.56
	Milled byproducts	0.62, 1.12, 0.41	0.62
	Aspirated grain fractions	38.46, 21.76, 44.12	38.46
Barley	Pearled/pot barley	0.16, 0.12, 0.08	0.12
	Flour	4.5, 3.67, 3.18	3.67
	Bran	4.25, 5.00, 5.45	5.00
	Brewing malt	0.50, 0.50, 0.30	0.50
	Malt sprouts	1.68, 0.96, 0.30	0.96
	Beer	<0.03, <0.04, <0.05	0.03
	Brewer's grain (dry)	2.38, 2.42, 2.14	2.38
	Brewer's yeast	0.19, 0.27, 0.19	0.19
Rice	Hulls	4.16, 4.92, 2.55	4.16
	Polished rice	0.06, 0.02, 0.01	0.02
	Bran	0.58, 1.42, 1.04	1.04
Maize, forage	Silage	1.32, 0.86, 0.56	0.86
Cotton	Hulls	0.82, 0.13, 0.08	0.13
	Meal	0.06, <0.02, 0.004	0.02
	Refined oil	<0.05, <0.02, 0.004	0.004
Coffee	Dried coffee cherry	13.6, 12.7, 1.03, 0.97	6.865
	Concentrated liquor	0.06, 0.10, 0.08, 0.07	0.075
	Instant coffee	0.18, 0.01, 0.16, 0.16	0.16
	Roasted ground coffee	0.60, 0.93, 0.63, 0.58	0.615

Livestock feeding studies

Dairy cow feeding study

Mefentrifluconazole was administered orally once daily to five groups of three lactating Holstein/Friesian/Ayrshire cross dairy cattle (2–14 years of age; 496–752 kg bw) by gelatine capsule for 28 days (Bancroft, 2015, BASF DocID 2016_1190690). Mean daily feed consumption for the dose groups during the exposure period were 7.71–20.13 kg dry matter/day. Mean daily milk yields for the dose groups during the dosing period were 6.7 to 16.9 kg/cow/day. Based on mean daily feed consumption, the dosing levels were equivalent to 1.5, 7.5, 50 and 150 ppm in the feed. Milk was collected twice daily (am and pm sampling pooled) throughout the 28 days of dosing. On day 21, milk was also separated into cream and skimmed milk. Muscle (loin, hindleg), liver, kidney and fat (peri-renal, mesenterial and subcutaneous) samples were collected at sacrifice 22–24 hours after the final dose, except for three of the six cows from the highest dose tested group which were sacrificed three, seven and fourteen days after the final dose to monitor the decline of residue levels post dosing. The maximum frozen storage intervals were 49 days for milk while the maximum storage intervals for tissues were 97, 110, 35 and 90 days for liver, kidney, muscle and fat, respectively. Samples were analysed for the parent compound using the LC-MS/MS analytical method L0272/01 and for the metabolite M750F022 using the GC-MS method L0309/01. The LOQ for each analyte was 0.010 mg/kg.

The methods were concurrently validated by fortifying samples of milk, cream, skimmed milk, muscle, liver, kidney and fat, with known amounts of mefentrifluconazole and M750F022. Mean

recoveries of the parent compound in milk (whole and skimmed), cream and tissues ranged from 70 percent to 92 percent with relative standard deviations of 3 percent to 11 percent. Likewise, mean recoveries of the metabolite in milk and tissues ranged from 73 percent to 105 percent with relative standard deviations of 5 percent to 14 percent. The validation results demonstrated the acceptability of the methods used in this study. The results of the study are shown in Tables 170 and 171.

Table 170 Residues of mefentrifluconazole and metabolite M750F022 in milk (including skimmed milk and cream)

	Dose Groups				
Study day	1.5 ppm	7.5 ppm	50 ppm	150 ppm	150 ppm
Mefentrifluconazole, mg/kg					
-1	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]
1	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.022, 0.025, 0.029 [0.025]	0.081, 0.089, 0.099 [0.090]	0.073, 0.104, 0.112 [0.096]
3	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <u0.01 [<0.01]	0.049, 0.063, 0.064 [0.058]	0.178, 0.202, 0.246 [0.209]	0.151, 0.166, 0.282 [0.200]
5	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.046, 0.047, 0.048 [0.047]	0.132, 0.177, 0.203 [0.171]	0.184, 0.230, 0.357 [0.257]
7	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.047, 0.055, 0.058 [0.053]	0.127, 0.183, 0.222 [0.177]	0.159, 0.224, 0.337 [0.240]
10	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.052, 0.063, 0.067 [0.061]	0.172, 0.207, 0.265 [0.215]	0.162, 0.221, 0.337 [0.240]
14	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.060, 0.071, 0.110 [0.080]	0.184, 0.192, 0.273 [0.216]	0.229, 0.247, 0.344 [0.273]
17	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.050, 0.065, 0.078 [0.064]	0.110, 0.127, 0.268 [0.168]	0.119, .0157, 0.210 [0.162]
21	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.050, 0.060, 0.083 [0.064]	0.173, 0.233, 0.354 [0.253]	0.168, 0.207, 0.368 [0.248]
24	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.046, 0.058, 0.063 [0.056]	0.161, 0.168, 0.280 [0.203]	0.205, 0.239, 0.283 [0.242]
28	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.047, 0.053, 0.059 [0.053]	0.169, 0.226, 0.248 [0.214]	0.159. 0.187. 0.258 [0.201]
29	Not analysed				0.060, 0.124, 0.128 [0.104]
30					0.024, 0.042, 0.046 [0.037]
31					<0.01, 0.015 [0.012]
32					<0.01, <0.01 [<0.01]
33					<0.01, <0.01 [<0.01]
34					<0.01
35					<0.01
36					<0.01
37					<0.01
38					<0.01
39					<0.01
40					<0.01
41					<0.01
Skimmed milk (day 21)	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, 0.010, 0.016 [0.012]	0.026, 0.076, 0.103 [0.099]	0.028, 0.033, 0.073 [0.044]
Cream (day 21)	<0.01, <0.01, <0.01 [<0.01]	0.043, 0.052, 0.061 [0.052]	0.382, 0.431, 0.459 [0.424]	0.563, 1.19, 1.95 [1.23]	0.919, 1.29, 2.16 [1.46]
M750F022, mg/k					
Milk (day 21)	n.a.	n.a.	<0.01, <0.01, 0.01 [0.010]	n.a.	0.020, 0.021, 0.022 [0.021]

Study day	Dose Groups				
	1.5 ppm	7.5 ppm	50 ppm	150 ppm	150 ppm
Cream (day 21)	n.a.	<0.01, <0.01, <0.01 [<0.01]	<0.01, 0.010, 0.014 [0.011]	n.a.	0.090, 0.100, 0.108 [0.099]

Note:

Underlined values represent the maximum individual residue.

Table 171 Residues of mefentrifluconazole and metabolite M750F022 in dairy cattle tissues

Dose Groups	Muscle	Liver	Kidney	Perirenal fat	Mesenterial fat	Subcutaneous fat
Mefentrifluconazole (mg/kg)						
1.5 ppm	<0.01, <0.01, <0.01 [<0.01]	0.029, 0.031, 0.034 [0.031]	<0.01, 0.013, 0.014 [0.012]	0.016, 0.017, 0.018 [0.017]	0.018, 0.018, 0.018 [0.018]	0.012, 0.016, 0.017 [0.015]
7.5 ppm	<0.01, <0.01, <0.01 [<0.01]	0.112, 0.155, 0.182 [0.150]	0.028, 0.043, 0.074 [0.048]	0.029, 0.058, 0.059 [0.049]	0.030, 0.051, 0.077 [0.053]	<0.01, 0.017, 0.041 [0.023]
50 ppm	0.051,0.063, 0.105 [0.073]	0.643, 0.936, 1.40 [0.993]	0.047, 0.320, 0.505 [0.291]	0.461, 0.586, 0.900 [0.649]	0.456, 0.563, 0.566 [0.528]	0.171, 0.493, 0.784 [0.483]
150 ppm	0.128, 0.141, 0.221 [0.163]	2.50, 3.01, 3.58 [3.03]	0.944, 1.06, 1.88 [1.29]	0.942, 0.190, 2.29 [1.71]	0.652, 0.961, 1.87 [1.16]	0.019, 0.562, 1.20 [0.594]
150 ppm – 3 day withdrawal	0.063	0.885	0.275	0.536	2.25	1.47
150 ppm– 7 day withdrawal	<0.01	0.021	<0.01	0.017	0.023	0.322
150 ppm – 14 day withdrawal	<0.01	<0.01	<0.01	<0.01	<0.01	0.023
M750F022 (mg/kg)						
1.5 ppm	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
7.5 ppm	n.a.	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, 0.011 [<0.01]	<0.01, <0.01, <0.01 [<0.01]
50 ppm	<0.01, <0.01, <0.01 [<0.01]	0.019, 0.022, 0.022 [0.021]	0.018, 0.020, 0.020 [0.019]	0.075, 0.086, 0.090 [0.083]	0.075, 0.086, 0.090 [0.083]	0.026, 0.053, 0.077 [0.052]
150 ppm	0.014,0.016, 0.018 [0.016]	0.031, 0.039, 0.044 [0.038]	0.040, 0.041, 0.043 [0.041]	0.134, 0.143, 0.212 [0.163]	0.058, 0.114, 0.203 [0.125]	0.027, 0.061, 0.130 [0.073]
150 ppm– 3 day withdrawal	<0.01	0.016	0.012	0.096	0.149	0.109
150 ppm – 7 day withdrawal	<0.01	<0.01	<0.01	0.051	0.054	0.068
150 ppm – 14 day withdrawal	<0.01	<0.01	<0.01	<0.01	<0.01	0.023

Note:

Underlined values represent the maximum individual residue.

Laying hen feeding study

Mefentrifluconazole was administered orally once daily to 4 groups of 12 *Gallus domesticus* hens per group (each group further divided into 3 subgroups of 4 hens per subgroup) (22 weeks of age; 1.3–2.4 kg bw) by gelatine capsule for 34 days (Schatz, 2015, BASF DocID 2015_1106667). Mean daily feed consumption for the dose groups during the exposure period was 0.13 kg dry matter/day. Average egg production during the dosing period ranged from 5–7 eggs/week. Based on mean daily feed consumption, the dose levels were equivalent to 1.5, 4.5 and 15 ppm in the feed. Eggs were collected twice daily (am and pm sampling pooled) throughout the 34 days of dosing. On day 24, egg samples from the 15 ppm

dosing level were separated into egg yolk and egg white. Muscle, liver, skin with fat and abdominal fat samples were collected at sacrifice 6 hours after the final dose, except for 12 hens from the 15 ppm group which were sacrificed two, seven and fourteen days after the final dose to monitor the decline of residue levels post dosing. The maximum frozen storage intervals were 86 days for eggs while the maximum storage intervals for tissues were 88, 88, 97 and 60 days for muscle, liver, fat and skin with fat, respectively.

Samples were analysed for the parent compound using the LC-MS/MS analytical method L0272/01 and for the metabolite M750F022 using the GC-MS method L0309/01. The LOQ reported for each method was 0.010 mg/kg. The methods were concurrently validated by fortifying samples of eggs, muscle, liver, skin with fat and abdominal fat, with known amounts of mefentrifluconazole and M750F022. Mean recoveries of the parent compound in eggs, muscle, liver, skin with fat and abdominal fat samples ranged from 84 percent to 102 percent with relative standard deviations of 2 percent to 11 percent. Likewise, mean recoveries of the metabolite in eggs and tissues ranged from 71 percent to 90 percent with relative standard deviations of 11 percent to 15 percent. The validation results demonstrated the acceptability of the methods used in this study. The results of the study are shown in Table 172 and 173.

Table 172 Residues of mefentrifluconazole and metabolite M750F022 in eggs

	Dose Groups[A]			
Study day	1.5 ppm	4.5 ppm	15 ppm	15 ppm
Mefentrifluconazole (mg/kg)				
-1	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]
1	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]
3	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.011, <0.01, <0.01 [0.01]	<0.01, <0.01, <0.01 [<0.01]
5	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.024, 0.016, 0.011 [0.017]	0.019, 0.011, 0.014 [0.015]
7	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.038, 0.024, 0.022 [0.028]	0.023, 0.016, 0.020 [0.020]
10	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.041, 0.025, 0.025 [0.030]	0.028, 0.025, 0.024 [0.026]
14	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.042, 0.034. 0.030 [0.035]	0.030, 0.025, 0.025 [0.026]
17	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.035, 0.032, 0.027 [0.031]	0.029, 0.020, 0.022 [0.024]
21	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.030, 0.021, 0.022 [0.024]	0.030, 0.019, 0.022 [0.024]
24	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	Yolk:0.091, 0.060, 0.078 [0.076] White:<0.01, <0.01, <0.01 [<0.01]	0.032, 0.024, 0.024 [0.027]
28	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.037, 0.023, 0.031 [0.030]	0.029, 0.022, 0.033 [0.028]
33	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.036, 0.025, 0.028 [0.030]	0.037, 0.017, 0.020 [0.025]
35	Not analysed			0.029, 0.020, 0.021 [0.023]
40				<0.01, <0.01
47				<0.01
M750F022 (mg/kg)				
-1	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]
1	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]
3	<0.01, <0.01, <u0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.012, <0.01, <0.01 [0.011]	<0.01, <0.01, <0.01 [<0.01]
5	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.032, 0.020, 0.013 [0.022]	0.012, 0.013, 0.013 [0.012]

	Dose Groups[A]			
Study day	1.5 ppm	4.5 ppm	15 ppm	15 ppm
7	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.035, 0.021, 0.023 [0.026]	0.047, 0.036, 0.030 [0.038]
10	<0.01, <0.01, <0.01 [<0.01]	0.012, 0.010, 0.013 [0.012]	0.028, 0.052, 0.059 [0.046]	0.049, 0.051, 0.040 [0.047]
14	<0.01, <0.01, <0.01 [<0.01]	0.015, 0.015, 0.015 [0.015]	0.094, 0.073, 0.071 [0.079]	0.064, 0.070, 0.053 [0.062]
17	<0.01, <0.01, <0.01 [<0.01]	0.016, 0.013, 0.015 [0.015]	0.059, 0.061, 0.064 [0.061]	0.062, 0.050, 0.049 [0.054]
21	<0.01, <0.01, <0.01 [<0.01]	0.019, 0.020, 0.017 [0.019]	0.062, 0.063, 0.051 [0.059]	0.054, 0.051, 0.047 [0.051]
24	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	Yolk: 0.021, 0.015, 0.015 [0.017] White: <0.01, <0.01, <0.01 [<0.01]	0.056, 0.060, 0.054 [0.057]
28	<0.01, <0.01, <0.01 [<0.01]	0.012, 0.010, 0.012 [0.012]	0.061, 0.044, 0.053 [0.056]	0.065, 0.051, 0.048 [0.055]
33	<0.01, <0.01, <0.01 [<0.01]	0.016, 0.016, 0.015 [0.016]	0.075, 0.073, 0.064 [0.071]	0.076, 0.048, 0.052 [0.059]
35	Not analysed	0.069, 0.056, 0.067 [0.064]		
40		0.014, 0.014 [0.014]		
47		<0.01		

Notes:

Underlined values represent the maximum individual residue.

[A] Eggs from group 0.15 ppm were not analysed since no quantifiable residues were found in the two next higher dose groups.

Table 173 Residues of mefentrifluconazole and metabolite M750F022 in hen tissues

Dose Groups	Muscle	Liver	Fat	Skin with fat
	Mefentrifluconazole (mg/kg)			
0.15 ppm	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]
1.5 ppm	<0.01, <0.01, <0.01 [<0.01]	0.017, 0.011, <0.01 [0.013]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]
4.5 ppm	<0.01, <0.01, <0.01 [<0.01]	0.021, 0.012, 0.013 [0.015]	0.019, 0.021, 0.025 [0.022]	0.010, 0.011, 0.011 [0.011]
15 ppm	0.027, 0.010, <0.01 [0.016]	0.20, 0.06, 0.035 [0.097]	0.25, 0.15, 0.10 [0.17]	0.15, 0.08, 0.066 [0.10]
15 ppm – 2 day withdrawal	<0.01	<0.01	<0.01	<0.01
15 ppm – 7 day withdrawal	<0.01	<0.01	<0.01	0.017
15 ppm – 14 day withdrawal	<0.01	<0.01	<0.01	<0.01
M750F022 (mg/kg)				
0.15 ppm	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	<0.01, <0.01, <0.01 [<0.01]	0.015, <0.01, <0.01 [0.012]
1.5 ppm	<0.01, <0.01, <0.01 [<0.01]	0.018, 0.017, 0.019 [0.018]	0.024, 0.044, 0.030 [0.033]	0.012, 0.021, 0.018 [0.017]
4.5 ppm	<0.01, <0.01, <0.01 [<0.01]	0.033, 0.020, 0.030 [0.028]	0.064, 0.070, 0.071 [0.069]	0.036, 0.041, 0.035 [0.037]
15 ppm	0.037, 0.030, 0.031 [0.033]	0.20, 0.13, 0.12 [0.15]	0.36, 0.27, 0.30 [0.31]	0.19, 0.18, 0.19 [0.18]

Dose Groups	Muscle	Liver	Fat	Skin with fat
15 ppm – 2 day withdrawal	<0.01	0.015	0.061	0.037
15 ppm – 7 day withdrawal	<0.01	<0.01	0.013	<0.01
15 ppm – 14 day withdrawal	<0.01	<0.01	<0.01	<0.01

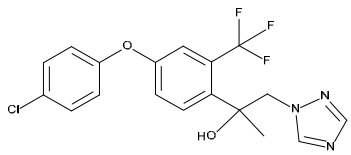
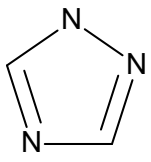
APPRAISAL

Mefentrifluconazole, (2RS)-2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl) propan-2-ol, is a triazole fungicide belonging to the group of the sterol biosynthesis inhibitors.

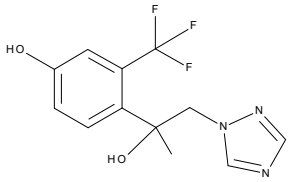
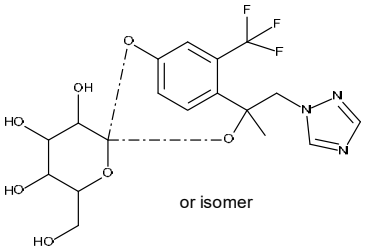
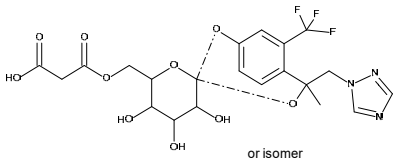
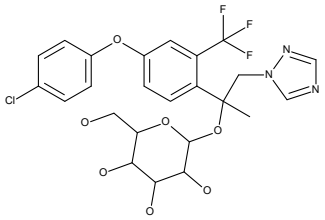
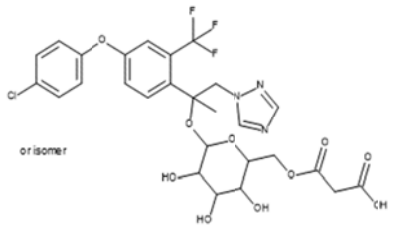
Mefentrifluconazole was scheduled at the Fifty-first Session of the CCPR for Evaluation for Residues and Toxicology by the 2020 JMPR, which was postponed to the 2021 JMPR Meeting, where an ADI of 0–0.04 mg/kg bw and an ARfD of 0.3 mg/kg bw were established. The Residue evaluation was rescheduled to the 2022 JMPR Meeting.

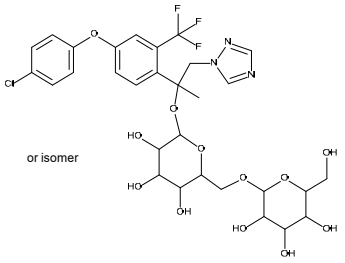
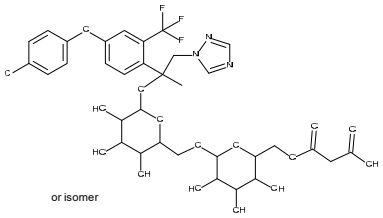
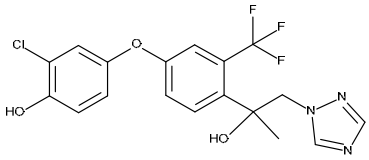
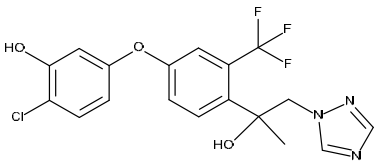
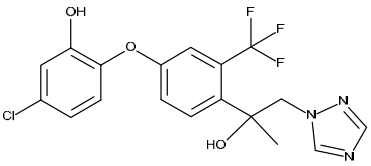
The Meeting received information from the manufacturer on physical and chemical properties, metabolism studies on plants and animals, environmental fate in soil, analytical method and stability in stored analytical samples, use patterns and supervised residue trials, processing studies and livestock feeding studies. A summary of metabolites found in metabolism studies are shown in Table 174.

Table 174 Summary information on mefentrifluconazole and its metabolites mentioned in this appraisal

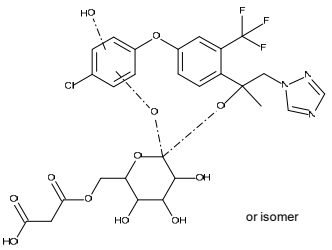
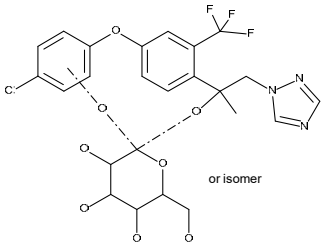
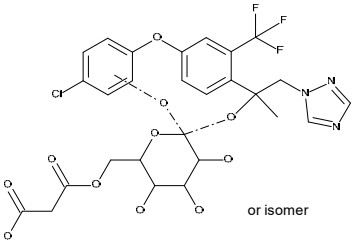
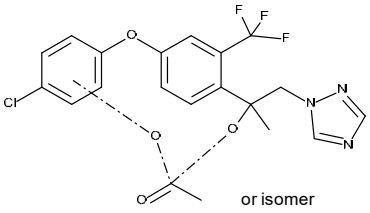
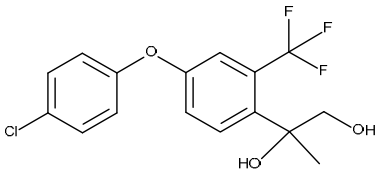
Code Number (Reg. Number)	Chemical Name	Molecular Structure	Occurrence in			
			Rat	Livestock (Hen & Goat)	Crop (Wheat, Grape & Soya bean)	Rotational Crops
Mefentrifluconazole (5834378)	(2RS)-2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-ol			x	x	
M750F001 (87084) 1,2,4-triazole	1,2,4-(1*H*)-triazole		x	x	x	x

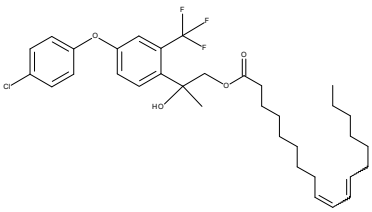
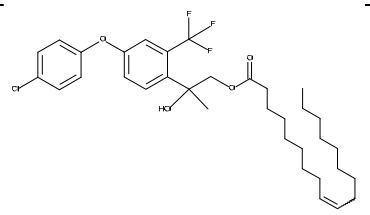
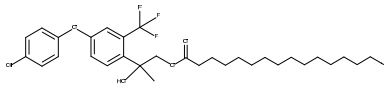
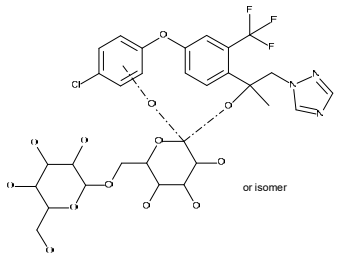
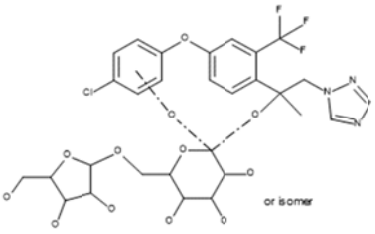
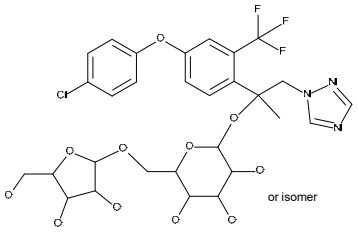
Mefentrifluconazole

Code Number (Reg. Number)	Chemical Name	Molecular Structure	Occurrence in			
			Rat	Livestock (Hen & Goat)	Crop (Wheat, Grape & Soya bean)	Rotational Crops
M750F003 (5924326)	4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenol			x		
M750F009					x	
M750F010					x	
M750F011	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl hexopyranoside				x	
M750F012	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl 6-O-(carboxyacetyl)hexopyranoside				x	

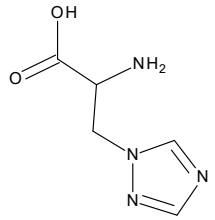
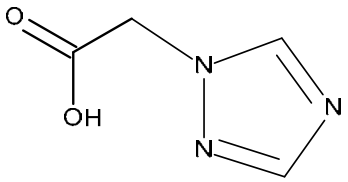
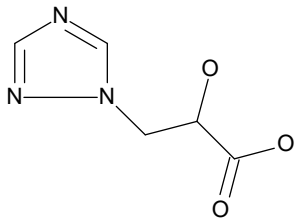
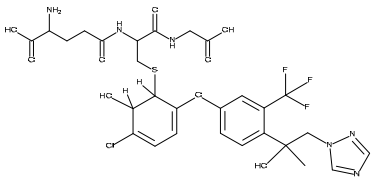
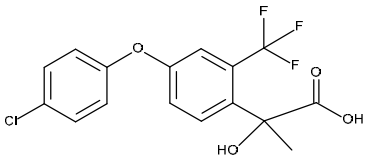
Code Number (Reg. Number)	Chemical Name	Molecular Structure	Occurrence in			
			Rat	Livestock (Hen & Goat)	Crop (Wheat, Grape & Soya bean)	Rotational Crops
M750F013	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl 6-O-hexopyranosylhexopyranoside				x	
M750F014	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl 6-O-[6-O-(carboxyacetyl)hexopyranosyl]hexopyranoside				x	
M750F015 (6011549)	2-chloro-4-{4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenoxy}phenol			x		
M750F016 (6010140)	2-chloro-5-{4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenoxy}phenol			x		
M750F017 (6010139)	5-chloro-2-{4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenoxy}phenol			x		

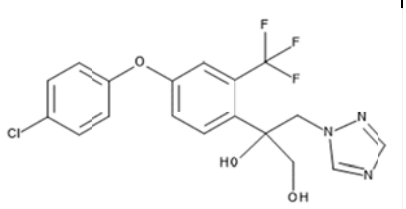
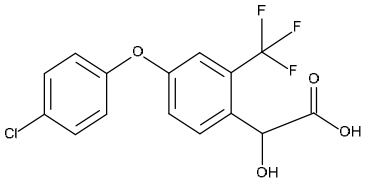
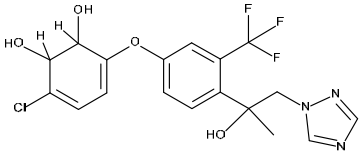
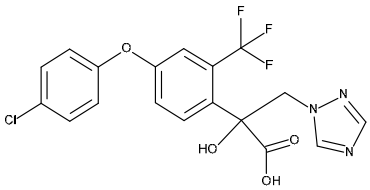
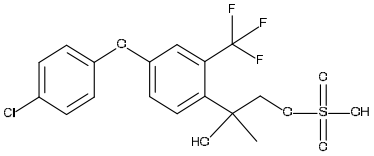
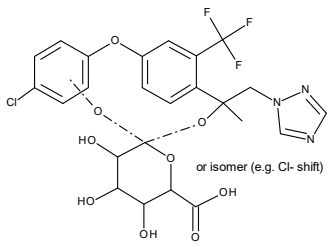
Mefentrifluconazole

Code Number (Reg. Number)	Chemical Name	Molecular Structure	Occurrence in			
			Rat	Livestock (Hen & Goat)	Crop (Wheat, Grape & Soya bean)	Rotational Crops
M750F018					x	
M750F019					x	
M750F020					x	
M750F021					x	
M750F022	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]propane-1,2-diol			x		

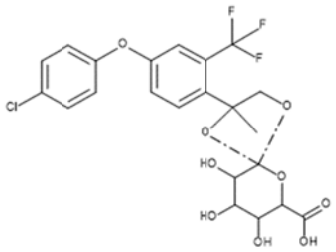
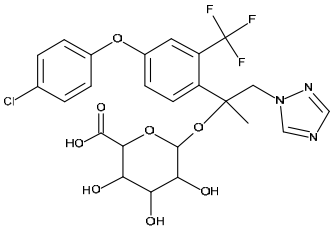
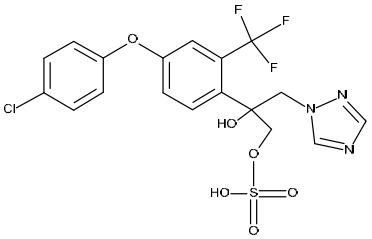
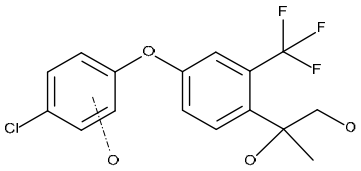
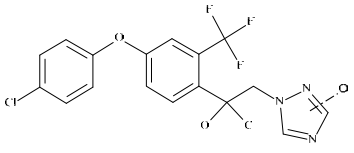
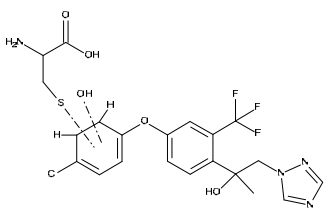
Code Number (Reg. Number)	Chemical Name	Molecular Structure	Occurrence in			
			Rat	Livestock (Hen & Goat)	Crop (Wheat, Grape & Soya bean)	Rotational Crops
M750F023	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxypropyl (9Z,11E)-octadeca-9,11-dienoate			x		
M750F024	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxypropyl (9Z)-octadec-9-enoate			x		
M750F025 (6056452)	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxypropyl hexadecanoate			x		
M750F026					x	
M750F027					x	
M750F028	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1H-1,2,4-triazol-1-yl)propan-2-yl 6-O-pentofuranosylhexopyranoside				x	

Mefentrifluconazole

Code Number (Reg. Number)	Chemical Name	Molecular Structure	Occurrence in			
			Rat	Livestock (Hen & Goat)	Crop (Wheat, Grape & Soya bean)	Rotational Crops
M750F029 (270412) Triazole alanine (TA)	2-amino-3-(1*H*-1,2,4-triazol-1-yl)propionic acid				x	
M750F030 (137281) Triazole acetic acid (TAA)	(1*H*-1,2,4-triazol-1-yl)acetic acid				x	
M750F031 (5050862) Triazole lactic acid, Triazole hydroxypropionic acid (TLA)	2-hydroxy-3-(1*H*-1,2,4-triazol-1-yl)propanoic acid				x	
M750F034	gamma-glutamyl-S-(5-chloro-6-hydroxy-2-{4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenoxy}cyclohexa-2,4-dien-1-yl)cysteinylglycine			x		
M750F038	(2R)-2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxypropanoic acid			x		

Code Number (Reg. Number)	Chemical Name	Molecular Structure	Occurrence in			
			Rat	Livestock (Hen & Goat)	Crop (Wheat, Grape & Soya bean)	Rotational Crops
M750F039	(2S)-2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-3-(1*H*-1,2,4-triazol-1-yl)propane-1,2-diol			x		
M750F040	(2S)-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl](hydroxy)acetic acid		x	x		
M750F041	3-chloro-6-{4-[2-hydroxy-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl]-3-(trifluoromethyl)phenoxy}cyclohexa-3,5-diene-1,2-diol			x		
M750F042	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxy-3-(1*H*-1,2,4-triazol-1-yl)propanoic acid			x		
M750F043	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxypropyl hydrogen sulfate		x	x		
M750F063				x		

Mefentrifluconazole

Code Number (Reg. Number)	Chemical Name	Molecular Structure	Occurrence in			
			Rat	Livestock (Hen & Goat)	Crop (Wheat, Grape & Soya bean)	Rotational Crops
M750F064				x		
M750F068	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-1-(1*H*-1,2,4-triazol-1-yl)propan-2-yl hexopyranosiduronic acid			x		
M750F072	2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]-2-hydroxy-3-(1*H*-1,2,4-triazol-1-yl)propyl hydrogen sulfate			x		
M750F078		 or isomer (e.g. Cl- shift)		x		
M750F086				x		
M750F091				x		

Based on its physical chemical properties, mefentrifluconazole is slightly soluble in water and moderately soluble in non-polar solvents. It is likely to sequester to fatty matrices based on its Log Kow. It has low potential for volatilization. Hydrolysis and aqueous photolysis are unlikely to be important routes of degradation at environmentally relevant pH levels.

Plant metabolism

Mefentrifluconazole metabolism data were provided for grape, soya bean and wheat.

Grape

Three grapevines (variety *Müller-Thurgau*), grown outdoors, received three foliar treatments with either a 1:1 mixture of [^{14}C-U-chlorophenyl:^{13}C-U-chlorophenyl]-mefentrifluconazole or a 2:1 mixture of [^{14}C-3(5)-triazole:^{13}C-3(5)-triazole]-mefentrifluconazole at a rate of 150 g ai/ha with re-treatment intervals of 10–11 days. Grape leaves and clusters were harvested 12 days following the last application.

Total radioactive residues (TRR) in grape berries, leaves and stalks following combustion were 0.40–0.44 mg eq/kg, 7.24–8.86 mg eq/kg and 0.67–1.21 mg eq/kg, respectively.

Extraction of grape berries, leaves, and stalk samples with methanol (3×) and water (2×) released 87–90 percent TRR, 89–91 percent TRR, and 93–94 percent TRR, respectively. Hydrolysis of the post-extraction solids (PES) following various enzymatic treatments released an additional 2–5 percent TRR, which were not further analysed.

Mefentrifluconazole was the major identified residue in all matrices, accounting for 64–70 percent TRR (0.22–0.30 mg/kg) in berries, 60–70 percent TRR (4.43–5.11 mg eq/kg) in leaves, and 86–92 percent TRR (0.56–1.04 mg eq/kg) in stalks. Metabolite M750F019 was identified in berries, leaves, and stalks at 6–7 percent TRR (0.024–0.026 mg eq/kg), 14–21 percent TRR (1.068–1.55 mg eq/kg), and 2 percent TRR (0.015 mg eq/kg), respectively. The chlorophenyl-label-specific metabolite M750F026 was identified as a minor metabolite in leaves only at 1 percent TRR (0.10 mg eq/kg).

Soya bean

Ten containers of soya bean plants (variety *Sultana*, 13 plants/container), cultivated indoors in a vegetation hall and subsequently moved into climatic chambers, received three foliar treatments with either a 1:1 mixture of [^{14}C-U-chlorophenyl:^{13}C-U-chlorophenyl]-mefentrifluconazole or a 2:1 mixture of [^{14}C-3(5)-triazole:^{13}C-3(5)-triazole]-mefentrifluconazole at a rate of 125 g ai/ha with re-treatment intervals of 17–19 days. Soya bean forage was harvested 19 days after the first application (just before the second application; BBCH growth stage of 71–72). At harvest, 47–48 days after the final application at BBCH growth stage 89, the mature pods were collected and manually opened in order to separate seeds from hulls. In addition green pods were also harvested and the remaining stems and leaves (matrix: rest of plant) were collected.

TRRs reported were highest in the rest of plant (16.0–19.9 mg eq/kg), followed by the green pods (8.86–16.0 mg eq/kg), forage (4.4–6.5 mg eq/kg), hulls (3.74–3.89 mg eq/kg) and seed (0.11–2.6 mg eq/kg).

Radioactivity released following extraction with methanol (3×) and water (2×) ranged between 91–93 percent, 69–74 percent, and 87–88 percent in soya bean forage, hull, and rest of plant, respectively. When extracted using acetonitrile:isohexane (1:1) and water, between 56–76 percent TRR were released from soya bean seeds and 78–83 percent from green pods. PES of forage, hulls, rest of plant and seeds were solubilized using various enzymatic treatments releasing 4–38 percent TRR.

Unextracted residues of green pods were not hydrolysed.

Mefentrifluconazole (free and conjugated) was a major residue in forage, hulls and rest of plant, accounting for 60–83 percent TRR (3.18–13.70 mg/kg). The minor metabolites, M750F012 and M70F018/M750F020 (both free and conjugated), were identified at levels ranging from 0.03–6 percent TRR (0.01–0.97 mg eq/kg).

In green pods, mefentrifluconazole represented 69 percent TRR (5.98 mg/kg) while M750F012 and M70F018/M750F020 were each ≤4 percent (≤0.33 mg eq/kg).

Mefentrifluconazole (free) was a minor residue in seed, accounting for only 0.4–4 percent TRR (0.005–0.013 mg/kg). Triazole alanine (free and conjugated) was a major metabolite, accounting for 48 percent TRR (1.46 mg eq/kg). 1,2,4-Triazole and triazole lactic acid (both free) were identified at levels of 0.3 percent TRR (0.008 mg eq/kg) and 1 percent TRR (0.04 mg eq/kg), respectively.

Chiral analysis of forage, hull and rest of plant samples (C-label and T-label) confirmed that the racemic mixture (1:1 ratio of S-enantiomer and R-enantiomer) is essentially maintained, and hence that there is no significant change in the ratio of the mefentrifluconazole enantiomers. Chiral analysis was not conducted for seed/green pod since mefentrifluconazole was not present in quantifiable amounts.

Wheat

Twenty containers of spring wheat (variety *Thassos*, 10 containers per label), cultivated indoors in a vegetation hall/greenhouse, received two foliar treatments with either a 1:1 mixture of [^{14}C-U-chlorophenyl:^{13}C-U-chlorophenyl]-mefentrifluconazole or a 2:1 mixture of [^{14}C-3(5)-triazole:^{13}C-3(5)-triazole]-mefentrifluconazole at a rate of 150 g ai/ha with a re-treatment interval of 21 days. Wheat forage was harvested 15 days after the first application (just before the second application; BBCH growth stage 61). Straw and grain were harvested 35 days after the final application at BBCH growth stage 89.

Total radioactive residues (TRR) were highest in straw (14.3–24.3 mg eq/kg), followed by forage (2.5–2.6 mg eq/kg) and grain (0.06–0.07 mg eq/kg).

Forage and straw samples were extracted with methanol (3×) and water (2×). Wheat grain was extracted with acetonitrile:isohexane (1:1) and water. Solvent extracted radioactivity ranged between 95–96 percent TRR, 44–78 percent TRR, and 83–86 percent TRR in wheat forage, grain, and straw, respectively. The acetone precipitate of grain and the forage and straw post-extraction solids were subjected to solvent and enzymatic treatments releasing an additional 20–40 percent TRR for grain and 2–9 percent TRR for forage and straw.

In forage, mefentrifluconazole (free) accounted for 84–89 percent TRR (2.01–2.06 mg/kg). The minor metabolites, M750F009, M750F012, M750F019 and M750F018/M750F020, all present in the free form, collectively represented 3–4 percent TRR (0.06–0.09 mg eq/kg). In straw, mefentrifluconazole (free and conjugated) accounted for 59–68 percent TRR (9.6–14.3 mg/kg). The total concentration of the same metabolites ranged from 19–21 percent TRR (2.7–5.1 mg eq/kg), accounting for up to 0.4-fold the concentrations of mefentrifluconazole (free and conjugated). Mefentrifluconazole was not found in grain, and triazole alanine and triazole acetic acid were major metabolites accounting for 46 percent TRR (0.28 mg eq/kg) and 22 percent (0.13 mg eq/kg), respectively. 1,2,4-triazole was present at 1 percent TRR (0.006 mg eq/kg).

In summary, the unchanged parent is the predominant residue (>60 percent TRR) in mefentrifluconazole treated plants, notably in forage (wheat, soya bean), leaf/stalks (grapes), straw/hulls (wheat, soya bean), green pods (soya bean) and grapes. The enantiomer ratio of the two mefentrifluconazole isomers remained unchanged (racemic mixture). In wheat grain and soya bean seed,

the unchanged parent is present at very low levels, and the predominant component of the residue is the group of triazole derived metabolites (triazole alanine [TA], triazole acetic acid [TAA], triazole lactic acid [TLA] and 1,2,4-triazole), formed via cleavage of the triazole bridge, with triazole alanine the most abundant compound. The metabolic pathway of mefentrifluconazole in plants is largely based on two main transformation steps: hydroxylation followed by conjugation and cleavage of the triazole bridge followed by conjugation.

Animal metabolism

The Meeting received animal metabolism studies with mefentrifluconazole in lactating goats, laying hens and rats.

Rats

Metabolism of mefentrifluconazole in rats was evaluated by the 2021 JMPR Meeting. Metabolism in the rat was extensive and complex with a total of 68 identified metabolites resulting from phase I and phase II reactions. Main metabolic pathways comprised hydroxylation, methylation and cleavage of the ether group or of the triazole ring from the parent molecule, often followed by conjugations. Most metabolites occurred at low concentrations and only very few of them, observed in the faeces, accounted for more than 30 percent of the dose. In urine, the most abundant metabolite was 1,2,4-triazole with a maximum abundance of 10 percent. In bile, the five main metabolites were all glucuronides which had been formed subsequent to hydroxylation. In faeces, there was a 1:1 ratio of the two isomers of mefentrifluconazole, whereas a shift towards the R-enantiomer was observed in methanolic liver and kidney extracts.

Lactating goats

The metabolism of chlorophenyl-U-^{14}C-labelled, triazole-3-(5)-^{14}C-labelled or trifluoromethylphenyl-ring-U-^{14}C-labelled mefentrifluconazole was investigated in lactating goats. Animals were dosed orally once daily for 12–14 consecutive days. The nominal daily doses were equivalent to 12 ppm in the diet. During the dosing period, urine and faeces were sampled once daily, while milk was collected twice daily. Liver, kidney, muscle and fat samples were collected after animal sacrifice, approximately 23 hours after administration of the last dose.

Most of the radioactivity was recovered in the excreta with urine containing 26–40 percent of the administered dose (AD) and faeces containing 34–50 percent of the AD. The radioactivity recovered in milk and tissues was low, each accounting for ≤ 2.2 percent of the AD. Plateau levels of radioactive residues in milk were reached within 5–6 days after administration of the first dose.

The calculated total radioactive residues (TRRs) in the pooled milk samples (144–288 h) ranged from 0.029–0.273 mg eq/kg for whole milk, 0.016–0.270 mg eq/kg for skimmed milk and 0.207–0.521 mg eq/kg for cream. For tissues, TRRs were highest in liver (0.650–1.332 mg eq/kg), followed by kidney (0.352–0.422 mg eq/kg), composite fat sample (0.213–0.532 mg eq/kg) and composite muscle sample (0.047–0.223 mg eq/kg). In general, levels of radioactivity were lower in milk and muscle.

Extraction of whole milk with acetonitrile released the majority of the radioactivity (86–96 percent TRR). Mefentrifluconazole was the major residue, accounting for 44.5–47.5 percent TRR (0.014–0.028 mg/kg) as were 1,2,4-triazole (78 percent TRR; 0.214 mg eq/kg) and the metabolite M750F043 (14–25 percent TRR; 0.004–0.016 mg eq/kg). Three additional minor metabolites were identified: M750F022 (1–2 percent TRR; 0.001 mg eq/kg), M750F041 (6–7 percent TRR; 0.002–0.004 mg eq/kg) and M750F072 (6–11 percent TRR; 0.002–0.004 mg eq/kg). A similar metabolic profile was observed in skimmed milk and cream.

Isohexane extraction of the composite fat samples released the majority of the radioactivity (> 91 percent TRR) for all three labels. Mefentrifluconazole was the main component of the residues (85–88 percent TRR; 0.18–0.47 mg/kg). Metabolite M750F022 and 1,2,4-triazole were the only other metabolites detected in fat, at up to 6 percent TRR (0.031 mg eq/kg) and 5 percent TRR (0.01 mg eq/kg), respectively.

Extraction of composite muscle samples with methanol released greater than 92 percent TRR. Mefentrifluconazole and the 1,2,4-triazole were the predominant residues, accounting for 12–96 percent TRR (0.03–0.09 mg/kg) and 87 percent TRR (0.19 mg eq/kg), respectively. Metabolite M750F022 was observed at much lower levels (7 percent TRR; 0.003 mg eq/kg).

EThe remaining unextracted radioactivity (up to 9 percent TRR) was subjected to protease hydrolysis which released an additional 2–3 percent TRR (0.01–0.04 mg eq/kg). Mefentrifluconazole represented one of the main components of the residue (26–50 percent TRR; 0.17–0.62 mg/kg), together with the metabolite M750F016 (10–15 percent TRR; 0.065–0.20 mg eq/kg) and 1,2,4-triazole (32 percent TRR; 0.21 mg eq/kg). Metabolite M750F068, resulting from glucuronidation of the parent compound, was also observed in liver but at lower levels (3–4 percent TRR; 0.03–0.06 mg eq/kg). In addition, the minor metabolite M750F022 and its glucuronide derivative M750F038 accounted for 5–11 percent TRR (0.05–0.15 mg eq/kg).

Methanol extraction of kidney released greater than 96 percent TRR. Mefentrifluconazole accounted for 10–46 percent TRR (0.04–0.20 mg/kg). Major predominant metabolites included 1,2-4-triazole (68 percent TRR; 0.27 mg eq/kg), M750F038/M750F064 (co-eluting in one peak, sum: 27 percent TRR; 0.09 mg eq/kg; present in a 1:1 ratio), M750F068 (18 percent TRR; 0.06 mg eq/kg), M750F022 (6–14 percent TRR; 0.02–0.06 mg eq/kg) and M750F038 (11 percent TRR; 0.05 mg eq/kg). The metabolites M750F003, M750F015, M750F016 and M750F072 were also observed but none represented greater than 4 percent TRR (0.02 mg eq/kg).

While the ratio of both mefentrifluconazole isomers was approximately 50:50 in the doses administered to the animals and in the extracts of faeces, the relative amount of the (S)-isomer was lower compared to the (R)-isomer in cream, liver, fat, kidney and muscle, ranging from 20 percent:80 percent to 30 percent:70 percent. These findings demonstrated that matrix-specific differences were observed. This shift towards the R-enantiomer was also observed in the methanol extracts of liver and kidney in the rat metabolism study.

Laying hens

The metabolism of chlorophenyl-U-^{14}C-labelled, triazole-3-(5)- ^{14}C-labelled or trifluoromethylphenyl-ring-U-^{14}C-labelled mefentrifluconazole was investigated in laying hens. The test item was administered once daily by gavage (ten animals per label) for 14 consecutive days at a nominal dose of 12 ppm feed. During the dosing period, excreta were collected once daily, while eggs were collected twice daily after which they were separated into egg whites and egg yolks. Liver, kidney, muscle and fat samples were collected after animal sacrifice, 3–6 hours after administration of the last dose.

The radioactive residues in excreta accounted for 75–89 percent AD. Only ≤ 0.3 percent AD was retained in edible tissues and < 1 percent AD in egg. ^{14}C-residues in egg yolk reached a plateau concentration within 7 days of dosing for all radiolabels. In egg white, ^{14}C-residues reached a plateau within 3–7 days depending on the radiolabel.

Total radioactivity was highest in composite fat samples (0.21–1.1 mg eq/kg), followed by kidney (0.42–0.64 mg eq/kg), liver (0.31–0.58 mg eq/kg) and composite muscle samples (0.053–0.36 mg eq/kg).

Following sequential extractions of egg yolks with methanol and water, greater than 89 percent TRR was released. Mefentrifluconazole (6–44 percent TRR; 0.03–0.12 mg/kg), M750F022 (39–47 percent TRR; 0.19–0.29 mg eq/kg) and 1,2,4-triazole (41 percent TRR; 0.11 mg eq/kg) represented the major residues. The fatty acid conjugates of the metabolite M750F022 (sum of M750F023, M750F024 and/or M750F025) were also present but at lower levels (6–15 percent TRR; 0.03–0.09 mg eq/kg).

The radioactivity in egg whites following administration of the C- and TFMP- radiolabelled-mefentrifluconazole was ≤ 0.009 mg/kg eq, therefore, these were not subjected to further analysis. Egg whites from the T-label study were extracted with methanol and water which, released almost all of the radioactivity (98 percent TRR; 0.350 mg eq/kg). 1,2,4-Triazole was the only metabolite identified, accounting for the majority of the radioactivity (83 percent TRR; 0.297 mg eq/kg).

Methanol and/or methanol/water extraction of muscle samples released greater than 85 percent TRR. M750F022 (C/TFMP-labels: 50–77 percent TRR; 0.02–0.05 mg/kg) and 1,2,4-triazole (T-label: 91 percent TRR; 0.322 mg eq/kg) represented the main components. Mefentrifluconazole and the fatty acid conjugates of M750F022 (sum of M750F023, M750F024 and/or M750F025) were present in lower amounts, accounting for 6–7 percent TRR (0.003–0.005 mg eq/kg) and 10–20 percent TRR (0.007–0.010 mg eq/kg), respectively.

Sequential extraction of liver with methanol/water released 83–100 percent TRR. The predominant metabolites observed included M750F022 (29–37 percent TRR; 0.12–0.17 mg eq/kg), the glutathione conjugate, M750F034 (up to 20 percent TRR; 0.12 mg eq/kg), and the 1,2,4-triazole (85 percent TRR; 0.41 mg eq/kg). Mefentrifluconazole and the fatty acid conjugates of M750F022 (sum of M750F023, M750F024 and/or M750F025) accounted for 6–7 percent TRR (0.03 mg/kg) and 7–12 percent TRR (0.02–0.07 mg eq/kg), respectively.

Extraction of kidney with methanol released 79–99 percent TRR, with limited radioactivity released following subsequent extractions using isohexane or water. M750F022 (20 percent TRR; 0.09–0.12 mg eq/kg) and 1,2,4-triazole (66 percent TRR; 0.37 mg eq/kg) were identified as major metabolites. Conversely, the parent and the fatty acid conjugates of M750F022 (sum of M750F023, M750F024 and/or M750F025) were present in low amounts, each accounting for 4 percent TRR (0.017 mg/kg).

Extraction of fat (C- and TFMP label) using acetonitrile released 83–112 percent TRR, while extraction using methanol (T-label) released 102 percent TRR, with limited radioactivity released following subsequent extractions with isohexane or water. 1,2,4-Triazole (73 percent TRR; 0.14 mg eq/kg), the fatty acid conjugates of M750F022 (sum of M750F023, M750F024 and M750F025) (~42 percent TRR; 0.287–0.380 mg eq/kg) and the metabolite M750F022 (25–41 percent TRR; 0.18–0.37 mg eq/kg) accounted for the majority of the radioactivity. Mefentrifluconazole was present at lower levels (5–20 percent TRR; 0.04–0.10 mg/kg).

In summary, the metabolism of mefentrifluconazole in livestock and rats was qualitatively similar, based on two main transformation steps: hydroxylation followed by conjugation and cleavage followed by conjugation. Most of the radioactivity was eliminated via excreta. In goat and poultry matrices, mefentrifluconazole and 1,2,4-triazole were the predominant components of the residue. Metabolite M750F022 (and its fatty acid conjugates) was also a major component in poultry matrices.

Environmental fate in soil

The Meeting received information on soil aerobic metabolism, hydrolysis and photolysis properties of mefentrifluconazole. Studies were also received on the nature of [^{14}C]-mefentrifluconazole in confined rotational crops and the magnitude of mefentrifluconazole in field rotational crops.

Aerobic soil metabolism (laboratory studies)

Mefentrifluconazole is persistent in soil with DT_{50} ranging from 156 to >1000 days. However, the Meeting noted that the predicted DT_{50} values were extrapolated well beyond the study duration (120 days) and should be treated with caution.

The metabolites 1,2,4-triazole and M750F003 were detected, reaching maximum amounts of 5.2 percent of the total applied radioactivity (TAR) and 2 percent TAR, respectively. A number of additional metabolites were detected, however, none exceeded 1 percent TAR at any sampling time.

Hydrolysis

Mefentrifluconazole is stable in aqueous solutions at environmentally relevant pHs of 5, 7 and 9.

Soil photolysis

Limited degradation of the parent compound was observed in both irradiated and dark control soil samples, hence photolysis was not an important route of dissipation.

Field Dissipation

The dissipation of mefentrifluconazole under field conditions has been studied in the United States (bare ground and turf-cropped soil), Europe (bare soil) and (bare soil). Quantifiable residues of mefentrifluconazole were detected only in the first 20 cm of the soils. No residues above the LOQ were detected below 20 cm in any sample at any site. For bare soil, the overall geometric mean (non-normalised) DT_{50} was estimated to be 149 days, indicating mefentrifluconazole is non-persistent to moderately persistent. Therefore, the Meeting decided that mefentrifluconazole shows limited potential to accumulate in soil following application in consecutive years.

Confined rotational crops

[Chlorophenyl-U-^{14}C] and [triazole-3(5)-^{14}C]-labelled mefentrifluconazole, formulated as EC formulations, were applied to bare sandy loam soil, in plastic containers maintained in either a glass roofed vegetation hall, phytotron or in a glass house, at an application rate of 300 g ai/ha. Spinach (variety *Corvette)*, white radish (variety *April Cross)* and spring wheat (*Thasos*) were sown 30/31, 120/122 and 364/365 days after the soil treatment. All crops were harvested at maturity and additional immature spinach samples as well as spring wheat forage samples (in part dried to hay) were collected 25–33 days and 49–55 days after planting (DAP), respectively.

Significant uptake and translocation of TRRs from soil into the secondary crops was observed over all plant-back intervals (PBI) and matrices (particularly spring wheat grain), which is due to the uptake and translocation of high amounts of triazole derivative metabolites (1,2,4-triazole, triazole alanine [TA], triazole acetic acid [TAA] and triazole lactic acid [TLA]). The highest levels of radioactive residues were found in spring wheat straw (30-day PBI) and in spring wheat grain, hay and straw after the 120/122–day PBI. The TRRs in spinach and white radish matrices were generally lower compared to those in wheat matrices. Overall, residues remained similar or decreased at longer PBIs, except for wheat grain where residues peaked at the 120-day PBI followed by a slight decrease by the 365-day PBI, yet still higher than the TRRs at the 30-day PBI.

In all tested matrices, except wheat grain, methanol released 26–96 percent TRR and water released an additional 1–34 percent TRR, while that released from wheat grain ranged from 7–49 percent and 8–53 percent TRR, respectively. PES underwent extensive hydrolysis using various solvents and enzymes, and analysis of the hydrolysates demonstrated that the radioactivity was associated with plant

constituents.

Mefentrifluconazole was the main component detected in all tested samples, except grain. At 30/31-day PBI, residues accounted for 14–91 percent TRR (0.008–0.013 mg/kg) in immature and mature spinach, and 4–70 percent TRR (0.006–0.101 mg/kg) in the other matrices. At 120/122-day PBI, residues of the parent were 36–61 percent TRR (0.006–0.055 mg/kg) in immature and mature spinach, wheat forage, hay and straw, and at 364/365-day PBI, accounted for 1–41 percent TRR (0.0008–0.018 mg/kg) in wheat samples. No parent or metabolites were detected in radish roots or tops beyond the 30-day PBI.

In spinach and wheat, metabolites accounting for 2–80 percent TRR (0.0003–0.018 mg eq/kg), 5–83 percent TRR (0.0004–0.032 mg eq/kg) and 22–65 percent TRR (0.004–0.022 mg eq/kg) at 30-, 120- and 365-day PBIs, respectively, were characterised based on their chromatographic properties.

At longer PBIs, the parent was only detected in wheat hay (122-day PBI: 1.3 percent TRR; [0.030 mg/kg]) and straw (122-day PBI; 1 percent TRR; [0.014 mg/kg] and 365-day PBI; 1 percent TRR [0.008 mg/kg]).

In most cases, the main metabolite in the crop matrices (T-label) was triazole alanine (13–94 percent TRR; 0.022–0.982 mg eq/kg), followed by triazole lactic acid 9–39 percent TRR (0.005–0.807 mg eq/kg). The only exceptions were spring wheat hay (122 DAT) and spring wheat straw (all PBIs), where, triazole lactic acid was the most abundant component among all the analytes identified. In spring wheat grain, triazole alanine (42–73 percent TRR; 0.98–2.36 mg eq/kg) was the main component followed by triazole acetic acid (20–24 percent TRR; 0.46–0.69 mg eq/kg). The sum of the triazole derivative metabolites and 1,2,4-triazole in all secondary crops ranged from 65–101 percent TRR.

In summary, when rotational crops were cultivated on mefentrifluconazole-treated soil, the residues included mainly two components, the parent and triazole derivative metabolites (1,2,4-triazole, TA, TAA, TLA), the latter being generated by cleavage of the parent molecule at the triazole bridge.

Field rotational crops

Field rotational crop studies were conducted in the United States and Europe during the 2014 and 2015 growing seasons. In the United States, treated plots received three broadcast soil directed spray applications, with a 7-day retreatment interval, for a total rate of 595 to 614 kg ai/ha/season. In Europe, a single application was made to bare soil at up to 327 kg ai/ha. At intervals of 1, 3 (Europe only), 4 and 11/12 months following the last application to the bare soil, wheat, lettuce or broccoli, and carrot or radish were planted.

In the United States, mefentrifluconazole residues in wheat forage/hay/straw reached 2.38/0.53/0.06, 0.69/0.87/< 0.01, 1.13/2.65/0.02, and 1.57/1.97/0.01 mg/kg at 1, 3, 4 and 11 month PBIs, respectively. In radish tops/root, the residues reached 0.07/0.03, 0.04/0.02, 0.02/0.02 and < 0.01/< 0.01 mg/kg at 1, 3, 4 and 12 month PBIs, respectively. Residues in wheat grain and lettuce were < 0.01 mg/kg at all PBIs.

In Europe, residues of mefentrifluconazole in all tested crops, including a cereal grain (wheat), root crop (carrot and radish), leafy (lettuce and spinach) and Brassica (broccoli and cauliflower) vegetables were consistently below the LOQ of 0.01 mg/kg at all PBIs of 1, 4 and 12 months.

In summary, the environmental fate data demonstrated that mefentrifluconazole is relatively persistent in soil and is stable in aqueous solutions at environmentally relevant pHs. Photolysis of mefentrifluconazole on the soil surface is not anticipated to be an important dissipation process. The metabolism in rotational crops showed to be similar to that in primary crops with no rotational crop specific metabolites. Uptake of mefentrifluconazole in food commodities and wheat straw was very

limited, however, measurable uptake into wheat forage and hay was observed.

No quantifiable residues of 1,2,4-triazole were observed in any of the tested crops. In general, residues of triazole alanine, triazole acetic acid and triazole lactic acid were higher in feed commodities than food commodities. In food commodities, triazole alanine was the highest among all triazole derivative metabolites, followed by triazole lactic acid and triazole acetic acid, all of which declined with increased PBI.

Methods of Analysis

The Meeting received descriptions and validation data for three analytical methods capable of quantifying residues of mefentrifluconazole in diverse plant matrices. The samples were extracted with methanol/water/2 mol/L HCl (70/25/5), acetonitrile or methanol/water (80/20), the samples with hexane, cleaned-up with salts and dispersive SPE (QuEChERs), or injected directly in the LC-MS/MS. Average recoveries were in the range of 70–120 percent (with a few isolated exceptions) with RSD of ≤ 20 percent. The methods were satisfactorily validated at LOQ of 0.01 mg/kg for all tested plant matrices, including cereals, citrus, coffee, soya beans, grapes and apple.

The Meeting also received descriptions and validation data for two method for analysis of mefentrifluconazole residues in animal matrices. In one method, samples were extracted with acetonitrile and iso-hexane (milk, cream, fat) or methanol/water/2N HCl (70/25/5) (muscle, kidney, liver and egg), and the other method uses QuEChERs. Residues were quantified by LC-MS/MS, and the methods were satisfactorily validated at a LOQ of 0.01 mg/kg.

Two methods to analyse the metabolite M750F022 and the fatty acid conjugate M750F025 (measured as M750F022) in hen matrices were provided. Samples were extracted with acetonitrile:isohexane, methanol/water/2 mol/L HCl (70/25/5) or methanol/water, the extracts cleaned up on SPE column, and residues quantified using GC-MS. The fatty acid conjugates of M750F022 were hydrolysed using NaOH (10 M). Recoveries of mefentrifluconazole and the metabolites M750F022 and M750F025 were acceptable, and the LOQ achieved for all animal commodities were 0.01 mg/kg for each analyte.

The methods for analysing mefentrifluconazole in plant matrices and mefentrifluconazole and the metabolites M750F022 and M750F025 in animal matrices were successfully validated by independent laboratories, demonstrating good reproducibility. Some of the plant-specific methods were also subjected to radiovalidation, where bioincurred residues of mefentrifluconazole were adequately recovered from samples of wheat forage, soya bean green pods and grapes collected from the metabolism studies, demonstrating the efficiency of the data collection analytical methods to extract incurred residues of mefentrifluconazole. The only exception was wheat straw, where the extraction efficiency was 59 percent likely due to the difference in extraction solvents between the metabolism study (methanol (3×) and water (2×)) and the analytical method (acetonitrile). For residues of mefentrifluconazole in animal matrices, extraction efficiencies were 80 percent or higher for milk, cream, muscle, kidney, fat, egg yolk and lower for liver (46 percent). For M750F022, extraction efficiencies were 90 percent or higher for milk, cream, kidney, fat and lower for egg yolk (66 percent), muscle (61 percent) and liver (46–50 percent). This was also likely due to the minor differences in extraction solvents between the metabolism study and the analytical method.

Stability of pesticide residues in stored analytical samples

Residues of mefentrifluconazole in samples fortified at 0.1 mg/kg were determined to be stable at ≤-18 ºC for at least 24 months in high water content commodities (tomato, apple, wheat whole plant), high oil

content commodities (rapeseed), high protein content commodities (dry bean seed, dry pea seed, dry soya bean seed), high starch content commodities (potato tuber), high acid content commodities (grape, lemon), and wheat straw.

Residues of mefentrifluconazole and M750F022 (at 0.1 mg/kg) were stable at ≤-18 ºC for at least 6 months in milk, cream, eggs and bovine and poultry tissues.

These demonstrated storage stability intervals covered the storage durations of the crop field trials, processing studies and animal feeding studies.

Definition of the residue

Plant commodities

The nature of the mefentrifluconazole residues was investigated in grapes (leaves, stalks and berries), soya beans (green pods, hulls, seed, and rest of plant) and wheat (forage, grain, straw) following foliar treatment.

Mefentrifluconazole was the major analyte in all tested plant matrices (60–92 percent TRR; 0.22–14.3 mg/kg), except wheat grain and soya bean seed where the predominant metabolite was triazole alanine (free and conjugated: 46–48 percent; 0.28–1.5 mg eq/kg), a common metabolite of several triazole fungicides. As suitable analytical methods are available to analyse the parent compound, the Meeting considered that mefentrifluconazole was a suitable marker for monitoring compliance.

In deciding which compounds should be included in the residue definition for risk assessment, the Meeting considered the likely occurrence and toxicological properties of M750F019, found in grapes, as well as the triazole derivative metabolites found in soya bean seed and wheat grain.

M750F019 was not observed in the rat, however, its toxicity was considered to be covered by the health based guidance value (HBGV) of the parent compound, as noted in the 2021 JMPR Report. Furthermore, in the grape metabolism study, the ratio of M750F019 to the parent compound in grapes was 0.1, demonstrating a low contribution to the dietary exposure, compared to the parent compound.

Triazole derivative metabolites (1,2,4-triazole, triazole alanine, triazole lactic acid and triazole acetic acid) were found in significant amounts in wheat grain and soya bean seed from primary plant metabolism studies. Moreover, these metabolites were also frequently detected in control and treated crops from supervised field trials. The Meeting noted that these metabolites can arise from other sources and have toxicities known to be different from mefentrifluconazole. The Meeting concluded that these metabolites should be assessed separately, and were not further considered in the current evaluation.

The Meeting decided the residue definition for dietary risk assessment for plant commodities should be mefentrifluconazole.

Animal commodities

The nature of the mefentrifluconazole residues was investigated in lactating goats and laying hens. The metabolism of mefentrifluconazole was qualitatively similar in both animals, yet more extensive in goats.

Mefentrifluconazole (free and/or conjugated) was a major component of the residue in the goat (milk 3–48 percent TRR; kidney 10–46 percent TRR; muscle 12–96 percent TRR; liver 31–53 percent TRR; fat 85–88 percent TRR), but a minor component in the hen (egg yolk 6–44 percent TRR; muscle 6–7 percent TRR; fat 5–20 percent TRR; liver 10–26 percent TRR and kidney 4 percent TRR). Nevertheless, mefentrifluconazole (free and/or conjugated) was present in all tested livestock matrices which would make it a suitable marker for monitoring compliance.

Validated analytical methods are available for the determination of mefentrifluconazole in tissues, milk and eggs. The Meeting agreed the residue for compliance monitoring for tissues, milk and eggs should be mefentrifluconazole (free and conjugated).

In deciding which compounds should be included in the residue definition for risk assessment, the Meeting considered the likely occurrence and toxicological properties of the metabolites potentially contributing to the dietary risk.

Specific toxicological studies reviewed at the 2021 JMPR Meeting were only available for the metabolite M750F022, which was considered to have equal or lower toxicity than the parent compound. The Meeting concluded that the HBGVs apply to mefentrifluconazole and its metabolites M750F015, M750F016, M750F017, M750F019, M750F022, M750F038, M750F043, and M750F068, expressed as mefentrifluconazole. The current Meeting also concluded that two additional livestock metabolites (M750F064 and M750F034) are not of concern from the genotoxicity perspective and the HBGVs for parent mefentrifluconazole are also applicable.

1,2,4-Triazole was found in measurable amounts in lactating goat and laying hen metabolism studies (32–87 percent TRR; ≤ 0.4 mg eq/kg). 1,2,4-Triazole and triazole alanine were also frequently detected in control and treated samples of milk, eggs and tissues in the animal feeding studies. The Meeting concluded that these metabolites can arise from other sources and have toxicities known to be different from mefentrifluconazole. These metabolites should be assessed separately, considering their source and respective toxicities, and were not further considered in the current evaluation.

Only the major metabolites, M750F016, M750F043, M750F064 and M750F068, identified in milk, liver and kidney of the lactating goat metabolism study are being considered for the residue definition for risk assessment as the nature of the residues in fat were not further elucidated considering the low TRRs.

In goat muscle and fat tissues, the majority of the TRR was identified as parent mefentrifluconazole (85–96 percent TRR; 0.03–0.47 mg/kg). In liver, kidney and milk, the sum of mefentrifluconazole and M750F022 (including their conjugates M750F068, M750F064 and M750F043) represent 60 percent, 64 percent and 70 percent of the TRR, respectively, which cover more than 80 percent of the total compounds for which the HBGVs apply. The less prominent metabolite M750F016 found in liver does not contribute significantly to the overall dietary exposure nor to the sum of mefentrifluconazole and M750F022 (free and conjugated) already taken into account for dietary risk assessment.

Only the major metabolites identified in the poultry metabolism study, M750F022 and its fatty acid conjugates (M750F023, M750F024 and M750F025) and M750F034 (sulfate conjugate of mefentrifluconazole) were considered.

The 2021 Meeting noted that the additional fatty acid side chains of the conjugates M750F023, M750F024 and M750F025 may cause differences in kinetics compared to the metabolite M750F022, and therefore, the Meeting concluded that the TTC approach for non-genotoxic compounds of Cramer Class III (1.5 µg/kg bw/day) should be considered for these metabolites.

For compounds covered by the parent HBGVs, metabolite M750F022 was the predominant component in all poultry tissues and eggs accounting for up to 10-fold the concentrations of the parent compound. In the laying hen feeding study, other than the parent compound, only M750F022 was investigated in eggs and tissues, where residues of this metabolite were equivalent to or higher than those of the parent compound at all feeding levels. Therefore, this metabolite will contribute to the overall dietary exposure to mefentrifluconazole and should be considered in combination with M750F034 (sulfate conjugate of mefentrifluconazole) in the residue definition for risk assessment.

The Meeting agreed that the suitable residue definition for risk assessment is the sum of mefentrifluconazole (free and conjugated) + M750F022 (free and conjugated), expressed as mefentrifluconazole.

Conclusions

Based on the above, the Meeting recommended the following residue definitions for mefentrifluconazole.

Definition of the residue for compliance with the MRL and for dietary exposure assessment for plant commodities: *Mefentrifluconazole*.

Definition of the residue for compliance with the MRL for animal commodities: *Mefentrifluconazole (free and conjugated).*

Definition of the residue for dietary exposure assessment for animal commodities: Sum of mefentrifluconazole (free and conjugated) + M750F022 (free and conjugated), expressed as mefentrifluconazole.

In deciding whether the residue for monitoring compliance is fat-soluble, the Meeting noted that the mean residues of mefentrifluconazole at the highest dose tested in the lactating cow feeding study were 0.16 mg/kg in muscle and 1.7 mg/kg in perirenal fat while residues were 5× higher in cream compared to whole milk. In the laying hen feeding study, total mefentrifluconazole residues at the highest dose tested were 0.05 mg/kg in muscle and 0.53 mg/kg in fat.

The Meeting considered the residue is fat-soluble.

Results of supervised residue trials in crops

Citrus fruits

The critical GAP for the citrus fruits crop group is from the United States: 3×146 kg ai/ha, 14-day RTI (days), 0-day PHI. The Meeting received supervised residue trials conducted on whole orange, grapefruit and lemon in the United States, as well as two trials on orange and one trial on lemon conducted in Mexico, all matching the critical GAP.

Mefentrifluconazole residues in whole oranges in ranked order were (n = 14); 0.15, 0.17, 0.18, 0.19 (2), 0.20 (2), 0.23, 0.24, 0.29, 0.33, 0.46, 0.66 and 0.70 mg/kg.

Noting that oranges are the representative crop of the subgroup of oranges, the Meeting estimated a maximum residue level of 1 mg/kg, an STMR of 0.215 mg/kg, and an HR of 0.70 mg/kg for the Subgroup of oranges, sweet, sour.

Mefentrifluconazole residues in whole grapefruits in ranked order were (n = 6); 0.10, 0.13, 0.16, 0.19, 0.20 and 0.24 mg/kg.

Noting that grapefruits are representative of the subgroup of pummelo and grapefruits, the Meeting estimated a maximum residue level of 0.5 mg/kg, an STMR of 0.16 mg/kg and an HR of 0.24 mg/kg for the Subgroup of pummelo and grapefruits.

Mefentrifluconazole residues in whole lemons in ranked order were (n = 7): 0.30, 0.33 (2), 0.37, 0.44, 0.60 and 0.98 mg/kg.

Noting that lemons are the representative crop of the subgroup of lemons and limes, the Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR of 0.37 mg/kg, and an HR of 0.98 mg/kg for the Subgroup of lemons and limes.

The GAP covers the group of citrus fruits, including use on mandarins. Although trials were not provided for mandarins, the Meeting noted that residues in lemons/limes have been shown to be similar to or greater than residues in mandarins. Therefore, the Meeting decided to extrapolate the residues from lemon and estimated a maximum residue level of 1.5 mg/kg, an STMR of 0.37 mg/kg, and an HR of 0.98 mg/kg, for the Subgroup of mandarins.

Pome fruits

The critical GAP for pome fruits is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 0-day PHI. The Meeting received supervised residue trials conducted on apple and pear in Canada and the United States matching the critical GAP.

Mefentrifluconazole residues in apples in ranked order were (n = 13): < 0.01, 0.23, 0.26, 0.30 (2), 0.37, 0.39, 0.43 (2), 0.45, 0.46, 0.47 and 0.55 mg/kg.

Mefentrifluconazole residues in pears in ranked order were (n = 10): < 0.01, 0.30, 0.32, 0.34 (2), 0.40, 0.52, 0.68, 0.73 and 0.92 (highest 1.12) mg/kg.

The Meeting noted that the GAP covers the group of pome fruits except persimmon, Japanese and that median residues in apples and pears are within a 5-fold difference. The Mann-Whitney U-test also determined that the datasets were from the same population. Therefore, the Meeting decided to combine the two datasets of apples and pears.

Combined mefentrifluconazole residues in apples and pears were (n = 23): < 0.01 (2), 0.23, 0.26, 0.30 (3), 0.32, 0.34 (2), 0.37, 0.39, 0.40, 0.43 (2), 0.45, 0.46, 0.47, 0.52, 0.55, 0.68, 0.73 and 0.92 (highest 1.12) mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR of 0.39 mg/kg, and an HR of 1.12 mg/kg (based on the highest residue of replicate samples) for the Group of pome fruits except persimmon, Japanese.

Stone Fruits

The critical GAP for stone fruits is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 0-day PHI. The Meeting received supervised residue trials conducted on whole peaches, cherries and plums in Canada and the United States matching the critical GAP.

Mefentrifluconazole residues in whole peaches in ranked order were (n = 12): 0.36, 0.41, 0.42, 0.47, 0.48, 0.52, 0.60, 0.70, 0.72, 0.81 and 0.96 (2) (highest 1.04) mg/kg.

Noting that peaches are the representative crop of the subgroup of peaches, the Meeting estimated a maximum residue level of 2 mg/kg, an STMR value of 0.56 mg/kg, an HR of 1.04 mg/kg (based on the highest residue of replicate samples) for the Subgroup of peaches.

Mefentrifluconazole residues in sweet and tart cherries in ranked order were (n = 7): 0.04, 0.94, 1.0, 1.1, 1.4, 2.0 and 2.2 (highest 2.4) mg/kg.

Noting that cherries are the representative crop of the subgroup of cherries, the Meeting estimated a maximum residue level of 5 mg/kg, an STMR of 1.1 mg/kg and an HR of 2.4 mg/kg (based on the highest residue of replicate samples) for the Subgroup of cherries.

Mefentrifluconazole residues in plums in ranked order were (n = 9): < 0.01, 0.03, 0.13, 0.21, 0.26, 0.30, 0.32, 0.37 and 0.98 (highest 1.0) mg/kg.

Noting that plums are the representative crop of the subgroup of plums, the Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR value of 0.26 mg/kg and an HR of 1.0 mg/kg (based on the

highest residue of replicate samples) and for the Subgroup of plums.

Cane berries

The critical GAP for cane berries is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 0-day PHI. The Meeting received supervised residue trials conducted on blackberries in the United States matching the critical GAP.

Mefentrifluconazole residues in blackberries in ranked order were (n=6): 0.25, 0.35, 0.71, 1.2 and 1.3 (2) (highest of 1.62) mg/kg.

Noting that blackberries are the representative crop of the subgroup of cane berries, the Meeting estimated a maximum residue level of 3 mg/kg, an STMR value of 0.96 mg/kg and an HR of 1.62 mg/kg (based on the highest residue of replicate samples) for the Subgroup of cane berries.

Bush berries

The critical GAP for bush berries is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 0-day PHI. The Meeting received supervised residue trials conducted on blueberries in the United States matching the critical GAP.

Mefentrifluconazole residues in blueberries in ranked order were (n=9): 0.06, 0.18, 0.51, 0.56, 0.58, 0.68, 0.74, 0.77 and 3.16 (highest 3.24) mg/kg.

Noting that blueberries are the representative crop of the subgroup bush berries, the Meeting estimated a maximum residue level of 5 mg/kg, an STMR value of 0.58 mg/kg and an HR of 3.24 mg/kg (based on the highest residue of replicate samples) for the Subgroup of bush berries and extrapolated these values to elderberries and Guelder rose.

Grapes

The critical GAP for wine grapes is from the United States; 3×146 g ai/ha, 10-day RTI (days) and a 14-day PHI. The Meeting received supervised residue trials conducted on wine grapes in Canada and the United States matching the critical GAP.

Mefentrifluconazole residues in grapes in ranked order were (n = 8): 0.25, 0.33, 0.38, 0.41, 0.67, 0.83, 1.0 and 1.1 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg, an STMR value of 0.54 mg/kg and an HR of 1.1 mg/kg for wine grapes.

The critical GAP for table grapes is from the United States; 2×112 g ai/ha, 10-day RTI (days) and a 14-day PHI. The Meeting received trials from the United States on table grapes where 3 foliar applications were made at 147–155 g ai/ha, 8–11-day RTI (days)s, seasonal application rates of 449–460 g ai/ha and 14–21-day PHIs.

As supervised field trials were conducted at rates 2-fold greater than the critical GAP and the RTI (days)s were longer, without suitable data to estimate half-lives, the Meeting concluded that the overall impact of these parameters on the residue was > 25 percent, and a maximum residue level could not be estimated for table grapes.

Low growing berries

The critical GAP for low growing berries is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 0-day PHI. The Meeting received supervised residue trials conducted on strawberries in the United States

matching the critical GAP.

Mefentrifluconazole residues in strawberries in ranked order were (n = 11): < 0.01 (2), 0.08, 0.15, 0.24, 0.29, 0.43, 0.44, 0.50, 0.62 and 1.1 mg/kg.

Noting that strawberries are the representative crop of the subgroup of low growing berries, the Meeting estimated a maximum residue level of 2 mg/kg, an STMR value of 0.29 mg/kg and an HR of 1.1 mg/kg for the Subgroup of low growing berries.

Assorted tropical and sub-tropical fruits – inedible peel

Avocado

The critical GAP for avocado is from El Salvador; 3×120 g ai/ha, 14-day RTI (days) and a 3-day PHI. The Meeting received supervised residue trials conducted on avocado in Brazil, Colombia and Mexico matching the critical GAP.

Mefentrifluconazole residues in pitted whole avocadoes, expressed as whole fruit, in ranked order were (n = 6): 0.10, 0.22, 0.32, 0.39, 0.42 and 0.50 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg, an STMR value of 0.36 mg/kg and an HR of 0.50 mg/kg for avocadoes.

Banana

The critical GAP for bananas is from Ecuador; 4×140 g ai/ha, 14-day RTI (days) and a 0-day PHI. The Meeting received supervised residue trials, conducted on bagged and unbagged bananas in Brazil, Colombia and Ecuador, where 5 foliar applications were made at a target rate of 140 g ai/ha, RTI (days)s of 14 days, seasonal application rates of 700 g ai/ha and a DALA of 0 days.

The Meeting agreed that the first application in the trials made 70 days prior to harvest would not contribute significantly to residues at the time of harvest and considered suitable for estimating a maximum residue level.

Mefentrifluconazole residues in unbagged whole bananas in ranked order were (n=10): 0.04, 0.12, 0.16, 0.24, 0.35, 0.47, 0.54, 0.57, 0.65 and 0.74 mg/kg.

Mefentrifluconazole residues in the pulp of unbagged bananas in ranked order were (n=10): < 0.01, 0.01, 0.04, 0.05 (2), 0.06, 0.09, 0.14, and 0.21 (2) mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR of 0.055 mg/kg and an HR of 0.21 mg/kg.

Mango

The critical GAP for mango is from China; 3×0.016 kg ai/hL, 10-day RTI (days) and a 14-day PHI. The Meeting received supervised residue trials conducted on mango in China approximating the critical GAP.

Mefentrifluconazole residues in whole mangoes in ranked order were (n = 6): 0.12, 0.16 (2), 0.20, 0.22 and 0.28 mg/kg. Residues in pulp were all < 0.01 m/kg (n=6).

The Meeting estimated a maximum residue level of 0.6 mg/kg and an STMR and HR of 0.01 mg/kg.

Papaya

The critical GAP for papaya is from El Salvador; 2×120 g ai/ha, 14-day RTI (days) and a 3-day PHI. The

Meeting received supervised residue trials conducted on papaya from Brazil, Colombia and Mexico, matching the critical GAP.

Mefentrifluconazole residues in whole papayas in ranked order were (n = 6): < 0.01, 0.04, 0.07 (2), 0.19 and 0.22 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg, an STMR value of 0.07 mg/kg and an HR of 0.22 mg/kg for papayas.

Bulb vegetables

The critical GAP for bulb vegetables is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 7-day PHI. The Meeting received supervised residue trials conducted on bulb onions and green onions in Canada and the United States matching the critical GAP.

Mefentrifluconazole residues in bulb onions in ranked order were (n=13): < 0.01, 0.01(2), 0.03 (2), 0.05 (2), 0.08, 0.09 (3), 0.10 and 0.11 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg, an STMR of 0.05 mg/kg and an HR of 0.14 mg/kg (based on the highest residue of replicate samples) for the Subgroup of bulb onions.

Mefentrifluconazole residues in green onions in ranked order were (n=5): 0.11, 0.28, 0.39, 0.42 and 2.1 mg/kg.

The Meeting estimated a maximum residue level of 4 mg/kg, an STMR of 0.39 mg/kg and an HR of 2.2 mg/kg (based on the highest residue of replicate samples) for the Subgroup of green onions.

Fruiting vegetables – Cucurbits

The critical GAP for fruiting vegetables-cucurbits is from the United States for "cucurbit vegetables"; 3×146 g ai/ha, 7-day RTI (days) and a 0-day PHI. The Meeting received supervised residue trials on cucumbers, summer squash and melons from Canada and the United States matching the critical GAP.

Mefentrifluconazole residues in cucumbers in ranked order were (n = 9): 0.01, 0.02, 0.03 (4), 0.04 (2) and 0.10 (highest 0.123) mg/kg.

Mefentrifluconazole residues in summer squashes in ranked order were (n = 8): 0.01(2), 0.04, 0.05 (3) and 0.09 (2) mg/kg.

The Meeting noted that the median residues of cucumbers and summer squashes were within 5-fold, and that the Mann-Whitney U-test determined the datasets were from the same population. Therefore, the Meeting decided to combine the two datasets of cucumbers and summer squashes.

The ranked order of the combined mefentrifluconazole residues in cucumbers and summer squashes were (n = 17): 0.01 (3), 0.02, 0.03 (4), 0.04 (3), 0.05 (3), 0.09 (2) and 0.10 (highest 0.123) mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg, an STMR of 0.035 mg/kg and an HR of 0.123 mg/kg (based on the highest residue of replicate samples) for the Subgroup of fruiting vegetables, cucurbits - cucumbers and summer squashes.

Mefentrifluconazole residues in whole muskmelons in ranked order were (n = 8): 0.11 (3), 0.14, 0.16, 0.17, 0.21 and 0.22 mg/kg.

Noting that melons is the representative crop of the melons, pumpkins and winter squashes crop subgroup, the Meeting estimated a maximum residue level of 0.5 mg/kg, an STMR of 0.15 mg/kg and an HR of 0.23 mg/kg (based on the highest residue of replicate samples) for the Subgroup of fruiting vegetables, cucurbits – melons, pumpkins and winter squashes.

Fruiting vegetables – Other than cucurbits

The critical GAP for fruiting vegetables-other than cucurbits is from the United States for "fruiting vegetables"; 3×146 g ai/ha, 7-day RTI (days) and a 0-day PHI. The Meeting received trials on cherry tomatoes, tomatoes, bell peppers and non-bell peppers from the United States matching the critical GAP.

Mefentrifluconazole residues in field tomatoes (including cherry tomatoes [CT]) in ranked order were (n= 19): 0.03, 0.04, 0.05 (2), 0.08, 0.09, 0.10, 0.11, 0.13[CT], 0.14, 0.15 (2), 0.17, 0.19, 0.23, 0.25, 0.36[CT], 0.37 and 0.41[CT] (highest 0.45) mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg, an STMR of 0.14 mg/kg and an HR of 0.45 mg/kg (based on the highest residue of replicate samples) for the Subgroup of tomatoes.

Mefentrifluconazole residues in field bell peppers and non-bell peppers [NB] in ranked order were (n= 14): 0.04, 0.05, 0.06 (2), 0.20, 0.22, 0.24[NB], 0.26 [NB], 0.30, 0.33[NB], 0.39[NB], 0.43, 0.60[NB] and 0.73 (highest 0.84) mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR of 0.25 mg/kg and an HR of 0.84 mg/kg (based on the highest residue of replicate samples) for the Subgroup of peppers (excluding martynia, okra and roselle).

The critical GAP from the United States for fruiting vegetables, other than cucurbits, also covers eggplants. The Meeting decided the pepper data could be used to extrapolate the maximum residue level of 1.5 mg/kg, the STMR of 0.25 mg/kg and the HR of 0.84 mg/kg for peppers to the Subgroup of eggplants.

Chili peppers, dried

Based on the estimated maximum residue level of 1.5 mg/kg for the Subgroup of peppers (excluding martynia, okra and roselle) and applying a default processing factor of 10, the Meeting estimated a maximum residue level of 15 mg/kg for peppers, chili, dried, together with an STMR of 2.5 mg/kg (0.25 × 10) and an HR of 8.4 (0.84 × 10) mg/kg.

Leafy vegetables (including Brassica leafy vegetables)

The critical GAP for leafy vegetables (including Brassica leafy vegetables) is from the United States for "leafy vegetables"; 3 × 146 g ai/ha, 7 day-RTI (days), 0-day PHI. The Meeting received trials from Canada and the United States on head lettuce, leaf lettuce, cos lettuce, spinach, radish leaves and mustard greens. All trials matched the critical GAP, except those for radish leaves, which were harvested at a DALA of 7 days.

Mefentrifluconazole residues in head lettuce with wrapper leaves, in ranked order were (n = 8): 0.12, 0.27, 0.32, 0.89, 1.30, 1.50, 2.1 and 2.2 mg/kg. Residues in head lettuce without wrapper leaves were < 0.01, 0.05, 0.09 and 1.6 mg/kg.

Mefentrifluconazole residues in leaf lettuce in ranked order were (n = 7): 2.4, 2.7, 3.0, 4.2, 4.4, 6.4 and 7.2 mg/kg.

Mefentrifluconazole in one sample of cos lettuce was 2.3 mg/kg.

Mefentrifluconazole residues in spinach in ranked order were (n = 8): 3.8, 4.6, 4.9, 5.2, 11, 12 (2) and 17 (highest 18) mg/kg.

The Meeting noted that the GAP in the United States covers the subgroup of leafy vegetables and decided to explore the possibility of estimating a subgroup maximum residue level for mefentrifluconazole. The median residues in head lettuce with wrapper leaves, leaf lettuce and spinach

differed by more than 5-fold and from the Kruskal-Wallis test, the datasets were not shown to be from the same residue population.

Therefore, the Meeting used the spinach dataset to estimate a maximum residue level of 30 mg/kg, an STMR of 8.1 mg/kg and an HR of 18 mg/kg (based on the highest residue of replicate samples) for the Subgroup of leafy greens.

The Meeting noted that the acute dietary exposure assessment showed that residues in leafy greens exceeded the ARfD of 0.3 mg/kg bw, at 140 percent for each amaranth leaves, chicory leaves and edible leaved chrysanthemums for Belgian toddlers, 130 percent for raw endive for Dutch children, 240 percent for cooked/boiled endive for Dutch toddlers, 140 percent for head lettuce for Dutch children and 120 percent for leaf lettuce for Dutch children. No alternative GAP was available.

Mefentrifluconazole residues in mustard greens in ranked order were (n = 4): 4.1, 5.0, 8.3 and 12 mg/kg.

The Meeting noted that the GAP in the United States for the subgroup of leafy vegetables includes Brassica leafy vegetables. The Meeting estimated a maximum residue level of 30 mg/kg, an STMR of 6.65 mg/kg and an HR of 12 mg/kg for the Subgroup of Leaves of Brassicaceae.

For dietary burden calculation, the Meeting estimated a median residue of 6.65 mg/kg and highest residue of 12 mg/kg for mustard greens and extrapolated the median and highest residues to kale leaves and rape forage.

The Meeting noted that the acute dietary exposure assessment showed that exposure from the consumption of mustard greens exceeded the ARfD of 0.3 mg/kg bw, at 240 percent for Chinese cabbage for Chinese children, 110 percent for kale for German children and 200 percent for mustard greens for Chinese children. No alternative GAP was available.

Legume vegetables

The critical GAP for legume vegetables, except soya bean and edamame, is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 21-day PHI. The Meeting received trials on beans and peas with pods and succulent beans and peas without pods from Canada and the United States matching the critical GAP.

Beans with pods

Mefentrifluconazole residues in beans with pods in ranked order were (n=6): < 0.01 (5) and 0.03 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.03 mg/kg for the Subgroup of beans with pods, except soya bean (succulent seeds in pods).

Peas with pods

In trials approximating the critical GAP, mefentrifluconazole residues in peas with pods in ranked order were (n=9): < 0.01 (5), 0.02, 0.03 (2) and 0.08 (highest 0.10) mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.10 (based on the highest residue of replicate samples) mg/kg for the Subgroup of peas with pods.

Succulent beans without pods

In trials approximating the critical GAP, mefentrifluconazole residues in succulent beans without pods in ranked order were (n=6): < 0.01 (5) and 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.03 mg/kg, an STMR of 0.01 mg/kg and an

HR of 0.02 mg/kg for the Subgroup of succulent beans without pods, except soya bean (succulent seeds).

Succulent peas without pods

In trials approximating the critical GAP, mefentrifluconazole residues in succulent peas without pods in ranked order were (n=9): < 0.01 (9) mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg, an STMR of 0.01 mg/kg and an HR of 0.01 mg/kg for the Subgroup of succulent peas without pods.

Pulses

The critical GAP for pulses, except dry soybeans, is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 21-day PHI. The Meeting received trials on dry beans, dry peas and dry lentils from Canada and the United States matching the critical GAP.

Dry beans

Mefentrifluconazole residues in dry beans in ranked order were (n=10): < 0.01 (7), 0.02 (2) and 0.05 mg/kg.

The Meeting estimated a maximum residue level of 0.07 mg/kg and an STMR of 0.01 mg/kg for the Subgroup of dry beans, except soya bean dry.

Dry peas

In trials approximating the critical GAP, mefentrifluconazole residues in dry peas in ranked order were (n=8): < 0.01, 0.01 (3), 0.02 (3) and 0.09 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg and an STMR of 0.015 mg/kg for the Subgroup of dry peas, except lentil (dry).

Dry lentils

In trials approximating the critical GAP, mefentrifluconazole residues in dry lentils in ranked order were (n=6): 0.04, 0.06, 0.14, 0.30, 0.55 and 0.68 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.22 mg/kg for lentils.

Dry soya beans

The critical GAP for dry soya beans is from the United States; 2×146 g ai/ha, 7-day RTI (days), 21-day PHI. The Meeting received supervised residue trials conducted on dry soya bean seeds in the United States matching the critical GAP.

Mefentrifluconazole residues in dry soya beans in ranked order were (n=17): < 0.01 (12), 0.01 (2), 0.03, 0.06 and 0.31 mg/kg.

The Meeting estimated a maximum residue level of 0.40 mg/kg and an STMR of 0.01 mg/kg for soya bean, dry.

Root and tuber vegetables

Root vegetables

The critical GAP for root and tuber vegetables, except sugar beets, is from the United States; 3×146 g

ai/ha, 7-day RTI (days), 7-day PHI. The Meeting received supervised residue trials conducted on carrots and radish in the United States matching the critical GAP.

Mefentrifluconazole residues in carrot roots in ranked order were (n=11): < 0.01 (2), 0.05, 0.06, 0.10, 0.11, 0.12, 0.15, 0.16, 0.22 and 0.24 mg/kg.

Mefentrifluconazole residues in radish roots in ranked order were (n=7): < 0.01, 0.03 (2), 0.08, 0.11, 0.13 and 0.38 (highest 0.40) mg/kg.

The Meeting noted that the GAP covers the group of root vegetables, except sugar beets. The median residues in carrot roots and radish roots are within a 5-fold difference and the Mann-Whitney U-test also determined that the datasets were from the same population. Therefore, the Meeting decided to combine the datasets for carrot roots and radish roots.

Combined mefentrifluconazole residues in carrot roots and radish roots were (n = 18): < 0.01 (3), 0.03 (2), 0.05, 0.06, 0.08, 0.10, 0.11, 0.11, 0.12, 0.13, 0.15, 0.16, 0.22, 0.24 and 0.38 (highest 0.40) mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg, an STMR of 0.105 mg/kg and an HR of 0.40 mg/kg for the Subgroup root vegetables, except sugar beets.

Sugar beet

The critical GAP for sugar beet is from the United States; 2×146 g ai/ha, 14-day RTI (days), 7-day PHI. The Meeting received supervised residue trials conducted on sugar beet roots in Canada and the United States where the RTI (days) was 7 days and the sugar beet roots were harvested at DALAs of 14–21days. Therefore, a maximum residue level could not be estimated for sugar beet roots.

Tuberous and corm vegetables

The critical GAP for tuberous and corm vegetables is from the United States; 3×146 g ai/ha, 7-day RTI (days), 7-day PHI. The Meeting received supervised residue trials conducted on potatoes in Canada and the United States matching the critical GAP.

Mefentrifluconazole residues in potatoes in ranked order were (n = 19): < 0.01 (18) and 0.04 (highest 0.05) mg/kg.

Noting that potatoes are the representative crop of the subgroup tuberous and corm vegetables, the Meeting estimated a maximum residue level of 0.05 mg/kg, an STMR of 0.01 mg/kg and HR of 0.05 mg/kg for the Subgroup of tuberous and corm vegetables.

Wheat

The critical GAP for wheat, triticale and rye is from the United States; 2×146 g ai/ha, 14-day RTI (days), 21-day PHI. The Meeting received supervised residue trials conducted on wheat in Canada and the United States matching the critical GAP. The Meeting also received trials conducted in Europe, but none matched the critical GAP.

Mefentrifluconazole residues in wheat in ranked order were (n = 23): < 0.01, 0.02, 0.03 (3), 0.04 (2), 0.06, 0.08 (2), 0.09 (3), 0.10 (2), 0.11 (2), 0.12 (3), 0.13, 0.14, and 0.27 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.09 mg/kg for wheat grain.

Noting that the GAP covers triticale and rye, and wheat grain is the representative crop of the similar grains, and pseudocereals without husks crop subgroup, the Meeting decided to extrapolate the residues from wheat grain and estimated a maximum residue level of 0.4 mg/kg and an STMR of

0.09 mg/kg for triticale and rye.

Barley

The critical GAP for barley and oats is from the United States; 2×146 g ai/ha, 14-day RTI (days), 21-day PHI. The Meeting received supervised residue trials conducted on wheat in Canada and the United States matching the critical GAP. The Meeting also received trials conducted in Europe, but none matched the critical GAP.

Mefentrifluconazole residues in barley in ranked order were (n = 10): < 0.01, 0.20, 0.25, 0.34, 0.37, 0.48, 0.56, 0.71, 0.80, and 1.7 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg and an STMR of 0.425 mg/kg for residues of mefentrifluconazole in barley grain.

Noting that the GAP covers oats, and barley grain is the representative crop of the similar grains, and pseudocereals with husks crop subgroup, the Meeting decided to extrapolate the residues from barley grain and estimated a maximum residue level of 3 mg/kg and an STMR of 0.425 mg/kg for oats.

Rice

The critical GAP for rice is from China; 2×12 g ai/ha, 5-day RTI (days), 21-day PHI. The Meeting received supervised residue trials conducted on rice in China matching the critical GAP. The Meeting also received trials conducted in Brazil and United States, but none matched the critical GAP.

Mefentrifluconazole residues in rice grain in ranked order were (n = 12): 0.029, 0.65 (2), 0.75, 1.0, 1.1, 1.3, 1.4, 1.8, 2.3, and 2.5 (2) mg/kg.

Mefentrifluconazole residues in husked rice in ranked order were (n = 12): < 0.01, 0.08, 0.09, 0.10 (2), 0.11 (2), 0.19, 0.24, 0.47, 0.50, and 0.79 mg/kg.

The Meeting estimated a maximum residue level of 5 mg/kg and an STMR of 1.2 mg/kg for residues of mefentrifluconazole in rice.

The Meeting also estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.11 mg/kg for residues of mefentrifluconazole in husked rice.

Sorghum

The critical GAP for sorghum and millet is from the United States; 2×146 g ai/ha, 14-day RTI (days), 21-day PHI. The Meeting received supervised residue trials conducted on sorghum in the United States matching the critical GAP.

Mefentrifluconazole residues in sorghum in ranked order were (n = 9): < 0.01, 0.18, 0.24, 0.31, 0.41, 0.42, 0.52, 0.78, and 1.2 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg and an STMR of 0.41 mg/kg for residues of mefentrifluconazole in sorghum grain.

Noting that the GAP covers millet, and sorghum grain is the representative crop of sorghum grain and millet crop subgroup, the Meeting decided to extrapolate the residues from sorghum grain and estimated a maximum residue level of 2 mg/kg and an STMR of 0.41 mg/kg for millet.

Maize

The critical GAP for maize and popcorn is from the United States; 2×146 g ai/ha, 14-day RTI (days), 21-day PHI. The Meeting received supervised residue trials conducted on maize in the United States

matching the critical GAP.

Mefentrifluconazole residues in maize in ranked order were (n = 17): < 0.01 (17) mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg and an STMR of 0.01 mg/kg for residues of mefentrifluconazole in maize.

Noting that the GAP covers popcorn, and maize is the representative crop of maize cereals crop subgroup, the Meeting decided to extrapolate the residues from maize and estimated a maximum residue level of 0.01(*) mg/kg and an STMR of 0.01 mg/kg for popcorn.

Sweet Corn

The critical GAP for sweet corn is from the United States; 3×146 g ai/ha, 14-day RTI (days), 21-day PHI. The Meeting received supervised residue trials conducted on sweet corn in the United States matching the critical GAP.

Mefentrifluconazole residues in sweet corn kernels plus cob with husks removed in ranked order were (n = 13): < 0.01 (9), and 0.02 (4) mg/kg.

The Meeting estimated a maximum residue level of 0.04 mg/kg and an STMR of 0.01 mg/kg and an HR of 0.02 mg/kg for residues of mefentrifluconazole in sweet corn (corn-on-the-cob) (kernels plus cob with husk removed).

Sugar cane

The critical GAP for sugar cane is from the United States; 2×146 g ai/ha, 14-day RTI (days) and a 14-day PHI. The Meeting received supervised residue trials conducted on sugar cane in the United States matching the critical GAP. The Meeting also received trials conducted in Brazil, but none matched the critical GAP.

Mefentrifluconazole residues in sugar cane in ranked order were (n=8): 0.10, 0.25, 0.31, 0.36, 0.38, 0.42, 0.48 and 0.97 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.37 mg/kg for sugar cane.

Tree Nuts

The critical GAP for tree nuts is from the United States; 3×146 g ai/ha, 7-day RTI (days) (all tree nuts, except pistachio) and 10-day RTI (days) (pistachio) and a 14-day PHI. The Meeting received supervised residue trials conducted on pecans and almonds in the United States matching the critical GAP. For pistachios, the application rate and PHI of the residue trials matched the critical GAP, however, the RTI (days)s were 7-days.

Mefentrifluconazole residues in pecans were in ranked order (5): < 0.01 (5) mg/kg.

Mefentrifluconazole residues in almonds were in ranked order (5): < 0.01 (4) and 0.02 mg/kg.

Mefentrifluconazole residues in pistachios were in ranked order (3): 0.01, 0.01 and 0.04 mg/kg.

The Meeting noted that the United States GAP covers the group of tree nuts. The median residues in pecans, almonds and pistachios are within a 5-fold difference and the Kruskal-Wallis test also determined that the datasets were from the same population. Despite the shorter RTI (days) in the crop field trials for pistachios, the Meeting decided the combined dataset was suitable to estimate the maximum residue level, STMR and HR.

Combined mefentrifluconazole residues in pecans, almonds and pistachios were (n=13): < 0.01 (9), 0.01 (2), 0.02 and 0.04 mg/kg.

The Meeting estimated a maximum residue level of 0.06 mg/kg, an STMR of 0.01 mg/kg, and an HR of 0.06 mg/kg (based on the highest residue of replicate samples) for the Group of tree nuts.

Oilseeds and oilfruits

Small seed oilseeds

The critical GAP for rapeseeds is from the United States; 2×146 g ai/ha, 14-day RTI (days) and a 21-day PHI. The Meeting received supervised residue trials conducted on rapeseed in Canada and the United States matching the critical GAP.

Mefentrifluconazole residues in rapeseed in ranked order were (n=13): < 0.01, 0.01 (2), 0.04 (2), 0.05, 0.06 (2), 0.12, 0.15, 0.25 (2) and 0.74 mg/kg.

Noting that the United States GAP covers the subgroup of rapeseeds and that rapeseeds are the representative crop of the subgroup small seed oilseeds, the Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.06 mg/kg for the Subgroup of small seed oilseeds.

Sunflower seeds

The critical GAP for sunflower seeds is from the United States; 2×146 g ai/ha, 7-day RTI (days) and a 21-day PHI. The Meeting received supervised residue trials conducted on sunflower seeds in Canada and the United States matching the critical GAP. The Meeting also received trials conducted in United States, but none matched the critical GAP.

Mefentrifluconazole residues in sunflower seeds in ranked order were (n=10): < 0.01 (4), 0.01 (2), 0.04, 0.05 and 0.06 (2) mg/kg.

Noting that the United States GAP covers the subgroup of sunflower seeds and that sunflower seeds are the representative crop of the subgroup sunflower seeds, the Meeting estimated a maximum residue level of 0.15 mg/kg and an STMR of 0.01 mg/kg for the Subgroup of sunflower seeds.

Cotton seeds

The critical GAP for cotton seeds is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 30-day PHI. The Meeting received supervised residue trials conducted on cotton seeds in the United States matching the critical GAP.

Mefentrifluconazole residues in cotton seeds in ranked order were (n=10): < 0.01, 0.01 (2), 0.03 (2), 0.04 (2), 0.05 (3), 0.10 and 0.12 mg/kg.

Noting that the United States GAP covers the subgroup of cotton seeds and that cotton seeds are the representative crop of the subgroup cotton seeds, the Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.04 mg/kg for the Subgroup of cottonseed.

Peanuts

The critical GAP for peanuts is from the United States; 3×202 g ai/ha, 14-day RTI (days) and a 14-day PHI. The Meeting received supervised residue trials conducted on peanuts from the United States matching the critical GAP.

Mefentrifluconazole residues in peanuts were (n = 11): < 0.01 (11) mg/kg.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and an STMR value of 0.01 mg/kg for peanuts.

Coffee bean

The critical GAP for coffee bean is from Ecuador; 3×160 g ai/ha, 60-day RTI (days) and a 45-day PHI. The Meeting received supervised residue trials conducted on coffee beans from South America matching the critical GAP.

Mefentrifluconazole residues in coffee beans in ranked order were (n = 19): < 0.01 (11), 0.01 (2), 0.02 (3), 0.07, 0.14 and 0.33 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.01 mg/kg for coffee beans.

Residues in animal feeds

Legume animal feeds

The critical GAP for legume vegetables, except soya bean, is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 21-day PHI for bean forage, bean hay, pea vines, and pea hay. The Meeting received trials on pea vines and hay from Canada and the United States matching the critical GAP.

Peas - Vines and hay

Mefentrifluconazole residues in pea vines in ranked order were (n=8): 0.8, 1.3, 2.2, 2.8, 4.3, 7.5, 9.5 and 10.2 mg/kg.

The Meeting estimated a median residue of 3.5 mg/kg and a highest residue of 10.3 mg/kg (based on the highest residue of replicate samples) for pea vines.

Mefentrifluconazole residues in pea hay in ranked order were (n=8): 4.4, 5.7, 6.3, 9.2, 10.3, 10.6, 11 and 12 (highest 13) mg/kg (dry weight).

The Meeting estimated a maximum residue level of 30 mg/kg, a median residue of 9.74 mg/kg and a highest residue of 13 mg/kg (based on the highest residue of replicate samples) for pea hay (dry weight).

Soya bean - Forage and hay

The critical GAP for soya beans is from the United States; 2×146 g ai/ha, 7-day RTI (days) and a 14-day PHI for forage and 21-day PHI for hay.

Mefentrifluconazole residues in soya bean forage from trials matching the critical GAP were: 1.2 and 2.9 mg/kg.

The Meeting noted there were an insufficient number of trials approximating the critical GAP to estimate median and highest residues for soya bean forage.

Mefentrifluconazole residues in soya bean hay from trials matching the critical GAP were in ranked order (n=17): 1.1, 1.7, 2.3, 3.0, 3.2, 4.0, 4.1, 4.3, 4.5, 5.7, 6.0, 6.1, 7.1, 8.2, 8.8, 9.0 and 11 (highest 12) mg/kg (dry weight).

The Meeting estimated a maximum residue level of 20 mg/kg, a median residue of 4.5 mg/kg and a highest residue of 12 (based on the highest residue of replicate samples) for soya bean hay (dry weight).

Non-grass forages

The critical GAP for non-grass forages is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 14-day PHI. The Meeting received trials on alfalfa forage and hay and clover forage and hay from the United States.

While supervised field trials were conducted according to the critical GAP, the RTI (days)s between the second and third applications were significantly longer (14–48 days), without reliable data to estimate half-lives, the Meeting concluded that the overall impact of these parameters on the residue was >25 percent, and maximum residue levels, median residues and highest residues could not be estimated for alfalfa forage and hay or clover forage and hay.

Straw and hay of cereal grains (including pseudocereals)

Wheat

The critical GAP for wheat forage, hay and straw is from the United States; 2×146 g ai/ha, 14-day RTI (days), and a 21-day PHI.

Mefentrifluconazole residues in wheat forage in ranked order were (n=24): < 0.01, 0.12, 0.28, 0.33, 0.56, 0.57, 0.63, 1.1, 1.2, 1.7, 2.5, 2.7, 2.8, 3.3, 3.6, 3.8, 4.0, 4.2 (2), 5.5, 5.9, 6.8, 7.8 and 9.6 mg/kg (dry weight).

Mefentrifluconazole residues in wheat hay in ranked order were (n=24): < 0.01, 0.12, 0.21, 0.23, 0.32, 0.54, 0.72, 1.0, 1.3, 1.5, 2.1 (2), 2.7, 3.5 (2), 3.6, 4.5, 4.7, 4.9, 5.4, 5.6, 5.7, 5.8 and 5.9 mg/kg (dry weight).

Mefentrifluconazole residues in wheat straw in ranked order were (n = 23): < 0.01, 2.8, 3.1, 3.5, 4.5, 5.3, 6.5, 6.8, 7.1, 8.7, 9.5, 10.3, 10.6, 10.8, 11.9, 12.1, 13.4, 14.3, 17.0, 17.1, 18.1, 18.4 and 25.7 mg/kg (dry weight).

Barley

The critical GAP for barley hay and straw is from the United States; 2×146 g ai/ha, 14-day RTI (days), and a 21-day PHI.

Mefentrifluconazole residues in barley hay in ranked order were (n=9): 0.57, 5.3, 5.5, 6.8, 7.2, 8.0, 8.1, 9.1 and 11.1 mg/kg (dry weight).

Mefentrifluconazole residues in barley straw in ranked order were (n=10): < 0.01, 3.0, 3.8, 7.3, 8.1, 11.3, 13.8, 18.1, 20.9 and 22.7 mg/kg (dry weight).

Rice

The critical GAP for rice is from China; 2×12 g ai/ha, 5-day RTI (days), 21-day PHI. The Meeting received supervised residue trials conducted on rice in China matching the critical GAP.

Mefentrifluconazole residues in rice straw in ranked order were (n = 12): 0.12, 0.14, 1.1, 1.6, 2.2, 2.7, 3.4, 4.0, 4.7, 5.0, 7.3 and 10 mg/kg based on a dry matter content of 90 percent derived from the OECD 2018 feed calculator.

Mefentrifluconazole residues in rice husks in ranked order were (n = 12): 0.08, 2.5, 2.6, 3.0, 3.1, 4.4, 4.5, 5.7, 7.4, 9.3 and 9.4 (2) mg/kg.

Sorghum

The critical GAP for sorghum is from the United States; 2×146 g ai/ha, 14-day RTI (days) and a 21-day PHI. The Meeting received supervised residue trials conducted on sorghum forage and fodder (stover) in the United States matching the critical GAP.

Mefentrifluconazole residues in sorghum forage in ranked order were (n = 9): < 0.01, 0.60, 0.76, 1.3, 1.7, 2.1, 3.4, 4.4, and 4.8 mg/kg (dry weight).

Mefentrifluconazole residues in sorghum stover in ranked order were (n=9): < 0.01, 1.2, 1.3, 3.2, 4.1, 4.4, 4.7, 6.3, and 9.1 mg/kg (dry weight).

Maize

The critical GAP for maize is from the United States; 2×146 g ai/ha, 14-day RTI (days) and a 21-day PHI. The Meeting received supervised residue trials conducted on maize forage and fodder (stover) in the United States matching the critical GAP.

Mefentrifluconazole residues in maize forage in ranked order were (n = 17): < 0.01, 1.0, 1.3 (2), 1.6, 1.8, 2.4, 2.5, 3.1, 3.5, 3.7, 4.0, 4.1, 4.4, 4.7, 5.4, and 7.2 mg/kg (dry weight).

Mefentrifluconazole residues in maize stover in ranked order were (n=17): < 0.01, 3.0, 3.2, 3.7, 5.0, 5.2 (2), 6.3, 8.0, 8.1, 8.2, 8.3, 9.7, 9.9, 10, 11, and 12 mg/kg (dry weight).

Sweet corn

The critical GAP for maize is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 21-day PHI. The Meeting received supervised residue trials conducted on sweet corn forage and fodder (stover) in Canada and the United States matching the critical GAP.

Mefentrifluconazole residues in sweet corn forage in ranked order were (n = 13): < 0.01, 1.6, 2.8, 3.5, 3.7, 4.1, 4.2, 4.3, 4.6, 5.5, 5.8, 6.4 and 6.6 mg/kg, based on a dry matter content of 48 percent derived from the OECD 2018 feed calculator.

Mefentrifluconazole residues in sweet corn stover in ranked order were (n=13): < 0.01 (2), 0.45, 0.84, 1.2, 1.3, 1.9, 2.4, 2.5, 3.1 (2), 3.3 and 4.2 mg/kg (dry weight).

The Meeting estimated residues in cereal forages as follows:

Wheat forage median residue of 2.75 mg/kg and highest residue of 9.6 mg/kg (dry weight),

Sorghum forage median residue of 1.7 mg/kg and highest residue of 4.8 mg/kg (dry weight),

Maize forage median residue of 3.1 mg/kg and highest residue of 7.2 mg/kg (dry weight), and

Sweet corn forage median residue of 4.2 mg/kg and highest residue of 6.6 mg/kg (dry weight).

Based on the similarity of residues and commodities within the group of straw and hay of cereal grains (including pseudocereals), the Meeting agreed to make a recommendation based on the trials giving the highest estimates. The Meeting estimated a maximum residue level of 50 mg/kg (dry weight; from barley straw), a median residue of 10.3 mg/kg (dry weight; from wheat straw) and a highest residue of 25.7 mg/kg (dry weight; from wheat straw) for straw and hay of cereal grains (including pseudocereals).

Grass forages

The critical GAP for grass forages is from the United States; 3×146 g ai/ha, 7-day RTI (days) and a 0-day PHI. The Meeting received trials on forages of Bermuda grass, blue grass, brome grass and fescue from

the United States. While supervised field trials were conducted according to the critical GAP in terms of application rate and harvest time, the RTI (days)s were longer (14 days). Without reliable data to estimate half-lives, the Meeting concluded that the overall impact of these parameters on the residue was >25 percent, and maximum residue levels, median residues and highest residues could not be estimated for Bermuda grass, blue grass, brome grass and fescue forage and hay.

Miscellaneous fodder and forage crop

Sugar beet tops

The critical GAP for sugar beets is from the United States; 2×146 g ai/ha, 14-day RTI (days), 7-day PHI. The Meeting received supervised residue trials conducted on sugar beet tops in Canada and the United States where the RTI (days) was 7 days and the sugar beet roots were harvested at DALAs of 14–21days. Therefore, a maximum residue level could not be estimated for sugar beet tops.

Almond hulls

The critical GAP for tree nuts is from the United States; 3×123 g ai/ha, 7-day RTI (days) (all tree nuts, except pistachio) and a 14-day PHI. The Meeting received supervised residue trials conducted on almonds in the United States matching the critical GAP.

Mefentrifluconazole residues in almond hulls were in ranked order (5): 1.1 (2), 1.2, and 1.7 (2) mg/kg.

The Meeting estimated a maximum residue level of 4 mg/kg and a median residue of 1.2 mg/kg.

Peanut hay

The critical GAP for peanuts is from the United States; 3×202 g ai/ha, 14-day RTI (days) and a 14-day PHI. The Meeting received supervised residue trials conducted on peanuts from the United States matching the critical GAP.

Mefentrifluconazole residues in peanut hay were (n = 11): 3.1, 3.5, 4.4, 7.0, 7.1, 8.9, 9.3, 10.6, 14.2, 14.3 and 28.8 mg/kg (dry weight).

The Meeting estimated a maximum residue level of 40 mg/kg, a median residue of 8.9 and a highest residue of 30 mg/kg (based on the highest residue of replicate samples) for peanut hay (dry weight).

Fate of residues during processing

Effects on the nature of the residue during processing

The Meeting received information on the hydrolysis of mefentrifluconazole under simulated processing conditions and the effects of processing on residues of mefentrifluconazole in several commodities.

High temperature hydrolysis

In studies on the hydrolytic stability of aqueous solutions of radiolabelled mefentrifluconazole, samples were incubated under three sets of conditions, each designed to simulate an appropriate process: 90 °C (pH 4, 20 minutes) to simulate pasteurisation, 100 °C (pH 5, 60 minutes), to simulate boiling, baking and brewing, and 120 °C (pH 6, 20 minutes in the dark) to simulate sterilisation.

Under conditions representative of industrial and household processing procedures such as pasteurisation (pH 4, 90 °C, 20 min), baking, boiling, brewing (pH 5, 100 °C, 60 min) and sterilisation (pH 6,

120 °C, 20 min), no degradation product exceeding 2 percent of total radioactivity was detected, demonstrating that mefentrifluconazole is hydrolytically stable. In addition, no change in the isomer ratio was observed. Therefore, the nature of the residue is not affected by processing operations.

Residues in processed commodities

The Meeting evaluated processing studies for oranges, apples, plums, peaches, grapes, strawberries, cucumbers, tomatoes, soya beans, sugar beets, potatoes, wheat, barley, rice, corn, cotton, and coffee. For field corn grain, no reliable processing factors can be calculated since the residues in the raw agricultural commodities (RAC) were <LOQ. Maximum residue levels (mrl) in processed commodities are only estimated when they are higher than the maximum residue levels for the RAC. Processing factors and residue estimates are summarized in Table 178. As the maximum residue level, STMR and HR in lemon were higher than orange, residues in lemons were used to calculate the residues in citrus processed commodities.

Table 178 Processing factors and residue estimates for mefentrifluconazole

	Residue in RAC, mg/kg				Processing Factors		Residue in processed commodity, mg/kg		
Raw commodity	mrl	STMR	HR	Processed commodity	Individual	Best estimate	mrl	STMR-P	HR-P
Lemon	1.5	0.37	0.98	Juice	< 0.02, < 0.01, < 0.02	0.02	-	0.007	
				Pulp	0.02, < 0.03, 0.02	0.02	-	0.007	0.020
				Dried pulp	0.15, 0.04, 0.11	0.11	-	0.04	
				Peel	2.6, 3.3, 2.6	2.6		0.96	2.5
				Oil	38, 71, 41	41	70	15.2	
				Marmalade	0.09, 0.12, 0.31	0.12	-	0.044	
Apple	1.5	0.39	1.12	Canned apples	0.05, < 0.13, 0.25	0.13	-	0.051	
				Fruit syrup	0.40, 0.88, 0.38	0.40	-	0.16	
				Apple sauce	0.05, < 0.13, 0.11	0.11	-	0.043	
				Dried apples	0.31, 0.25, 0.33	0.31	-	0.12	0.35
				Juice	0.09, < 0.13, 0.16	0.13	-	0.051	
				Wet pomace	3.10, 3.25, 2.36	3.10	-	1.2	
				Dried pomace	11.5, 9.88, 7.51	9.88	15	3.9	
Plum	1.5	0.26	1.0	Dried prune	2.57, 4.26, 4.08	4.1	7	1.1	4.1
				De-pitted plum	0.98, 1.16, 1.12	1.1	-	0.29	1.1
				Juice	0.08, 0.20, 0.15	0.15	-	0.039	
				Puree	0.76, 0.43, 0.56	0.56	-	0.15	
Grape-Wine	2	0.54	1.1	Rosé Wine Process					
				Dry pomace	3.13, 3.93, 3.09	3.13	-	1.7	
				Pasteurized juice	0.04, 0.05, 0.05	0.05	-	0.027	
				Rosé wine	0.02, 0.02, 0.03	0.02	-	0.011	
				Red Wine Process					
				Dry Pomace	5.21, 4.26, 3.55	4.26	9	2.3	
				Pasteurized juice	0.12, 0.13, 0.13	0.13	-	0.070	
				Red wine	0.03, 0.02, 0.03	0.03	-	0.016	
Strawberry	2	0.29	1.1	Canned strawberries	0.93, 0.77, 1.18	0.93	-	0.27	
				Fruit syrup	0.20, 0.17, 0.30	0.20	-	0.058	
				Jam before cooking	0.48, 0.21, 0.38	0.38	-	0.11	
				Jam after cooking	0.48, 0.25, 0.43	0.43	-	0.12	
Cucumber	0.15	0.035	0.12	Canned gherkins	1.73, 0.52, 0.88	0.88	-	0.031	0.106
				Pickled gherkins	0.73, 0.26, 0.84	0.73	-	0.026	0.088
Tomato	0.7	0.14	0.45	Canned tomatoes	0.06, 0.08, 0.05	0.06	-	0.0084	0.027
				Ketchup after	0.35, 0.68, 0.56	0.56	-	0.078	-

Mefentrifluconazole

Raw commodity	Residue in RAC, mg/kg			Processed commodity	Processing Factors		Residue in processed commodity, mg/kg		
	mrl	STMR	HR		Individual	Best estimate	mrl	STMR-P	HR-P
				pasteurization					
				Paste	1.00, 0.49, 0.46	0.49	-	0.069	-
				Peeled tomatoes	0.07, 0.06, 0.03	0.06	-	0.0084	0.027
				Puree	0.31, 0.28, 0.20	0.28	-	0.039	
				Raw juice	0.11, 0.08, 0.08	0.08	-	0.011	
				Sundried tomatoes	6.67, 9.17, 15.97	9.17	7	1.3	4.1
				Wet pomace	2.93, 1.75, 7.14	2.93	-	0.41	
Soya bean	0.4	0.01		Aspirated grain fraction	93	93		0.93	-
				Hulls	< 0.83	0.83	-	0.0083	-
				Meal (toasted)	< 0.83	0.83	-	0.0083	-
				Crude oil	1.0	1.0	-	0.01	-
				Tofu	< 0.83	0.83	-	0.0083	-
				Soy sauce	< 0.83	0.83	-	0.0083	-
				Pollards	< 0.83	0.83	-	0.0083	-
				Flour	< 0.83	0.83	-	0.0083	-
				Miso	< 0.83	0.83	-	0.0083	-
				Soy milk	< 0.83	0.83	-	0.0083	-
				Refined oil	< 0.83	0.83	-	0.0083	-
				Meal (untoasted)	< 0.83	0.83	-	0.0083	-
Sugar beet	0.6	0.06		Molasses	0.88, 1.1, 0.53	0.88	-	0.053	-
				Raw sugar	< 0.06, < 0.05, 0.10	0.06	-	0.0036	-
				Affinated sugar	0.11, 0.11, 0.18	0.11	-	0.0066	-
				Refined sugar	< 0.06, < 0.05, 0.10	0.06	-	0.0036	-
				Dried pulp	4.75, 5.24, 3.24	4.75	3	0.29	-
				Ensiled pulp	0.88, 1.14, 0.68	0.88	-	0.053	-
Potato	0.05	0.01	0.05	Peeled tuber	< 0.5, < 0.33	0.33	-	0.0033	0.0165
				Wet peel	1.5, 1.67	1.6	-	0.016	-
				Stove boiled-without peel	< 0.5, < 0.33	0.33	-	0.0033	0.0165
				Microwaved boiled-with peel	< 0.5, < 0.33	0.33	-	0.0033	0.0165
				Baked-with peel	<1.0, < 0.33	0.33	-	0.0033	0.0165
				Fried-without peel	< 0.5, < 0.33	0.33	-	0.0033	0.0165
				Crisps/chips-without peel	< 0.5, < 0.33	0.33	-	0.0033	0.0165
				Granules/flakes	< 0.5, < 0.33	0.33	-	0.0033	-
				Starch	< 0.5, < 0.33	0.33	-	0.0033	-
				Dried pulp-with peel	3.5, 1.33	2.4	-	0.024	0.096
Wheat forage		2.75 (dw)	9.6 (dw)	Wet silage	1.10, 1.44, 1.19	1.2	-	3.3	12
				Wilted silage	8.0, 4.7, 6.5	6.5	-	18	62
Wheat grain	0.4	0.09		Unprocessed bran	2.38, 3.71, 2.94	2.94	1.5	0.26	-
				Germ	0.85, 1.82, 1.12	1.12	0.5	0.10	-
				Shorts	2.62, 4.53, 3.53	3.53	1.5	0.32	-
				Gluten	0.55, < 0.59, 0.44	0.55	-	0.050	-
				Gluten meal	0.29, < 0.59, < 0.29	0.29	-	0.026	-
				Starch	< 0.08, < 0.59, < 0.29	0.29	-	0.026	-
				Whole meal flour	0.77, 1.00, 0.79	0.79	-	0.071	-
				Whole grain bread	0.54, < 0.59, 0.56	0.56	-	0.050	-
				Milled by-products	0.62, 1.12, 0.41	0.62	-	0.056	-
				Aspirated grain	38.46, 21.76, 44.12	38.46	16	3.5	-

Raw commodity	Residue in RAC, mg/kg			Processed commodity	Processing Factors		Residue in processed commodity, mg/kg		
	mrl	STMR	HR		Individual	Best estimate	mrl	STMR-P	HR-P
				fractions					
Barley	3	0.425		Pearled/pot barley	0.16, 0.12, 0.08	0.12	-	0.051	-
				Flour	4.5, 3.67, 3.18	3.67	15	1.6	-
				Unprocessed bran	4.25, 5.00, 5.45	5.00	15	2.1	-
				Brewing malt	0.50, 0.50, 0.30	0.50	-	0.21	-
				Malt sprouts	1.68, 0.96, 0.30	0.96	-	0.41	-
				Beer	< 0.03, < 0.04, < 0.05	0.03	-	0.013	-
				Brewer's grain (dry)	2.38, 2.42, 2.14	2.38	8	1.0	-
				Brewer's yeast	0.19, 0.27, 0.19	0.19	-	0.081	-
Rice	5	1.2		Hulls	4.16, 4.92, 2.55	4.16	15	1.8	-
				Polished rice	0.06, 0.02, 0.01	0.02	-	0.0085	-
				Bran	0.58, 1.42, 1.04	1.04	-	0.44	-
Maize, forage		2.9 (dw)	7.2 (dw)	Silage	1.32, 0.86, 0.56	0.86	-	2.5	6.2
Cotton	0.2	0.04		Hulls	0.82, 0.13, 0.08	0.13	-	0.0052	-
				Meal	0.06, < 0.02, 0.004	0.02	-	0.0008	-
				Refined oil	< 0.05, < 0.02, 0.004	0.004	-	0.00016	-
Coffee	0.4	0.01		Concentrated liquor	0.06, 0.10, 0.08, 0.07	0.075	-	0.00075	-
				Instant coffee	0.18, 0.01, 0.16, 0.16	0.16	-	0.0016	-
				Roasted ground coffee	0.60, 0.93, 0.63, 0.58	0.615	-	0.0062	-

Using the estimated maximum residue level of 1.5 mg/kg for the subgroup of lemons and applying the processing factors of 2.6 for citrus dried peel and 41 for citrus oil, the Meeting estimated maximum residue levels of 4 mg/kg and 70 mg/kg for citrus dried peel and citrus oil, respectively.

Using the estimated maximum residue level of 1.5 mg/kg for pome fruits and applying the processing factor of 9.9 for dried pomace, the Meeting estimated a maximum residue level of 15 mg/kg for apple dried pomace.

Using the estimated maximum residue level of 1.5 mg/kg for the subgroup of plums and applying the processing factor of 4.1 for dried prune, the Meeting estimated a maximum residue level of 7 mg/kg for dried prune plum.

Using the estimated maximum residue level of 2 mg/kg for wine grapes and applying the processing factors of 4.26 for dry pomace, the Meeting estimated a maximum residue level of 9 mg/kg for grape dried pomace.

Using the estimated maximum residue level of 0.7 mg/kg for the subgroup of tomatoes applying the processing factor of 9.2 for sundried tomatoes, the Meeting estimated a maximum residue level of 7 mg/kg for dried tomatoes.

Using the estimated maximum residue level of 0.6 mg/kg for sugar beets and applying the processing factor of 4.75 for dried pulp, the Meeting estimated a maximum residue level of 3 mg/kg for sugar beet dried pulp.

Using the estimated maximum residue level of 0.4 mg/kg for wheat grain and applying the processing factors of 2.94 for wheat unprocessed bran, 1.12 for wheat germ, 3.53 for shorts and 38.5 for aspirated grain fractions, the Meeting estimated maximum residue levels of 1.5 mg/kg for wheat

unprocessed bran, 0.5 mg/kg for wheat germ, 1.5 mg/kg for wheat shorts and 16 mg/kg for wheat aspirated grain fractions.

Using the estimated maximum residue level of 3 mg/kg for barley grain and applying the processing factors of 3.67 for barley flour, 5 for unprocessed barley bran and 2.38 for dry brewer's grain, the Meeting estimated maximum residue levels of 15 mg/kg for each barley flour and barley bran and 8 mg/kg for brewer's grain (dry).

Residues in animal commodities

The following adjustment factors were applied to the mefentrifluconazole residues in the livestock feeding studies, to account for both free and conjugated residues of mefentrifluconazole, which are only present in animal offal, for compliance with the MRL.

Ruminant liver: mefentrifluconazole residues × 1.1

Ruminant kidney: mefentrifluconazole residues × 1.6

Poultry liver: mefentrifluconazole residues × 4.5

Similarly, the following adjustment factors were applied to the residues of mefentrifluconazole and M750F022 in the livestock feeding studies, to account for free and conjugated residues, where applicable, for dietary risk assessment:

Milk: mefentrifluconazole residues + M750F022 residues × 7.45 × 1.15 (MW factor)

Ruminant liver: mefentrifluconazole residues × 1.1 + M750F022 residues × 1.15 (MW factor)

Ruminant kidney: mefentrifluconazole residues × 1.6 + M750F022 residues × 5.6 x 1.15 (MW factor)

Poultry commodities: mefentrifluconazole residues × 4.5 + M750F022 residues × 1.15 (MW factor)

Farm animal feed studies

The Meeting received a feeding study in dairy cattle where mefentrifluconazole was administered orally once daily to five groups of three lactating cattle for 28 days.

Based on mean daily feed consumption, the dosing levels were equivalent to 1.5, 7.5, 50 and 150 ppm (2 groups) in the feed. Milk was collected twice daily (am and pm sampling pooled) throughout the 28 days of dosing. On day 21, milk was also separated into cream and skimmed milk. Muscle, liver, kidney and fat samples were collected at sacrifice 22-24 hours after the final dose, except for one of the 150 ppm dose groups which were sacrificed three, seven and fourteen days after the final dose to monitor the decline of residue levels post dosing. Samples were analysed for the parent compound by LC-MS/MS (LOQ of 0.010 mg/kg).

At the lowest feeding level of 1.5 ppm, residues of mefentrifluconazole were below the LOQ in milk and muscle, however, they were measurable in liver, kidney and fat. The Metabolite M750F022 was not analysed in milk or any of the tissues at this lowest feeding level. A summary of the results are shown in Table 179. For the depuration study, measurable residues of mefentrifluconazole and M750F022 were observed in milk and tissues after three days of withdrawal, however, after seven and fourteen days of withdrawal, low or no residues above the limit of quantification of 0.01 mg/kg were detected, demonstrating a rapid excretion of mefentrifluconazole and M750F022.

Table 179 Residues of mefentrifluconazole in milk and tissues from lactating goats dosed at 7.5, 50 and

150 ppm mefentrifluconazole daily for 28 days (n=3 at each dose level)

	Mefentrifluconazole (free + conjugated) residues [a] (mg/kg)					
	Feeding level: 7.5 ppm		Feeding level: 50 ppm		Feeding level: 150 ppm	
	Mean	Max	Mean	Max	Mean	Max
Muscle	< 0.01	< 0.01	0.07	0.11	0.16	0.22
Liver	0.16	0.19	1.05	1.48	3.21	3.79
Kidney	0.08	0.12	0.47	0.82	2.10	3.06
Fat	0.05	0.08	0.65	0.9	1.71	2.29
Milk (Day 28)	< 0.01	< 0.01	0.05	0.06	0.21	0.25
	Mefentrifluconazole (free + conjugated) + M750F022 (free + conjugated) residues [b] (mg/kg)					
	Mean	Max	Mean	Max	Mean	Max
Muscle	< 0.01	< 0.01	0.08	0.12	0.18	0.24
Liver	0.17	0.20	1.08	1.51	3.26	3..85
Kidney	0.09	0.13	0.60	0.95	2.37	3.34
Fat	0.06	0.09	0.75	1.00	1.85	2.52
Milk (Day 28)	< 0.01	< 0.01	0.14	0.15	0.39	0.44

Notes:

[a] For MRL determination.

[b] For HR and STMR determination for dietary risk assessment.

The Meeting also received a feeding study in laying hens where mefentrifluconazole was administered orally once daily to 4 groups of 12 hens per group by gelatine capsule for 34 days, at dose levels equivalent to 1.5, 4.5 and 15 ppm (2 groups) in the feed. Eggs were collected twice daily (am and pm sampling pooled) throughout the 34 days of dosing. On day 24, egg samples from the 15 ppm level were separated into egg yolk and egg white. Muscle, liver, skin with fat and abdominal fat samples were collected at sacrifice 6 hours after the final dose, except for the hens of one of the 15 ppm group which were sacrificed two, seven and fourteen days after the final dose to monitor the decline of residue levels post dosing.

Samples were analysed for the parent compound by LC-MS/MS and for the metabolite M750F022 by GC-MS, with LOQs of 0.010 mg/kg.

Quantifiable residues of mefentrifluconazole were observed in eggs and muscle at the highest dose tested only. In fat and skin with fat, parent residues were measurable at the 4.5 ppm and 15 ppm feeding levels and in liver, residues were quantifiable at all feeding levels. Similarly, residues of M750F022 were measurable in eggs at 4.5 ppm and 15 ppm dosing levels, in muscle at the highest dose tested and in liver, fat and skin with fat at all feeding levels (except 0.15 ppm for liver and fat, where no residues were quantifiable). A summary of the results are shown in Table 180. For the depuration study, no measurable residues of mefentrifluconazole were observed in tissues after two days of withdrawal. In liver and skin no residues of M750F022 above the LOQ could bedetermined after seven days of withdrawal. In fat residue concentrations decreased steadily to below the LOQ (0.01 mg/kg) after 14 days of withdrawal. The results are shown in Table 175.

Table 175 Residues of mefentrifluconazole (free and conjugated) in eggs and tissues from laying hens dosed at 1.5 ppm, 4.5 ppm and 15 ppm mefentrifluconazole daily for 28 days (n= 12 hens per dosing level)

Matrix	Maximum Residues of Mefentrifluconazole (free and conjugated)[a] (mg/kg)		
	Feeding level: 1.5 ppm	Feeding Level :4.5 ppm	Feeding level: 15 ppm
Muscle	< 0.01	< 0.01	0.03
Liver	0.08	0.09	0.90
Fat	< 0.01	0.025	0.25

Matrix	Maximum Residues of Mefentrifluconazole (free and conjugated)[a] (mg/kg)					
	Feeding level: 1.5 ppm		Feeding Level :4.5 ppm		Feeding level: 15 ppm	
Eggs	< 0.01		< 0.01		0.04	
	Total Residues of Mefentrifluconazole (free and conjugated) and M750F022 (free and conjugated)[b] (mg/kg)					
Matrix	Feeding level: 1.5 ppm		Feeding Level :4.5 ppm		Feeding level: 15 ppm	
	Mean	Max	Mean	Max	Mean	Max
Muscle	< 0.01	< 0.01	< 0.01	< 0.01	0.05	0.07
Liver	0.07	0.10	0.10	0.13	0.61	1.13
Fat	0.05	0.06	0.10	0.11	0.53	0.66
Eggs	< 0.01	< 0.01	0.028	0.03	0.11	0.12

Notes:

[a] For MRL determination.

[b] For HR and STMR determination for dietary risk assessment.

Farm animal dietary burden

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the current JMPR Meeting. The dietary burdens, estimated using the most recent version of the OECD livestock dietary burden calculator, are presented in Annex 6 and summarised in Table 176.

Table 176 Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden: mefentrifluconazole, ppm of dry matter diet							
	United States-Canada		**EU**		**Australia**		**Japan**	
	max	**mean**	**max**	**mean**	**max**	**mean**	**max**	**Mean**
Beef cattle	4.7	2.3	36	18	60	22	15	6.1
Dairy cattle	23	10	36	17	67 ❶	28 ❷	10	4.0
Broilers	0.81	0.81	0.81	0.58	0.92	0.92	0.34	0.34
Layers	0.66	0.66	12 ❸	5 ❹	0.92	0.92	0.54	0.54

Notes:

❶ Highest maximum dairy cattle dietary burden suitable for MRL for milk and mammalian tissues and HR estimates for tissues.

❷ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk and mammalian tissues.

❸ Highest maximum poultry dietary burden suitable for MRL and HR estimates for poultry tissues and eggs.

❹ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Animal commodity maximum residue levels

Cattle

The calculations used to estimate maximum residue levels, STMR and HR values for cattle matrices are shown in Table 177.

Table 177 Estimated residues for maximum residue levels, HRs and STMRs for mefentrifluconazole in cattle commodities

Mefentrifluconazole feeding study	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
MRL [a] beef or dairy cattle							
Feeding study	50.00	0.05	50.00	0.11	1.48	0.82	0.90

Mefentrifluconazole feeding study	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
	150.00	0.21	150.00	0.22	3.79	3.06	2.29
Dietary burden	67.00	0.08	67.00	0.13	1.87	1.20	1.14
HR[B] beef or dairy cattle							
Feeding study			50.00	0.12	1.51	0.95	1.00
			150.00	0.24	3.85	3.34	2.52
Dietary burden			67.00	0.14	1.91	1.36	1.26
STMR [b] beef or dairy cattle							
Feeding Study	7.50	< 0.01	7.50	< 0.01	0.17	0.09	0.06
	50.00	0.14	50.00	0.08	1.08	0.60	0.75
Dietary burden	28.00	0.07	28.00	0.04	0.61	0.34	0.39

Notes:

[a] For MRL determination: Residues of mefentrifluconazole (free and conjugated).

[b] For HR and STMR determination: Total residues of mefentrifluconazole (free and conjugated) and M750F022 (free and conjugated), expressed as parent equivalents.

Based on the calculated residues of mefentrifluconazole (free and conjugated) in milk and cattle tissues, the Meeting estimated maximum residue levels of 0.10 mg/kg in milks, 0.15 mg/kg in meat from mammals other than marine mammals, 1.5 mg/kg in mammalian fats and 2.0 mg/kg in mammalian offal (based on liver).

Based on the highest total residues of mefentrifluconazole (free and conjugated) and M750F022 (free and conjugated) in cattle tissues, the Meeting estimated HR values of 0.14 mg/kg in mammals other than marine mammals, 1.26 mg/kg in mammalian fats and 1.91 mg/kg in mammalian offal (based on liver).

Based on the mean total residues of mefentrifluconazole (free and conjugated) and M750F022 (free and conjugated) in milk and cattle tissues, the Meeting estimated STMR values of 0.07 mg/kg in milks, 0.04 mg/kg in mammals other than marine mammals, 0.39 mg/kg in mammalian fats and 0.61 mg/kg in mammalian edible offal (based on liver).

Poultry

The calculations used to estimate maximum residue levels, STMR and HR values for poultry matrices are shown in Table 178.

Table 178 Estimated residues for maximum residue levels, HRs and STMRs for mefentrifluconazole in poultry commodities

Mefentrifluconazole feeding study	Feed level (ppm) for egg residues	Residues (mg/kg) in egg	Feed level (ppm) for tissue residues	Residues (mg/kg)		
				Muscle	Liver	Fat
MRL [a] broiler or layer poultry						
Feeding study	4.50	< 0.01	4.50	< 0.01	0.09	0.03
	15.00	0.04	15.00	0.03	0.90	0.25
Dietary burden	12.00	0.031	12.00	0.024	0.67	0.187
HR[B] broiler or layer poultry						
Feeding study	4.50	0.03	4.50	< 0.01	0.13	0.11
	15.00	0.12	15.00	0.07	1.13	0.66
Dietary burden	12.00	0.094	12.00	0.053	0.844	0.503
STMR [b] broiler or layer poultry						
Feeding study	4.50	0.03	4.50	< 0.01	0.10	0.10
	15.00	0.11	15.00	0.05	0.61	0.53

Mefentrifluconazole feeding study	Feed level (ppm) for egg residues	Residues (mg/kg) in egg	Feed level (ppm) for tissue residues	Residues (mg/kg)		
				Muscle	Liver	Fat
Dietary burden	5.00	0.032	5.00	0.012	0.124	0.12

Notes:

[a] For MRL determination: Residues of mefentrifluconazole (free and conjugated)

[b] For HR and STMR determination: Total residues of mefentrifluconazole (free and conjugated) and M750F022 (free and conjugated), expressed as parent equivalents

Based on these calculated residues of mefentrifluconazole (free and conjugated) in eggs and poultry tissues, the Meeting estimated maximum residue levels of 0.04 mg/kg in eggs and 0.03 mg/kg in poultry meat, 0.2 mg/kg in poultry fatsand 0.7 mg/kg in poultry edible offal.

Based on the highest total residues of mefentrifluconazole (free and conjugated) and M750F022 (free and conjugated) in eggs and poultry tissues, the Meeting estimated HR values of 0.094 mg/kg in eggs, 0.053 mg/kg in poultry meat, 0.503 mg/kg in poultry fat and 0.844 mg/kg in poultry offal.

Based on the mean total residues of mefentrifluconazole (free and conjugated) and M750F022 (free and conjugated) in eggs and poultry tissues, the Meeting estimated STMR values of 0.032 mg/kg in eggs and 0.012 mg/kg in poultry meat, 0.124 mg/kg in poultry fat and 0.12 mg/kg in poultry edible offal.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *mefentrifluconazole*

Definition of the residue for compliance with the MRL for animal commodities: *mefentrifluconazole (free and conjugated)*

Definition of the residue for dietary risk assessment for animal commodities: sum of *mefentrifluconazole (free and conjugated) +* 2-[4-(4-chlorophenoxy)-2-(trifluoromethyl)phenyl]propane-1,2-diol *(*M750F022*), free and conjugated, expressed as mefentrifluconazole equivalents.* The molecular weight conversion factor to express M750F022 in mefentrifluconazole equivalents = 1.15.

The residue is fat soluble.

Table 179 Recommendations for residues of mefentrifluconazole from the 2022 JMPR

CCN	Commodity name	Recommended Maximum residue levels (mg/kg)[a]		STMR (-P) (mg/kg)	HR (-P) (mg/kg)
		New	Previous		
AM 0660	Almond, hulls	4	1.2		
	Apple fruit syrup			0.16	0.45
	Apple sauce			0.043	
DF 0226	Apple, dried			0.12	0.35
JF 0226	Apple, juice			0.051	
AB 1230	Apple wet pomace			1.2	
AB 0226	Apple dried pomace	15		3.9	
FI 0326	Avocado	1		0.36	0.50
FI 0327	Banana	1.5		0.055 (pulp)	0.21 (pulp)

CCN	Commodity name	Recommended Maximum residue levels (mg/kg)[a]		STMR (-P) (mg/kg)	HR (-P) (mg/kg)
		New	Previous		
	Barley, beer			0.13	
GC 0640	Barley	3		0.425	
CM 3510	Barley bran, unprocessed	15		2.1	
	Barley, brewing malt			0.21	
CF 3511	Barley, flour	15		1.6	
CM 0640	Barley, pearled			0.051	
VP 2060	Beans with pods, except soya bean (succulent seeds in pods), Subgroup of	0.05		0.01	0.03
VA 2031	Bulb Onions, Subgroup of	0.2		0.05	0.14
FB 2006	Bush berries, Subgroup of	5		0.58	3.24
	Carrot culls			0.10	0.38
FB 2005	Cane berries, Subgroup of	3		0.96	1.62
FS 0013	Cherries, Subgroup of	5		1.1	2.4
	Citrus marmalade			0.044	
OR 0001	Citrus oil, edible	70		15.2	
JF 0001	Citrus juice			0.007	
	Citrus peel			0.96	2.5
	Citrus pulp			0.007	0.020
AB 0001	Citrus pulp, dried			0.04	
SB 0716	Coffee bean	0.4		0.01	
SO 0691	Cottonseed, Subgroup of	0.2		0.04	
SM 0716	Coffee beans, roasted			0.0062	
	Coffee beans, concentrated liquor			0.00075	
	Coffee beans, instant coffee			0.0016	
OR 0691	Cotton seed oil, edible			0.00016	
AS 3564	Dried distiller's grain from barley	8		1	
VD 2065	Dry beans, except soya bean (dry), Subgroup of	0.07		0.01	
VD 2066	Dry peas, except lentil (dry), Subgroup of	0.15		0.015	
MO 0105	Edible offal (mammalian)	2.0		0.61 (based on liver)	1.91 (based on liver)
VO 2046	Eggplants, Subgroup of	1.5		0.25	0.84
PE 0112	Eggs	0.04		0.032	0.094
FB 0267	Elderberries	5		0.58	3.2
VC 2039	Fruiting vegetables, Cucurbits - Cucumbers and Summer squashes, Subgroup of	0.15		0.035	0.123
VC 2040	Fruiting vegetables, Cucurbits – Melons, Pumpkins and Winter Squashes, Subgroup of	0.5		0.15	0.23
VA 2032	Green Onions, Subgroup of	4		0.39	2.2
AB 0269	Grape, dried pomace	9		2.3	
JF 0269	Grape, juice			0.070	
-	Grape, wine (red)			0.016	
	Grape, wine (white)			0.011	

Mefentrifluconazole

CCN	Commodity name	Recommended Maximum residue levels (mg/kg)[a]		STMR (-P) (mg/kg)	HR (-P) (mg/kg)
		New	Previous		
FB 2254	Guelder rose	5		0.58	3.2
VL 2050	Leafy greens[a], Subgroup of	30		8.1	18
VL 0054	Leaves of Brassicaceae[a], Subgroup of	30		6.65	12
FC 0002	Lemons and Limes (including Citron), Subgroup of	1.5		0.37	0.98
VD 0533	Lentil (dry)	1.5		0.22	
FB 2009	Low growing berries, Subgroup of	2		0.29	1.1
MF 0100	Mammalian fats (except milk fats)	1.5		0.39	1.26
GC 0645	Maize	0.01*		0.01	
FC 0003	Mandarins (including Mandarin-like hybrids), Subgroup of	1.5		0.37	0.98
FI 0345	Mango	0.6		0.01	0.01
MM 0095	Meat (from mammals other than marine mammals)	0.15 (fat)		0.04 (muscle) 0.39 (fat)	0.14 (muscle) 1.26 (fat)
GC 0646	Millet	2		0.41	
ML 0106	Milks	0.10		0.07	
VL 0485	Mustard greens			6.65	12
GC 0647	Oats	3		0.425	
FC 0004	Oranges, Sweet, Sour (including Orange-like hybrids), Subgroup of	1		0.215	0.70
FI 0350	Papaya	0.5		0.07	0.22
FS 2001	Peaches (including Nectarine and Apricots), Subgroup of	2		0.56	1.04
AL 0072	Pea, hay and/or straw	30 (dry weight)		Median: 9.74	Highest: 13
VP 2061	Peas with pods, Subgroup of	0.15		0.01	0.10
SO 0697	Peanut	0.01*		0.01	
AL 0697	Peanut, hay and/or straw	40 (dry weight)		Median: 8.9	Highest: 30
VO 0051	Peppers, except martynia, okra and roselle, Subgroup of	1.5		0.25	0.84
HS 0444	Peppers, Chili, dried	15		2.5	8.4
FS 0014	Plums (including fresh Prunes), Subgroup of	1.5		0.26	1.0
FP 0009	Pome fruits except persimmon, Japanese, Group of	1.5		0.39	1.12
GC 0656	Popcorn	0.01*		0.01	
DV 0589	Potato flakes/granules			0.0033	
	Potato, baked with peel			0.0033	0.0165
	Potato, crisps/chips – without peel			0.0033	0.0165
	Potato, fried without peel			0.0033	0.0165
	Potato, peeled tuber			0.0033	0.0165
	Potato, starch			0.0033	
	Potato, stove boiled -without peel			0.0033	0.0165
PO 0111	Poultry, edible offal	0.7		0.12	0.844
PF 0111	Poultry, fats	0.2		0.124	0.503
PM 0110	Poultry, meat	0.03 (fat)		0.012 (muscle) 0.124 (fat)	0.053 (muscle) 0.503 (fat)
DF 0014	Prune, dried	7		1.1	4.1

CCN	Commodity name	Recommended Maximum residue levels (mg/kg)[a]		STMR (-P) (mg/kg)	HR (-P) (mg/kg)
		New	Previous		
	Prune juice			0.039	
	Prune puree			0.15	
FC 0005	Pummelo and Grapefruits (including Shaddock-like hybrids, among others Grapefruit), Subgroup of	0.5		0.16	0.24
GC 0649	Rice	5		1.2	
CM 1206	Rice bran, unprocessed			0.44	
CM 0649	Rice, husked	1.5		0.11	
CM 1205	Rice, polished			0.0085	
VR 2070	Root vegetables, except sugar beet, Subgroup of	0.5		0.105	0.40
GC 0650	Rye	0.4		0.09	
SO 2090	Small seed oilseeds, Subgroup of	1		0.06	
GC 0651	Sorghum Grain	2		0.41	
	Strawberries, canned			0.27	
	Strawberry fruit syrup			0.058	
	Strawberry, jam			0.12	
VD 0541	Soya bean (dry)	0.40		0.01	
	Soya bean, flour			0.0083	
AL 0541	Soya bean, hay and/or straw	20 (dry weight)		Median: 4.5	Highest: 12
	Soya bean, miso			0.0083	
OC 0541	Soya bean oil, crude			0.01	
OR 0541	Soya bean oil, refined			0.0083	
	Soya bean, soya sauce			0.0083	
	Soya bean, tofu			0.0083	
AS 0081	Straw and hay of cereal grains	50 (dry weight)		10.3	25.7
VP 2062	Succulent beans without pods, except soya bean (succulent seeds), Subgroup of	0.03		0.01	0.02
VP 2063	Succulent peas without pods, Subgroup of	0.01*		0.01	0.01
GS 0659	Sugar cane	1.5		0.37	
SO 2091	Sunflower seeds, Subgroup of	0.15		0.01	
GC 0447	Sweet corn (Corn-on-the-cob) (kernels plus cob with husk removed)	0.04		0.01	0.02
VO 2045	Tomatoes, Subgroup of	0.7		0.14	0.45
	Tomato, canned			0.0084	0.027
VW 0448	Tomato, paste			0.069	
DM 0448	Tomato puree			0.039	
JF 0448	Tomato, juice			0.011	
DV 0448	Tomato, dried	7		1.3	4.1
	Tomato, wet pomace			0.41	
TN 0085	Tree nuts, Group of	0.06		0.01	0.06
GC 0653	Triticale	0.4		0.09	
VR 2071	Tuberous and corm vegetables, Subgroup of	0.05		0.01	0.05
	Pickled gherkins	0.026		0.088	
FB 1236	Wine-grapes	2		0.54	

CCN	Commodity name	Recommended Maximum residue levels (mg/kg)[a]		STMR (-P) (mg/kg)	HR (-P) (mg/kg)
		New	Previous		
GC 0654	Wheat	0.4		0.09	
CF 3521	Wheat aspirated grain fractions	16		3.5	
CM 0654	Wheat bran, unprocessed	1.5		0.26	
CF 1210	Wheat, germ	0.5		0.10	
	Wheat gluten			0.05	
	Wheat gluten meal			0.026	
CF 3515	Wheat, shorts (cereal grain milling by-product)	1.5		0.32	
	Wheat starch			0.026	
CF 1212	Wheat, whole meal flour			0.071	
	Whole grain bread			0.050	

a. On the basis of the information provided to the JMPR it was concluded that the estimated acute dietary exposure to residues of mefentrifluconazole for the consumption of commodities from the subgroups of Leafy greens and Leaves of Brassicaceae may present a public health concern.

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for mefentrifluconazole is 0–0.04 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for mefentrifluconazole were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 6–40 percent of the maximum ADI.

Acute dietary exposure

The ARfD for mefentrifluconazole is 0.3 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for mefentrifluconazole were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs were at or less than 100 percent of the ARfD, except for:

Amaranth leaves, chicory leaves and edible leaved chrysanthemums (140 percent each for Belgian toddlers)

Raw endive (130 percent for Dutch children)

Cooked/boiled endive (240 percent for Dutch toddlers)

Cos lettuce (140 percentfor Dutch children)

Head lettuce (140 percent for Dutch children)

Leaf lettuce (120 percent for Dutch children)

Chinese cabbage (240 percent for Chinese children)

Kale (110 percent for German children)

Mustard greens (200 percent for Chinese children)

The meeting concluded that acute dietary exposure to residues of mefentrifluconazole in commodities where the ARfD is exceeded may present a public health concern.

Threshold of toxicological concern (TTC) consideration for metabolites

The Meeting agreed that the fatty acid conjugates of M750F022 (M750F023, M750F024 and M750F025) identified in poultry matrices could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day).

The current Meeting estimated dietary exposures for metabolite M750F023 of 0.056 µg/kg bw/day, for M750F024 of 0.034 µg/kg bw/day and for M750F025 of 0.033 µg/kg bw/day.

The Meeting concluded that the estimated dietary exposure to residues of M750F023, M750F024 and M750F025 from uses considered by the JMPR is below the TTC for Cramer Class III compounds and is unlikely to present a public health concern. Should further uses be considered in the future, these conclusions may need to be re-evaluated.

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2018/7003950	Crawford, L.	2018	Magnitude of the residue of BAS 750 F in peach following applications of BAS 750 02 F SC. 820558, 137G1678. 2018/7003950. Landis International Inc., Valdosta GA, United States of America. GLP. Unpublished
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2014/1315283	Plier, S.	2015	Determination of residues of BAS 750 F (Reg.No. 5834378) in wheat and its processed products after two applications of BAS 750 01 F in Germany, 2014 433295, P 3551 G; 14 10 47 006. 2014/1315283. BioChem agrar Labor fuer biologische und chemische Analytik GmbH, Gerichshain, Germany Fed.Rep. GLP. Unpublished
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2017/3002104	Castro, M.	2017	Residue study of BAS 750 02 F in rice (paddy and upland, with hulls, without hulls and polished), after treatment with BAS 750 02 F, under field conditions in Brazil. 805168, GENCS 2758-15. 2017/3002104. BASF S.A. - GENCS-S-APS/RG. Unpublished
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2019/2041531	Jose, W	2019	Residue study of mefentrifluconazole in coffee (dried coffee cherry, beans and processed fractions) after treatment with BAS 751 01 F under field conditions in Brazil. 742829. 2019/2041531. BASF SA, Guaratingueta, Brazil. GLP. Unpublished
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2019/7002361	Wyatt, D.	2019	Magnitude of the residues of BAS 750 F in leafy vegetables raw agricultural commodities following applications of BAS 750 02 F. 744167, 558G1835 2019/7002361. The Carringers Inc., Apex NC, United States of America. GLP Unpublished
2016/7010852	Webber, T.	2018	Magnitude of the residue of BAS 750 F in carrot and radish following applications of BAS 750 03 F. 763680.RL, PSM-16-04-02, 137G1604 2016/7010852. Precision Study Management LLC, Amarillo TX, United States of America. GLP. Unpublished
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2017/1018091	Moreno, S.	2017	Study on the residue behaviour of BAS 750 F (Reg. No. 5834378) on sunflower after treatment with BAS 750 05 F under field conditions in North and South Europe, season 2016. 811522, 16/09/PF; S16-04324. 2017/1018091 Agricultura y Ensayo S.L., Alcala de Guadaira, Spain. GLP. Unpublished
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METALAXYL (138)/METALAXYL-M (212)

First draft prepared by Professor E Dutra Caldas, University of Brasilia, Brazil

BACKGROUND

Metalaxyl and metalaxyl-M are fungicides. Metalaxyl is a racemic mixture of R- and S-enantiomers, where the R-enantiomer is the biologically active form, and metalaxyl-M consists of a minimum of 97 percent of the R-enantiomer.

Metalaxyl was evaluated under the periodic review and metalaxyl-M was evaluated for new uses by the JMPR in 2021. At the same Meeting, both were evaluated for toxicology, when a ADI and a ARfD were established, which apply to both metalaxyl and metalaxyl-M

The current Meeting received GAP information, analytical method, and residue trials on metalaxyl in pineapple from the Government of Thailand. Furthermore, data provided to the 2021 on dried ginseng was re-evaluated.

ANALYTICAL METHOD

A summary report of the analytical method to analyse metalaxyl in pineapple was submitted. Pineapple samples were extracted with acetone and dichloromethane, sodium chloride and sodium sulfate were added, and the extract cleaned-up with silica gel column. Metalaxyl was determined by GC-NPD. Recovery at concentrations of 0.01, 0.1 and 1.0 mg/kg (n=10) ranged from 90 to 91 percent, with a RSD of <15 percent. The LOQ was set at 0.01 mg/kg.

The method used to analyse the field trial samples include extraction with acetone and dichloromethane, with no cleanup step. One trial reported recoveries ranging from 89.9 to 95 percent at 0.02, 0.05 and 0.5 mg/kg with RSD <5 percent. The LOQ was set at 0.02 mg/kg.

USE PATTERNS

Table 1 Registered use of metalaxyl in pineapple in Thailand

WP formulation, ai g/kg	Application					PHI, days
	Method	No	Rate, kg ai/ha	Spray concentration, kg ai/hL	Interval, days	
250	Foliar	2	0.25	0.05	60	14

RESIDUES RESULTING FROM SUPERVISED TRIALS

A summary report of the residue trials conducted in pineapple was provided, with information of analytical method used. The results are shown in Table 2

Table 2 Residues of metalaxyl in pineapple treated with metalaxyl in supervised trials conducted in Thailand in 2010 using 2 foliar applications of a WP formulation (60 days interval).

District, province (Variety)	Application			DAT, days	Residues (mg/kg)	Trial Application
	Rate (kg ai/ha)	Conc (kg ai/hL)	No			
Tha takiap, Chachoengsao (Pattavia)	0.25	0.05	2	0	0.43	Metalaxpine-01
				1	0.32	
				3	0.13	
				5	0.07	
				7	0.03	
				10	0.03	

District, province (Variety)	Application			DAT, days	Residues (mg/kg)	Trial Application
	Rate (kg ai/ha)	Conc (kg ai/hL)	No			
				14	0.03	
				21	0.02	
				28	<0.02	
Sriracha, Chonburi (Sriracha)	0.25	0.05	2	0	0.21	Metalaxpine-02
				1	0.23	
				3	0.13	
				5	0.13	
				7	<0.02	
				10	<0.02	
				14	<0.02	
				21	<0.02	
				28	<0.02	
Nong Ya Plong, Petchaburi (Pattavia)	0.25	0.05	2	0	0.45, 0.57, 0.38	Metalaxpine-03
				1	0.20, 0.25, 0.16	
				3	0.10, 0.09, 0.06	
				5	0.06, 0.05, 0.02	
				7	0.02 (2), 0.05	
				10	0.02 (2), 0.05	
				14	<0.02 (3)	
				21	<0.02 (3)	
Central, Ratchaburi	0.26	0.05	2	0	0.58, 0.93, 0.75	Metalaxpine-04
				1	0.15 (2), 0.08	
				3	0.04, 0.06, 0.05	
				5	0.03, 0.07, 0.04	
				7	0.05 (2), 0.4	
				10	<0.02 (2), 0.06	
				14	<0.02, 0.02(2) (0.02)	
				26	<0.02 (2), 0.03	
Central, Prajubkerikan (Pattavia)	0.23	0.05	2	0	0.22, 0.30, 0.38	Metalaxpine-05
				1	0.29, 0.27 (2)	
				3	0.19, 0.15, 0.13	
				5	0.13 (2), 0.15	
				7	0.10 (2), 0.09	
				10	0.05 (2), 0.06	
				14	0.06 (2), 0.05 (0.06)	
				21	0.04 (2), 0.03	
				28	0.03 (2), 0.02	

APPRAISAL

Metalaxyl is a racemic mixture of R- and S-enantiomers, where the R-enantiomer is the biologically active form, and metalaxyl-M consists of a minimum of 97 percent of the R-enantiomer.

The 2021 JMPR evaluated metalaxyl under the periodic review and metalaxyl-M for new uses. A single ADI and ARfD were also established by the same Meeting.

The residue definitions for metalaxyl and metalaxyl-M for plant commodities are:

Compliance with MRL: metalaxyl (sum of enantiomers)

Dietary risk assessment: metalaxyl (sum of enantiomers) and N-(2-hydroxymethyl-6-methylphenyl)-N-(methoxyacetyl)alanine methyl ester (M8; free and conjugated; sum of enantiomers), expressed as metalaxyl.

The present Meeting received analytical method, GAP information and residue trials for pineapple from the Government of Thailand. Additionally, data evaluated by the 2021 JMPR for dried ginseng was re-assessed.

Method of analysis

A summary report, provided to the Meeting, describe a GC-NPD method to analyse metalaxyl in pineapple. The method included extraction with acetone and dichloromethane, clean-up with silica gel column and was validated at a LOQ of 0.01 mg/kg. The method used in the analysis of field trial samples, however, does not include a clean-up step and has a reported LOQ of 0.02 mg/kg, with very limited validation data.

Results of supervised residue trials on crops

The estimated values for metalaxyl are intended to cover uses of metalaxyl-M. For dietary risk assessment, a factor of 1.3 is applied to STMR and HR values found in the fruits to account for the presence of metabolite M8 (free and conjugated). No factor is needed for bulb, root, and tuber crops or for crops when no residues are expected to be found.

Pineapple

Five residues trials were conducted with metalaxyl in Thailand in 2010 according to GAP (2×0.25 kg ai/ha, 60 days interval, and 14 days PHI). Residues were < 0.02 (2), 0.02, 0.03 and 0.06 mg/kg.

The Meeting estimates a maximum residue level of 0.1 mg/kg, a STMR of 0.026 mg/kg and a HR of 0.78 mg/kg for metalaxyl in pineapple.

Ginseng

The 2021 JMPR evaluated trials conducted with metalaxyl-M in South Korea (GAP of 0.08 kg ai/ha, 10-day interval, 14-day PHI) and recommended a MRL of 0.03(*) mg/kg for metalaxyl in ginseng.

Fresh ginseng samples were washed, dried in hot air to reach the water content under 14 percent and analysed. Residues were < 0.06 mg/kg (n= 9).

Based on the data evaluated by the 2021 JMPR, the Meeting estimates a maximum residue level of 0.06(*) mg/kg, and a STMR and HR of 0.06 mg/kg for metalaxyl in ginseng, dried including red ginseng.

RECOMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for metalaxyl and metalaxyl-M for compliance with the MRL for plant commodities: *metalaxyl (sum of enantiomers).*

Definition of the residue for metalaxyl and metalaxyl-M for dietary risk assessment in plant commodities: *metalaxyl (sum of enantiomers) and N-(2-hydroxymethyl-6-methylphenyl)-N-(methoxyacetyl)alanine methyl ester (M8; free and conjugated; sum of enantiomers), expressed as metalaxyl.*

Definition of the residue for for metalaxyl and metalaxyl-M for compliance with the MRL in animal commodities: *the sum of metalaxyl (sum of enantiomers) and metabolites (free + conjugated) M3 (N-(2,6-*

dimethylphenyl)-N-(hydroxyacetyl)alanine methyl ester) and M8 (N-(2-hydroxymethyl-6-methylphenyl)-N-(methoxyacetyl)alanine methyl ester (sum of enantiomers), expressed as metalaxyl.

Definition of the residue for metalaxyl and metalaxyl-M for dietary risk assessment in animal commodities: *the sum of metalaxyl (sum of enantiomers) and metabolites (free + conjugated) M1 (N-(2,6-dimethylphenyl)-N-(methoxyacetyl) alanine), M3 (N-(2,6-dimethylphenyl)-N-(hydroxyacetyl)alanine methylester), M6 (N-(2,6-dimethylphenyl)-N-(hydroxyacetyl)alanine), M7 (N-(2,6-dimethyl- 5-hydroxyphenyl)-N-(methoxyacetyl)alanine methyl ester) and M8 (N-(2-hydroxymethyl-6-methylphenyl)-N-(methoxyacetyl)alanine methyl ester (sum of enantiomers), expressed as metalaxyl*.

The residue is not fat-soluble.

CCN	Commodity	Maximum residue levels, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New (basis)[a/]	Previous		
FI 0353	Pineapple	0.1 (M)		0.026	0.078
DV 0604	Ginseng, dried including red ginseng	0.06* (MM)		0.06	0.06

[a/] residue data that was the basis for the estimation: metalaxyl (M), metalaxyl-M (MM)

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for metalaxyl and metalaxyl-M (alone or in combination) is 0–0.08 mg/kg bw. The International Estimated Daily Intakes (IEDIs) estimated for the 17 GEMS/Food Consumption Cluster Diets estimated by the 2021 JMPR was not impacted by the recommendation made for pineapple by the current Meeting. No consumption data is available for ginseng, dried.

The Meeting concluded that long-term dietary exposure to residues of metalaxyl residues from the uses considered the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for metalaxyl is 0.5 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for metalaxyl were calculated for pineapple and ginseng, dried. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs varied from 0–1 percent of the ARfD for children and 0–1 percent for the general population. The Meeting concluded that acute dietary exposure to residues of metalaxyl from uses considered by the current Meeting is unlikely to present a public health concern.

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METHIDATHION (051)

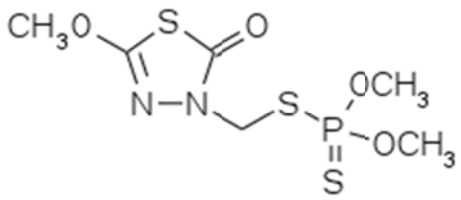
First draft prepared by Dr H Kobayashi, Ministry of Agriculture, Forestry and Fisheries, Japan

EXPLANATION

Methidathion is an organic thiophosphate insecticide. It has a role as an acetylcholinesterase inhibitor. It is registered in some countries. Methidathion was evaluated by JMPR in 1992 in the periodic review programme and re-examined several MRLs in 1994.

At the Fifty-first Session of CCPR (2019), methidathion was scheduled for consideration by the 2020 JMPR in the periodic review programme. The Meeting received the data for methidathion on plant and animal metabolism, method of analysis, use pattern, and residues resulting from supervised trials in apple, cherry, grape, mandarin, mango and peach. It is noted that the current Meeting did not receive some information that had been provided to 1992 JMPR on plant and animal metabolism, environmental fate and processing study.

IDENTITY

ISO common name	Methidathion
IUPAC name	S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O,O-dimethyl phosphorodithioate
Chemical Abstract name	S-[(5-methoxy-2-oxo-1,3,4-thiadiazol-3(2*H*)-yl)methyl] O,O-dimethyl phosphorodithioate
CAS No.	950-37-8
CIPAC No.	193
Synonyms	DMTP, GS-13005
Molecular formula	
Structural formula	$C_6H_{11}N_2O_4PS_3$
Molecular weight	302.33 g/mol

Physical and chemical properties

Property	Results	Test material purity	Reference
Methidathion			
Melting point	40.0-40.9 °C	99.1 percent	Das, 70672
Boiling point	150 °C (thermal decomposition) 99.9 °C at 1.3 Pa	99.1 percent	Das, 70673
Relative density	1.54 g/cm^3 (22 °C)	99.1 percent	Füldner, PP-98/143P.DES
Vapour pressure	2.25 x 10^{-4} Pa (25 °C)	99.1 percent	Rordorf, AG-87/34P
Description of the physical state, colour and odour, purity of the ai, and of technical grade	White solid (fine powder)	99.7 percent	Suda, MS0070C Suda, MS0070K

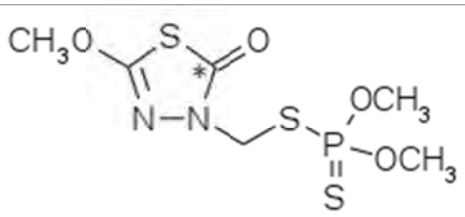
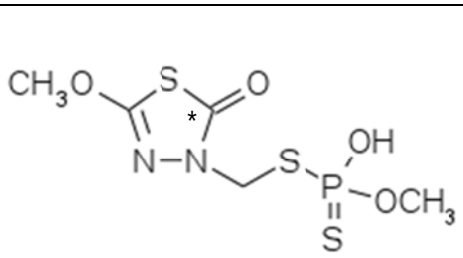
Property	Results	Test material purity	Reference
Solubility of purified ai in water	200 mg/L (25 °C)	99.7 percent	Rodler, EA 149867
Solubility in organic solvent	Acetone: >500 g/L Dichloromethane: >500 g/L Ethyl acetate: >500 g/L Hexane: 14 g/L Methanol: >500 g/L Octanol: 99 g/L Toluene: >500 g/L	91.7 percent	Kettner, 77698
n-Octanol / water partition coefficient (log P_{ow})	2.2 (25 °C)	96.6 percent	Daly, 35827
Hydrolysis rate under sterile conditions in the absence of light	$t_{1/2}$ = 37 days (pH5, 24-25 °C) $t_{1/2}$ = 48 days (pH7, 24-25 °C) $t_{1/2}$ = 13 days (pH9, 24-25 °C)	^{14}C-Methidathion Radiochem. purity: (1) 97.6 percent (2) 98.9 percent	Saxena, HLA 6117-134
Photolysis	Irradiated: $t_{1/2}$ = 11.6 days (corresponding to 8.2 natural days) Dark: $t_{1/2}$ = 45.9 days (pH 7, 25 °C, Xenon light 11.3 W/m^2 (300-400 nm)) Irradiated: $t_{1/2}$ = 40.1 days Dark: $t_{1/2}$ = 29.5 days (pH 7, 23 °C, Xenon light: 40.4 W/m^2 (300-400 nm))	98.9 percent 99.5 percent	Saxena, HLA 6117-137 Mamouni, 848529
Dissociation constant in water	No dissociation in aqueous solutions in the pKa range of 1-12	99.7 percent	Hörmann, PP-98/144P.DCW
Thermal stability	Stable up to 150 °C		Schürch, PP-93/5T.TSA

Specifications

Specifications for Methidathion have not been established by JMPS.

METABOLISM AND ENVIRONMENTAL FATE

The studies for plant metabolism (oranges, tomatoes, common beans and alfalfa) and animal metabolism (lactating cows and goats) were conducted with the test materials with the label position indicated in Figure 1.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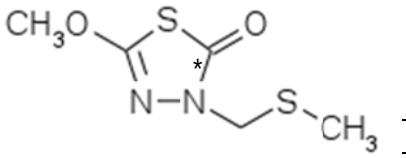
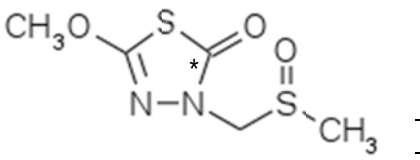
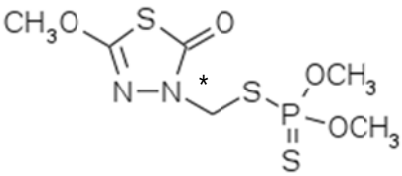
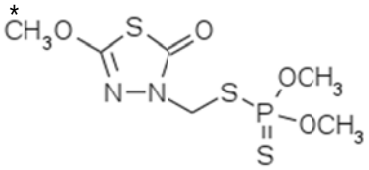
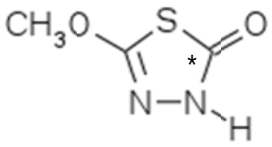
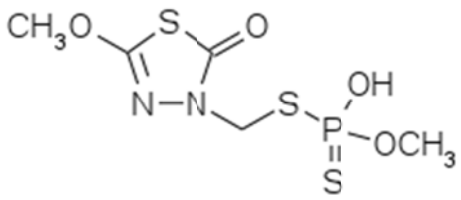
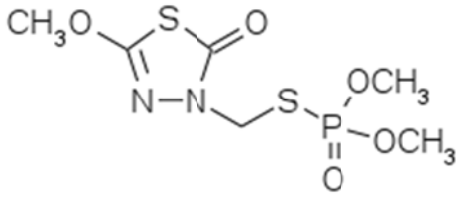
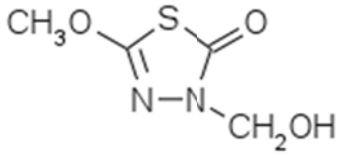
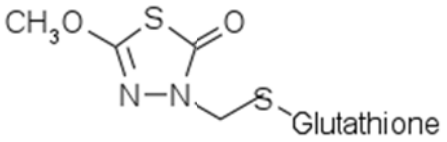
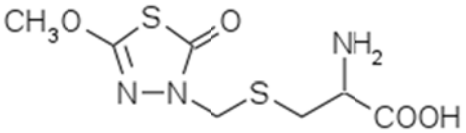
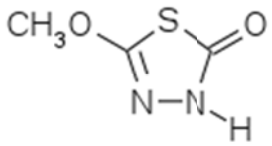
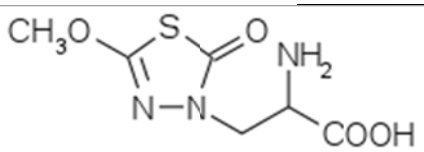
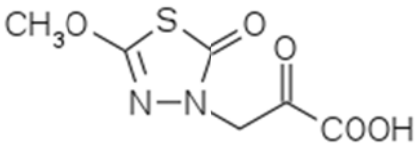
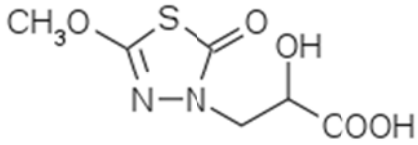
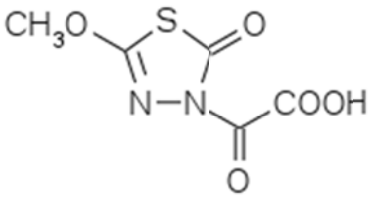
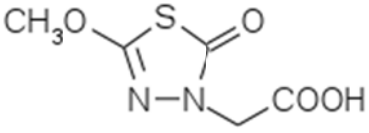
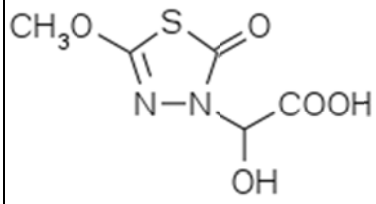
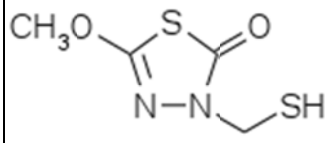
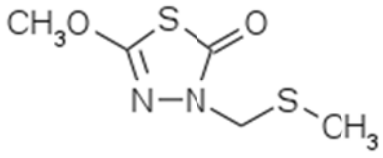
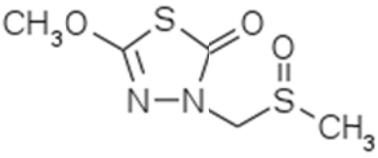
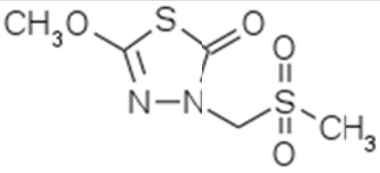
	
^{14}C-RH-sulfone	^{14}C-oxygen analogue
	
3-methylene-^{14}C-methidathion	O-methyl-^{14}C-methidathion
	
2-carbonyl-^{14}C-RH	

Figure 1 Radiolabelled test materials used in the metabolism studies

Table 3 Metabolites/degradates found in metabolism studies

Compound code number, chemical name	Structure	Found in

Compound code number, chemical name	Structure	Found in
Desmethyl methidathion S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O-methyl phosphorodithioate		Orange, tomato, rat
Oxygen analogue of methidathion Methidathion oxon GS 13007 S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O,O-dimethyl phosphorothioate		Tomato, bean, alfalfa
GS-20685 2,3-dihydro-3-hydroxymethyl-5-methoxy-1,3,4-thiadiazol-2-one		(orange, rat)
Glutathione conjugate of methidathion 2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl-thioglutathione		(orange, tomato, rat)
Cysteine conjugate of methidathion 2-amino-3-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethylthioxy) propionic acid		Orange, tomato, rat
RH GS 12956 2,3-dihydro-5-methoxy-1,3,4-thiadiazol-2-one		(orange), bean, alfalfa, (rat)
RH-Alanine conjugate 2-amino-3-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl) propionic acid		Orange
RH-Keto acid conjugate 2-oxo-3-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl) propionic acid		Orange
RH-Lactic acid conjugate 3-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl)-2-hydroxypropionic acid		Orange

Compound code number, chemical name	Structure	Found in
RH-glyoxylic acid conjugate 2-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-yl) -2-oxo-acetic acid		Orange
RH-Acetic acid conjugate 2-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-yl) acetic acid		(orange)
RH-Hydroxy acetic acid conjugate 2-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-yl)-2-hydroxy-acetic acid		Orange
RH-thiol 2,3-dihydro-5-methoxy-4-sulfanyl-1,3,4-thiadiazol-2-one		Tomato, (orange, bean, alfalfa), (rat)
RH-sulfide 2,3-dihydro-5-methoxy-4-methylsulfanyl-1,3,4-thiadiazol-2-one		Tomato, bean, alfalfa, rat
RH-sulfoxide GS 28370 2,3-dihydro-5-methoxy-4-methylsulfinyl-1,3,4-thiadiazol-2-one		Tomato, bean, alfalfa, rat
RH-sulfone GS28369 2,3-dihydro-5-methoxy-4-methylsulfonyl-1,3,4-thiadiazol-2-one		Tomato, bean, alfalfa, rat

Note:

(): Supposed to exist but not identified

Plant metabolism

The Meeting received information on plant metabolism of methidathion using some compounds in Figure 1 in oranges, tomatoes, beans and alfalfas. In the following texts, total radioactive residues (TRR) or applied radio activity (AR) is expressed in mg-methidathion equivalents/kg.

Oranges (Burnett 1992, ML-91-704)

The metabolism of methidathion under field condition in a commercial orchard was studied on a navel orange tree (Beck, 4 years old, 1.8 m high). The soil was a sandy loam (73.3 percent sand, 20.0 percent silt, 6.7 percent clay, 0.4 percent organic matter, pH 6.5, CEC 6.7 meq/100 g, bulk density 1.48 kg/L, 17.3 percent moisture hold capacity at 33 kPa). The EC-formulated 2-carbonyl-^{14}C-methidathion (0.06 kg ai/hL, 500 mL) was twice applied to an orange tree at 11 days after bloom and 43 days after the first application at rates of 0.067 kg ai/ha. A control tree was in the same orchard and 235 m away from the treated tree.

Oranges were sampled at maturity 159 days after last application (DALA). Leaves were sampled after the first application, 28 days after the first application and at maturity of fruits (159 DALA). All fruits and leaves harvested were stored at -17 °C.

Oranges were washed with deionized water, scrubbed gently with a soft-bristled brush using a surfactant commonly used during commercial orange processing (Brogdex #567-10, 1.25 mL/L), and then rinsed with deionized water. The washes and rinse were combined. The fruits were peeled. The peeled fruit was processed in a blender, centrifuged and the juice decanted. The pulp was washed once with deionized water and centrifuged again. The pulp rinse and juice were weighed and volumes determined. The pulp was air-dried. The peel was processed by a food processor with dry ice added.

Subsamples of each solid matrix were homogenized to determine the TRR. Other samples were extracted with methanol-water (9:1, v/v) and then partitioned with chloroform. The radioactivity of each fraction was analysed by liquid scintillation counting (LSC). The organic fraction was analysed by TLC and the aqueous fraction by anion ion-exchange column chromatography and TLC.

The total radioactive residues in leaves were 39 mg eq/kg after the first treatment, 36 mg eq/kg at 28 days after the first treatment and 45 mg eq/kg at 159 DALA. The TRR in mature orange fruit harvested 159 DALA was 0.56 mg eq/kg, with 66 percent TRR (1.0 mg eq/kg) in orange peel, 11 percent TRR (0.40 mg eq/kg) in orange pulp and 22 percent TRR (0.25 mg eq/kg) in orange juice. In the wash, 0.5 percent TRR (< 0.01 mg eq/kg) was detected.

Table 4 Distribution of radioactivity in orange tissues (Application rate: 0.067 kg ai/ha)

Sample	% TRR	mg eq/kg
Leaves		
After 1st treatment	na	39
28 days after the first treatment	na	36
159 DALA	na	45
Orange	100	0.56
Peel	66	1.0
Pulp	11	0.40
Juice	22	0.25
Wash	0.5	<0.01

Note:

na= Not analysed.

The extractability of radioactivity in the leaves and fruits is summarized in Table 5 and Table 6, respectively. In orange peel, 65 percent TRR (0.67 mg eq/kg) was extracted with methanol-water (9:1, v/v) and 27 percent TRR (0.28 mg eq/kg) was unextracted. When this extract was partitioned into water-chloroform, 29 percent TRR (0.31 mg eq/kg) was water soluble and 24 percent TRR (0.25 mg eq/kg) was partitioned into chloroform, and 6.4 percent TRR (0.067 mg eq/kg) was unknown volatile components in the rotary evaporation distillate. For pulp, majority was unextracted residue (57 percent TRR,

0.23 mg eq/kg) and only 17 percent TRR was extracted (0.068 mg eq/kg), in which 12 and 2.5 percent TRR (0.048 and 0.010 mg eq/kg) were partitioned in aqueous and chloroform phase, respectively. Almost all residues in juice (94 percent TRR, 0.24 mg eq/kg) was partitioned into the aqueous phase.

Table 5 Radioactivity in fractions of orange leaves (Application rate: 0.067 kg ai/ha)

Sample	**Leaves 28 DAT1**		**Leaves 159 DALA**	
	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR	-	36	-	45
Aqueous methanol	86	31	78	35
Part. in water	26	9.2	46	21
Part. in $CHCl_3$	48	38	38	17
unextracted residue	18	6.4	15	6.7
Total recovered	104	36	93	42

Table 6 Radioactivity in fractions of orange fruits (Application rate: 0.067 kg ai/ha)

Sample	**Peel**		**Pulp**		**Juice**	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR		1.0		0.40		0.25
Aqueous methanol	65	0.67	17	0.068	94	0.24
Part. in water	29	0.31	12	0.048	94	0.24
Part. in chloroform	24	0.25	2.5	0.010	Nd	<0.01
Distillate[a]	6.4	0.067	nd	nd	Na	na
Unextracted residue	27	0.28	57	0.23	Na	na
Total recovered	92	0.95	74	0.29	94	0.24

Note:

[a] Radioactivity recovered in rotary evaporation distillate trap. nd: not detected, na: not applicable.

Unextracted residues in the orange peel were further treated by sequential acid and base hydrolysis as shown in Table 7. Low percentages were hydrolysed with either acid or base (0.11–3.5 percent TRR, 0.001–0.036 mg eq/kg).

Table 7 Radioactive residues in unextracted fraction from orange peel

Sample	Percent of unextracted [%]	[% TRR]	[mg eq/kg]
Orange peel, unextracted	100	27	0.28
0.1 mol/L HCl extraction			
$CHCl_3$ partition	0.4	0.11	0.001
Aqueous partition	2.1	0.57	0.006
1 mol/L HCl hydrolysis			
$CHCl_3$ partition	1.9	0.51	0.005
Aqueous partition	39	11	0.11
Crude cellulose	13	3.5	0.036
5 mol/L NaOH hydrolysis			
$CHCl_3$ partition	1.0	0.27	0.003
Aqueous partition	13	3.5	0.036
Crude lignin	1.1	0.30	0.003

Residues in plant extracts were further analysed by TLC and anion ion-exchange chromatography. In all samples, organic soluble fractions consisted of methidathion. From aqueous soluble fractions, three clusters of compounds, designated cluster A, B and C, were separated. Cluster A contained RH-alanine conjugate, cluster B contained RH-acetic acid conjugate, RH-lactic acid conjugate,

RH-hydroxy acetic acid conjugated and cluster C contained desmethyl-methidathion (Table 8). In this study, oxygen analogue of methidathion was not detected.

Table 8 identification of radioactive residues in aqueous and organic extracts from orange leaves, peel and juice

	Orange leaves 28 DAT1		**Orange leaves 159 DALA**		**Orange peel**		**Orange juice**	
TRR [mg eq/kg]	36		45		1.0		0.25	
	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]
Total extractable	73	26	84	37	53	0.56	94	0.24
Organic soluble	*48*	*17*	38	17	24	0.25	nd	<0.01
Methidathion	48	17	38	17	24	0.25	nd	nd
Aqueous soluble	26	9.2	46	21	29	0.31	94	0.24
Cluster A								
RH-alanine conjugate	2.7	1.0	2.2	1.0	14	0.14	72	0.18
Cluster B								
RH-acetic acid conjugate	-	-	-		4.2	0.04	-	-
RH-lactic acid conjugate	6.9	2.5	16	7.1	3.7	0.04	10	0.03
RH-hydroxy acetic acid conjugate	-	-	-	-	3.5	0.04	-	-
Cluster C								
Desmethyl-Methidathion	16	5.6	21	9.7	1.7	0.02	3.1	<0.01
Aqueous total identified	25	9.1	39	18	27	0.28	86	0.22

The Meeting also received a supplementary study (Simoneaux, 1993, ABR-93021).

Mature orange leaves (159 DALA) from study ML-91-704 were extracted with methanol-water (9:1, v/v), concentrated by rotary evaporation to remove methanol, and then partitioned with chloroform. The aqueous fraction was separated into three clusters (cluster A, B and C) by anion exchange column chromatography. These fractions were further cleaned up with chromatography using non-polar adsorbent resin column and analysed by TLC. The radioactivity was analysed by LSC.

Clusters A, B and C of mature leaves prepared by this study, and of orange peel and orange juice prepared by study ML-01-704 were derivatized for identification. Cluster A was incubated with beta-glucosidase, cellulase or N-glycosidase at 37 °C. Clusters A, B and C were butylated by adding 3 mol/L HCl in n-butanol and heating at 115 °C for 15–30 minutes. These were evaporated to remove solvent, dissolved in ethyl acetate and partitioned with water. The organic fraction was separated by preparative TLC for purification.

The butyl esters were further esterized. Purified butyl esters of cluster B and C were dried up under N_2, dissolved in acetic anhydride and heated at 155 °C for 5 minutes for acetylation. Purified butyl esters of cluster A were dissolved in trifluoroacetic anhydride and heated at 155 °C for 5 minutes. Each reaction mixture was dissolved in water, adjusted pH to 7 and partitioned with ethyl acetate. The ethyl acetate fractions were dried over sodium sulphate, filtered and analysed by TLC, HPLC-MS/MS. The distribution of TRR in each cluster was shown in Table 9 (Radioactive residue of aqueous solution = 100 percent).

Table 9 Characterisation of radioactive residues in anion exchange clusters from analysis of aqueous soluble residues of selected mature orange plant parts

	Leaves 159 DALA		Orange peel		Orange juice	
Cluster	[% TRR of aqueous soluble]	[mg eq/kg]	[% TRR of aqueous soluble]	[mg eq/kg]	[% TRR of aqueous soluble]	[mg eq/kg]
A	3.8	1.4	41	0.42	42	0.11
B	19	6.6	18	0.19	26	0.06
C	37	13	11	0.12	20	0.05
Total	59		70		88	

Clusters A, B and C accounted for almost all of the aqueous soluble fractions in orange leaves, peel and juice. For cluster A from juice, attempt to further clean-up with non-polar adsorbent resin column was unsuccessful because of the presence of large amount of sugar. Derivatisation of the sugar co-extractants and/or the proposed alanine conjugate in juice was impossible as no purified sample for TLC was obtained. Therefore, for further identification and characterization, clusters A, B and C isolated from leaves (159 DALA) were used. In clusters A, B and C, TRRs were 2.4, 10 and 23 percent, respectively (Table 10).

Table 10 TRR Characterisation in orange leaves (159 DALA)

	Leaves 159 DALA (% TRR)		
	Organic soluble	Aqueous soluble	Bound residues
	26	38	23
Cluster A	-	1.4	-
Cluster B	-	7.2	-
Cluster C	-	14	-

When clusters A, B and C were subject to non-polar adsorbent resin column clean-up followed by being re-chromatographed on anion ion-exchange column, most of the radioactive residue were retained on the resin and subsequently released by methanol (Table 11).

Table 11 Characterisation of radioactive residues in the water and methanol fraction of non-polar adsorbent resin column of aqueous extracts from orange leaves (159 DALA)

	[%] of total radioactivity in Clusters	
Cluster	aqueous fraction	Methanol fraction
A	27	73
B	6.5	92
C	33	74

In the Cluster A aqueous fraction, there was no evidence of release of the radioactive ring of methidathion that may be associated as an aglycone of a sugar conjugate. An attempt at derivatizing the water fraction from the non-polar adsorbent resin column with the butylating agent went only to approx. 23 percent completion. Butylated cluster A was partitioned with ethyl acetate and then analysed by TLC. Spot was at the origin for water fraction, while an Rf was given for ethyl acetate fraction.

The Cluster A methanol fraction showed a spot with an R_f value consistent with where the alanine conjugate would be expected. The ethyl acetate fraction after esterification shows a much higher R_f spot consistent with a derivatised alanine conjugate. The ethyl acetate fraction further separated with HPLC resulted in 2 compounds (namely A1 and A2). For A1, a structure could not be determined from the

spectra obtained because of the high background. For A2, the observed molecular ion was *m/z* 275 and the daughter spectra showed fragments at *m/z* 259, 220, 202, 185 and 174, consistent with the butyl ester of RH-alanine conjugate.

Cluster B was retained quantitatively on the resin and therefore only the methanol was available for chromatography. The chromatogram of Cluster B showed two main spots and one minor spot. The methanol fraction was butylated and separated by preparatory TLC. Two bands B1 and B2 were isolated, acetylated and cleaned up by preparative TLC.

Band B1 was purified by HPLC and split in two peaks B1A and B1B. These two fractions were collected and analysed by mass spectral analysis. Peak B1A showed only one component with a mass of *m/z* 318 and daughter fragments of *m/z* 277, 259, 245, 217, 203, 185, 175 and 143. These were consistent with lactic acid conjugate of methidathion with the attached butyl and acetyl groups from the derivatisation. Peak B1B showed two components from butylated products. The first component had a mass of *m/z* 276 with daughter fragments of *m/z* 221, 203, 175 and 133, consistent with the butyl ester of the lactic acid conjugate of RH. The second component had a mass of *m/z* 262 with daughter fragments of *m/z* 245, 207, 189, 161, 147 and 133, consistent with the butyl ester of the hydroxyl acetic acid conjugate of RH.

Band B2 was purified by preparative TLC and gave two components which were analysed by mass spectral analysis. The component B2A had a mass of *m/z* 318 with daughter fragments of *m/z* 277, 259, 245, 221, 203, 175, 157 and 146, consistent with the structure for B1A. The second component B2B had a mass of *m/z* 304 with daughter fragments of *m/z* 263, 231, 245, 203 and 161, consistent with the structure of the hydroxyl acetic acid conjugate of RH that is both butylated and acetylated.

For Cluster C, the methanol fraction after the resin chromatography was butylated and separated by preparatory TLC. The butylated product was acetylated and purified by TLC and HPLC resulting in two peaks C1 and C2. The larger peak C1 was purified using another HPLC method and separated in two peaks C1A and C1B, which were analysed by mass spectral analysis. No further work was done on C2.

Peak C1A showed one component that had a mass of *m/z* 318 with daughter fragments of *m/z* 277, 259, 245, 217, 203, 185, 175 and 101, consistent with the double derivative of the lactic acid conjugate of RH, which is the same compound as in peak B1A. Peak C1B gave two components by mass spectral analysis. The first component had a mass of *m/z* 276 with daughter fragments of *m/z* 221, 203, 175, 147 and 133, consistent with the butyl ester of the lactic acid conjugate of RH. The second component had a mass of *m/z* 262 with daughter fragments of *m/z* 245, 207, 189, 161, 147 and 133, consistent with the butyl ester of the hydroxyl acetic acid conjugate of RH.

TLC analysis showed the Cluster B and C methanol fractions contain the same components and Cluster C water and methanol fractions contained the same components with obvious spots off the diagonal consistent with the aforementioned degradation. These data indicate that when Cluster B and C components are identified in a more definitive manner that the same metabolites will be present in each cluster as a result of Cluster C degrading to Cluster B component during the isolation process.

Samples of methanol/water non-extractables from orange peel and pulp were incubated successively with pectinase, cellulase and protease enzymes prior to acid hydrolysis with 0.1N HCl and 1.0N HCl. Anion exchange chromatography of the combined pectinase and cellulase hydrolysate from orange peel and combined pectinase, cellulase and protease hydrolysates from orange pulp contained prominent A and B clusters released by the enzymes. Pectinase released primarily Cluster B components, whereas cellulase released primarily Cluster A components. No Cluster C components were released by enzyme treatment because of degradation of Cluster C to B components. As there were the small

amounts of radioactivity available in relation to the large amounts of co-extractants present, further analysis was impossible (Table 12, Table 13).

Table 12 Characterisation of radioactive residues in hydrolysates from extracted orange peel

Sample description	Normalised recovery [%]	Recovery [%]	[% TRR]	[mg eq/kg]
Orange peel residue	100	100	27	0.28
Pectinase hydrolysate	25	20	5.2	0.055
Cellulase hydrolysate	41	32	8.6	0.089
Protease hydrolysate	8.5	6.6	1.8	0.018
0.1 mol/L HCl hydrolysate	6.7	5.2	1.4	0.015
1.0 mol/L HCl hydrolysate	2.7	2.1	0.6	0.006
Total hydrolysates	84	65	18	0.18
Bound (remaining solids)	6.6	5.2	1.4	0.014
Total recovery	90	71	19	0.20
Other filtered solids	9.7	7.6	2.0	0.021
Total peel recovery	100	78	21	0.22

Table 13 Characterisation of radioactive residues in hydrolysates from extracted orange pulp

Sample description	Normalised recovery [%]	Recovery [%]	[% TRR]	[mg eq/kg]
Orange pulp residue	100	100	57	0.23
Pectinase hydrolysate	20	27	15	0.062
Cellulase hydrolysate	37	49	28	0.11
Protease hydrolysate	20	26	15	0.061
0.1 mol/L HCl hydrolysate	5.1	6.7	3.8	0.015
1.0 mol/L HCl hydrolysate	2.8	3.7	2.1	0.008
Total hydrolysates	86	113	64	0.26
Bound (remaining solids)	5.6	7.4	4.3	0.017
Total recovery	91	120	69	0.28
Other filtered solids	8.9	12	6.7	0.027
Total pulp recovery	100	132	76	0.30

In conclusion, the organic soluble residues of orange plant parts were mostly methidathion. Parent compound was not detected in orange juice. Predominant metabolites were: RH-alanine conjugate; RH-lactic acid conjugate and RH-hydroxy acetic acid conjugate; and RH-keto acid conjugate and RH-glyoxylic acid conjugate, respectively. Enzyme treatment of unextracted residues released the same components.

The proposed metabolic pathway of methidathion was shown in Figure 2.

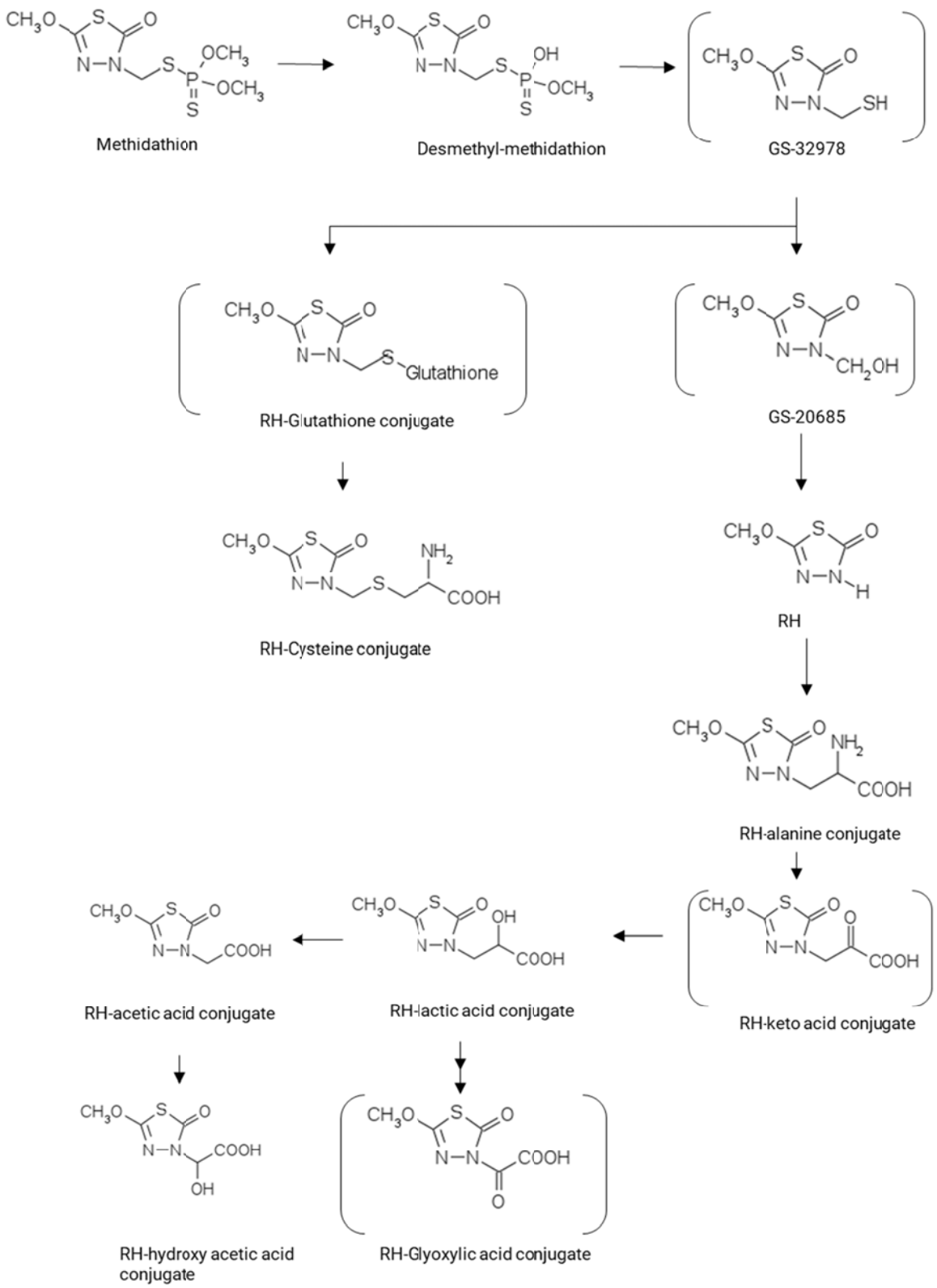


Figure 2 Proposed metabolic pathway of methidathion in orange

Tomatoes (Chopade et al, 1981)

The metabolism of methidathion was studied on tomato fruits (Manapal). To the surface of detached semi-ripe tomato fruits (100–150 g), 2-carbonyl-^{14}C-methidathion (1, 7 and 14 mg/kg) and the following labelled metabolites (7 mg/kg) were applied: ^{14}C-desmethyl methidathion, ^{14}C-RH sulfide, ^{14}C-RH sulfoxide, and ^{14}C-RH sulfone (Figure 1). Treated and control tomatoes were stored at room temperature (22–24 °C) and sampled at 0, 3, 7 and 14 days after treatment (DAT) with 2-carbonyl-^{14}C-methidathion and at 0 and 7 days after the application of labelled metabolites.

Sampled fruits were homogenized, extracted twice with acetone-water (9:1, v/v) and centrifuged. The supernatants were combined, and acetone was removed under vacuum. The extracts were partitioned with chloroform. The chloroform extract was concentrated under N_2 and the aqueous phase extracts were lyophilized. Both were stored at -15 °C until further analysis by TLC.

The chloroform extract was analysed with TLC. The lyophilized aqueous phase was dissolved in water and separated by chromatography using non-polar adsorbent resin column. The fractions that eluted with the potassium bromide gradient that represented an individual peak were pooled and lyophilized. The radioactive compounds were extracted from the residue into methanol and concentrated under N_2. The concentrated methanol extracts were analysed by TLC.

Treatment with 2-carbonyl-^{14}C-methidathion

In the analysis of tomatoes treated with 2-carbonyl-^{14}C-methidathion at 14 DAT, most of radioactivity was recovered (91–97 percent AR), with 82–92 percent TRR in aqueous phase and 3.8–14 percent TRR in chloroform phase.

TLC analyses of the chloroform extracts revealed four compounds: methidathion (2.5–9.8 percent TRR, 0.02–1.1 mg eq/kg) the oxygen analogue of methidathion 0.51–3.2 percent TRR (0.004–0.37 mg eq/kg), RH-sulfoxide and GS-20685 comprised ≤1.3 percent TRR (≤0.08 mg eq/kg).

There were 4 peaks separated by the chromatography from water soluble extract. The largest peaks corresponded to RH-cysteine conjugate (33–47 percent TRR, 0.37–3.8 mg eq/kg), followed by desmethyl-methidathion (24–41 percent TRR, 0.19–4.8 mg eq/kg). The other two (1.8–6.3 percent TRR, 0.37–3.8 mg eq/kg; and 5.4–6.6 percent TRR, 0.19–4.8 mg eq/kg) were not further identified. The summary is shown in Table 14.

According to the time-course experiment using 2-carbonyl-^{14}C-methidathion, the data indicated extensive degradation of methidathion over a 14-day period. At day 14, more than 80 percent TRR was found in the aqueous phase. Non-extracted residues were in the order of 3-4 percent TRR over the entire time course studied, indicating little if any breakdown to [^{14}C]-carbon dioxide and incorporation into the carbon pool. Some loss of radioactivity from the treated tomato fruit occurred during the storage in the chamber, which increased with time and decreased with increasing dose level, varying between 3-9 percent of the initial applied dose of 2-carbonyl-^{14}C-methidathion over a 14-day period. The amount of desmethyl methidathion relative to the RH-cysteine conjugate increased with the increased dose.

Table 14 Characterisation of residues in tomatoes 14 DAT with 1, 7 and 14 mg/kg ^{14}C-methidathion

Fraction or metabolite	Residue [mg eq/kg] ± s.d. and (percent TRR)		
	Methidathion applied at 1 mg/kg fruit	Methidathion applied at 7 mg/kg fruit	Methidathion applied at 14 mg/kg fruit
Methidathion applied	0.87±0.09	6.73±0.34	12.07±0.70
Recovery[1]	0.79 (91 percent AR)	6.13 (91 percent AR)	11.69 (97 percent AR)
Chloroform phase	*0.03±0.002 (3.8)*	*0.80±0.080 (13)*	*1.68±0.73 (14)*
Methidathion	0.02±0.001 (2.5)	0.56±0.002 (9.1)	1.14±0.007 (9.8)

Fraction or metabolite	Residue [mg eq/kg] ± s.d. and (percent TRR)		
	Methidathion applied at 1 mg/kg fruit	Methidathion applied at 7 mg/kg fruit	Methidathion applied at 14 mg/kg fruit
Oxygen analogue	0.004±0.001 (0.51)	0.12±0.006 (2.0)	0.37±0.004 (3.2)
RH-Sulfoxide	0.003±0.001 (0.38)	0.08±0.001 (1.3)	0.08±0.02 (0.68)
GS-20685	0.001±0.001 (0.13)	0.02±0.001 (0.33)	0.05±0.004 (0.43)
Unknown	0.003±0.001 (0.38)	0.02±0.006 (0.33)	0.04±0.03 (0.34)
Aqueous phase	***0.73 (92)***	***5.14 (84)***	***9.64 (82)***
RH-Cysteine conjugate	0.37 (47)	2.08 (34)	3.83 (33)
Unknown 2	0.05 (6.3)	0.16 (2.6)	0.21 (1.8)
Unknown 3	0.05 (6.3)	0.32 (5.2)	0.65 (5.6)
Desmethyl-Methidathion	0.19 (24)	2.32 (38)	4.78 (41)
Non-extractable	**0.03±0.005 (3.8)**	**0.19±0.03 (3.1)**	**0.37±0.07 (3.2)**

Note:

[1] recovery has been calculated as sum of extractions (chloroform and aqueous phase) and non-extractables.

s.d: standard deviation (n=3).

Treatment with ^{14}C-desmethyl methidathion

After treatment of tomatoes with ^{14}C-desmethyl methidathion, most of the radioactivity (93–99 percent AR) was recovered, with the distribution of 90–96 percent TRR in aqueous phase and ≤1.6 percent TRR in chloroform phase. The amount of non-extractables increased from 4.2 percent to 8.4 percent TRR (0.29 and 0.52 mg eq/kg, respectively) from 0 to 7 DAT, which indicated some breakdown of the compounds and incorporation in the carbon pool.

In the chloroform extract of samples 7 DAT, two compounds, RH-sulfide (1.1 percent TRR, 0.07 mg eq/kg) and unchanged desmethyl methidathion (0.32 percent AR, 0.02 mg eq/kg), were identified.

In the aqueous phase of samples 7 DAT, the largest amount of unchanged desmethyl methidathion was found (52 percent TRR, 3.2 mg eq/kg), followed by RH-cysteine conjugate (21 percent TRR, 1.3 mg eq/kg) and two unknown compounds (5.3–7.1 percent TRR, 0.33–0.44 mg eq/kg). A similar pattern was observed for 0 DAT (Table 15).

Table 15 Characterisation of residues in tomatoes 0 and 7 DAT with 7 mg/kg ^{14}C-desmethyl methidathion

Fraction or metabolite	Residue [mg eq/kg] ± s.d. and (percent TRR)	
	Day 0	Day 7
Desmethyl-Methidathion applied	7.00±0.08	6.64±0.42
Recovery[1]	6.9 (99 percent AR)	6.19 (93 percent AR)
Chloroform phase	*0.01±0.005 (0.14)*	*0.10±0.02 (1.6)*
Desmethyl-Methidathion	0.008 (0.12)	0.02±0.000 (0.32)
RH-Sulfide	-	0.07±0.000 (1.1)
Aqueous phase	***6.60±0.07 (96)***	***5.57±0.44 (90)***
RH-Cysteine conjugate	0.12 (1.7)	1.30±0.40 (21)
Unknown 2	0.11 (1.6)	0.33±0.09 (5.3)
Unknown 3	0.07 (1.0)	0.44±0.04 (7.1)
Desmethyl-Methidathion	5.94 (86)	3.21±0.57 (52)
Non-extractable	**0.29±0.02 (4.2)**	**0.52±0.07 (8.4)**

Note:

[1] recovery has been calculated as sum of extractions (chloroform and aqueous phase) and non-extractables.

s.d: standard deviation (n=3).

Treatment with ^{14}C-RH-sulfide

Radioactivity in samples 7 DAT with ^{14}C-RH-sulfide was recovered at 96 percent AR and observed in chloroform phase (82 percent TRR, 4.6 mg eq/kg), aqueous phase (18 percent TRR, 0.99 mg eq/kg) and non-extractable (0.36 percent TRR, 0.02 mg eq/kg). In the chloroform extract, two compounds were identified: RH-sulfoxide (60 percent TRR, 3.4 mg eq/kg) and unchanged sulfide (20 percent TRR, 1.1 mg eq/kg). In the aqueous phase, three peaks were separated with the largest being RH-cysteine conjugate (14 percent TRR, 0.78 mg eq/kg) and other two (1.1–1.4 percent TRR, 0.06–0.08 mg eq/kg) being unknown (Table 16).

Table 16 Characterisation of residues in tomatoes 0 and 7 DAT with 7 mg/kg ^{14}C-RH-sulfide

Fraction or metabolite	Residue [mg eq/kg] ± s.d. and (% TRR)	
	Day 0	Day 7
RH-Sulfide applied	7.09±0.09	5.80±0.52
Recovery[1]	6.99 (99 percent AR)	5.56 (96 percent AR)
Chloroform phase	*6.71±0.06 (96)*	*4.55±0.28 (82)*
RH-Sulfide	6.62±0.01 (95)	1.11±0.10 (20)
RH-Sulfoxide	-	3.35±0.15 (60)
Unknown	0.09±0.01 (1.3)	0.09±0.01 (1.6)
Aqueous phase	***0.05*±0.03 (0.72)**	***0.99*±0.13 (18)**
RH-Sulfide	0.046 (0.66)	-
RH-Cysteine conjugate	-	0.78 (14)
Unknown 2	-	0.08 (1.4)
Unknown 3	-	0.06 (1.1)
Non-extractable	**0.23±0.03 (3.3)**	**0.02±0.01 (0.36)**

Note:

[1] recovery has been calculated as sum of extractions (chloroform and aqueous phase) and non-extractables.

s.d: standard deviation (n=3).

Treatment with ^{14}C-RH-sulfoxide

The recovery of radioactivity of samples 7 DAT with ^{14}C-RH-sulfoxide was 95 percent of applied radioactivity (AR), with the distribution of 86 percent TRR in chloroform phase (5.8 mg eq/kg), 10 percent TRR in aqueous phase (0.68 mg eq/kg) and 3.9 percent TRR in unextracted (0.26 mg eq/kg). The predominant compound in chloroform phase was unchanged RH-sulfoxide (70 percent TRR, 4.7 mg eq/kg), followed by RH-sulfone (16 percent TRR, 1.1 mg eq/kg). In aqueous phase, RH-cysteine conjugate (7.7 TRR, 0.52 mg eq/kg) and three unknown compounds (0.74 percent AR, 0.05 mg eq/kg) were separated (Table 17).

Table 17 Characterisation of residues in tomatoes 0 and 7 DAT with 7 mg/kg ^{14}C-RH-sulfoxide

Fraction or metabolite	Residue [mg eq/kg] ± s.d. and (% TRR)	
	Day 0	Day 7
RH-Sulfoxide applied	7.29±0.05	7.10±0.16
Recovery[1]	7.18 (98 percent AR)	6.75 (95 percent AR)
Chloroform phase	*6.27±0.10 (87)*	*5.81±0.27 (86)*
RH-Sulfoxide	6.24±0.01 (87)	4.73±0.01 (70)
RH-Sulfone	-	1.06±0.01 (16)
Unknown	0.02±0.01 (0.28)	0.01±0.002 (0.15)
Aqueous phase	***0.69±0.09 (9.6)***	***0.68±0.14 (10)***
RH-Sulfoxide	0.66 (9.2)	-
RH-Cysteine conjugate	-	0.52 (7.7)
Unknown 2	-	0.05 (0.74)

Fraction or metabolite	Residue [mg eq/kg] ± s.d. and (% TRR)	
	Day 0	Day 7
Unknown 3	-	0.05 (0.74)
Unknown 4	-	0.05 (0.74)
Non-extractable	**0.22±0.03 (3.1)**	**0.26±0.06 (3.9)**

Notes:

[1] recovery has been calculated as sum of extractions (chloroform and aqueous phase) and non-extractables.

s.d: standard deviation (n=3).

Treatment with ^{14}C-RH-sulfone

The recovery of radioactivity of samples 7 DAT with ^{14}C-RH-sulfone was 96 percent AR, with the distribution of 91 percent TRR in chloroform phase (5.8 mg eq/kg), 6.4 percent TRR in aqueous phase (0.41 mg eq/kg) and 3.1 percent TRR in unextracted (0.20 mg eq/kg). In chloroform phase, unchanged RH-sulfone (89 percent TRR, 5.8 mg eq/kg) and an unknown compound (1.4 percent TRR, 0.09 mg eq/kg) were observed. In aqueous phase, RH-cysteine conjugate (2.8 percent TRR, 0.18 mg eq/kg) and three unknown compounds (0.16–2.3 percent TRR, 0.01–0.15 mg eq/kg) were separated.

Table 18 Characterisation of residues in tomatoes 0 and 7 DAT with 7 mg/kg ^{14}C-RH-sulfone

Fraction or metabolite	Residue [mg eq/kg] ± s.d. and (% TRR)	
	Day 0	Day 7
RH-Sulfone applied	7.05±0.27	6.71±0.16
Recovery[1]	6.81 (97 percent AR)	6.45 (96 percent AR)
Chloroform phase	*6.46±0.23 (95)*	*5.84±0.19 (91)*
RH-Sulfone	6.14±0.10 (90)	5.75±0.05 (89)
Unknown	0.32±0.10 (4.7)	0.09±0.05 (1.4)
Aqueous phase	***0.16±0.02 (2.3)***	***0.41±0.04 (6.4)***
RH-Sulfone	0.16 (2.3)	-
RH-Cystein conjugate	-	0.18 (2.8)
Unknown 2	-	0.01 (0.16)
Unknown 3	-	0.05 (0.78)
Unknown 4	-	0.15 (2.3)
Non-extractable	0.19±0.03 (2.8)	0.20±0.03 (3.1)

Notes:

[1] recovery has been calculated as sum of extractions (chloroform and aqueous phase) and non-extractables.

s.d: standard deviation (n=3)

The proposed metabolic pathway in tomato was shown in Figure 3.

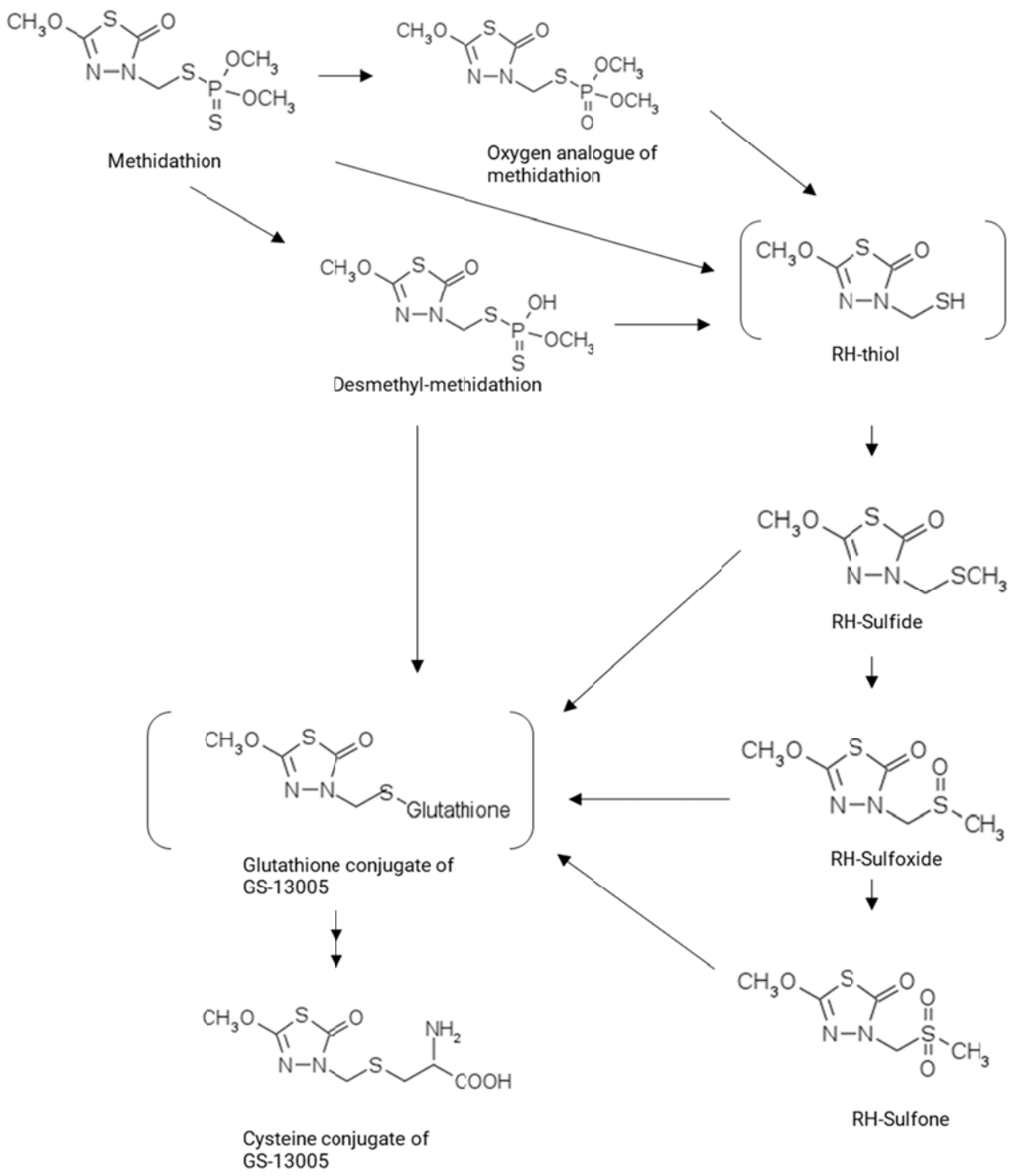


Figure 3 Proposed metabolic pathway of methidathion in tomato

Common beans and alfalfa (Dupuis et al, 1971)

Common bean plants (*Phaseolus vulgaris*) were cultivated in pots and then transferred into the metabolism chamber with conditions of temperature 22 °C, relative humidity 60 percent and 14 hour/day illumination when the second trifoliate leaf appeared. Radioactive materials, 2-carbonyl-^{14}C-methidathion, 3-methylene-^{14}C-methidathion, O-methyl-^{14}C-methidathion, ^{14}C-oxygen analogue, and 2-carbonyl-^{14}C-RH (Figure 1), were dissolved in 100 µL of acetone and topically applied to leaves (1 primary leaf or 8 young leaves, application rates shown in Table 20**)**. No phytotoxic symptom was observed after treatment. Normal air was drawn through the system at a rate of 2 L/min and CO_2 was absorbed in two wash bottles with NaOHaq (2 mol/L).

Alfalfa plants (*Medicago salvia*) were grown in the field and acclimated for 3 weeks in the experimental room. One plant was transferred into the above-mentioned metabolism chamber. Others were cultivated in the experimental room. The height of the plants at the time of treatment was 30-40 cm. A radioactive material, 2-carbonyl-^{14}C-methidathion, was dissolved in 100 µL of acetone and topically applied to leaves (40 leaves, application rates shown in Table 21). No phytotoxic symptom was observed after treatment.

Generally, the radioactivity detected in the untreated parts of the plant was small, and in most experiments present in the form of polar metabolites. Only in the case of stem of alfalfa and bean plants, trace amounts of methidathion were found after 1 and 2 weeks representing approximately 0.2-0.5 percent AR.

The rate of release of $^{14}CO_2$ after topical application of methidathion labelled with ^{14}C at 3 different positions was measured. With regard to common beans, the release was faster for 2-carbonyl-^{14}C-methidathion (22 percent AR was excreted as $^{14}CO_2$ at 8 DAT), followed by 3-methylene-^{14}C-methidathion (20 percent AR excreted at 8 DAT), and O-methyl-^{14}C-methidathion (5 percent AR excreted at 8 DAT). The rate of release of $^{14}CO_2$ in alfalfa was slower (7 percent AR of 2-carbonyl-^{14}C-methidathion excreted at 8 DAT) than that in beans.

Plants were homogenized and extracted with acetone:water (8:2, v/v). After evaporation of the acetone, the aqueous extract was partitioned with chloroform. The chloroform phase was further separated by TLC and methidathion, oxygen analogue and RH were identified by co-chromatography with reference materials.

Both in common bean and alfalfa, high radioactivity was observed in water phase. At least 4 metabolic fractions were found in the aqueous phase, but no satisfactory separation was achieved by TLC with neutral solvent systems. The use of systems containing formic acid or ammonium hydroxide allowed the separation of distinct zones which, however, were partially the result of decomposition of the original constituents. The pattern of products on the TLC was almost the same among the 3 types of labelled methidathion (Table 19). RH was originated not only compounds in Zone 1 (Table 19) but also those in Zone 2, indicating the presence of several metabolites resulting in the same compound after hydrolysis.

The final product of this degradation was a stable compound which also liberated from 1/3 of the water-soluble radioactivity by acid hydrolysis (7 mol/L HCl, 23 °C, 24 h) of the total aqueous phase and was identified as RH. In chloroform phase, parent methidathion was predominant (Tables 20 and 21).

In this study, dosage was described based on the number of leaves or plant, not based on the weight, and it was impossible to calculate the concentration of residues.

Table 19 Radioactivity in the aqueous phase of common bean leaves 7 DAT with 3 types of labelled methidathion

	Rf of the radioactive fraction	
	0.05 0.35 0.5	0.8
	Zone 1	Zone 2
Type of label	[%] of radioactivity in aqueous extract	
3-methylene-^{14}C-methidathion	81.5	18.5
2-carbonyl-^{14}C-methidathion	85.3	14.7
O-methyl-^{14}C-methidathion	83.4	16.6

Table 20 Characterisation and identification of radioactivity in common bean plants after topical application of ^{14}C-methidathion

Compound Applied	3.9Dosage	DAT	Chloroform phase				Water phase %AR	Non-extractable %AR	$^{14}CO_2$ %AR
			Methidathion % AR	Oxygen analogue % AR	RH % AR	Polar fraction % AR			
2-carbonyl-^{14}C-methidathion	83.9 ug/plant[a]	7	6.03	0.22	0.66	1.95	20.2	1.55	20.4
2-carbonyl-^{14}C-methidathion	70.2 ug/plant	14	6.9				14.9	12.4	27.4
O-methyl-^{14}C-methidathion	122 ug/plant	16	1.34	0.23	0.55	1.81	47.0	10.65	8.0
3-methylene-^{14}C-methidathion	642 ug/plant	13	3.33	0.15	traces	0.15	25.7	10.9	23.3
2-carbonyl-^{14}C-RH	476 ug/plant	15	-	-	5.4	1.8	55.3	6.7	2.3

Table 21 Characterisation and identification of radioactivity in alfalfa plants after topical application of ^{14}C-methidathion

Compound applied	Dosage	DAT	Chloroform phase				Water phase %AR Methidathion % AR	Non-extractable %AR Oxygen analogue % AR	$^{14}CO_2$ %AR RH % AR
			Methidathion % AR	Oxygen analogue % AR	RH % AR	Polar fraction % AR			
2-carbonyl-^{14}C-methidathion	15.9 ug/leaf[b]	14[a]	39.35	0.86	1.00	1.80	32.0	6.4	13.7
2-carbonyl-^{14}C-methidathion	16.9 ug/leaf[b]	7[b]	53.2	0.33	1.74	0.33	14.5	2.9	-
2-carbonyl-^{14}C-methidathion	16.9 ug/leaf[b]	13[b]	31.9	0.57	0.43	0.39	17.25	4.9	-

Notes:

[a] Plants cultivated in metabolism chamber.

[b] Plants cultivated under open-air conditions.

The proposed metabolic pathway in beans and alfalfa is shown in Figure 4.

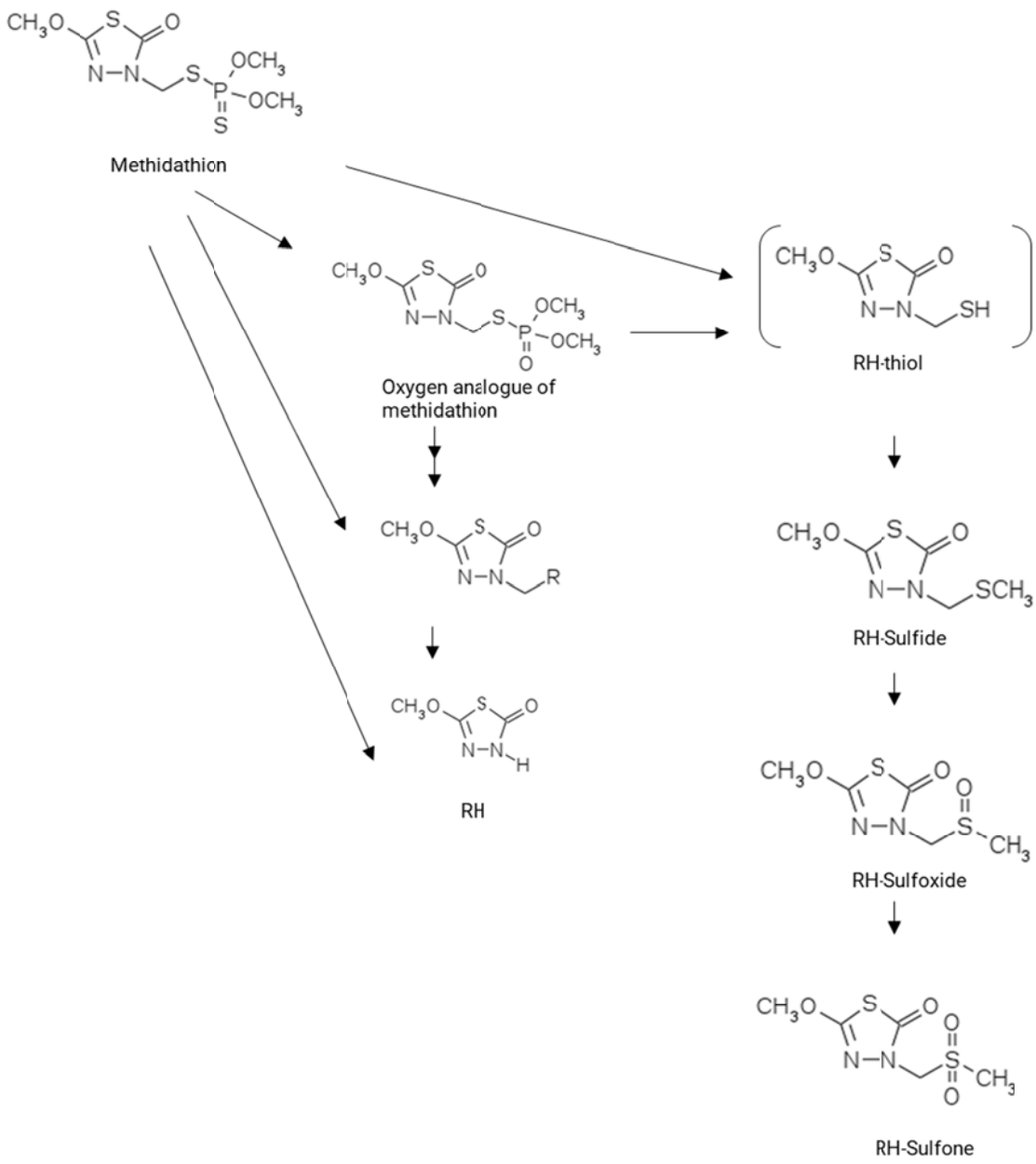


Figure 4 Proposed metabolic pathway of methidathion in bean and alfalfa

Conclusion

Metabolic pathways in orange, tomato, bean and alfalfa were qualitatively similar.

Animal metabolism

Information was available on metabolism of methidathion in lactating cows and lactating goats.

Lactating cows (Cassidy et al., 1969)

The metabolism of methidathion in a lactating cow (Holstein, 500 kg, age unknown) was studied using 2-carbonyl-^{14}C-methidathion administered orally in gelatine capsules (containing 165 mg of 2-carbonyl-^{14}C-methidathion), three times daily for 5 days. The daily administration rate was 1 mg/kg bw/day and

equivalent to 32 ppm (feed intake: 15.5 kg/day). A cow (Holstein, 455 kg, age unknown) was used as a control. Blood samples were taken 1 hour after the animal received the second dose capsule in the day. Milk was collected twice daily using milking machines. Excreta (urine, faeces) were collected daily.

At the end of the 10 days post dosing period, the animals were sacrificed. The following tissues subsampled and frozen: brain, heart, kidney, liver, round muscle, tenderloin muscle, spleen, blood and fat (omental, tail head, perirenal, subcutaneous).

Urine samples were radioassayed without further treatment. Faeces samples were extracted with acetone-methanol (1:1, v/v), concentrated and radioassayed. Insoluble faecal residues and blood samples were combusted and then analysed.

Milk was radioassayed using a wetting reagent and anthracene. Milk samples extracted by acetone were further fractionated in benzene soluble fraction and water-soluble fraction. The oxygen analogue of methidathion was analysed using silica gel TLC developed with chloroform-acetone (9:1, v/v) to be identified by inhibition of fly head cholinesterase.

Total radioactivity in tissues except fat were analysed by combustion using six replicates. Mean (±SD) recoveries for spiked samples at 0.02 and 0.1 mg/kg of seven tissue types were 93±6 percent and 93±2 percent, respectively.

Total radioactivity in fat was determined by pre-treating a homogenized subsample with NCS toluene quaternary ammonium base. The samples were heated in closed vial on the steam bath for several hours, cooled, diluted with toluene and analysed by LSC.

Milk

The total amount of radioactivity throughout the study period in milk was found at 0.6 percent AR (0.5 mg eq/kg), with 70 percent TRR (0.35 mg eq/kg) in aqueous fraction and 20 percent TRR (0.1 mg eq/kg) in the non-extractables.

The radioactivity observed in milk was increased by day 6 (1 DALA, 0.5 mg eq/kg) and no plateau was observed. The radioactivity was decreased to 0.004 mg eq/kg at 10 DALA, with 50 percent TRR in aqueous fraction and the rest in the non-extractables. Oxygen analogue of methidathion was not detected (LOD=0.01 mg eq/kg) in any of the milk samples. No further identification or characterization was conducted.

Tissues

Following daily oral administration of ^{14}C-methidathion to a cow at a dose of 1 mg/kg bw/d for 5 consecutive days and 10 days post-treatment period, residues in tissues were 0.11 mg eq/kg in liver, 0.04 mg eq/kg in kidney and 0.02-0.03 mg eq/kg in other tissues (Table 22). Metabolites were neither characterized nor identified.

Table 22 TRR in tissues from a cow fed ^{14}C-methidathion for 5 consecutive days

Tissue	Total radioactive residues [mg eq/kg]
Liver	0.11
Kidney	0.04
Heart	0.02
Muscle	
round	0.02
tenderloin	0.02
Spleen	0.03

Tissue	Total radioactive residues [mg eq/kg]
Brain	0.02
Fat	
perirenal	0.03
subcutaneous	0.02
omental	0.03
tail head	0.02

Urine, faeces

In the urine, 24 percent AR was recovered. The largest amount was observed in the specimen collected at 3 days after the first treatment. After the last capsule was administered (day 5), the radioactivity decreased. Predominant compound (about 85 percent) in urine was very polar and had different Rf value on paper chromatography with acetonitrile-water-ammonia (40:9:1, v/v/v) from that of methidathion, oxygen analogue of methidathion or RH. No further identification was conducted.

In the faeces, 34 percent AR was recovered, with 18 percent in acetone-methanol extracts and 16 percent in the residues. Radioactivity peaked at 4 days after the first treatment. After the last capsule was administered, the radioactivity decreased rapidly. The metabolite in faeces was polar and different from the reference materials of methidathion, oxygen analogue of methidathion or RH on TLC. No further identification was conducted.

Lactating cows (Polan and Chandler, 1970)

The metabolism of methidathion in lactating cows was studied. Cow 290 (Jersey, 413 kg, 4 years old) was administered 2-carbonyl-^{14}C-methidathion orally in capsules (containing 206 mg of 2-carbonyl- ^{14}C-methidathion) twice daily for 30 days. The daily administration rate was 1 mg/kg bw/day and equivalent to 65 ppm (feed intake: 6.4 kg/day). On the 31st day, the cow received 700 mg of 2-carbonyl-^{14}C-methidathion orally in a capsule (1.7 mg/kg bw, 110 ppm).

Cow 282 (Jersey, 469 kg, 4 years old, producing 11.6 kg milk daily) orally received 768 mg (1.7 mg/kg bw, 120 ppm) of 2-carbonyl-^{14}C-methidathion (single dose). Blood samples were taken 0.5, 1, 2, 3, 4, 6, 8, 10, 16, 24, 36, 48, 72 and 96 hours after the animal received the dose. Milk and excreta (urine, faeces) were totally collected for 96 hours after administration and individual samples were taken at various intervals. Exhaled CO_2 was trapped for four-minute intervals in NaOH (concentration not mentioned) periodically through 24 hours.

Total radioactivity of blood, urine and faeces were aliquoted for direct counting in liquid scintillation systems. For CO2, since expired CO2 was not quantitatively collected during the four-minute collection period, an aliquot of the trapping alkali was counted and the radioactivity corrected for counting efficiency. Another aliquot was reacted completely with BaCl2, the resulting precipitate was dried and $BaCO_3$ determined gravimetrically.

In the case of single dose, almost all radioactivity applied was collected (99.1 percent AR). Radioactivity was mainly found in exhaled CO_2 (50.8 percent AR) and urine (43.2 percent AR). For multi-dose experiment, the majority was found in urine (37.9 percent AR) followed by CO_2 (16.0 percent AR) among the total recovery of 60.6 percent AR (Table 23). In both cases, radioactivity was at low level in milk (0.8–1.2 percent AR).

Table 23 Recovery of labelled carbon from excretory routes in lactating cows for 96 hours after administration of 2-carbonyl- ^{14}C-methidathion

	Radioactive residues (% AR)	
	Cow 290 (administered for 31 days)	Cow 282 (single dose)
Exhaled CO_2	16.0	50.8
Urine	37.9	43.2
Faeces	5.5	4.3
Milk	1.2	0.8 (0.53 mg eq/kg)
Total	60.6	99.1

In addition, Cow 446 (Holstein, 556 kg, 4 years old) was given orally 1.25 mg/kg bw daily of methidathion (non-labelled) for 16 days. On 17th day, the cow was dosed with 945 mg of 2-carbonyl-^{14}C-methidathion (1.7 mg/kg bw, equivalent to 61 ppm). Milk was sampled at the end of 12 and 24 hours after administration.

Milk was extracted with acetone and filtered, and the residue on the filter was washed with benzene. All extracts were combined and left to stand for a few minutes to allow separation.

The benzene phase was evaporated to an oily residue and dissolved in hexane, followed by extraction with acetonitrile. The acetonitrile phase was dried up and dissolved in benzene. It was chromatographed on silica gel TLC with toluene-acetone (45:15) as mobile phase. The aqueous phase was partitioned with dichloromethane. The dichloromethane phase was dehydrated with anhydrous sodium sulphate, evaporated and dissolved in benzene. Final determinations for methidathion, RH-sulfone and RH-sulfoxide were conducted on GC-FID and by TLC (mobile phase (1): toluene-acetone=3:1, (2): acetonitrile-water-NH3 aq=40:9:1).

Radioactivity in the milk for 0–12 and 12–24 hours was 1.2 and 0.7 percent AR, respectively. Methidathion was not detected in either milk samples. RH-sulfone and RH-sulfoxide accounted for 3.4 and 0.9 percent, respectively, of the total radioactivity in milk during the first 12 hr. Distribution of milk extract radioactivity is in Table 24.

Table 24 Methidathion, RH-sulfone and RH-sulfoxide in extracted milk by TLC (Counts per minutes per spot)

	Mobile phase (1)		Mobile phase (2)	
	0-12 hr after dose	12-24 hr after dose	0-12 hr after dose	12-24 hr after dose
RH-sulfoxide	450	0	480	0
RH-sulfone	2050	155	2175	145
Methidathion	0	0	0	0

Lactating goats (Dupuis, et al., 1971)

The metabolism of methidathion in a lactating goat (55 kg, 2-year-old) was studied using 2-carbonyl-^{14}C-methidathion once administered by stomach tube (48.2 mg of 2-carbonyl-^{14}C-methidathion dissolved in 50 mL water-ethanol (1:4, v/v)). The dose was 0.88 mg/kg bw and equivalent to 38 ppm (assuming the feed intake of lactating goat as 1.27 kg). Milk was extracted with methanol-acetone (1:1, v/v), removed acetone, and then partitioned with chloroform.

Radioactivity in the range of 1 percent AR was found in the milk 72 hours after a single dose. Approximately 95 percent of radioactivity in milk was found in aqueous phase. Methidathion or its oxygen

analogue was not found in milk. No further identification or characterization of metabolites was conducted.

Conclusion

In milk, methidathion and its oxygen analogue were not detected. RH-sulfone and RH-sulfoxide, metabolites of methidathion identified, were detected, but they were accounted only for <5 percent TRR, which means other metabolites should exist but not identified. Radioactivity disappeared from milk in 10 DALA. In tissues, radioactivity remained in 10 DALA. As no further information was available, it was not possible to estimate metabolic pathway in livestock.

ENVIRONMENTAL FATE

Degradation under sunlight in soil (Lourdes et al, 2005)

Four types of 10 g of soil samples, namely unamended soil (S), amended with biosolid (4 percent w/w, SB), tetradecyltrimethylammonium (TDTMA, SS) as surfactant and both biosolid and surfactant (SBS), were placed on petri dishes (9.7 cm i.d.), added 10 mL of water and air dried for 48 h to level the surface of solid (0.89±0.09 mm thickness). Three mL of methidathion solution (33 mg/L) in water (S and SB) or in a TDTMA solution (SS and SBS) was dripped uniformly, and then heated under pressure for 1 h/d for three consecutive days to suppress the microbial activity. All petri dishes were covered with a glass disk and sealed with silicon. The experiment was conducted outdoors in Granada between July and August (2000) and air temperatures ranged between 15–38 °C, and irradiation from 0 (night) – 9870 (day) Wm^{-2}. At different time intervals, two Petri dishes of each type of soil (0, 1, 3, 7, 10, 14 and 21 days) and their corresponding dark controls (0, 3, 10, 14 and 21 days) were analysed.

For analysis, soil samples were extracted using Soxhlet with 75 mL of acetone for 6 h. The extract was concentrated using rotary evaporator, added 2 mL of hexane-toluene (1:1, v/v) and extracted. Samples were analysed by GC-FPD for methidathion and GC-MS (column: DB5-MS, 0.25 mm id × 30 m, EI mode) for photoproducts.

Degradation of methidathion was observed in all samples, and it was considered first order kinetic degradation. In all soil types, the degradation of methidathion was faster under sunlight (Table 25). In this study, neither methidathion oxygen analogue nor RH was detected after 3 days of sunlight irradiation of SS.

Table 25 First-order kinetic parameters for methidathion degradation under sunlight and in the dark in unamended soil (S) and soil amended with biosolid (SB), TDTMA (SS) and both amendments (SBS)

Soil	Sunlight			Dark controls		
	$k \times 10$ (d^{-1})	$t_{1/2}$ (days)	R^2	$k \times 10$ (d^{-1})	$t_{1/2}$ (days)	R^2
S	3.9±0.3	1.7	0.8869	1.7±0.1	4.1	0.9244
SS	4.3±0.3	1.6	0.8611	1.2±0.1	5.8	0.9401
SB	3.8±0.3	1.8	0.8960	2.7±0.3	2.6	0.8531
SBS	3.9±0.3	1.8	0.8898	2.3±0.1	3.0	0.9532

RESIDUE ANALYSIS

Analytical methods

The Meeting received seven methods of analysis for supervised residue trials.

Mandarin-1972 (Gotoh (1603[1]), 1979a; Gotoh (1604), 1979b; Suzuki (1607), 1971; and Kato (1608), 1972)

Pulp or peel of mandarin (50 g) was homogenized, extracted by 250 mL of ethyl acetate and cleaned up with florisil. Analysis was conducted by GC-ECD with a 5 percent QF-1 column (3 mm i.d. × 1.5 m). LOD reported was 0.05 mg/kg. Information on linearity of calibration curve was not available. Recovery for methidathion in mandarin was 73–94 percent (pulp) and 79–87 percent (peel), noting that only one sample was analysed for lower concentrations (0.02 and 0.04 mg/kg). Since LOQ was not reported, the Meeting determined LOQ as 0.1 mg/kg, the lowest fortification level with more than three trials (Table 26).

Table 26 Recovery data for Method Mandarin-1972

Matrix	Analyte	Fortification mg/kg	n	Recovery range mg/kg (mean)	RSD percent
Mandarin, flesh	Methidathion	0.02	1	73	-
		0.04	1	79	-
		0.1	4	63-80 (73)	9.9
		1	4	85-100 (94)	7.1
Mandarin, peel		0.02	1	83	-
		0.04	1	79	-
		1	4	75-106 (87)	16

Mandarin-1991 (Odanaka (1601), 2007; Higuchi and Matsuzawa (1602), 2008; Matano and Kobayashi (1605), 1991a; and Kuroda and Higuchi (1606), 1991)

Flesh or peel of mandarin (20 g) was homogenized, extracted with 100 mL of acetone and cleaned up with C18 cartridge column. Analysis was conducted with GC-FPD with DB-5 column (0.55 mm i.d. × 15 m). LOD was reported to be 0.005 mg/kg. Since LOQ was not reported, it was determined by the meeting as the lowest fortification level at 0.1 mg/kg that has acceptable recoveries. The calibration curve was linear (R^2>0.99) between 0.025 and 1 mg/kg. Recovery for methidathion in mandarin was 87–104 percent (pulp) and 92–98 percent (peel) (Table 27).

Table 27 Recovery data for Method Mandarin-1991

Matrix	Analyte	Fortification mg/kg	N	Recovery range mg/kg (mean)	RSD percent
Mandarin, flesh	Methidathion	0.1	2	103-105 (104)	-
		0.2	4	84-90 (87)	3.0
Mandarin, peel		0.2	2	93-94 (94)	-
		0.4	2	90-93 (92)	-
		20	2	97-98 (98)	-

Apple-1976 (Kato (1340), 1977 and Kaneuchi (1341), 1977)

Apples (50 g) were homogenized and extracted with 200 mL of ethyl acetate. It was cleaned up with silica gel column chromatography and dried under N_2. The solution was analysed by GC-flame thermionic detector (FTD) with GE XE-60 column (3 mm i.d. × 1.5 m). LOD was 0.01 mg/kg. Since LOQ was not reported, it was determined by the meeting as the lowest fortification level at 0.2 mg/kg that has acceptable recoveries. Information on linearity of calibration curve was not available. Recovery for methidathion in apples was 94 percent (Table 28). Sufficient information was not available for validation of the method.

[1] Report code number for reference

Table 28 Recovery data for Method Apple-1976

Matrix	Analyte	Fortification mg/kg	N	Recovery range mg/kg (mean)	RSD percent
Apple	Methidathion	0.2	3	91-97 (94)	3.2
		0.3	4	87-100 (94)	6.1

Apple-1988 (Kuroda and Higuchi (1395), 1992; Matano and Kobayashi (1396), 1991b; Kuroda and Higuchi (1397), 1990b; Matano and Kobayashi (1398), 1991c; Kuroda and Higuchi (1400), 1991; Kuroda and Higuchi (1401), 1990a; Kato (1402), 1988; Kuroda and Higuchi (1403), 1988; Kato (1501), 1974; Tsuchiya (1502), 1974; Gotou et al (1503), 1991; Kuroda and Higuchi (1504), 1990e; Tamai (1505), 1999; Kato (1506), 1979 and Tsuchiya (1507), 1979)

Apples or grapes (20 g) was homogenized, extracted with 100 mL of acetone and cleaned up by diatomite. Analysis was conducted with GC-flame photometric detector (FPD) with 5 percent OV-17 column (2.6 mm i.d. × 0.3 m). LOD reported was 0.002 mg/kg. Since LOQ was not reported, it was determined by the meeting as the lowest fortification level at 0.1 mg/kg that has acceptable recoveries.. Information on linearity of calibration curve was not available. Recovery for methidathion was 89–98 percent and 86–100 percent in apples and grapes, respectively (Table 29). Sufficient information was available for validation of the method for the analysis of methidathion in apple, but not in grape.

Table 29 Method recovery data for Method Apple-1988

Matrix	Analyte	Fortification mg/kg	N	Recovery range mg/kg (mean)	RSD percent
Apple	Methidathion	0.1	3	97-100 (98)	1.6
		0.12	6	86-93 (90)	3.2
Grape		0.04	2	87-92 (89)	-
		0.1	2	96-104 (100)	-
		0.2	4	72-98 (90)	13
		1.0	2	82-90 (86)	-

Peach-1981 (Goto (1431), 1981; Kato (1432), 1981; Goto (1455), 1990a; Goto (1456), 1990b; Kuroda and Higuchi (1457), 1990c; Kuroda and Higuchi (1458), 1990d; Ishitsuka (1459), 2014; Kato (1901), 1981; Imano and Shouji (1902), 1981 and Iijima et al (2013), 2013)

Peaches (50 g for pulp or 20 g for peel) or cherries (50 g) was homogenized and extracted with 150 mL of acetone. Analysis was conducted with GC-FID with 1.95 percent OV-2 + 1.5 percent OV-17 column (2 mm i.d. × 0.3 m). LOD was reported to be 0.002 mg/kg for peach pulp and cherries and 0.005 mg/kg for peach peel. Since LOQ was not reported, it was determined by the meeting as the lowest fortification level at 0.1 mg/kg that has acceptable recoveries. The calibration curve was linear (R^2>0.99) between 0.05 and 2 mg/kg. Recovery for methidathion was 88-100 percent, 93-114 percent and 82-94 percent in peach, pulp; peach, peel and cherry, respectively (Table 30).

Table 30 Method recovery data for Method Peach-1981

Matrix	Analyte	Fortification mg/kg	n	Recovery range mg/kg (mean)	RSD percent
Peach, pulp	Methidathion	0.1	4	90-109 (100)	8.6
		0.2	4	86-92 (88)	3.0
Peach, peel	Methidathion	0.1	2	112-118 (114)	-
		0.2	6	87-101 (93)	5.2

Matrix	Analyte	Fortification mg/kg	n	Recovery range mg/kg (mean)	RSD percent
Cherry	Methidathion	0.06	2	85-88 (87)	-
		0.1	4	80-84 (82)	2.0
		0.2	4	90-102 (94)	6.0
		1.0	4	85-90 (88)	2.4

Mango-2003 (Ebisu (2001), 2003 and Hashimoto (2002), 2005)

Mangos (20 g) was homogenized and extracted by 100 mL of acetone. The extract was cleaned up by diatomite followed by florisil. Analysis was conducted with GC-FID with DB-1 column (0.53 mm i.d. × 15 m). LOD and LOQ were 0.002 and 0.005 mg/kg, respectively. The recovery was 73 and 85 percent at 0.1 and 0.005 mg/kg, respectively (Table 31). The calibration curve was linear (R^2>0.99) between 0.04 and 2 mg/kg. Recovery for methidathion in mango was between 73–100 percent (Table 31).

Table 31 Recovery data for Method Mango-2003

Matrix	Analyte	Fortification mg/kg	n	Recovery range mg/kg (mean)	RSD percent
Mango	Methidathion	0.005	3	77-91 (85)	8.3
		0.025	3	90-109 (100)	9.5
		0.1	3	70-77 (73)	5.2
		2.5	3	77-79 (78)	1.1

LC-MS/MS Method (Nakatsuji (1903), 2014; Nakatsuji (1904), 2015; Sugimoto (2003), 2016; Shimamura and Fujita (2019c), 2019; Ogiyama (JP2017C303), 2018; Morita (JP2018C021), 2019a; and Morita (JP2018C023), 2019b)

Samples (20 g) was homogenized and extracted with 100 mL of acetone. After filtration with Celite, acetone was removed under N_2, added saturated sodium chloride aqueous solution and extracted with n-hexane/ethyl acetate (1:1, v/v). The extract was cleaned up with a graphite carbon/ethylenediamine-N-propyl silylation silica gel column. Analysis was conducted by LC-MS/MS with C_{18} column (m/z 303.0 > 145.1). The LOD and LOQ were 0.0025 and 0.005 mg/kg, respectively. The calibration curve was linear (R^2>0.999) between 0.025 and 0.05 mg/kg. Recovery for methidathion was 89–103 percent in peach (fruit), 89–94 percent in peach (pulp), 76–80 percent in cherry, 83–86 percent in grapes and 98–114 percent in mango (Table 30).

Table 32 Method recovery data for LC-MS/MS Method

Matrix	Analyte	Fortification mg/kg	n	Recovery range mg/kg (mean)	RSD percent
Peach (fruit)	Methidathion	0.005	5	100-105 (103)	2.1
		0.1	5	92-98 (94)	4.2
		0.5	5	81-96 (89)	6.8
Peach, pulp		0.005	5	91-97 (94)	2.9
		0.1	5	81-95 (89)	6.6
Cherry		0.005	5	78-83 (80)	2.3
		1	5	75-77 (76)	1.1
Grape		0.005	5	79-86 (83)	3.2
		0.1	5	80-87 (85)	3.4
		0.5	5	81-89 (86)	4.2
Mango		0.01	6	112-116 (114)	2.0
		0.5	6	93-107 (98)	5.0

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

The Meeting received storage stability studies of methidathion in commodities with high water content, apple, cherry, mango, and peach and high acid content and high-water content, grape and mandarin. In all studies, samples fortified was not analysed at day 0; however, mean procedural recovery was available.

Mandarin (Higuchi and Matsuzawa (1602), 2008; Matano and Kobayashi (1605), 1991a; and Kuroda and Higuchi (1606), 1991)

Mandarin (20 g) was homogenized and fortified with methidathion at 0.2 and 1.0 mg/kg for pulp and 0.2, 1.0 and 5.0 mg/kg for peel. Samples were stored at -20 °C for 21–207 days and analysed with method Mandarin-1972 or Mandarin-1991.

The summary of data is shown in Table 34.

Table 33 Storage stability data for methidathion in mandarin

Matrix	Spike level [mg/kg]	Storage period [days]	Recovery of Methidathion [%]	Mean recovery after storage [%]	Mean procedural recovery [%]	Study
Mandarin, flesh	0.2	136	75, 74	74	104 (@0.1 mg/kg)	Ref. 1605[1]
	0.2	137	77, 75	76		
	1.0	97	94, 93	93	87 (@0.2 mg/kg)	Ref. 1606[1]
	1.0	98	90, 90	90		
	1.0	207	91, 91	91	92 (@0.4 mg/kg)	Ref. 1602[2]
	1.0	21	98, 97	98	97 (@0.5 mg/kg)	
Mandarin, peel	0.2	136	70, 64	68	93	Ref. 1605[1]
	1.0	98	85, 81	83	91 (@0.4 mg/kg) 97 (@20 mg/kg)	Ref. 1606[1]
	1.0	99	91, 88	89		
	5.0	207	75, 74	75	97	Ref. 1602[2]
	5.0	21	71, 71	71	92	

Notes:

[1] Method Mandarin-1972.

[2] Method Mandarin-1991.

Apple (Kuroda and Higuchi (1395), 1992; Matano and Kobayashi (1396), 1991b; Kuroda and Higuchi (1397), 1990b; Matano and Kobayashi (1398), 1991c; Kuroda and Higuchi (1400), 1991; Kuroda and Higuchi (1401), 1990a; Kato (1402), 1988; and Kuroda and Higuchi (1403), 1988)

Apples (25 g), after removal of core, were homogenized and fortified with methidathion at 0.2 or 1.0 mg/kg. Samples were stored at -20 °C for 14–120 days and analysed with method Apple-1988.

In one study (Ref 1400), samples of apple, after removal of core, were not homogenized but chopped to about 1 cm^3 and fortified with methidathion at 1.0 mg/kg. Samples were stored at -20 °C for 106-208 days and analysed with method Apple-1988.

The summary of data is shown in Table 34.

Table 34 Storage stability data for methidathion in apples

Matrix	Spike level [mg/kg]	Storage period [days]	Recovery of Methidathion [%]	Mean recovery after storage [%]	Mean procedural recovery [%]	Study
Apple	1.0	21	92, 82	87	86 (@0.4 mg/kg)	Ref. 1402
	1.0	57	98, 91	94		
	0.2	1	94, 89	92	94	Ref. 1403
	0.2	29	87, 85	86		
	1.0	106	79, 74	76	98 (@0.1 mg/kg)	Ref. 1400[1]
	1.0	148	76, 70	73		
	1.0	208	74, 73	74		
	1.0	29	89, 88	89	90 (@0.12 mg/kg)	Ref. 1401
	1.0	59	89, 89	89		
	1.0	95	86, 85	85		
	0.2	14	98, 95	96	93	Ref. 1396
	0.2	24	92, 92	92		
	1.0	23	87, 83	85	94 (@0.2 mg/kg)	Ref. 1397
	1.0	35	84, 83	83		
	0.2	13	93, 85	89	87	Ref. 1398
	1.0	120	89, 88	88	89 (@0.2 mg/kg)	Ref. 1399
	1.0	34	87, 89	93	96 (@0.2 mg/kg)	Ref. 1395

Notes:

[1/] Samples were chopped (approximately 1 cm^3) and then fortified

Cherry (Nakatsuji (1903), 2014; Nakatsuji (1904), 2015; Iijima et al (2013), 2013; and Shimamura and Fujita (2019c), 2019c)

Cherries (20 g) were homogenized and fortified with methidathion at 0.1 or 0.25 mg/kg. Samples were stored at -20 °C for 20-73 days and analysed with method Peach-1981(Ref 1903 and 2013) or LC-MS/MS Method (Ref 1904 and 2019c) (Table 35).

Table 35 Storage stability data for methidathion in cherries

Matrix	Spike level [mg/kg]	Storage period [days]	Recovery of Methidathion [%]	Mean recovery after storage [%]	Mean procedural recovery [%]	Study
Cherry	0.25	37	94, 93	94	97	Ref. 2013[1]
	0.25	44	95, 94	94		
	0.25	57	76, 73	75	80 (@0.005 mg/kg) 76 (@1.0 mg/kg)	Ref. 1903[1]
	0.25	20	85, 85	85	95 (@0.005 mg/kg) 85 (@1.0 mg/kg)	Ref. 1904[1]
	0.1	53	91, 89	90	98 (@0.005 mg/kg) 95 (@0.2 mg/kg)	2019c[2]
	0.1	59	89, 89	89		
	0.1	60	92, 90	91		
	0.1	73	90, 87	89		

Notes:

[1/] analysed by Method Peach-1981.

[2/] Analysed by LC-MS/MS Method.

<u>Peach</u> (Goto (1455), 1990a; Goto (1456), 1990b; Kuroda and Higuchi (1457), 1990c; Kuroda and Higuchi (1458), 1990d; Ishitsuka (1459), 2014; Ogiyama (JP2017C303), 2018; and Morita (JP2018C021), 2019a)

Peach pulp (Refs 1455, 1457 and 1459; JP2017C303; and JP2018C021), peach peel (Refs 1456, 1458 and 1459) or peach fruit (JP2017C303 and JP2018C021) were used for storage stability study. Sample (20 g) was homogenized (20 g) and fortified with methidathion at 0.2, 0.25 or 0.1 mg/kg. Samples were stored at -20 °C for 19-274 days and analysed by method Peach-1981 (Refs 1455-1459) or LC-MS/MS Method (JP2017C303 and JP2018C021) (Table 36).

Table 36 Storage stability data for methidathion in peaches

Matrix	Spike level [mg/kg]	Storage period [days]	Recovery of Methidathion [%]	Mean recovery after storage [%]	Mean procedural recovery [%]	Study
Peach, pulp	0.2	33	93, 93	93	90	Ref. 1455[1]
	1.0	72	94, 92	93	87 (@0.2 mg/kg)	Ref. 1457[1]
	0.25	42	Na	92	89	Ref. 1459[1]
	0.25	274	Na	94		
	0.1	19	93, 91	92	93 (@0.05 mg/kg)	JP2017C303[2]
	0.1	60	91, 90	91	88 (@0.05 mg/kg)	
	0.1	78	91, 90	91	93 (@0.05 mg/kg)	
	0.1	57	75, 73	74	80 (@0.05 mg/kg)	JP2018C021[2]
Peach, peel	0.2	38	102, 97	99	96	Ref. 1456[1]
	1.0	72	92, 92	92	90 (@0.2 mg/kg)	Ref. 1458[1]
	0.25	42	na	95	91	Ref. 1459[1]
	0.25	274	na	92		
Peach, fruit	0.1	19	93, 91	92	93 (@0.05 mg/kg)	JP2017C303[2]
	0.1	60	92, 89	91	92 (@0.05 mg/kg)	
	0.1	78	90, 82	86	90 (@0.05 mg/kg)	
	0.1	57	81, 78	80	81 (@0.05 mg/kg)	JP2018C021[2]

Notes:

[1/] Analysed by Method Peach-1981.

[2/] Analysed by LC-MS/MS Method.

<u>Grape</u> (Gotou et al (1503), 1991; Kuroda and Higuchi (1504), 1990e; Tamai (1505), 1999; and Morita (JP2018C023), 2019b)

Grapes (20 g) were homogenized and fortified with methidathion at 0.1, 0.2 or 1.0 mg/kg. Samples were stored at -20 °C for 18-301 days and analysed with method Apple-1988 (Refs 1503, 1504 and 1505) or LC-MS/MS Method (JP2018C023) (Table 37).

Table 37 Storage stability data for methidathion in grapes

Matrix	Spike level [mg/kg]	Storage period [days]	Recovery of Methidathion [%]	Mean recovery after storage [%]	Mean procedural recovery [%]	Study
Grapes	0.2	18	98, 72	85	94	Ref. 1503[1]
	1.0	60	93, 89	90	95 (@0.2 mg/kg)	Ref. 1504[1]
	0.2	277	-	96	102	Ref. 1505[1]
	0.2	301	-	104	102	
	0.1	35	93, 92	93	82 (@0.05 mg/kg)	JP2018C023[2]
	0.1	62	88, 85	87	82 (@0.05 mg/kg)	
	0.1	85	88, 85	87	95 (@0.05 mg/kg)	
	0.1	90	87, 87	87	82 (@0.05 mg/kg)	
	0.1	111	92, 84	88	95 (@0.05 mg/kg)	

Notes:

[1/] Analysed by Method Apple-1988

[2/] Analysed by LC-MS/MS Method

<u>Mango</u> (Hashimoto (2002), 2005 and Sugimoto (2003), 2016)

Mango, after removal of seed, was homogenized (20 g) and fortified with methidathion at 0.25 or 0.5 mg/kg. Samples were stored at -20 °C for 34–123 days and analysed by method Mango-2003 (Ref 2002) or LC-MS/MS Method (Ref 2003) (Table 38).

Table 38 Storage stability data for methidathion in mangoes

Matrix	Spike level [mg/kg]	Storage period [days]	Recovery of Methidathion [%]	Mean recovery after storage [%]	Mean procedural recovery [%]	Study
Mango	0.25	34	105, 102	104	100 (@0.025 mg/kg) 78 (@2.5 mg/kg)	Ref. 2002[1]
	0.5	115	95, 88	92	103 (@0.1 mg/kg)	Ref. 2003[2]
	0.5	119	88, 83	86		
	0.5	123	85, 82	84		
	0.5	7[a]	96, 94	94		
	0.5	7[a]	103, 102	102		

Notes:

[1/] Analysed by Method Mango-2003.

[2/] Analysed by LC-MS/MS Method.

[a] Samples stored refrigerated at 4 °C.

USE PATTERN

The Meeting received the cGAP for mandarins, cherries, peaches, apples, grapes and mangoes (Table 39). The label provided covers a broader spectrum of uses. In all Japanese GAPs, interval period is not specified.

It is noted that in Japan, retreatment interval between applications is not specified for crops to which methidathion could be applied more than once on the label, but that an interval of 7 days is common in practice.

Table 39 Registered uses of methidathion on various crops considered by the Meeting

Crop	Country	Formulation	Application						Timing/PHI (days)
			Method	Rate (kg ai/ha)	Dilution rate	Spray conc. (kg ai/hL)	Water volume[3] (L/ha)	Max number of applications	
Mandarins	Japan	EC 400 g ai/L	foliar spray		x 1500	0.027	7000	4	14
		EC 300 g ai/L+ EC 400 g ai/L	Spray at tree trunk (2) + foliar spray(2)		x50 x 1500	0.6 0.027	1800 7000	2 + 2	Egg laying timing of longicorn 14
Apples	Japan	EC 300 g ai/L	Spray at tree trunk		x100	0.3	1800	2	30
	Japan	WP 360 g ai/L	Foliar spray		x1500	0.024	7000	2	30
Cherries	Japan	EC 400 g ai/L	Foliar spray		x1500	0.027	7000	3	7
Peaches	Japan	WP 360 g ai/L+ EC 300 g ai/L	Foliar spray Spray at tree trunk and main branch		x1500 x 200	0.024 0.15	7000 1800	2[1] 2[1]	21 60
Grapes	Japan	WP 360 g ai/L	Foliar spray		x1500	0.024	7000	2[2]	14
		WP 360 g ai/L	Soil irrigation		x500	0.072	100000	2[2]	90
		EC 300 g ai/L	Spray at tree trunk and main branch		x200	0.15	1800	1[2]	Before bud burst
Mangos	Japan	EC 400 g ai/L	Foliar spray		x1500	0.027	7000	2	45

Notes:

[1/] Number of applications can be two for each formulation, and can be up to four applications in total..

[2/] Use of methidathion on grape, regardless of formulation type, can be up to twice in a year. Within the limit, before bud burst, methidathion can be used only once.

[3/] Recommendation.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received supervised trials on mandarins, cherries, peaches, apples, grapes and mangoes. In many trials, samples from the same supervised trial were analysed in two different laboratories. As many of the analytical results from replicated analyses were significantly different (i.e. differences are larger than the expanded measurement uncertainty estimated by Horwitz equation), the larger value between two analyses was taken for conservative estimation of residues.

Table 40 Summary table for supervised field trials

Commodity	Table
Citrus fruits	
Mandarin	Table 41
Pome fruits	
Apple	Table 42
Stone fruits	
Cherry	Table 44
Peach	Table 45
Berries and other small fruits	
Grape	Table 46, Table 47
Assorted tropical and sub-tropical fruits – inedible peel	
Mango	Table 48

Mandarins (Gotoh (1603), 1979a; Gotoh (1604), 1979b; Suzuki (1607), 1971; and Kato (1608), 1972; Odanaka (1601), 2007; Higuchi and Matsuzawa (1602), 2008; Matano and Kobayashi (1605), 1991a; and Kuroda and Higuchi (1606), 1991)

The Meeting received 18 supervised trials conducted between 1971 and 2006 on mandarins in Japan. In eight trials, mandarins received four foliar applications of methidathion diluted with water at 0.040 kg ai/hL; in two trials, three foliar applications at 0.027 kg ai/hL; in six trials, two foliar applications at 0.027–0.040 kg ai/hL; and in two trials, two foliar applications at 0.027 kg ai/hL and two sprays at tree trunk at 0.012 kg ai per tree.

Mandarin was peeled and peel and pulp were separately analysed. The concentration in whole fruit was calculated from the analytical values for peel and pulp. Methidathion formulation was 0.40 kg/L EC (foliar application) or 0.30 kg/L EC (sprays at tree trunk). The first application was made at 35–131 days before harvest (BBCH 60–81). The longest storage interval was 136 days. The results are shown in Table 41.

Table 41 Residues in mandarins resulting from supervised trials in Japan (foliar spray of methidathion 400 EC or 300 EC formulations)

Mandarins Study reference Location, year (variety)	Application					DALA	Methidathion[2] (mg/kg)			Storage interval
	No	Interval (days)	Conc (kg ai/hL) [1]	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)	Whole fruit	Pulp	Peel	
GAP, Japan	4		0.027 (spray)	4 x 70		14				

Mandarins Study reference Location, year (variety)	Application					DALA	Methidathion[2] (mg/kg)			Storage interval
	No	Interval (days)	Conc (kg ai/hL)[1]	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)	Whole fruit	Pulp	Peel	
			Or 0.027 (spray) + 0.6 (trunk application)	 Or 2 x 70 + 2 x 18						
1607, 1608 Nagasaki, Japan[3] 1971 (Unshiu)	2	82	0.040	2 x 40	2 x 1.6	21 39	0.60 0.51	<0.05 <0.05	2.8 2.4	115
	4	56, 26, 21	0.040	2 x 40	4 x 1.6	18 28	2.0 1.8	<0.05 <0.05	9.2 8.4	115
1607, 1608 Wakayama, Japan[3] 1971 (Unshiu)	2	92	0.040	2 x 50	2 x 2.0	17 31	1.2 0.70	<0.05 <0.05	5.6 3.2	39
	4	54, 38, 17	0.040	4 x 50	4 x 2.0	14 29	3.7 2.6	<0.05 <0.05	17 12	39
1603, 1604 Miyazaki, Japan[3] 1978 (Unshiu)	2+2	12, 14, 126	0.040	50 0.3/tree 0.3/tree 50	2.0 0.012/tree 0.012/tree 2.0	14	1.0	0.003	4.8	49
1603, 1604 Hiroshima, Japan[3] 1978 (Unshiu)	2+2	10, 25, 136	0.040	50 0.3/tree 0.3/tree 50	2.0 0.012/tree 0.012/tree 2.0	14	1.2	0.004	5.6	33
1605, 1606 Shizuoka, Japan[4] 1990 (Unshiu)	2	7	0.027	2 x 50	2 x 1.3	21 28	1.1 0.98	<0.1 <0.1	4.1 3.3	107
	3	7, 7	0.027	3 x 50	3 x 1.3	21 28	1.0 1.1	<0.1 <0.1	3.7 3.8	107 136
	4	7, 7, 7	0.027	4 x 50	4 x 1.3	21 28	1.4 1.6	<0.1 <0.1	5.4 5.5	107
	2	7	0.040	2 x 50	2 x 2.0	21 28	1.2 1.0	<0.1 <0.1	4.3 3.4	107
	4	7, 7, 7	0.040	4 x 50	4 x 2.0	21 28	2.2 1.9	<0.1 <0.1	9.2 7.1	107
1605, 1606 Wakayama, Japan[4] 1990 (Unshiu)	2	7	0.027	2 x 50	2 x 1.3	21 28	0.99 0.89	<0.1 <0.1	4.4 4.5	111
	3	7, 7	0.027	3 x 50	3 x 1.3	21 28	1.6 1.2	<0.1 <0.1	8.0 5.2	111

Mandarins Study reference Location, year (variety)	Application					DALA	Methidathion[2] (mg/kg)			Storage interval
	No	Interval (days)	Conc (kg ai/hL)[1]	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)	Whole fruit	Pulp	Peel	
	4	7, 7, 7	0.027	4 x 50	4 x 1.33	21 28	2.4 2.2	<0.1 <0.1	11 7.9	111
	2	7	0.040	2 x 50	2 x 2.0	21 28	1.7 2.2	<0.1 <0.1	6.3 7.4	111
	4	7, 7, 7	0.040	4 x 50	4 x 2.0	21 28	3.2 3.4	<0.1 <0.1	13 13	111
1601, 1602 Kanagawa, Japan[4] 2006 (Unshiu)	4	7, 7, 7	0.040	4 x 40	4 x 1.6	14 21 28	5.9 3.4 3.7	<0.1 <0.1 <0.1	26 19 20	2
1601, 1602 Oita, Japan[4] 2006 (Unshiu)	4	7, 7, 7	0.040	4 x 40	4 x 1.6	14 21 28	2.8 3.8 3.2	<0.1 <0.1 <0.1	18 21 15	23 2 23

Notes:

[1/] Recommended water volume to spray is 7000 L/ha.

[2/] Each sample was analysed twice in each of two different laboratories. The higher mean analytical result was shown in the table.

[3/] Analyzed by Method Mandarin-1972.

[4/] Analyzed by Method Mandarin-1991 (LOQ: 0.1 mg/kg for pulp and 0.2 mg/kg for peel).

Apples (Kato (1340), 1977 and Kaneuchi (1341), 1977; Kuroda and Higuchi (1395), 1992; Matano and Kobayashi (1396), 1991b; Kuroda and Higuchi (1397), 1990b; Matano and Kobayashi (1398), 1991c; Kuroda and Higuchi (1400), 1991; Kuroda and Higuchi (1401), 1990a; Kato (1402), 1988; and Kuroda and Higuchi (1403), 1988)

The Meeting received 57 supervised trials conducted between 1976 and 1990 on apples in Japan. In two trials, EC formulation of methidathion diluted with water at 0.024 kg ai/hL applied to apples twice, followed by the injection of WP formulation of methidathion to the trunk once (0.72 kg ai/hL). In other trials, apples received 1–5 foliar application(s) of WP formulation as aqueous solution with the concentrations of 0.018–0.024 kg ai/hL. The longest storage interval was 189 days. The result is shown in Table 42.

Apples were analysed after removal of core and the analytical results were shown as the weight of analysed portion basis. The weight of core is estimated 9.8-14 percent of fruit, using the data of apples (Golden delicious, Granny Smith, Gold Spur, Spartan, Red Gold and Royal Delicious) in *Satish et al. (2017)* and assuming that the shapes of apples are flat sphere and the density of each part of apple is uniform (Table 43).

Table 42 Residues in apples without core resulting from supervised trials in Japan (foliar spray of methidathion 360 WP or 300 EC formulations)

Apples Study reference Location, year (variety)	Application					DALA	Methidathion[1] (mg/kg)	Storage interval
	No	Interval (days)	Conc (kg ai/hL)	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)		
GAP, Japan	2		0.024 (WP, spray) Or 0.3 (trunk application)	2 x 70 Or 2 x 18		30		
1340, 1341 Nagano, Japan[2] 1976 (Kogyoku)	3	15 13	0.024	3 x 70	3 x 1.7 (WP)	7 14 21	0.25 0.16 0.05	189
	5	16 12 15 13	0.024	5 x 70	5 x 1.7 (WP)	7 14 21	0.14 0.12 0.06	189
1340, 1341 Yamagata, Japan[2] 1976 (Fuji)	3	13 14	0.024	3 x 70	3 x 0.96	7 14 21	0.28 0.28 0.18	144
	5	14 15 13 16	0.024	5 x 70	5 x 0.96 (WP)	7 14 21	0.26 0.12 0.16	144
1402, 1403 Akita, Japan[3] 1988 (Fuji)	1		0.018	60	1 x 1.1 (WP)	21	0.15	24
	1		0.018	60	1 x 1.1 (WP)	28 42	<0.1 <0.1	24
	2	7	0.018	2 x 60	2 x 1.1 (WP)	28 42	<0.1 <0.1	24
	2	7	0.018	2 x 60	2 x 1.1 (WP)	56	<0.1	24
	1		0.024	60	1 x 1.4 (WP)	21	0.12	24
	1		0.024	60	1 x 1.4 (WP)	28 42	<0.1 <0.1	24
	2	7	0.024	2 x 60	2 x 1.4 (WP)	28 42	<0.1 <0.1	24
	2	7	0.024	2 x 60	2 x 1.4 (WP)	56	<0.1	24
1402, 1403 Nagano, Japan[3] 1988 (Orin)	1		0.018	60	1 x 1.1 (WP)	20	<0.1	38
	1		0.018	60	1 x 1.1 (WP)	28 42	<0.1 <0.15	38
	2	6	0.018	2 x 60	2 x 1.1 (WP)	28 42	<0.1 <0.1	38
	2	7	0.018	2 x 60	2 x 1.1	56	<0.1	59

Apples Study reference Location, year (variety)	Application					DALA	Methidathion[1] (mg/kg)	Storage interval
	No	Interval (days)	Conc (kg ai/hL)	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)		
					(WP)			
	1		0.024	60	1 x 1.4 (WP)	20	<0.1	59
	1		0.024	60	1 x 1.4 (WP)	28 42	<0.1 <0.1	59
	2	6	0.024	2 x 60	2 x 1.4 (WP)	28 42	<0.1 <0.1	59
	2	7	0.024	2 x 60	2 x 1.4 (WP)	56	<0.1	59
1400, 1401 Iwate, Japan[3] 1989 (Fuji)	1		0.024	60	1 x 1.4 (WP)	45 60	<0.1 <0.1	76
	1		0.024	60	1 x 1.4 (WP)	90	<0.1	76
	2	6	0.024	2 x 60	2 x 1.4 (WP)	45 60	<0.1 <0.1	76
	2	7	0.024	2 x 60	2 x 1.4 (WP)	90	<0.1	76
1400, 1401 Miyagi, Japan[3] 1989 (Fuji)	1		0.024	60	1 x 1.4 (WP)	34 49	<0.1	51
	1		0.024	60	1 x 1.4 (WP)	88	<0.1	51
	2	9	0.024	2 x 60	2 x 1.4 (WP)	34 49	<0.1 <0.1	51
	2	10	0.024	2 x 60	2 x 1.4 (WP)	88	<0.1	51
1400, 1401 Nagano, Japan[3] 1989 (Fuji)	1		0.024	60	1 x 1.4 (WP)	45 60	<0.1 <0.1	99
	1		0.024	60	1 x 1.4 (WP)	90	<0.1	99
	2	7	0.024	2 x 60	2 x 1.4 (WP)	45 60	<0.1 <0.1	99
	2	7	0.024	2 x 60	2 x 1.4 (WP)	90	<0.1	99
1396, 1397 Yamagata, Japan[3] 1990 (Tsugaru)	1		0.024	60	1 x 1.4 (WP)	21 27	<0.1 <0.1	47
	3	7 6	0.024 0.36 (trunk application)	2 x 60 0.02/tree	2 x 1.4 (EC) 1 x 0.72/tree (WP)	21 27	<0.1 <0.1	47
1396, 1397 Toyama, Japan[3] 1990 (Senshu)	1		0.024	60	1 x 1.4 (WP)	21 30	<0.1 <0.1	34

Apples Study reference Location, year (variety)	Application					DALA	Methidathion[1] (mg/kg)	Storage interval
	No	Interval (days)	Conc (kg ai/hL)	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)		
	3	7 10	0.024 0.36 (trunk application)	2 x 60 0.02/tree	2 x 1.4 (EC) 1 x 0.72/tree (WP)	21	<0.1	34
1398, 1399 Akita, Japan[3] 1990 (Fuji)	1		0.024	60	1 x 1.4 (WP)	21	<0.1	140
	1		0.024	60	1 x 1.4 (WP)	29 45	<0.1 <0.1	140
	2	7	0.024	2 x 60	2 x 1.4 (WP)	29 45	<0.1 <0.1	30
	2	7	0.024	2 x 60	2 x 1.4 (WP)	60	<0.1	30
1398, 1399 Iwate, Japan[3] 1990 (Fuji)	1		0.024	60	1 x 1.4 (WP)	21	0.15	135
	1		0.024	60	1 x 1.4 (WP)	30	0.10	135
	2	7	0.024	2 x60	2 x 1.4 (WP)	30	<0.1	135
	2	7	0.024	2 x 60	2 x 1.4 (WP)	45	<0.1	135
1398, 1399 Ibaraki, Japan[3] 1990 (Fuji)	1		0.024	60	1 x 1.4 (WP)	21	0.17	135
	1		0.024	60	1 x 1.4 (WP)	30	0.11	135
	2	7	0.024	2 x 60	2 x 1.4 (WP)	30	<0.1	135
	2	7	0.024	2 x 60	2 x 1.4 (WP)	45	<0.1	135
1398, 1399 Nagano, Japan[3] 1990 (Fuji)	1		0.024	60	1 x 1.4 (WP)	21	<0.1	135
	1		0.024	60	1 x 1.4 (WP)	29	<0.1	135
	2	7	0.024	2 x 60	2 x 1.4 (WP)	30	<0.1	135
	2	7	0.024	2 x 60	2 x 1.4 (WP)	45	<0.1	135
1395 Akita, Japan[3] 1991	1		0.024	70	1 x 1.7 (WP)	20	0.32	44

Apples Study reference Location, year (variety)	Application					DALA	Methidathion[1] (mg/kg)	Storage interval
	No	Interval (days)	Conc (kg ai/hL)	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)		
(Fuji)								
	1		0.024	70	1 x 1.7 (WP)	28	<0.1	44
	2	7	0.024	2 x 70	2 x 1.7 (WP)	45	<0.1	44
1395 Iwate, Japan[3] 1991 (Fuji)	1		0.024	50	1 x 1.2 (WP)	21	<0.1	60
	1		0.024	50	1 x 1.2 (WP)	30	<0.1	60

Notes:

[1/] Each sample was analysed twice in each of two different laboratories. The higher mean analytical result was shown in the table.

[2/] Analysed by Method Apple-1976 (validation data not available).

[3/] Analysed by Method Apple-1988 (LOQ: 0.1 mg/kg).

Table 43 Estimated weight ratio of seed to whole fruit for apple[1]

Cultivar	Horizontal diameter (cm)	Vertical diameter (cm)	Core length (cm)	Core diameter (cm)	Fruit volume (cm3)	Core volume (cm3)	Ratio (percent)
Golden Delicious	6.21	5.70	5.34	1.79	115	13.4	12
Granny Smith	7.21	6.89	6.75	2.19	188	25.4	14
Gold Spur	6.23	6.03	5.85	1.79	123	14.7	12
Spartan	6.20	5.60	5.23	1.78	113	12.8	11
Red Gold	6.31	5.52	5.20	1.77	115	12.8	11
Royal Delicious	6.76	6.04	5.73	2.10	145	14.1	9.8

Notes:

[1/] Assuming that (1) the shape of apples is flat sphere, (2) the shape of core is cylinder, and (3) the density is uniform.

Cherry (Kato (1901), 1981; Imano and Shouji (1902), 1981; Nakatsuji (1903), 2014; Iijima et al. (2013), 2013; and Shimamura and Fujita (2019c), 2019)

The Meeting received 20 supervised trials conducted between 1981 and 2018 on cherries in Japan. Cherries received EC formulation of methidathion (0.40 kg ai/L) diluted with water at 0.018-0.027 kg ai/hL three times. In trials in Refs 1901 and 1902, every sample was analysed in two different laboratories and the higher values were shown in the table. The longest storage interval was 54 days.

Cherries were analysed after removal of seeds and the results were shown as the weight of whole fruit basis, except in trials of 1901 and 1902 in which they were analysed portion basis. For these two trials, the analytical values were converted to whole fruit basis using the information that seed weight is 7.6 percent of whole fruit according to *Kalyoncu et al. (2009)*.

The result is shown in Table 44.

Table 44 Residues in cherries resulting from supervised trials in Japan (foliar spray of methidathion 400 EC formulation)

Cherries Study reference Location, year (variety)	Application					DALA	Methidathion[1] (mg/kg)	Storage interval
	No	Interval (days)	Conc (kg ai/hL)	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)		(days)
GAP, Japan	3		0.027	3 x 70		7		
1901, 1902 Yamagata (Shima, Sagae), Japan[2,4] 1981 (Napoleon)	3	7 7	0.024	3 x 50	3 x 1.2	7	<0.1	9
	3	7 7	0.024	3 x 50	3 x 1.2	14	<0.1	9
	3	7 7	0.024	3 x 50	3 x 1.2	21	<0.1	9
	3	7 7	0.018	3 x 50	3 x 0.90	7	<0.1	9
	3	7 7	0.018	3 x 50	3 x 0.90	14	<0.1	9
	3	7 7	0.018	3 x 50	3 x 0.90	21	<0.1	9
1901, 1902 Yamagata (Tassho, Sagae), Japan[2,4] 1981 (Napoleon)	3	7 7	0.024	3 x 40	3 x 0.96	7	<0.1	8
	3	7 7	0.024	3 x 40	3 x 0.96	14	<0.1	8
	3	7 7	0.024	3 x 40	3 x 0.96	21	<0.1	8
	3	7 7	0.018	3 x 40	3 x 0.72	7	<0.1	8
	3	7 7	0.018	3 x 40	3 x 0.72	14	<0.1	8
	3	7 7	0.018	3 x 40	3 x 0.72	21	<0.1	8
2013 Fukushima, Japan[2] 2013 (Sato Nishiki)	3	7 7	0.024	3 x 50	3 x 1.2	1 3 7	1.2 0.49 <0.1	35
2013 Nagano, Japan[2] 2013 (Seiko Nishiki)	3	7 7	0.024	3 x 45	3 x 1.1	1 3 7	3.5 0.95 <0.1	42
1903 Yamanashi, Japan[3] 2014 (Sato Nishiki)	3	7 7	0.027	3 x 60	3 x 1.6	1 3 7 14	0.62 0.31 0.04 <0.01	29
1904 Yamanashi, Japan[3] 2015 (Sato Nishiki)	3	7 7	0.027	3 x 60	3 x 1.6	1 3 7 14	0.67 0.19 0.02 <0.01	14
2019c	3	7	0.027	3 x 60	3 x 0.95	3	0.52	20

Cherries Study reference Location, year (variety)	Application					DALA	Methidathion[1] (mg/kg)	Storage interval
	No	Interval (days)	Conc (kg ai/hL)	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)		(days)
Aomori, Japan[3] 2018 (Sato nishiki)		7				7 10	0.01 <0.01	
2019c Iwate, Japan[3] 2018 (Beni shuho)	3	7 7	0.027	3 x 60	3 x 0.96	3 7 10	0.28 <0.01 <0.01	53
2019c Yamagata, Japan[3] 2018 (Beni shuho)	3	7 7	0.027	3 x 60	3 x 1.1	3 7 10	0.60 0.05 0.03	54
2019c Fukushima, Japan[3] 2018 (Seiko nishiki)	3	7 7	0.027	3 x 60	3 x 0.88	3 7 10	0.13 <0.01 <0.01	27

Notes:

[1/] Each sample was analysed twice in each of two different laboratories. The higher mean analytical result was shown in the table.

[2/] Analysed by Method Peach-1981 (LOQ: 0.1 mg/kg)

[3/] Analysed by LC-MS/MS Method (LOQ: 0.01 mg/kg)

[4/] These trials were conducted in the same city and 10 km away from each other. Therefore, these data are not considered independent.

Peaches (Goto (1431), 1981; Kato (1432), 1981; Goto (1455), 1990a; Goto (1456), 1990b; Kuroda and Higuchi (1457), 1990c; Kuroda and Higuchi (1458), 1990d; Ishitsuka (1459), 2014; Ogiyama (JP2017C303), 2018; and Morita (JP2018C021), 2019a)

The Meeting received 17 supervised trials conducted between 1981 and 2018 on peaches in Japan. Peaches were received foliar application of WP formulation of methidathion diluted with water at 0.024 kg ai/hL. In some trials EC formulation (0.15–0.30 kg ai/hL) was injected to the trunk twice before foliar application. In several trials, samples were analysed in two different laboratories. The longest storage interval was 136 days. Samples were analysed after removal of seeds and analytical results were shown as the weight of analysed portion basis.

In trials with footnote 5, peel and pulp were analysed separately and the analytical values for fruit were calculated from the analytical values of peel and pulp, based on the weight of fruit after removal of seed. In other trials, whole fruit and pulp after removal of seed were analysed. The result is shown in Table 45.

Table 45 Residues in peaches resulting from supervised trials in Japan (foliar spray of methidathion 360 WP and/or 400 EC formulation)

Peaches Study reference Location, year (variety)	Application					DALA	Methidathion[1] (mg/kg)			Storage interval
	No	Interval (days)	Conc (kg ai/hL)	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)	Fruit	Pulp	Peel	(days)
GAP, Japan	2+2		2 x 0.024 + 2 x 0.15 (EC)[2]	2 x 70 + 2 x 1.8		21 60				
1431, 1432 Yamagata, Japan[4] 1981 (Okubo)	2+2	10 12 48	0.024 2 x 0.3	2 x 45 2 x 0.6/tree	0.96 (WP) 2 x 1.8 (EC) 1.1 /tree(WP)	21	<0.1	<0.1	<0.1	26
1431, 1432 Ishikawa, Japan[4] 1981 (Okubo)	2+2	11 21 34	-[4] 0.024	2 x 50 2 x 0.6/tree	-[4] (EC) 1.2 /tree(WP) -[4] (EC) 1.2 /tree(WP)	21	<0.1	<0.1	0.10	30, 21, 30
1455, 1456, 1457, 1458 Nagano, Japan[4] 1990 (Okubo)	2	7	0.024	2 x 45	2 x 1.1 (WP)	21 30	<0.1 <0.1	<0.1 <0.1	<0.1 <0.1	39, 84
	3	6 7	0.024	3 x 45	3 x 1.1 (WP)	30	<0.1	<0.1	<0.1	30
	4	7 6 7	0.024	4 x 45	4 x 1.1 (WP)	30	<0.1	<0.1	<0.1	30
1459 Fukushima, Japan[4] 2012 (Akatsuki)	2	7	0.024	2 x 36	2 x 0.86 (WP)	7 14	0.16 <0.1	<0.1 <0.1	0.88 <0.1	136
	2	7	0.024	2 x 36	2 x 0.86 (WP)	21 28	<0.1 <0.1	<0.1 <0.1	<0.1 <0.1	136
1459 Yamanashi, Japan[4] 2013 (Hikawa Hakuho)	2	7	0.024	2 x 31	2 x 0.75 (WP)	7 14	0.13 <0.1	<0.1 <0.1	0.58 0.20	27
	2	7	0.024	2 x 31	2 x 0.75 (WP)	21 28	<0.1 <0.1	<0.1 <0.1	<0.1 <0.1	27
JP2017C303 Fukushima, Japan[5] 2017 (Akatsuki)	2+2	7 46 7	0.15 0.024	2 x 13 2 x 40	2 x 2.0 (EC) 2 x 0.96 (WP)	7 14	0.20 0.04	0.04 0.01	- -	11
	2+2	7 32 7	0.15 0.024	2 x 13 2 x 40	2 x 2.0 (EC) 2 x 0.96 (WP)	21 28	0.01 <0.005	<0.005 <0.005	- -	11
JP2017C303 Yamanashi, Japan[5] 2017	2+2	7 46 6	0.15	2 x 13	2 x 2.0 (EC) 2 x 0.96 (WP)	7 14	0.26 <0.005	0.07 <0.005	- -	11

Peaches Study reference Location, year (variety)	Application					DALA	Methidathion[1] (mg/kg)			Storage interval
	No	Interval (days)	Conc (kg ai/hL)	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)	Fruit	Pulp	Peel	(days)
(Hakuhoo)			0.024	2 x 40						
	2+2	7 46 6	0.15 0.024	2 x 13 2 x 40	2 x 2.0 (EC) 2 x 0.96 (WP)	21 28	0.006 <0.005	<0.005 <0.005	- -	11
JP2017C303 Wakayama, Japan[5] 2017 (Hikawa Hakuhou)	2+2	7 46 7	0.15 0.024	2 x 16 2 x 42	2 x 2.4 (EC) 2 x 1.0 (WP)	7 14	0.26 0.04	0.03 0.02	- -	16
	2+2	7 46 7	0.15 0.024	2 x 16 2 x 42	2 x 2.4 (EC) 2 x 1.0 (WP)	21 28	0.01 <0.005	<0.005 <0.005	- -	16
JP2018C021 Yamanashi, Japan[5] 2018 (Hikawa Hakuhou)	2+2	7 47 6	0.15 0.024	2 x 13 2 x 47	1.9 (EC) 1.1 (WP) 1.9 (EC) 1.1 (WP)	7 14	0.38 0.06	0.15 0.02	- -	17
	2+2	7 47 6	0.15 0.024	2 x 13 2 x 47	1.9 (EC) 1.1 (WP) 1.9 (EC) 1.1 (WP)	21 28	0.020 0.008	0.007 <0.005	- -	17

Notes:

[1/] Each sample was analysed twice in each of two different laboratories. The higher mean analytical result was shown in the table.

[2/]Trunk application.

[3/]Application rate not recorded.

[4/] Analysed by Method Peach-1981 (LOQ: 0.1 mg/kg).

[5/] Analysed by LC-MS/MS Method (LOQ: 0.005 mg/kg).

Grapes (Kato (1501), 1974; Tsuchiya (1502), 1974; Gotou et al (1503), 1991; Kuroda and Higuchi (1504), 1990e; Tamai (1505), 1999; Kato (1506), 1979 and Tsuchiya (1507), 1979; and Morita (JP2018C023), 2019b)

The Meeting received 15 supervised trials using foliar application conducted between 1973 and 2018 on grapes in Japan. In four trials, grapes received a foliar application of EC formulation (0.30 kg ai/L) diluted with water at 0.15 kg ai/hL with 127-143 DALA. In 11 trials, WP formulation (0.36 kg ai/L) diluted with water at 0.024 kg ai/hL was applied to grapes twice by foliar application. Samples were analysed after removal of stem and the analytical results were shown as the weight of analysed portion basis. In several trials, samples were analysed in two different laboratories and the higher values were shown. The longest storage interval was 150 days. The result is shown in Table 46.

The Meeting also received four trials in conjunction with drenching or steam fog applications (Table 47).

Table 46 Residues in grapes resulting from supervised trials in Japan (foliar spray of methidathion 360 WP or 300 EC formulations)

Grapes Study reference Location, year (variety)	Application					DALA	Methidathion[2] (mg/kg)	Storage interval
	No	Interval (days)	Conc (kg ai/hL)	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)	Fruit	
GAP, Japan	2		0.024	2 x 70		14		
1501, 1502 Akita, Japan[4] 1973 (Delaware)	1		0.15	18	2.7 (EC)	127	<0.2	81
	1		0.15	18	2.7 (EC)	135	<0.2	92
1501, 1502 Kagawa, Japan[4] 1973 (Cambell Early)	1		0.15	18	2.7 (EC)	133	<0.2	150
	1		0.15		2.7 (EC)	143	<0.2	150
1506, 1507 Yamanashi, Japan[4] 1979 (Delaware)	2	7	0.024	2 x 30	2 x 0.72 (WP)	7	<0.2	69
	2	7	0.024	2 x 30	2 x 0.72 (WP)	14	<0.2	140
	2	7	0.024	2 x 30	2 x 0.72 (WP)	21	<0.2	69
1506, 1507 Okayama, Japan[4] 1979 (Muscat)	2	7	0.024	2 x 30	2 x 0.72 (WP)	7	<0.2	22
	2	7	0.024	2 x 30	2 x 0.72 (WP)	14	<0.2	22
	2	7	0.024	2 x 30	2 x 0.72 (WP)	21	<0.2	93
JP2018C023 Iwate[3], Japan[5] 2018 (Rosario Bianco)	2	7	0.024	2 x 33	2 x 0.80 (WP)	7 14 21	0.28 0.18 0.12	30
JP2018C023 Niigata[3], Japan[5] 2018 (Kyoho)	2	7	2 x 0.024	2 x 35	2 x 0.85 (WP)	7 14 21	0.04 0.01 0.01	48
JP2018C023 Ishikawa[3], Japan[5] 2018 (Delaware)	2	7	2 x 0.024	2 x 33	2 x 0.80 (WP)	7 14 21	0.17 0.05 0.009	97
JP2018C023 Ibaraki[3], Japan[5] 2018 (Deleware)	2	7	2 x 0.024	2 x 30	2 x 0.72 (WP)	7 14 21	0.03 0.008 0.005	58
JP2018C023 Yamanashi[3], Japan[5] 2018 (Delaware)	2	6	2 x 0.024	2 x 36	2 x 0.85 (WP)	7 14 21	0.04 0.02 0.01	71

Notes:

[1/] Recommended water volume to spray is 7000 L/ha.

[2/] Each sample was analysed twice in each of two different laboratories. The higher mean analytical result was shown in the table.

[3/] Trials in the greenhouse.

[4/] Analysed by Method Apple-1988 (validation data not available).

[5/] Analysed by LC-MS/MS Method (LOQ: 0.005 mg/kg).

Table 47 Residues in grapes resulting from supervised trials in Japan (various application methods of methidathion 360 WP or 300 EC formulation)

Grapes Study reference	Application				DALA	Methidathion[2] (mg/kg)	Storage interval
Location, year (variety)	No	Interval (days)	Conc (kg ai/hL) [1]	Rate (kg ai/ha)	(days)	Fruit	
GAP, Japan	2		2 x 0.072 (soil irrigatiion) 1 x 0.15 (trunk application)		21 60		
1503, 1504 Ishikawa, Japan 1990 (Delaware)	2[4]	14	0.072	2 x 72 (WP)[4]	53	<0.2	21
	2[3]+2[4]	14 32 7	2 x 0.072 2 x 0.024	2 x 72 (WP)[3] 2 x 0.72 (WP)[4]	14 21	<0.2 <0.2	63
1505 Yamanashi, Japan 1998 (Delaware)	2[5]	7	0.8	2 x 0.72 (WP)	14 21 28	<0.2 <0.2 <0.2	294[6]
1505 Yamanashi, Japan 1998 (Kyohou)	2[5]	7	0.8	2 x 0.72 (WP)	14 21 28	<0.2 <0.2 <0.2	277[6]

Notes:

In all trials analytical method was Method Apple-1988. (Validation data not available).

[1/] Recommended water volume to spray is 7000 L/ha.

[2/] Each sample was analysed twice in each of two different laboratories. The higher mean analytical result was shown in the table.

[3/] Trunk application.

[4/] Soil drenching.

[5/] Steam fog application.

[6/] Longer storage interval than stability study.

Mango (Ebisu (2001), 2003 and Hashimoto (2002), 2005; and Sugimoto (2003), 2016)

The Meeting received five supervised trials using foliar application conducted between 2003 and 2015 on mangoes in Japan. Mangoes received a foliar application of EC formulation (0.30 kg ai/L) diluted with water at 0.027 kg ai/hL with 30–60 DALA twice (four trials) or three times (one trial). The longest storage interval was 116 days. The result is shown in Table 48.

Samples were analysed after removal of seeds and concentration was expressed as analysed portion basis. In the study 2003, the weights of seed ranged from 5.1 to 8.3 percent of whole fruit (Table 50).

Table 48 Residues in mangoes resulting from supervised trials in Japan (methidathion 400 EC formulation)

Mangoes Study reference	Application					DALA	Methidathion (mg/kg)	Storage interval
Location, year (variety)	No	Interval (days)	Conc (kg ai/hL)	Spray volume (hL/ha)	Rate (kg ai/ha)	(days)	Fruit	
GAP, Japan	2		0.027	2 x 70		45		
2001 Okinawa, Japan[1] 2003 (Irwin)	3	7 7	0.027	3 x 15	3 x 0.4	45 59 73	0.007 <0.005 <0.005	0
2002 Ogasawara, Tokyo, Japan[1] 2005 (Irwin)	2	7	0.027	2 x 40	2 x 1.1	30 45 60	<0.03(3) <0.03(3) <0.03(3)	61
2003 Miyazaki, Japan[2] 2015 (Irwin)	2	7	0.027	2 x 55	2 x 1.5	30 45 60	0.11 0.08 0.03	116
2003 Nago, Okinawa, Japan[2,4] 2015	2	7	2 x 0.027	2 x 50	2 x 1.3	30 45 60	0.04 0.02 0.02	110
2003 Miyako, Okinawa, Japan[2,4] 2015	2	7	2 x 0.027	2 x 30	2 x 0.80	29 45 60	0.07 0.04 <0.01	116

Notes:

[1/] Analysed by Method Mango-2003 (LOQ: 0.005 mg/kg).

[2/] Analysed by LC-MS/MS Method (LOQ: 0.01 mg/kg).

[3/] Although samples were analysed by Method Mango-2003 (LOQ: 0.005 mg/kg), data in the study report indicated <0.03 mg/kg.

[4/] Although these trials are conducted in the same year and prefecture, these trials are considered independent because the plots locate in different islands and 340 km away from each other.

Table 49 Weight ratio of seed to whole fruit for mango

Sample number	Total weight (g)	Weight of seed (g)	Ratio (seed/total) (percent)
1	567	39.2	6.9
2	410	26.2	6.4
3	422	24.5	5.8
4	508	32.7	6.4
5	383	24.3	6.3
6	390	32.2	8.3
7	410	26.2	6.4
8	363	22.2	6.1
9	498	25.2	5.1
10	458	25.2	5.5
11	530	36.6	6.9
12	446	28.6	6.4

FATE OF RESIDUES IN STORAGE AND PROCESSING

No processing study was provided by the sponsor. However, some information was available from published literature.

Dried grape (Özbey et al., 2017)

Sultana grape was treated with 45 µL/kg of methidathion through spraying onto surface and stood still for 12 h at room temperature. Grapes were separated from stems and dried under several conditions: under sunlight for 21 days, and in a ventilated oven at various temperatures (at 50 °C for 72 hours, at 60 °C for 60 hours, at 70 °C for 48 hours and at 80 °C for 36 hours).

Methidathion was analysed as follows: grape samples were homogenized, mixed well with acetonitrile, added anhydrous $MgSO_4$ and NaCl and centrifuged. Extracts were analysed by GC/MS. Moisture content was analysed by AOAC 930.15.

In all trials, methidathion reduced significantly, in particular, when grapes were dried under high temperature (**Table** 50). During oven-drying, methidathion followed the first order kinetic model. Half-life time of methidathion was 19.8 hours at 50 °C and 10.8 hours at 60 °C.

Table 50 Processing factors of methidathion while producing dried grapes under various conditions

Condition	Processing factor
80 °C, 36 h	0.02
70 °C, 48 h	0.06
60 °C, 60 h	0.13
50 °C, 72 h	0.35
Sun-drying, 21 days	0.18

Degradation in fruit juices (Kyriakidis et al, 2000)

Methidathion (final concentration of 1.6 mg/kg) was added to orange and peach juices that were obtained at a market. Juices were stored at 0 ±1 °C, 15 °C or 40±1 °C. Samples were taken every 15 days for juices stored at 0 °C (up to 105 days), every 10 days for juices stored at 15 °C (up to 110 days), and every day up to the 4th day and then every 2 days for juices stored at 40 °C (up to 20 days). Samples were analysed by GC-NPD.

Methidathion degradation followed the first order kinetic model. The half-lives of methidathion depended on the temperature, but not on the type of juice (Table 51).

Table 51 Half-life times of methidathion in orange and peach juices

Storage temperature	Half-life of methidathion (days)	
	Orange juice	Peach juice
40 °C	4.1	3.8
15 °C	115	114
0 °C	330	385

Farm animal feeding studies

No feeding study was provided by the sponsor. However, some information was available from the published literature.

Cows (Polan et al., 1969)

Three groups of four dairy cows (4 years old, Holstein) were orally treated methidathion in gelatin capsules twice daily for at least 55 consecutive days (another group of four cows for control). The dose rates were 7.5, 15 and 30 ppm. The cows were fed with 1 kg of a 16 percent crude protein concentrate per 3 kg of milk produced and permitted ad libitum consumption of an alfalfa-orchard grass hay (1.5-1.8 percent of body weight). Milk samples were taken on three consecutive days before initial treatment, twice daily at five-day intervals during and for eight weeks following cessation of pesticide administration. One cow from each treatment group was slaughtered on Day 55 or 56 and samples of heart, brain, spleen, kidney, liver, Longissimus dorsi, Biceps femoris, omental, perirenal and subcutaneous fat were collected for residue analysis. Samples were analysed by GC.

Milk samples were analysed from cows treated at all doses collected between 30 and 54 days and at 15 pm collected at 54 and 60 days. All milk samples contained <0.01 mg/kg of methidathion and <0.025 mg/kg of oxygen analogue. In tissues analysed, all samples contained <0.01 mg/kg of methidathion and <0.025 mg/kg of oxygen analogue.

Laying hens (Wiseman and Young, 1970)

Ten groups of two laying hens (40 weeks old, S.C. White Leghorn) were orally fed *ad libitum* containing methidathion at 0 (control), 10, 50, 100 and 500 ppm. Eggs were collected each third day after administration for residue analysis. For 500 ppm-group, feed consumption (94–111 g/day for other groups and 71–80 g/day for 500 ppm group) and egg production rate (69-81T for other groups and 52–60 percent for 500 ppm group) decreased.

Methidathion was not detected (LOQ 0.002 mg/kg) in egg yolk from 10 ppm-group hens. In 50 ppm and 100 ppm group hens, low concentration (<0.02 mg/kg) of methidathion were detected after 18 or 21 days, respectively, of consecutive administration.

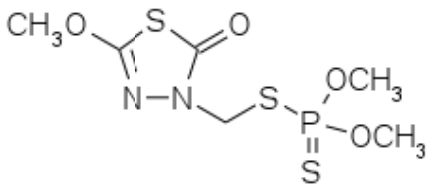
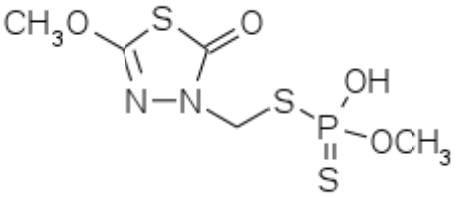
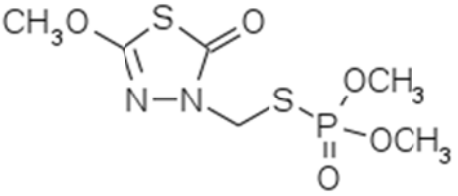
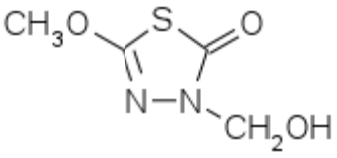
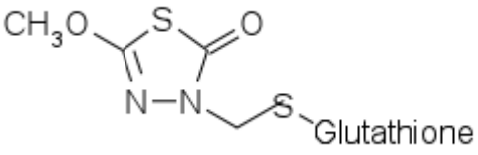
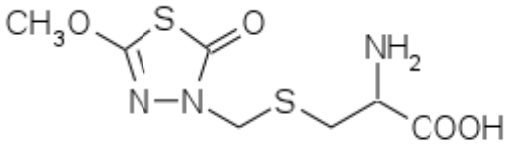
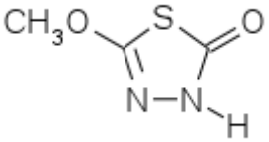
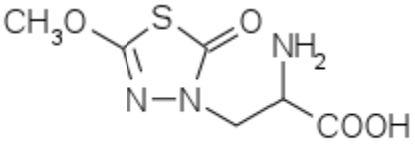
APPRAISAL

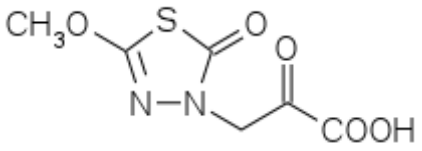
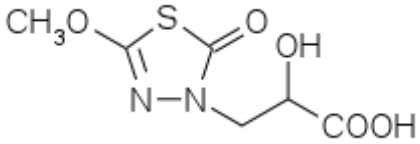
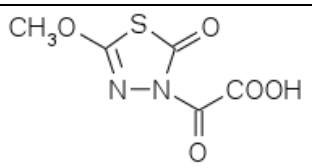
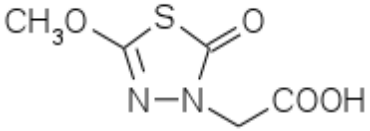
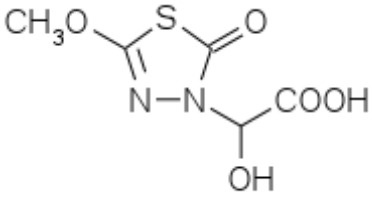
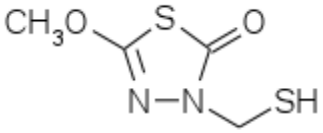
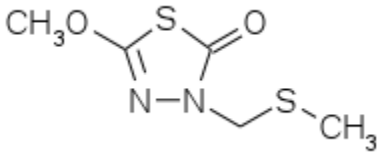
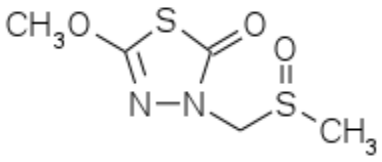
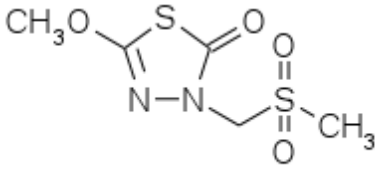
Methidathion, whose IUPAC name is S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O,O-dimethyl phosphorodithioate, is an organophosphate insecticide. Its mode of action is by inhibition of acetylcholinesterase. Methidathion was last evaluated for residues in 1994.

At the Fifty-first Session of CCPR (2019) methidathion was scheduled for periodic re-evaluation by the 2020 JMPR but was postponed to the 2022 JMPR. The Meeting received the data for methidathion on plant and animal metabolism, methods of analysis, GAP information, and residues resulting from supervised trials on apple, cherry, grape, mandarin, mango and peach.

The code names, chemical names and chemical structures of the compounds are as follows.

Table 52 Summary information on compounds referred to in the appraisal

Compound code name and chemical name	Structure
Methidathion S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O,O-dimethyl phosphorodithioate	
Desmethyl methidathion S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O-methyl phosphorodithioate	
Oxygen analogue of methidathion S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O,O-dimethyl phosphorothioate	
GS-20685 2,3-dihydro-3-hydroxymethyl-5-methoxy-1,3,4-thiadiazol-2-one	
Glutathione conjugate of GS-13005 RH-Glutathione conjugate 2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl-thioglutathione	
Cysteine conjugate of GS-13005 RH-Cysteine conjugate 2-amino-3-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol- 3-ylmethylthioxy) propionic acid	
RH 2,3-dihydro-5-methoxy-1,3,4-thiadiazol-2-one	
RH-Alanine conjugate 2-amino-3-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol- 3-ylmethyl) propionic acid	

Compound code name and chemical name	Structure
RH-Keto acid conjugate 2-oxo-3-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol- 3-ylmethyl) propionic acid	
RH-Lactic acid conjugate 3-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl)-2-hydroxypropionic acid	
RH-glyoxylic acid conjugate 2-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-yl) -2-oxo-acetic acid	
RH-Acetic acid conjugate 2-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-yl) acetic acid	
RH-Hydroxy acetic acid conjugate 2-(2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-yl)- 2-hydroxy-acetic acid	
RH-thiol 2,3-dihydro-5-methoxy-4-sulfanyl-1,3,4-thiadiazol-2-one	
RH-sulfide 2,3-dihydro-5-methoxy-4-methylsulfanyl-1,3,4-thiadiazol-2-one	
RH-sulfoxide 2,3-dihydro-5-methoxy-4-methylsulfinyl-1,3,4-thiadiazol-2-one	
RH-sulfone 2,3-dihydro-5-methoxy-4-methylsulfonyl-1,3,4-thiadiazol-2-one	

With respect to the physical and chemical properties that may impact on residues in crops, methidathion is not regarded as volatile, it is unlikely to be fat soluble as the log P_{ow} is 2.2, and the hydrolysis half-lives range from 13 days at pH 9 to 48 days at pH 7.

Plant metabolism

The Meeting received information on the fate of methidathion in oranges, tomatoes, common beans and alfalfa. In the metabolism studies, total radioactive residues (TRR) are expressed in mg methidathion equivalents/kg.

In a metabolism study on oranges, 2-carbonyl-^{14}C-methidathion was applied twice to an orange tree under outdoor conditions as foliar spray at rates of 0.067 kg ai/ha. Oranges were harvested at maturity 159 days after last application (DALA), washed by water with surfactant, and peeled. In plant metabolism study, juice was prepared from the peeled fruit. Subsamples of each matrix were homogenized to determine the TRR.

Radioactivity was mainly located in peel (66 percent TRR, 1.0 mg eq/kg), followed by juice (22 percent TRR, 0.25 mg eq/kg) and pulp (11 percent TRR, 0.40 mg eq/kg). Residues in the aqueous surface wash were insignificant (0.5 percent TRR, < 0.01 mg eq/kg). Peel and pulp were extracted with methanol-water (9:1). From orange pulp, 17 percent TRR (0.068 mg eq/kg) was extracted and when partitioned with water-chloroform, 12 and 2.5 percent TRR (0.048 and 0.010 mg eq/kg) were found in water and chloroform phase, respectively. Residues in pulp were not further characterized.

Since only radioactivity in peel and juice was subject to identification, the Meeting could not calculate the metabolite composition for the whole fruit.

In orange peel, 65 percent TRR was extracted by aqueous methanol and the following compounds were identified methidathion (24 percent TRR, 0.25 mg eq/kg), RH-alanine conjugate (14 percent TRR, 0.14 mg eq/kg), conjugates of RH with acetic acid, lactic acid or hydroxy acetic acid (3.5–4.2 percent TRR, 0.04 mg eq/kg), and desmethyl-methidathion (1.7 percent TRR, 0.02 mg eq/kg). Unextracted residues in the orange peel were further treated by sequential acid (0.1 and 1 mol/L HCl) and base (5 mol/L NaOH) hydrolysis. After hydrolysis with 0.1 mol/L HCl, 1 mol/L HCl and base, 0.68 percent TRR (0.007 mg eq/kg), 12 percent TRR (0.12 mg eq/kg) and 3.8 percent TRR (0.039 mg eq/kg), respectively, were extracted.

In orange juice, residues were predominantly aqueous soluble (94 percent TRR, 0.24 mg eq/kg). Parent methidathion was not detected. RH-alanine conjugate was predominant (72 percent TRR, 0.18 mg eq/kg), followed by RH-lactic acid conjugate (10 percent TRR, 0.03 mg eq/kg) and desmethyl-methidathion (3.1 percent TRR, < 0.01 mg/kg). In a metabolism study on tomatoes, the following compounds ^{14}C-labelled at 2-carbonyl in thiadiazol ring were used: methidathion, desmethyl methidathion, RH-sulfide, RH-sulfoxide and RH-sulfone. Labelled compounds, at rates of 1–14 mg/kg for ^{14}C-methidathion or a rate of 7 mg/kg for other compounds, were directly applied on the surface of detached semi-ripe tomato fruits. The fruit was stored at room temperature for 3–14 days (^{14}C-methidathion) or 7 days (other compounds) and then extracted with acetone-water (9:1). When ^{14}C-methidathion at a rate of 14 mg/kg was applied, 97 percent of applied radioactivity (AR, 12 mg eq/kg) was recovered 14 days after treatment (DAT). Desmethyl methidathion was the main residue (41 percent TRR, 4.8 mg eq/kg), followed by RH-cysteine conjugate (33 percent TRR, 3.8 mg eq/kg), methidathion (9.8 percent TRR, 1.1 mg eq/kg), its oxygen analogue (3.2 percent TRR, 0.37 mg eq/kg) and GS-20685 (0.43 percent TRR, 0.05 mg eq/kg).

In tomatoes treated with ^{14}C-desmethyl methidathion (7 DAT), 93 percent AR (6.2 mg eq/kg) was recovered. Identified compounds were: unchanged desmethyl methidathion (52 percent TRR, 3.2 mg eq/kg), RH-cysteine conjugate (21 percent TRR, 1.3 mg eq/kg) and RH-sulfide (1.1 percent TRR, 0.07 mg eq/kg). In tomatoes treated with ^{14}C-RH-sulfide (7 DAT), 96 percent AR (5.6 mg eq/kg) was recovered. Identified compounds were RH-sulfoxide (60 percent TRR, 3.4 mg eq/kg) and RH-cysteine conjugate (14 percent TRR, 0.78 mg eq/kg) and unchanged RH-sulfide (20 percent TRR, 1.1 mg eq/kg). In tomatoes treated with ^{14}C-RH-sulfoxide (7 DAT), 95 percent AR (6.8 mg eq/kg) was recovered. Identified compounds were unchanged RH-sulfoxide (70 percent TRR, 4.7 mg eq/kg), RH-sulfone (16 percent TRR, 1.1 mg eq/kg), and RH-cysteine conjugate (7.7 percent TRR, 0.52 mg eq/kg). When ^{14}C-RH-sulfone was applied, 96 percent AR (6.4 mg eq/kg) was recovered in the fruit, with 89 percent TRR (5.8 mg eq/kg) of unchanged RH-sulfone and 2.8 percent TRR (0.18 mg eq/kg) of RH-cysteine conjugate.

In a metabolism study on common beans, plants were cultivated in pots and methidathion ^{14}C-labelled at 2-carbonyl, 3-methylene, O-methyl or 2-carbonyl-RH was topically applied to leaves. Samples were collected 7–16 days after the treatment. The samples were extracted with acetone-water (8:2) and partitioned with chloroform and radioactivity in the chloroform phase was identified by TLC. Aqueous phase was not further identified. During the experiment, $^{14}CO_2$ was collected in 2 mol/L NaOH traps. Radioactivity was found in chloroform phase (3.6–8.9 percent AR), water phase (20–55 percent AR), non-extracted (1.6–12 percent AR) and $^{14}CO_2$ (2.3–27 percent AR). When methidathion or methidathion oxygen analogue was applied, methidathion, oxygen analogue and RH were recovered in chloroform phase (1.3–6.0 percent AR, 0.15–up to 5.4 percent AR, respectively). When RH was applied, radioactivity was mainly found in water phase (55.3 percent AR) and 5.4 percent AR of RH was recovered. Further information is not available in the study on common beans.

In a metabolism study on alfalfa, plants were cultivated in field and 2-carbonyl-14C-methidathion was topically applied to leaves. Samples were collected 7–16 days after the treatment. The sample was extracted by acetone-water (8:2) and partitioned with chloroform and chloroform phase was identified by TLC. During the experiment, $^{14}CO_2$ was collected in 2 mol/L NaOH traps. Radioactivity was found in chloroform phase (33–56 percent AR), water phase (14–32 percent AR), non-extracted (2.9–6.4 percent AR) and $^{14}CO_2$ (14 percent AR). In chloroform phase, methidathion, its oxygen analogue and RH were 32–53, 0.33–0.86 and 0.43–1.7 percent AR, respectively. In water phase, RH conjugate was found. Further information is not available in the study on alfalfa.

Summary of plant metabolism

When methidathion was applied to oranges, the parent methidathion was found in peel (24 percent TRR, 0.25 mg eq/kg), but not in juice. Major metabolites were RH-alanine conjugate (14 percent TRR, 0.14 mg eq/kg in peel and 72 percent TRR, 0.18 mg eq/kg in juice) and RH-acetic acid, lactic acid or hydroxy acetic acid conjugate (3.7 percent TRR, 0.04 mg eq/kg in peel and 10 percent TRR, 0.03 mg eq/kg in juice). When methidathion was applied to tomato fruits, desmethyl-methidathion (39–42 percent TRR, 2.3–4.8 mg eq/kg), RH-cysteine conjugate (34–35 percent TRR, 2.1–3.8 mg eq/kg), parent methidathion (9.8 percent TRR, 1.1 mg eq/kg), and its oxygen analogue (3.2 percent TRR, 0.37 mg eq/kg). When tomatoes were treated with ^{14}C-desmethyl methidathion, the major residues were unchanged desmethyl methidathion (52 percent TRR, 3.2 mg eq/kg) and RH-cysteine conjugate (21 percent TRR, 1.3 mg eq/kg) were the major metabolites. When methidathion was applied to common beans and alfalfa, parent methidathion (1.3–53 percent AR), its oxygen analogue (0.15–0.86 percent AR) and RH (≤1.7 percent AR) were found.

The Meeting noted that metabolites found in various plants were desmethyl-methidathion, the oxygen analogue of methidathion and RH or its conjugate, although some of them might not be observed

depending on plant species. The Meeting concluded that the metabolic profiles between the species were qualitatively similar.

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating cows and lactating goats.

Rats

The metabolism of methidathion in rats was reviewed in the framework of the toxicological evaluation by the WHO Core Assessment Group of the 2022 JMPR.

Cattle

In a metabolism study on lactating cow, 2-carbonyl-^{14}C-methidathion was administered orally in capsules three times per day for 5 consecutive days at 1 mg/kg bw per day (equivalent to 32 ppm) and the cow was sacrificed 10 days after the last application (DALA). Radioactivity in milk increased to 0.5 mg eq/kg at 1 DALA and then decreased to 0.004 mg eq/kg at 10 DALA, and no clear plateau was observed. Through the study period, accumulated total radioactivity recovered from milk was 0.6 percent AR (0.5 mg eq/kg) and oxygen analogue of methidathion was not found in milk (LOD: 0.01 mg eq/kg). Radioactivity was detected in liver at 0.11 mg eq/kg, kidney at 0.04 mg eq/kg, omental fat at 0.03 mg eq/kg, and muscle, at 0.02 mg eq/kg after 10 days of post-dosing period. No identification or characterization was conducted.

In another study of metabolism on lactating cow, 2-carbonyl-^{14}C-methidathion was administered orally once at 1.7 mg/kg bw (equivalent to 120 ppm). Radioactivity in milk collected after administration for 96 hours was 0.8 percent AR (0.53 mg eq/kg).

In the same study, an additional metabolism study on lactating cow was conducted. A cow was given orally 1.25 mg/kg daily of methidathion (non-labelled) for 16 days, and then 2-carbonyl-^{14}C-methidathion was orally administered once at 1.7 mg/kg bw (equivalent to 61 ppm). Milk was collected at the end of 12 and 24 hours after administration. Radioactivity in the milk for 0–12 and 12–24 hr after the application of labelled methidathion was 1.2 and 0.7 percent AR, respectively. In both milk samples, methidathion was not detected, and RH-sulfone and RH-sulfoxide accounted for 3.4 and 0.9 percent of TRR in milk, respectively, of the total radioactivity in milk collected within 12 hours after application. Further identification for the rest of radioactivity (95.7 percent TRR of milk) was not conducted.

Goat

In a metabolism study on lactating goats, 0.88 mg/kg bw (equivalent to 38 ppm) of 2-carbonyl-^{14}C-methidathion was once administered by stomach tube. At 3 DAT (72 hours after the single dose), 1 percent AR of radioactivity was found in milk. Methidathion or its oxygen analogue was not found in milk. No further identification or characterization was conducted.

Summary of animal metabolism

When single or multi dose of methidathion was orally administered to cattle, radioactivity was detected in the most of tissues and milk. In milk, RH-sulfone and RH-sulfoxide were detected (<5 percent AR total), and methidathion and its oxygen analogue were not detected. Since information on metabolites was insufficient in milk or tissues, it was not possible to estimate metabolic pathway in livestock.

Environmental fate

On soil surface, degradation of methidathion in soil followed first order kinetic degradation with half-lives of 1.7 days under the sunlight and 2.6–5.8 days in the dark, depending on the existence of microorganisms and surfactant. Neither RH nor the methidathion oxygen analogue was produced by light degradation. Further identification was not conducted. The Meeting concluded that degradation of methidathion was rapid in soil.

Methods of analysis

The Meeting received seven methods of analysis used for the determination of methidathion in the supervised field trials. Six of them were similar GC methods with some modifications and one was an LC-MS/MS method. In the validation studies, spiked samples were not analysed at day zero. However, since analysis of at least one sample within 1 month after spiking resulted in satisfactory recovery, the Meeting considered that the analytical values at day zero would be almost 100 percent of spike.

For GC methods, methidathion was extracted with acetone or ethyl acetate, cleaned up, and analysed using GC coupled with FID, ECD or FPD. The Meeting confirmed that the methods were validated for methidathion in mandarin, apple, peach and cherry with an LOQ of 0.1 or 0.2 mg/kg and mango with an LOQ of 0.005 mg/kg.

For the LC-MS/MS method, methidathion was extracted with acetone, cleaned up, and analysed using LC-MS/MS. The Meeting confirmed that the method was validated for methidathion in peach, cherry and grape with an LOQ of 0.005 mg/kg and mango with a LOQ of 0.01 mg/kg.

Stability of pesticide residues in stored analytical samples

Stability studies on methidathion residues in fortified mandarin (up to 7 months), apple (up to 7 months), cherry (up to 2.5 months), peach (up to 9 months), grape (up to 10 months) and mango (up to 4 months) were available. Noting the various storage periods for matrices in the same group, the Meeting concluded that methidathion in high water and high acid content commodities stored at ≤ -20 °C was stable for at least 9 and 7 months, respectively. In supervised trials received at the current Meeting, all samples were kept frozen at ≤ -20 °C and analysed within 7 months from sampling except in two trials, where the data were not used in the assessment.

Definition of the residue

Plant commodities

In the metabolism studies on orange, tomato, common beans and alfalfa, parent methidathion was found in all primary crop commodities analysed (7.2 percent TRR in orange, 8.4–9.4 percent TRR in tomato, 1.3–6.0 percent AR in bean plant and 32–53 percent AR in alfalfa plant). The Meeting noted that suitable analytical methods exist to determine methidathion in plant commodities. The Meeting considered that parent methidathion was suitable marker for MRL-compliance.

Based on the plant metabolism studies, the following compounds could be included in the residue definition for dietary risk assessment: conjugate of RH with alanine, acetic acid, hydroxyacetic acid, lactic acid, methanol (GS-20685), cysteine and sulfoxide; desmethyl-methidathion and the oxygen analogue of methidathion.

For RH and its conjugates of alanine, lactic acid, acetic acid, hydroxyacetic acid, cysteine, methanol (GS-20685) or sulfoxide, the Meeting concluded that the same ADI as methidathion should apply (0-0.002 mg/kg bw) and no ARfD was necessary due to lack of phosphate moiety. The Meeting

noted that the total levels of RH (free and conjugated) in the metabolism studies were significant: 7.2 percent TRR (0.26 mg eq/kg) in peel and 82 percent TRR (0.21 mg eq/kg) in juice in the study on oranges; 36–37 percent TRR (2.2–4.0 mg eq/kg) in the study on tomatoes; 0.55–0.66 percent AR in the study on beans; and 0.33–0.86 percent AR in the study on alfalfa. The Meeting concluded that these RH conjugated compounds should be included in the residue definition.

For desmethyl methidathion, the Meeting concluded that the same ADI and ARfD as methidathion should apply (ADI: 0–0.002 mg/kg bw, ARfD: 0.01 mg/kg bw). The Meeting noted that the level of desmethyl methidathion in the study on tomato was significant (39–42 percent TRR, 2.3–4.8 mg eq/kg). The Meeting concluded that desmethyl methidathion should be included in the residue definition.

For oxygen analogue, it was found in the metabolism study of tomato, bean and alfalfa but not in the study on orange. In the case of tomato, as the ratio of oxygen analogue (0.37 mg eq/kg) to the parent compound (1.14 mg eq/kg) was higher than 10 percent, the Meeting concluded that oxygen analogue should be included in the residue definition.

The Meeting concluded that since the mechanism of toxicity was similar to that of methidathion, oxygen analogue should be considered together with the parent methidathion for dietary risk assessment. As the potency of the oxygen analogue as a AChE inhibitor was considered to be about four times that of methidathion, the Meeting agreed to apply a potency factor of 4 to the residues of the oxygen analogue for long-term and short term risk assessment.

In conclusion, the definition of the residue for compliance with the MRL for plant commodities: *methidathion*.

The definition of the residue for long-term dietary exposure assessment for plant commodities: *sum of methidathion, S-2,3,-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O-methyl phosphorodithioate (desmethyl methidathion) and 2,3-dihydro-5-methoxy-1,3,4-thiadiazol-2-one (RH; free and conjugate), and 4x S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O,O-dimethyl phosphorothioate (oxygen analogue), expressed as methidathion*.

Definition of the residue for acute dietary exposure assessment for plant commodities: *sum of methidathion and 4× S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O,O-dimethyl phosphorothioate, expressed as methidathion.*

Animal commodities

For cattle, parent methidathion was detected in muscle, kidney and fat but not in liver and milk. In addition, methidathion may be detected in egg yolk (See animal feeding study). The Meeting considered that parent methidathion alone was not a suitable marker for enforcement.

In the metabolism studies, a large portion of radioactivity was not identified. Due to lack of further information on metabolites, the Meeting could not establish residue definitions for animal commodities.

Results of supervised residue trials on crops

Since all the metabolites included in the residue definition for dietary risk assessment were not analysed in the supervised trials, and the information in the metabolism studies was not sufficient to estimate conversion factors, the Meeting could not estimate STMRs and HRs. The Meeting agreed to estimate maximum residue levels but not to recommend them for adoption.

Citrus fruits

Mandarins

The critical GAP for methidathion on mandarin in Japan is four foliar applications of 0.027 kg ai/hL and a PHI of 14 days.

No trials matched the Japanese GAP. The Meeting could not estimate a maximum residue level.

Pome fruits

Apple

The critical GAP for methidathion on apple in Japan is for two foliar applications of 0.024 kg ai/hL with a PHI of 30 days.

In trials matching the GAP in Japan, residues of methidathion in apple were (n=7): < 0.1 (7) mg/kg. In trials with one application at 0.024 kg ai/hL and a PHI of 30 days at the same location as the trials with two applications, residues of methidathion in apple were higher than with two applications in 2 trials: 0.10 and 0.11 mg/kg. Therefore, residues of methidathion in apple were ((n=7): < 0.1 (5), 0.10 and 0.11 mg/kg.

In these supervised trials, the concentration was expressed on the basis of fruit weight after removal of core. The Meeting considered that the contribution of the core to the weight of the whole fruit is 9.8–14 percent and concluded that correcting the residue levels using this weight/weight ratio would lead to the same maximum residue level.

The Meeting estimated a maximum residue level of 0.2 mg/kg.

Since the analytical data for compounds in the residue definition were not sufficient in the supervised trials and metabolism study was not sufficient to estimate conversion factors, the Meeting could not conclude the assessment.

Pear

The Meeting could not estimate maximum residue level for pears since no data were available.

Stone fruits

Cherries

The critical GAP for methidathion on cherries in Japan is three foliar applications at 0.027 kg ai/hL and a PHI of 7 days. In trials matching the GAP conducted in Japan, residues of methidathion in cherries were (n=9): < 0.01 (2), 0.01, 0.02, 0.04, 0.05 and < 0.1(3) mg/kg.

In the supervised trials, the concentration was expressed on the basis of fruit weight after removal of seeds. The Meeting considered that the contribution of the seed to the weight of the whole fruit is less than 10 percent and concluded that correcting the residue levels using this weight/weight ratio would lead to the same maximum residue level.

The Meeting estimated a maximum residue level of 0.3 mg/kg.

Since analytical data for compounds in the residue definition were not sufficient in the supervised trials and metabolism study was not sufficient to estimate conversion factors, the Meeting could not conclude the assessment.

Peaches

In the critical GAP for methidathion on peaches in Japan is for 2 trunk injections (0.15 kg ai/hL) followed by two foliar applications of 0.024 kg ai/hL with a PHI of 21 days.

In trials matching the GAP, residues of methidathion in peaches were (n=4): 0.006, 0.011, 0.020 and < 0.1 mg/kg. The Meeting could not estimate a maximum residue level due to insufficient number of trials.

Berries and other small fruits

Grapes

The critical GAP for methidathion on grapes in Japan is two foliar applications at 0.024 kg ai/hL and a PHI of 14 days.

In trials matching the critical GAP conducted in Japan, residues of methidathion in grapes were (n=5): 0.008, 0.01, 0.02, 0.05 and 0.18 mg/kg. The Meeting could not estimate a maximum residue level due to insufficient number of trials.

Tropical and sub-tropical fruits – inedible peel

Mango

The critical GAP for methidathion on mangoes is in Japan and consists of two foliar applications of 0.027 kg ai/hL and a PHI of 45 days. In trials matching the GAP conducted in Japan, residues of methidathion in mango was (n=4) 0.02, < 0.03, 0.04 and 0.08 mg/kg.

In one supervised trial, mango was treated with three foliar applications at the same concentration and PHI as cGAP. In the trial, the residue of methidathion in mango was 0.007 mg/kg, which was lower than residue data from 3 applications. The Meeting assumed that the trial was considered to approximate GAP since the first application applied 59 days before harvest did not contribute to the residues at harvest.

In the trials on mango conducted in Japan approximating the GAP residue level were (n=5): 0.007, 0.02, < 0.03, 0.04 and 0.08 mg/kg.

The Meeting noted that in the supervised trials, the concentration was expressed as the basis of fruit weight after removal of seeds. The Meeting considered that the contribution of the seed to the weight of the whole fruit is less than 10 percent and concluded that correcting the residue levels using this weight/weight ratio would lead to the same maximum residue level.

The Meeting estimated a maximum residue level of 0.15 mg/kg.

Since the analytical data for compounds in the residue definition were not sufficient in the supervised trials and metabolism study was not sufficient to estimate conversion factors, the Meeting could not conclude the assessment.

Tea, green, black

The Meeting could not estimate a maximum residue level for tea, green, black since no data were available.

Fates of residues during processing

Processing study on grape to dried grape was available. Degradation of methidathion was observed in the process and the processing factors were 0.02–0.35, depending on the temperature of drying. The Meeting assumed the worst case scenario (50 °C, 72 hours) and the processing factor from grape to dried grape was estimated 0.35.

A study on degradation of methidathion in fruit juices (orange and peach) was available. When juices were stored at 40 °C, methidathion was reduced (half-life of 3.8–4.1 days), but degraded more slowly at lower temperatures (half-lives of =114–115 days at 15 °C and 330–385 days at 0 °C). At ambient temperature, the Meeting considered that the degradation of methidathion in juices at the shelf could be negligible.

Residues in animal commodities

Farm animal feeding studies

A dairy cow feeding study was available to the Meeting. Methidathion in gelatine capsule was administered orally twice daily to three groups of dairy cow (four for each) for 66 days at levels equivalent to 7.5, 15 or 30 ppm. The residue levels of methidathion and oxygen analogue of methidathion in milk and any tissues were < 0.01 mg/kg and < 0.025 mg/kg, respectively.

A laying hen feeding study was available in open literature. Feed containing methidathion at levels of 10, 50, 100 or 500 ppm were fed *ad libitum*. The residue was not detected in egg yolk from 10 ppm group hens (LOQ: 0.002 mg/kg). In 50 and 100 ppm group, low level of methidathion residues (< 0.02 mg/kg) were detected in egg yolk. No information on residues was available.

Farm animal dietary burden

From the commodities evaluated, the only processed commodity appearring in the OECD feed table was apple pomace. Since processing factors from apple to apple pomace for methidathion, desmethyl methidathion, oxygen analogue and RH (free+conjugate) were not available, the Meeting could not estimate the concentration of methidathion and its metabolites in animal commodities.

Animal commodity maximum residue levels

Since no conclusion could be reached on a residue definition for animal commodities, the Meeting withdrew all previous recommendations for animal commodity maximum residue levels for methidathion.

RECOMMENDATIONS

Definition of the residue for compliance with the MRL for plant commodities: methidathion

Definition of the residue for long-term dietary exposure assessment for plant commodities: sum of methidathion, S-2,3,-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O-methyl phosphorodithioate and 2,3-dihydro-5-methoxy-1,3,4-thiadiazol-2-one (free and conjugate), and 4× S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O,O-dimethyl phosphorothioate, expressed as methidathion

Definition of the residue for acute dietary exposure assessment for plant commodities: sum of methidathion and 4× S-2,3-dihydro-5-methoxy-2-oxo-1,3,4-thiadiazol-3-ylmethyl O,O-dimethyl phosphorothioate, expressed as methidathion.

Definition of the residue for animal products (for compliance with the MRL and for dietary exposure assessment): **a conclusion could not be reached**.

The residue is not fat soluble.

Table 53 Recommendations for residues of methidathion from the 2022 JMPR

CCN	Commodity	Maximum residue level (mg/kg)		STMR (mg/kg)	HR (mg/kg)
		New	Previous		
FC0226	Apple	W	0.5		
FS0013	Cherries	W	0.2		
FB0269	Grapes	W	1		
FC0206	Mandarins (including mandarin like hybrids)(subgroup)	W	5		
FP0230	Pear	W	1		
DT1114	Tea, green, black (black, fermented and dried)	W	0.5		

DIETARY RISK ASSESSMENT

No recommendations were made at the present meeting as the Meeting could not reach a conclusion on the residue definitions for animal commodities and sufficient residue data for metabolites were not available. No dietary risk assessment was conducted.

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PYRIDATE (315)

First draft prepared by Mr C Sieke and Dr J Heidler, Federal Institute for Risk Assessment, Germany

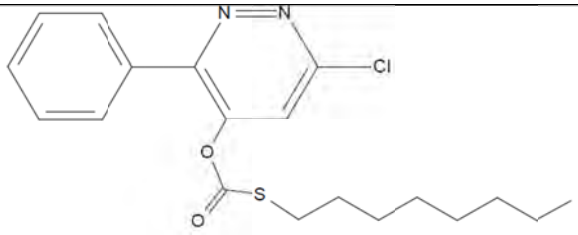
EXPLANATION

Pyridate is an herbicide of the phenylpyridazine class used to control annual broad-leaved weeds. It acts by inhibiting the photosynthetic electron transport at the photosystem II. The IUPAC name of pyridate is *O*-6-chloro-3-phenylpyridazine-4-yl *S*-octyl thiocarbonate

Pyridate was scheduled at the Fiftieth Session of the CCPR for evaluation as a new compound by the 2020 JMPR, reviewed by the 2019 JMPR for toxicology, where an ADI of 0–0.2 mg/kg bw and an ARfD of 2 mg/kg were established. The residue assessment was rescheduled to the current JMPR.

The Meeting received information on identity, physicochemical properties, metabolism (plant, confined rotational crops and animals), environmental fate, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials, fate of residues in processing, and livestock feeding studies.

IDENTITY

ISO common name:	Pyridate
IUPAC name:	*O*-6-chloro-3-phenylpyridazine-4-yl *S*-octyl thiocarbonate
CA nomenclature:	*O*-(6-chloro-3-phenylpyridazinyl) *S*-octyl thiocarbonate
CAS No.:	55512-33-9
CIPAC No.:	447
Synonyms:	CL 11344
Structural formula	
Molecular formula:	$C_{19}H_{23}ClN_2O_2S$
Molecular mass:	378.91 g/mol

Specifications

Specifications for pyridate were not yet developed by FAO.

PHYSICAL AND CHEMICAL PROPERTIES

Table 1 Physical and chemical properties of pure pyridate

Property	Results	Method (test material)	Reference
Appearance	Colourless solid, slightly aromatic odour	Batch RS-0033: 98.9 percent purity (purified ai)	Kettner, 1995, PYRIDATE_001

Property	Results	Method (test material)	Reference
Melting point	26.5-27.8 °C	OECD 102, EEC A1 Batch RS-0033: 98.9 percent purity (purified ai)	Bates, 1996, PYRIDATE_002
Boiling point	Not applicable, decomposes from 250 °C without boiling	OECD 103, EEC A2 Batch RS-0033: 98.9 percent purity (purified ai)	Bates, 1996, PYRIDATE_002
Relative density	1.28 g/cm^3 at 21 °C	OECD 109, EEC A3 Batch AMS 890/1: 98.9 percent purity (purified ai)	Füldner, 1998, PYRIDATE_003
Vapour pressure	Pyridate: 1.0 × 10^{-6} Pa CL 9673: 5.7 × 10^{-8} Pa at 25°C	EPA Series 163-2 No batch information provided	Landvoigt, 1988, PYRIDATE_004
Partition coefficient n-octanol / water	Log P_{ow} = 4.01±0.16 at room temperature Pyridate is hydrolytically unstable	OECD A 80/8 (^{14}C-pyridate)	Zohner, 1982, PYRIDATE_005
Solubility in water	1.49 mg/L at 20 °C Pyridate is hydrolytically unstable (conversion to CL 9673)	OECD A 80/6 (^{14}C-pyridate)	Zohner, 1980, PYRIDATE_006
Solubility in organic solvents	Solvent: g/L at 20 °C heptane: >250 dichloromethane: >250 methanol: >250 acetone: >250 xylene: >250 ethyl acetate: >250	CIPAC method MT 181 Batch H1012008: 91.4 percent purity (technical)	Seck & Jein, 2011, PYRIDATE_007
Hydrolysis	pH / DT_{50} at 25 °C / DT_{50} at 50 °C 4 / 117 h / 10.7 h 5 / 89 h / - 7 / 58.5 h / 4.66 h 9 / 6.2 h / 0.306 h Pyridate is hydrolytically unstable – hydrolysis proceeds via cleavage of the ester bond leading to CL 9673.	EEC C7, EPA Series 161-1 Batch ^{14}C-CL-11344-M8915-D: 97.9 percent purity (^{14}C-pyridate)	Lutringer, 1997, PYRIDATE_008
Photolysis	pH / DT_{50} 5 / 3.5 d 7 / 1.8 d 9 / 2.2 d No significant absorption at λ ≥290 nm. DT_{50} values indicate degradation via hydrolysis to CL 9673.	US-EPA, FIFRA 40 CFR part 158, Subdivision N Series 161-2 Batch WH-66: 99.2 percent (purified as) Batch ^{14}C-CL-11344-M8915-A: >98 percent purity (^{14}C-pyridate)	Van Dijk, 1992, PYRIDATE_009
Dissociation constant	Using two different models, no dissociation constant was estimated to lie within the environmentally relevant pH range of 4 to 10. Outside the environmentally relevant pH range the dissociation of the protonated nitrogen ring atoms is observed with pKa of -0.84.	Model 1: ACD Ilab2.0 ACD pKa algorithm version 12.1.0.50374 Model 2: VCCLAB, Virtual Computational Chemistry Laboratory, 2005	Weidenauer, 2012, PYRIDATE_010

Property	Results	Method (test material)	Reference
Thermal stability	Auto-ignition temperature: 245 °C Not explosive Not oxidising	EEC A15 Batch CL-11344/AR995: 91.4 percent purity (technical) EEC A14 Batch KL27: 92.1 percent purity (technical) EEC A17 Batch KL27: 92.1 percent purity (technical)	Krips, 1995, PYRIDATE_011 Angly, 1997, PYRIDATE_012 Kaul, 2012, PYRIDATE_013

METABOLISM AND ENVIRONMENTAL FATE

Metabolism studies were conducted using [^{14}C]-labelled pyridate at the pyridazine ring. The position of the label for the test substances is presented in the following figure:

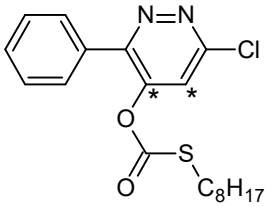
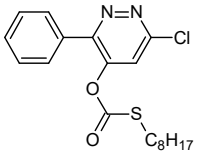
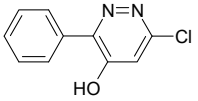
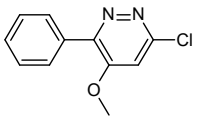


Figure 1 Structure of pyridate and position of radiolabels

Chemical names, structures and code names of metabolites and degradation products of pyridate are shown in Table 2.

Table 2 Known metabolites of pyridate

Code Names	Chemical Names (IUPAC)	Structure	Where found
Pyridate (CL 11344)	6-chloro-3-phenylpyridazine-4-yl-S-octyl-thiocarbonate	 378.9 g/mol	Plants matrices: broccoli (leaves), maize (leaves), peanut (forage, hay, hulls) Soil
Pyridafol (CL 9673)	6-chloro-4-hydroxy-3-phenylpridazine	 206.6 g/mol	Plants matrices: broccoli (leaves), maize (leaves), peanut (forage, hay) Animal matrices: Cow (kidney), goat (kidney, milk) Soil

Code Names	Chemical Names (IUPAC)	Structure	Where found
Pyridafol-O-methyl (Pyridafol-OMe; CL 9869)	6-chloro-4-methoxy-3-phenylpridazine	 220.7 g/mol	Plants matrices: peanut (forage, hay, hulls) Animal matrices: Goat (kidney) Soil

Plant metabolism

The metabolic fate in plants was investigated following foliar application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate to broccoli, maize and peanut.

In all studies, pyridate did undergo rapid hydrolytic cleavage into pyridafol, which is considered the herbicidal active compound. Detoxification of pyridafol occurred mainly by glucosidic conjugation, yielding in the CL9673-N-glucoside and CL9673-O-glucoside. Further degradation led to highly polar metabolites, before the radioactivity was incorporated into the carbon pool of natural plant constituents.

Broccoli

A metabolism study with broccoli (variety: Italica) under combined greenhouse and outdoor conditions was performed with [4,5-pyridazine-^{14}C]-radiolabelled pyridate (Zohner, 1988, PYRIDATE_014). Broccoli plants received one foliar application at the 2–3 leaf stage using a rate of 1.8 kg ai/ha. Plant samples were taken at 0 (immediately after the treatment), 14, 45, 73, 94 and 108 DAT.

The homogenized sample material was extracted once with acetone, followed by four times with acetone/water (8:2). The radioactivity of each extract was determined by liquid scintillation counting (LSC), while for the separation of the metabolites the extracts were combined and analysed by thin layer chromatographs (TLC). Identification was accomplished by co-chromatography with non-radiolabelled standards. Conjugates were hydrolysed with 2 mol/L hydrochloric acid and β-glucosidase. The post extraction solids (PES) were subjected to combustion and determination of the radioactivity by LSC, as well as to hydrolysis with 2 mol/L acetonic hydrochloric acid in order to characterize the PES. The unextracted residue was also characterized by means of a cell wall fractionation using α-amylase, protease, pectinase and dioxane/2 mol/L hydrochloric acid (9:1).

The TRR and the extracted radioactivity are shown in Table 3. The TRR was highest at 20 mg eq/kg in leaves immediately after treatment and declined to 0.011 mg eq/kg in samples taken at 108 DAT (days after treatment). At 0.009 mg eq/kg, similar levels were found in the combined edible samples from 75, 96 and 108 DAT. Extraction with acetone and acetone/water (8:2) liberated at least 75 percent TRR, except for stems and roots where the extractability was lower with 55 percent and 34 percent TRR, respectively.

Table 3 Total radioactive residues and extracted residues after one foliar application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate to broccoli, expressed as pyridafol equivalents

Matrix	DAT	TRR mg eq/kg	Extract mg eq/kg (% TRR)	PES mg eq/kg (% TRR)
Leaves	0	20	20 (100)	0.05 (0.31)
	14	6.1	5.7 (94)	0.35 (6.3)
	45	0.31	0.28 (86)	0.03 (14)
	108	0.011	0.008 (75)	0.003 (25)

Matrix	DAT	TRR mg eq/kg	Extract mg eq/kg (% TRR)	PES mg eq/kg (% TRR)
Edible parts (flowers)	75, 96, 108[1]	0.009	0.008 (84)	0.001 (16)
Stems	108	0.014	0.008 (55)	0.007 (45)
Roots	108	0.093	0.032 (34)	0.061 (66)

Notes:

[1] Edible parts were harvested at three intervals and combined.

The identification/characterization of radioactivity in broccoli leaves (0, 14, 45 DAT) and root is presented in Table 4. Characterization of the broccoli flowers was not performed due to their low radioactivity in the extract. Parent pyridate was a major identified residue in leaf samples from 0 and 14 DAT, accounting for 42–60 percent TRR (2.6–12 mg eq/kg), while in leaves from 45 DAT and in the root parent pyridate was not detected at all. As a major metabolite pyridafol was detected as well in in leaf samples from 0 and 14 DAT, accounting for 7.4–18 percent TRR (0.45–3.5 mg eq/kg). Additionally, the N- and O-glucoside of pyridafol was detected in leaf samples from 14 and 45 DAT accounting for 19–25 percent TRR (0.06–1.5 mg eq/kg). Two unknown components, M6 and M7, were detected at significant levels in leaf samples from 45 DAT and in the roots. Fractionation of the unextracted residue from selected leaf samples (14 or 45 DAT) demonstrated that the remaining radioactivity could be assigned to natural constituents such as starch, proteins, pectin and lignin.

Table 4 Summary of identified/characterized residues in broccoli leaf samples after one foliar application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate, expressed as pyridafol equivalents

Fraction	Leaves						Roots	
	0 DAT		14 DAT		45 DAT		108 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	20	100	6.1	100	0.31	100	0.093	100
Solvent extract	20	100	5.7	94	0.28	86	0.032	34
Pyridate	12	60	2.6	42	0.01	3.2	-	-
Pyridafol	3.5	18	0.45	7.4	0.01	3.2	-	-
Pyridafol-N- and O-glucoside	-	-	1.5	25	0.06	19	-	-
Unknowns M6 + M7	-	-	0.43	7.1	0.07	23	0.01	11
Polar fraction (“start”)	-	-	0.55	9.0	0.11	36	0.02	22
Total identified	16	78	4.6	74	0.08	25	-	-
Total characterized	-	-	0.98	16	0.18	59	0.03	33
Unextracted	0.05	0.31	0.35	6.3	0.03	14	0.061	66
Total	16	78	5.9	96	0.29	98	0.091	99

Maize

A metabolism study with maize (variety: not stated) under combined greenhouse and outdoor conditions was performed with [4,5-pyridazine-^{14}C]-radiolabelled pyridate (Zohner, 1988, PYRIDATE_015). Maize plants received one foliar application at BBCH 16–17 using a rate of 1.8 kg ai/ha. Plant samples were taken at 0 (immediately after the treatment), 14, 45, 90 and 118 DAT. Over daytime, plants were exposure to natural environmental conditions and kept in greenhouse overnight.

The homogenized sample material was extracted with acetone (1st and 5th extraction) and with acetone/water (8:2) (2nd to 4th extraction). The radioactivity of each extract was determined by LSC, while for the separation of the metabolites the extracts were combined and analysed by TLC. Identification was accomplished by co-chromatography with non-radiolabelled standards. Conjugates were hydrolysed with 2 mol/L hydrochloric acid and β-glucosidase. The PES was subjected to combustion and determination of

the radioactivity by LSC, as well as to hydrolysis with 2 mol/L acetonic hydrochloric acid in order to characterize the PES. The unextracted residue was also characterized by means of a cell wall fractionation using α-amylase, protease, pectinase and dioxane/2 mol/L hydrochloric acid (9:1).

The TRR and the extracted radioactivity are shown in Table 5. The TRR was highest with 17 mg eq/kg in leaves immediately after treatment and declined to 0.5 mg eq/kg in treated leaf samples taken at 108 DAT. In newly grown plant parts, the radioactivity was significantly lower at maximum 0.04 mg eq/kg, while in maize grain it was even lower at around the LOQ. Extractability of the residue with acetone and acetone/water (8:2) was good, liberating at least 75 percent TRR for the treated leaves from 0, 14 and 45 DAT. It was lower at later sampling time points, newly grown plant parts and grains.

Table 5 Total radioactive residues and extracted residues after one foliar application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate to maize, expressed as pyridafol equivalents

Matrix	DAT	TRR mg eq/kg	Extract mg eq/kg (% TRR)	PES mg eq/kg (% TRR)
Treated leaves	0	17	17 (100)	0.05 (0.29)
	14	2.8	2.3 (82)	0.53 (19)
	45	1.0	0.76 (75)	0.26 (25)
	90	0.48	0.18 (38)	0.30 (62)
	118	0.50	0.12 (24)	0.38 (76)
New Plant Parts	45	0.02	0.01 (50)	0.01 (50)
	90	0.03	0.01 (33)	0.01 (33)
	118	0.04	0.01 (24)	0.03 (75)
Grains	118	<0.01	<0.01 (32)	0.01 (68)

The identification/characterization of radioactivity in treated maize leaves (0, 14, 45, 90 and 108 DAT) is presented in Table 6. Characterization of the residue in all other matrices was not performed due to their low radioactivity in the extracts. Parent pyridate was a major identified residue in leaf samples from 0 and 14 DAT, accounting for 11–82 percent TRR (0.31–17 mg eq/kg), while in samples from later time points parent pyridate was around the LOD or lower. Pyridafol was detected as a major metabolite in treated leaf samples from 0 and 14 DAT, accounting for 15–16 percent TRR (0.44–2.6 mg eq/kg), but was significantly lower in samples from later time points. Additionally, the N- and O-glucoside of pyridafol was detected in treated leaf samples from 14 and 45 DAT accounting for 5.4–7.0 percent TRR (0.07–0.15 mg eq/kg). Two unknown components, M7 and M8, were detected in leaf samples from 14, 45 and 90 DAT at significant levels of 15–38 percent TRR (0.07–0.69 mg eq/kg). Fractionation of the unextracted residue from a 90 DAT leaf sample demonstrated that the remaining radioactivity could be assigned to natural constituents such as starch, proteins, pectin and lignin. The proposed metabolic pathway of pyridate is shown in Figure 2.

Table 6 Summary of identified/characterized residues in maize leaf samples after one foliar application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate, expressed as pyridafol equivalents

Fraction	Treated leaves									
	0 DAT		14 DAT		45 DAT		90 DAT		108 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	17	100	2.8	100	1.0	100	0.48	100	0.50	100
Solvent extract	17	100	2.3	82	0.76	75	0.18	38	0.12	24
Pyridate	14	82	0.31	11	0.01	1.0	<LOD	1.5	<LOD	1.1
Pyridafol	2.6	15	0.44	16	0.02	2.0	<LOD	1.1	<LOD	2.1
Pyridafol-N- and O-glucoside	-	-	0.15	5.4	0.07	7.0	<LOD	4.1	<LOD	1.8
Unknowns M7 + M8	-	-	0.69	25	0.38	38	0.07	15	0.03	6

Fraction	Treated leaves									
	0 DAT		14 DAT		45 DAT		90 DAT		108 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
ROI 2-5[1]	0.14	1.0	0.60	21	0.23	23	<LOD	14	<LOD	11
Polar fraction ("start")	0.16	0.8	0.07	2.5	0.04	4.0	<LOD	2.6	0.01	2
Total identified	16	97	0.90	32	0.10	10	0	7	0	5
Total characterized	0.30	2	1.4	49	0.65	65	0.07	31	0.04	19
Unextracted	0.05	0.29	0.53	19	0.26	25	0.30	62	0.38	76
Total	17	99	2.8	100	1.0	100	0.37	100	0.42	100

Notes:

[1] Sum of an unresolved region.

A second study with maize (variety: LG 9) under outdoor conditions was performed with [4,5-pyridazine-^{14}C]-radiolabelled pyridate (Ellgehausen, 1987, PYRIDATE_016). Plants received one foliar application at BBCH 14–15 using a rate of 1.73 kg ai/ha. Plant samples were taken at 0 (immediately after the treatment), 14, 45, 90 and 148 DAT.

The TRR in the homogenized sample material from all sampling time points was determined by combustion followed by LSC. Thereafter, aliquots of the homogenized treated leaves from 14, 45 and 148 DAT were sequentially extracted with (1) acetone, (2) acetone/water (8:2), (3) acetone in a Soxhlet overnight and (4) with acetone/0.1 mol/L hydrochloric acid (8:2). The radioactivity of each extract was determined by LSC, while for the characterization of the metabolites the extracts from steps 1–3 were combined for the treated leaves from 14 DAT and analysed by TLC and GC-FID. Identification was accomplished by co-chromatography with non-radiolabelled standards. Extracts from the 14 and 45 DAT samples were also hydrolysed with 2 mol/L and 4–6 mol/L hydrochloric acid. The PES was subjected to combustion and determination of the radioactivity by LSC.

The TRR and the extracted radioactivity are shown in Table 7. The TRR was highest with 168 mg eq/kg in leaves immediately after treatment and declined to 0.27 mg eq/kg at 148 DAT. At harvest, the TRR in maize grain was equal to 0.014 mg eq/kg. Extractability of the residue was good for leaves from 14 DAT, liberating at least 72 percent TRR, but was lower at later sampling time points. Maize grain was not extracted due to the low level of radioactivity in the samples.

Table 7 Total radioactive residues and extracted residues after one foliar application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate to maize, expressed as pyridate equivalents

Matrix	DAT	TRR mg eq/kg[1]	Extract mg eq/kg[1] (% TRR)	PES mg eq/kg[1] (% TRR)
Treated leaves	0	168	Not extracted	
	14	27	24 (89)	3.1 (11)
	45	0.58	0.28 (48)	0.30 (52)
New plant parts (leaves and stems)	14	0.73	0.52 (72)	0.21 (28)
	45	0.03	0.01 (44)	0.02 (56)
	90	0.015	Not extracted	
Treated leaves and new plant parts	148	0.27	0.08 (30)	0.19 (70)
Stems	148	0.03	0.02 (61)	0.01 (39)
Grains	148	Not extracted		
Husks/Stalks	148	Not extracted		

The identification/characterization of radioactivity was only performed in 14 DAT treated maize leaves and in new plant parts (leaves and stems) (Table 8). For the new plant parts,

identification/characterization was only reported for the water-soluble radioactivity, while the dichloromethane-soluble fraction was not further analysed. This was justified by a low level of radioactivity and a high amount of natural constituents in the dichloromethane-soluble fraction, despite having a similar level of radioactivity as the water-soluble fraction. Hence, a full mass balance could not be calculated. Parent pyridate was a minor residue in treated leaves and in new plant parts, accounting for 1.7–3.0 percent TRR (0.01–0.80 mg eq/kg). Pyridafol was detected as a minor metabolite only in treated leaf samples, accounting for 5.3 percent TRR (1.4 mg eq/kg). With M2 and M3 two major unknown metabolites were detected at 20–41 percent TRR (5.3–11 mg eq/kg). Further characterization of both metabolites by acid hydrolysis demonstrated that only M2 was susceptible and was assumed to be a conjugated derivative of pyridafol with an additional hydroxyl group. Unknown M3, not susceptible to acid hydrolysis, was assumed to be a neutral or weakly acidic metabolite susceptible to alkylating reagents. Additional unknown components were metabolite M1 and M5, ranging between 5.2–9.5 percent TRR (1.4–2.5 mg eq/kg). The proposed metabolic pathway of pyridate is shown in Figure 2.

Table 8 Distribution of radioactivity found in treated leaves of maize plants after one foliar application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate, expressed as pyridate equivalents

Fraction	Treated leaves		New plant parts (leaves and stems)	
	14 DAT		14 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	27	100	0.73	100
Solvent extract (steps 1-3)	24	89	0.52	72
Pyridate	0.80	3.0	0.01[2]	1.7[2]
Pyridafol	1.4	5.3	no data	
Unknown M1	2.5	9.5	0.17[2]	22[2]
Unknown M2	5.3	20	no data	
Unknown M3	11[1]	41[1]	Not detected	
Unknown M5	1.4	5.2	0.05[2]	7.0[2]
Total identified	2.2	8.3	0.01[2]	1.7[2]
Total characterized	20	76	0.17[2]	22[2]
Unextracted	3.1	11	0.21	28
Total	25	95	0.39[2]	52[2]

Notes:

[1] Sum of M3 found in the water- and dichloromethane phase.

[2] Individual components found in the water phase, only. The dichloromethane phase was not reported. Therefore a full mass balance could not be conducted.

Peanut

A metabolism study with peanut (variety: not stated) under combined greenhouse and outdoor conditions was performed with [4,5-pyridazine-^{14}C]-radiolabelled pyridate (Zohner, 1988, PYRIDATE_017). Peanut plants received one foliar application when the plants reached a height of 12.7 cm, using a rate of 3.6 kg ai/ha. Plant samples were taken at 0 (immediately after the treatment), 14, 45 and 219 DAT.

The homogenized sample material was extracted once with acetone, followed by four times with acetone/water (8:2). The radioactivity of each extract was determined by LSC, while for the separation of the metabolites the extracts were combined and analysed by TLC. Identification was accomplished by co-chromatography with non-radiolabelled standards. Conjugates were hydrolysed with 2 mol/L hydrochloric acid and β-glucosidase. PES were subjected to combustion and determination of the radioactivity by LSC, as well as to hydrolysis with 2 mol/L acetonic hydrochloric acid in order to characterize the PES. The

unextracted residue was also characterized by means of a cell wall fractionation using α-amylase, protease, pectinase and dioxane/2 mol/L hydrochloric acid (9:1).

The TRR and the extracted radioactivity are shown in Table 9. The TRR was highest with 59 mg eq/kg in leaves immediately after treatment and declined to 0.22 mg eq/kg in treated leaf samples taken at 219 DAT. In the nut meat, radioactivity was low at 0.04 mg eq/kg. Extractability of the residue with acetone and acetone/water (8:2) was good, liberating at least 78 percent TRR from the treated leaves at 0 and 14 DAT, but was lower in all other matrices.

Table 9 Total radioactive residues and extracted residues after one foliar application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate to peanut plants, expressed as pyridafol equivalents

Matrix	Sampling interval DAT	TRR mg eq/kg	Extract mg eq/kg (% TRR)	PES mg eq/kg (% TRR)
Forage	0	59	59 (100)	0.11 (0.19)
	14	28	22 (78)	6.2 (22)
	45	10	5.3 (54)	4.9 (48)
	219	0.22	0.09 (44)	0.13 (59)
Hay	45	38	24 (64)	14 (36)
	219	1.5	0.55 (43)	0.92 (63)
Hulls	219	0.36	0.13 (38)	0.23 (64)
Nut meat	219	0.04	0.01 (33)	0.03 (75)

The identification/characterization of radioactivity in peanut forage, hay and hulls (0, 14, 45 and 219 DAT) is presented in Table 10. Characterization of the residue in the nut meat was not performed due to its low radioactivity in the extract. Parent pyridate was a major identified residue only in forage samples from 0 DAT, accounting for 86 percent TRR (51 mg eq/kg), while in all other samples levels were significantly lower. Among major metabolites, only pyridafol in 0 DAT forage samples and the sum of the N- and O-glucoside of pyridafol in 14 DAT forage samples occurred at 10 percent TRR (6.2 mg eq/kg) and 20 percent TRR (5.5 mg eq/kg), respectively. Further, two unknown components, M6 and M7, were detected as major residues in basically all samples, except forage, from 0 DAT. Further characterization of both metabolites by acid hydrolysis as well as treatment with β-glucosidase demonstrated that only M7 was susceptible and was assumed to be a conjugate. A minor metabolite, only identified in the 215 DAT samples was CL 9869, accounting for up to 4.7 percent TRR (0.07 mg eq/kg). The cell wall fractionation of the unextracted residue from a 45 DAT forage sample demonstrated that the remaining radioactivity could be assigned to natural constituents such as starch, proteins, pectin and lignin. The proposed metabolic pathway of pyridate in plants is shown in Figure 2.

Table 10 Summary of identified/characterized residues in peanut forage, hay and hulls after one foliar application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate, expressed as pyridafol equivalents

Fraction	Forage								Hay				Hulls	
	0 DAT		14 DAT		45 DAT		219 DAT		45 DAT		215 DAT		215 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	59	100	28	100	10	100	0.22	100	38	100	1.5	100	0.36	100
Solvent extract	59	100	22	78	5.3	54	0.09	41	24	64	0.55	37	0.13	36
Pyridate	51	86	0.83	3.0	0.12	1.2	0.01	4.6	0.45	1.2	0.03	2.0	0	0
Pyridafol -OMe	0	0	0	0	0	0	0.01	4.6	0	0	0.07	4.7	0.01	2.8
Pyridafol	6.2	10	0.77	2.8	0.04	0.39	0	0	0.45	1.2	0	0	0	0
Pyridafol-N- and O-glucoside	0	0	5.5	20	0.35	3.4	0.01	4.6	1.1	3.2	0.06	4.0	0.02	5.6
Unknowns M6 + M7	0	0	5.4	19	1.4	14	0.02	9.1	6.9	18	0.15	10	0.04	11

Fraction	Forage								Hay				Hulls	
	0 DAT		14 DAT		45 DAT		219 DAT		45 DAT		215 DAT		215 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
ROI 2-5[1]	0	0	4.8	17	1.4	14	0.03	14	7.3	19	0.17	11	0.04	11
Polar fraction ("start")	1.3	2.2	3.7	13	1.4	14	0.01	4.6	7.8	21	0.05	3.3	0.02	5.6
Total identified	57	96	7.1	25	0.51	5	0.03	14	2.0	5.5	0.16	11	0.03	8.3
Total characterized	1.3	2.2	14	50	4.2	42	0.06	27	22	58	0.37	25	0.10	28
Unextracted	0.11	0.19	6.2	22	4.9	48	0.13	59	14	36	0.92	63	0.23	64
Total	58	98	27	97	10	95	0.22	100	38	99	1.5	98	0.36	100

Notes:

[1] Sum of an unresolved region.

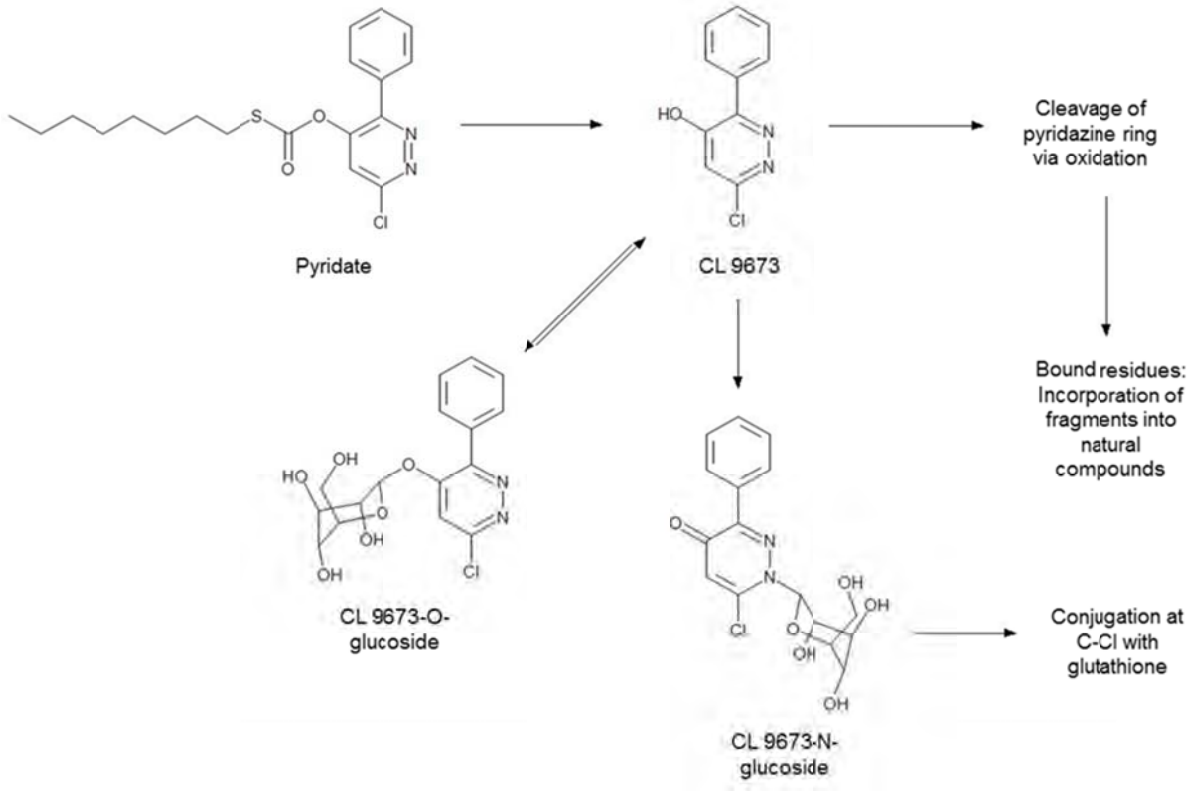


Figure 2 Proposed metabolic pathway of pyridate in broccoli, maize and peanut.

ENVIRONMENTA FATE

The Meeting received studies on aerobic soil degradation, soil photolysis and on the behaviour in confined rotational crops.

Aerobic soil degradation

The rate of degradation of pyridate and its metabolite pyridafol was studied in four aerobic soils using [4,5-pyridazine-^{14}C]-radiolabelled pyridate at a nominal application rate of 3.6 mg/kg oven dry soil, corresponding to 2690 g ai/ha (Morgenroth, 1995, PYRIDATE_022). One soil (Speyer 2.2 SP 211) was treated at a rate of 1.8 mg/kg, equivalent to a field application rate of 1350 g ai/ha. Soil characteristics are shown in Table 11.

Table 11 Characteristics of the soils used in study by Morgenroth (1995)

Soil	Classification (USDA)	Sand (%)	Silt (%)	Clay (%)	pH (KCl)	OC (%)	CEC (mval/kg)	MWHC (g/100 g)	C_{mic} (mg/kg)
Speyer 2.2	Sand	91.3	4.7	4.0	6.0	2.58	100	39.4	318
Speyer 2.2 SP 211	Loamy sand[a]	81.9	13.0	5.1	6.0	2.29	97	44.3	642
Auboden	Silt loam	67.3	26.5	6.2	7.5	1.80	202	47.7	294
Collombey	Sand/loamy sand	94.3	4.4	1.3	7.7	1.33	112	42.0	194
Les Evouettes	Silt loam/loam	59.4	29.3	11.3	6.1	1.40	155	55.3	364

Notes:

[a] = DIN classification

The test system was maintained in the dark at a nominal temperature of 20 ± 2 °C for 350 days (Speyer 2.2) or 96 days (all other soils). Volatile organics and CO_2 were trapped with ethanediol and 2 mol/L NaOH, respectively. Samples were taken at increasing intervals after the application.

The soil samples were sequentially extracted with acetonitrile (+0.5 percent acetic acid), acetonitrile/water (+0.5 percent acetic acid) (4:1), methanol (+0.5 percent acetic acid) and a Soxhlet extraction with acetone. Extracts were analysed by LSC for total radioactivity and by TLC and/or HPLC against reference standards to identify metabolites. The PES was further extracted with 0.5 mol/L NaOH to liberate unextracted radioactivity associated with the humin, fulvic acid and humic acid fractions. The soil remaining after the extraction was combusted followed by LSC.

Parent pyridate declined from 92–104 to <0.1–1.3 percent of applied radioactivity (AR) over the study time (Tables 12 and 13). Levels of metabolite pyridafol increased to a maximum of 72–90 percent AR over a course of 2–3 days, before declining to 3.1–13 percent AR at the end of study. Similar, levels of pyridafol-OMe peaked between days 7–64 at 3.5–6.1 percent AR Additionally, some unidentified radioactivity was detected at up to 6 percent AR. Further analysis of the bound residues from Speyer 2.2 soil after 28 and 64 days showed 4.1–7.0, 5.6–8.4, and 13–31 percent of the applied radioactivity was associated with the fulvic acid, humic acid and humin fractions, respectively.

Table 12 Biotransformation of [4,5-pyridazine-^{14}C]-radiolabelled pyridate in Speyer soil, expressed as percentage of applied radioactivity

Degradate	Sampling time (days)										
	0	1	2	3	7	15	28	64	126	252	350
Speyer 2.2											
Pyridate	104	53	34	23	14	12	1.9	5	5.2	3.8	1.3
Pyridafol	-	48	59	72	62	53	46	29	14	8.2	6.2
Pyridafol-OMe	-	<0.1	<0.1	0.3	5.7	4.9	2.4	3.4	4.3	3.8	2.7
Unknown M1	-	<0.1	<0.1	<0.1	<0.1	<0.1	6	1.2	0.8	2.2	2
Unknown M2	-	<0.1	<0.1	<0.1	<0.1	<0.1	2.1	1.3	1.9	1.7	1.5
Total extracted radioactivity	**104**	**102**	**95**	**100**	**88**	**74**	**69**	**50**	**34**	**25**	**18**
CO_2	-	<0.1	0.2	0.9	0.3	0.5	1.7	3.9	5.4	8.1	9.7
Volatiles organics	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unextracted	0.5	1.3	3.5	4.4	10	16	22[1]	46[2]	53	60	67
Total	**105**	**103**	**99**	**105**	**99**	**90**	**93**	**100**	**93**	**93**	**95**
Speyer 2.2 (SP 211)											
Pyridate	100	32	18	14	8.7	1.9	3.7	2.6	1.8	0.6	0.6
Pyridafol	2.1	67	75	73	64	52	24	8.7	4.7	4.7	3.1
Pyridafol-OMe	-	<0.1	<0.1	1.6	2.4	4.6	6.1	6	4.4	3	2.2

Degradate	Sampling time (days)										
	0	1	2	3	7	15	28	64	126	252	350
Speyer 2.2											
Unknown M1	-	<0.1	<0.1	1.4	1.8	1.9	1.7	1.8	1.9	1.6	1.5
Unknown M2	-	<0.1	<0.1	<0.1	0.9	2.4	1.4	1.2	0.8	0.6	0.6
Unknown M3	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.2
Unknown M4	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.2	0.2
Total extracted radioactivity	**102**	**102**	**97**	**95**	**85**	**72**	**44**	**26**	**18**	**15**	**12**
CO_2	-	0.2	0.6	1	1.6	4.9	11	18	23	26	26
Volatiles organics	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unextracted	1	3.4	5.9	9.5	16	26	43	49	48	52	56
Total	**103**	**106**	**104**	**106**	**103**	**103**	**98**	**92**	**89**	**93**	**94**

Notes:

[1] Consisting of 4.1 percent fulvic acid, 5.6 percent humin acid and 13 percent humin.

[2] Consisting of 7.0 percent fulvic acid, 8.4 percent humin acid and 31 percent humin.

Table 13 Biotransformation of [4,5-pyridazine-^{14}C]-radiolabelled pyridate in various soils, expressed as percentage of applied radioactivity

Degradate	Sampling time (days)							
	0	1	2	3	7	28	64	98
Auboden soil								
Pyridate	92	17	5.7	6.5	2.6	13	0.5	0.3
Pyridafol	11	82	91	86	81	31	19	13
Pyridafol-OMe	-	<0.1	<0.1	<0.1	<0.1	3.5	2.6	2.9
Unknown M1	-	<0.1	0.9	<0.1	2.0	2.1	1.0	1.3
Unknown M2	-	<0.1	<0.1	<0.1	<0.1	2.1	0.9	2.0
Total extracted radioactivity	**103**	**101**	**101**	**96**	**92**	**58**	**31**	**24**
CO_2	-	0.2	0.5	0.3	2.6	9.4	14	20
Volatiles organics	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unextracted	1.3	2.1	4.2	5.6	11	31	50	56
Total	**104**	**103**	**106**	**102**	**106**	**98**	**95**	**99**
Collombey soil								
Pyridate	92	13	10	6.2	5.5	4.6	0.9	0.5
Pyridafol	16	88	83	83	68	30	3.6	3.9
Pyridafol-OMe	-	<0.1	<0.1	<0.1	<0.1	<0.1	0.6	0.6
Unknown M1	-	<0.1	1.6	4.7	2.7	0.5	2.4	1.2
Unknown M2	-	<0.1	<0.1	<0.1	<0.1	1.6	0.4	0.3
Total extracted radioactivity	**108**	**102**	**99**	**96**	**77**	**40**	**9.7**	**9.5**
CO_2	-	0.2	0.5	0.8	3.7	16	26	26
Volatiles organics	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	n/d
Unextracted	0.5	2.4	5.4	7.6	14	32	52	52
Total	**108**	**105**	**105**	**104**	**95**	**88**	**88**	**87**
Les Evouettes soil								
Pyridate	94	28	4.7	3.8	2.4	<0.1	0.9	<0.1
Pyridafol	7.4	72	90	88	89	46	14	7.2
Pyridafol-OMe	-	<0.1	<0.1	<0.1	<0.1	<0.1	5.9	1.8
Unknown M1	-	<0.1	<0.1	<0.1	1.5	3.6	1.4	2.0
Unknown M2	-	<0.1	<0.1	<0.1	<0.1	<0.1	0.9	2.7
Total extracted radioactivity	**102**	**101**	**99**	**98**	**100**	**57**	**28**	**18**
CO_2	-	0.2	0.2	0.2	0.8	12	17	19
Volatiles organics	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unextracted	1.4	2.0	3.5	6.2	11	34	49	60
Total	**103**	**103**	**102**	**105**	**113**	**102**	**93**	**96**

Calculated $DegT_{50/90}$ values for pyridate and its metabolite pyridafol are summarised in Table 14. Pyridate was rapidly degraded in soil under aerobic conditions with normalised $DegT_{50}$ values, ranging from 0.3 to 3.3 days. Normalised $DegT_{50}$ values for the major metabolite pyridafol (pyridafol) ranged from 17 to 43 days.

Table 14 Calculated $DegT_{50/90}$ values for [4,5-pyridazine-^{14}C]-radiolabelled pyridate and its metabolite pyridafol in aerobic soils

Soil	Pyridate				Pyridafol			
	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Kinetics	Normalised $DegT_{50}$ (days)	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Kinetics	Normalised $DegT_{50}$ (days)
Speyer 2.2[a]	1.3	4.3	SFO	nc	47	155	SFO→SFO	43
	0.9	11	FOMC	3.3[b]	n/a			
Speyer 2.2 SP 211[a]	0.7	2.4	SFO	nc	17	56	SFO→SFO	17
	0.4	4.6	FOMC	1.4[b]	n/a			
Auboden	0.4	1.4	SFO	0.3	22	72	SFO→SFO	18
Collombey	0.4	1.2	SFO	0.4	17	55	SFO→SFO	17
	<0.1	1.5	FOMC	ns	n/a			
Les Evouettes	0.5	1.7	SFO	0.5	24	79	SFO→SFO	22

Notes:
nc = Not calculated.
ns = Not significant (no reliable fit).
[a] Including data up to 126 DAT.
[b] Based on pseudo SFO $DegT_{50}$ (i.e. FOMC $DegT_{50}$/3.32).

In a second study, the degradation of the pyridate metabolite pyridafol was investigated in four soils under aerobic conditions. Soils were treated with [4,5-pyridazine-^{14}C]-radiolabelled pyridafol at a nominal application rate of 2.9 mg/kg dry soil, corresponding to a field application rate of 1800 g ai/ha (Zohner, 1985, PYRIDATE_023). Soil characteristics are shown in Table 15.

Table 15 Characteristics of the soils used in study by Zohner, 1985

Soil	Classification (USDA)	Sand (%)	Silt (%)	Clay (%)	pH	OC (%)[a]	CEC (mval/kg)
Auboden	Silty loam	11.8	79.1	9.1	7.5	1.68	150
Pararendzina	Silty loam	21.3	61.3	17.4	7.2	2.61	303
Rendzina	Silty loam	14.0	58.3	27.7	5.4	1.74	251
Ranker	Sandy loam	67.3	20.9	11.8	5.8	0.99	104

Notes:
[a] OC = OM/1.724.

Test systems were maintained in the dark at a nominal temperature of 18–23 °C for 70 days. Since in a preliminary study no volatile ^{14}C-compounds were detected, only CO_2 was trapped with 2 mol/L KOH. Samples were taken at 0, 8, 16, 23, 30 and 70 days after application.

The soil samples were sequentially extracted with acetone followed by water saturated n-butanol in a Soxhlet apparatus. Liquid samples were analysed by LSC for total radioactivity and by TLC against reference standards to identify metabolites. The soil remaining after extraction was combusted followed by LSC.

Pyridate metabolite pyridafol declined in all soils from 83–94 percent AR to 21–28 percent AR over 70 days while levels of metabolite pyridafol-O-methyl generally increased at up to 16 percent AR

(Table 16). Similar, levels of $^{14}CO_2$ peaked at day 70, accounting for up to 25 percent AR. Unextracted radioactivity was determined at day 70 only and accounted for 31–37 percent AR (Table 16).

Table 16: Metabolism of pyridate metabolite pyridafol, expressed as percentage of applied radioactivity in soils (Zohner, 1985)

Degradate	Sampling time (days)					
	0	8	16	23	30	70
Auboden soil						
Pyridafol	93	84	69	61	49	22
Pyridafol-OMe	-	1.3	1.2	3.3	3.4	3.9
Unidentified degradates	4.3	4.6	5.1	6.8	4.0	4.9
Total extracted radioactivity	**103**	**93**	**83**	**73**	**60**	**36**
CO_2	0.7	2.7	6.6	9.6	13	25
Unextracted	-	-	-	-	-	34
Total	**n/a**	**n/a**	**n/a**	**n/a**	**n/a**	**94**
Pararendzina soil						
Pyridafol	94	74	71	59	49	21
Pyridafol-OMe	-	1.8	3.7	4.7	4.9	5.6
Unidentified degradates	5.8	6.4	5.7	3.1	5.6	5.7
Total extracted radioactivity	**106**	**86**	**84**	**70**	**63**	**37**
CO_2	0.0	0.4	1.6	4.3	7.7	14
Unextracted	-	-	-	-	-	33
Total	**n/a**	**n/a**	**n/a**	**n/a**	**n/a**	**84**
Rendzina soil						
Pyridafol	83	79	66	56	45	27
Pyridafol-OMe	-	6.3	11	13	16	16
Unidentified degradates	6.1	4.9	5.3	5.7	8.4	4.0
Total extracted radioactivity	**98**	**95**	**87**	**79**	**75**	**52**
CO_2	0.4	1.2	2.5	3.7	5.1	10
Unextracted	-	-	-	-	-	31
Total	**n/a**	**n/a**	**n/a**	**n/a**	**n/a**	**94**
Ranker soil						
Pyridafol	84	78	65	59	50	28
Pyridafol-OMe	0.5	4.5	9.3	10	12	10
Unidentified degradates	4.5	4	4.7	5.5	6	6.2
Total extracted radioactivity	**104**	**90**	**83**	**77**	**71**	**49**
CO_2	0.6	2.1	4.2	5.7	7.3	13
Unextracted	-	-	-	-	-	37
Total	**n/a**	**n/a**	**n/a**	**n/a**	**n/a**	**98**

Calculated $DegT_{50/90}$ values for pyridafol and pyridafol-OMe are summarised in Table 17. Normalised $DegT_{50}$ values for pyridafol) ranged from 20 to 28 days. Normalised $DegT_{50}$ values for pyridafol-OMe ranged from 14 to 25 days. For one soil (Auboden), no reliable $DegT_{50}$ value could be derived.

Table 17 Calculated $DegT_{50/90}$ values for [4,5-pyridazine-^{14}C]-radiolabelled pyridafol and pyridafol-OMe in aerobic soils

Soil	Pyridafol				Pyridafol-OMe			
	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Kinetics	Normalised $DegT_{50}$ (days)	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Kinetics	Normalised $DegT_{50}$ (days)
Auboden	33	110	SFO	20	62	205	SFO→SFO	nc
Pararendzina	33	111	SFO	20	40	134	SFO→SFO	25
Rendzina	39	128	SFO	24	30	101	SFO→SFO	19

Soil	Pyridafol				Pyridafol-OMe			
	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Kinetics	Normalised $DegT_{50}$ (days)	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Kinetics	Normalised $DegT_{50}$ (days)
Ranker	42	140	SFO	28	21	70	SFO→SFO	14

In a third study, the degradation of the pyridate metabolite pyridafol-OMe (CL 9869) was investigated in three soils under aerobic conditions. Soils were treated with [4,5-pyridazine-^{14}C]-radiolabelled pyridafol-OMe at a nominal application rate of 0.14 mg/kg dry soil, corresponding to a field application rate of 107 g ai/ha (Mewes, 1996, PYRIDATE_024). Soil characteristics are shown in Table 18.

Table 18 Characteristics of the soils used in study by Mewes, 1996

Soil	Gramastetten	Flaach	Feldkirchen
USDA Classification	Sandy loam	Sandy clay loam	Sandy loam
Clay (< 0.002 mm) (%)	16	25	10
Silt (0.002 - 0.05 mm) (%)	22	26	23
Sand (> 0.05 - 2 mm) (%)	62	49	67
Water content at 1/3 bar w/w	18.7	16.5	13.5
Water content at pF = 2.2 w/w	21.0	18.5	14.7
Bulk density (g/mL)	1.01	1.07	1.05
Microbial biomass (µg ATP/kg soil)	359	153	54
pH (H_2O / KCl / $CaCl_2$)	6.2 / 5.4 / 5.6	8.0 / 7.1 / 7.3	8.1 / 7.3 / 7.4
Organic carbon (%)	2.6	1.3	1.1
CEC (mmol/z/kg soil)	135	122	54

Test systems were maintained in the dark at a nominal temperature of 20±1 ˚C for 129 days. Volatile organics and CO_2 were trapped with ethanediol and 0.25 or 2.5 mol/L NaOH, respectively. Samples were taken at 0, 2, 4, 8, 18, 35 and 64 days after application for all soils and additionally at 88 and 129 days for Gramastetten soil.

The soil samples were sequentially extracted with acetone/water (8:2) or methanol/water (8:2) followed by acetone/0.2 mol/L HCl (1:1), acetone/1 mol/L HCl (1:1) and water. Extracts from the first and second extraction were further cleaned up by SPE on a C18 cartridge. Liquid samples were analysed by LSC for total radioactivity and by TLC and HPLC against reference standards to identify metabolites. The soil remaining after extraction was combusted followed by LSC.

Pyridate metabolite pyridafol-OMe declined in all soils from 90–98 percent AR to 4.7–7.2 percent AR over the study time, while levels of metabolite pyridafol generally increased up to 2.7–6.3 percent AR at day 8, before decreasing to 0.2–1.5 percent AR at the end of the study (Table 19). Levels of $^{14}CO_2$ increased over the study time, accounting for up to 12–43 percent AR. Similar, unextracted radioactivity increased as well, accounting for 35–45 percent AR at the end of the study.

Table 19 Metabolism of pyridate metabolite pyridafol-OMe, expressed as percentage of applied radioactivity in soils (Mewes, 1996)

Degradate	Sampling time (days)								
	0	2	4	8	16	32	64	90	120
Gramastetten soil									
Pyridafol-OMe	98	89	81	73	33	18	12	9.4	7.2
Pyridafol	<0.1	2.2	3.2	5.5	3.9	2.3	1	1.4	1.5
Total extracted radioactivity	**99**	**98**	**90**	**90**	**75**	**60**	**60**	**56**	**53**
CO_2	-	<0.1	0.2	0.5	1.9	4.1	7.5	9.4	12
Volatiles organics	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

Degradate	Sampling time (days)								
	0	2	4	8	16	32	64	90	120
Gramastetten soil									
Unextracted	1.5	4.2	11	15	30	36	36	36	35
Total	**100**	**102**	**101**	**105**	**107**	**100**	**103**	**102**	**100**
Flaach soil									
Pyridafol-OMe	90	87	77	67	33	15	5.4		
Pyridafol	<0.1	0.2	0.1	2.7	0.2	0.5	0.2		
Total extracted radioactivity	94	93	88	81	47	30	17		
CO_2	-	<0.1	0.2	0.6	2.6	8.9	24		
Volatiles organics	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1		
Unextracted	1.6	4.0	11	20	49	59	45		
Total	96	97	100	101	98	98	85		
Feldkirchen soil									
Pyridafol-OMe	95	93	82	69	38	11	4.7		
Pyridafol	<0.1	0.3	0.6	6.3	5.7	1.4	0.3		
Total extracted radioactivity	**100**	**93**	**90**	**87**	**55**	**20**	**9.4**		
CO_2	-	0.1	0.2	1.1	6.1	24	43		
Volatiles organics	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1		
Unextracted	1.0	3.2	5.5	13	38	46	35		
Total	**101**	**96**	**96**	**102**	**99**	**90**	**87**		

The calculated $DegT_{50/90}$ values for pyridafol-OMe (CL 9869) are summarised in Table 20. Pyridafol-OMe degraded rapidly in the test soils under aerobic conditions with SFO $DegT_{50}$ values ranging from 12 to 13 days.

Table 20 Calculated $DegT_{50/90}$ values for [4,5-pyridazine-^{14}C]-radiolabelled pyridafol-OMe in aerobic soils

Soil	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Kinetics
Gramastetten	13.0	43.1	SFO
	12.0	60.5	FOMC[a]
	11.9	58.4	DFOP[a]
Flaach	12.6	41.9	SFO
Feldkirchen	12.0	39.9	SFO

Notes:

[a] No reliable fit (not significant).

Soil photolysis

The soil surface photolytic behaviour of pyridate was investigated in a sandy loam soil using [4,5-pyridazine-^{14}C]-radiolabelled pyridate at a nominal application rate of 14 mg/kg, equivalent to 2000 g ai/ha. (Van Dijk & Baranowski, 1992, PYRIDATE_025). Soil characteristics are shown in Table 21.

Table 21 Characteristics of the soil used in study by Van Dijk & Baranowski, 1992

Soil	Classification (USDA)	Sand (%)	Silt (%)	Clay (%)	pH[a]	OC[b] (%)	CEC (mval/kg)	MWHC (g/100 g)
Ripperdan	Sandy loam	72.0	23.0	5.0	5.8	1.8	53	27.1

Soil samples were prepared on glass plates (~1 mm thick) and subjected to intermittent irradiation (12 hours light/dark cycles) for 31 days at 22 ± 2 °C using a xenon irradiation source with filters to eliminate wavelengths of 290 nm. Dark control samples were prepared in parallel. Volatile

organics and CO_2 were trapped with ethanediol and NaOH (molarity not specified), respectively. The irradiated and non-irradiated soil samples were analysed at 0, 2, 4, 8, 17 and 31 days.

The soil samples were extracted twice with acetonitrile/acetic acid (99.5+0.5), once with methanol/acetic acid (99.5+0.5) and once with methanol/0.5 percent acetic acid in water (8:2). Additionally samples were extracted exhaustively with acetonitrile/acetic acid (99.5+0.5) by refluxing for 16 hours. Extracts were analysed by LSC and TLC to determine the radioactivity and metabolite pattern, respectively. The soil remaining after extraction was combusted followed by LSC.

The percentage recovery of the applied radioactivity in irradiated moist and dark soil is presented in Table 22. Parent pyridate declined from 81 percent to 1.8 percent AR over the irradiation time, while metabolite pyridafol increased from 7.8 percent to 51 percent AR at day 4, before declining to 25 percent AR at day 31. At day 31, ultimate degradation via mineralisation to CO_2 accounted for 12 percent AR and unextracted radioactivity for 27 percent AR). In the dark controls, pyridate was hydrolysed to pyridafol as well, peaking at 57 percent AR at day 31. However, from day 4 onwards, no further significant degradation of pyridate was observed and no CO2 or volatiles were detected.

Table 22 Phototransformation of [4,5-pyridazine-^{14}C]-radiolabelled pyridate, expressed as percentage of applied radioactivity, on moist irradiated soil samples and incubation in the dark

Degradate	Incubation period [days]											
	Moist irradiated soil						Incubation in the dark					
	0	2	4	8	17	31	0	2	4	8	17	31
Pyridate	81	40	19	5.6	1.4	1.8	81	33	27	51	40	26
Pyridafol	7.8	41	51	50	46	25	7.8	50	54	36	47	57
Unidentified degradates[a,b]	2.4	1.6	5.6	8.6	3.7	3.1	2.4	0.3	0.4	-	-	-
Total extracted radioactivity	93	83	76	66	55	37	93	84	81	88	87	83
Refluxing with acetonitrile	-	6.2	5.6	7.2	11	19	-	6.1	9.9	2.8	2.8	12
CO_2	-	0.2	0.6	2.0	5.7	12	-	<0.05	<0.05	<0.05	<0.05	<0.05
Volatiles organics	-	<0.05	<0.05	<0.05	<0.05	<0.05	-	<0.05	<0.05	<0.05	<0.05	<0.05
Unextracted	2.2	3.7	6.2	13	20	27	2.2	2.2	2.3	1.4	3.6	3.4
Total	95	93	89	88	92	96	95	92	93	92	94	98

Notes:

[a] Maximum individual occurrence of seven individual unidentified fractions individually accounting for <8.6 % AR in irradiated soil and < 2.4 percent AR in dark soils **Inda dark soilIn dark**

Calculated photolysis $DegT_{50}$ values for pyridate and pyridafol were 1.8 and 19 days, respectively. The results of the kinetic assessment are summarized in Table 23. Kinetic evaluation of the dark samples was not possible due to strong data scattering.

Table 23 Calculated photolysis $DegT_{50/90}$ values for [4,5-pyridazine-^{14}C]-radiolabelled pyridate and its metabolite pyridafol in soil

Substance	Conditions	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Kinetics
Pyridate	Irradiated	1.8	5.9	SFO
	Dark	No reliable fit owing to data scattering		
Pyridafol (pyridafol)	Irradiated	19	65	SFO→SFO
	Dark	No reliable fit owing to data scattering		

Confined rotational crops

A confined rotational crop study under mixed outdoor and indoor conditions was conducted with [4,5-pyridazine-^{14}C]-radiolabelled pyridate applied at a rate of 1.8 kg ai/ha to a silty loam soil (Zohner, 1985, PYRIDATE_026). After plant-back intervals (PBIs) of 28 and 56 days, the nature and level of radioactive residues were investigated in lettuce (variety Kagraner Sommer (1st rotation); Grüner Escariol (2nd rotation)), carrots (variety Nantaise) and spring barley (variety not stated). Crops were harvested from the 28 day PBI, at 97 DAT for lettuce, 133 DAT for carrots and 163 DAT for barley, while crops from the 56 day PBI were harvested at 156 DAT for lettuce, 169 DAT for carrots and 209 DAT.

Ground soil samples were combusted prior to the determination of total radioactivity by LSC, while liquid samples such as extracts were directly measured by LSC. In order to characterize and identify the radioactivity present, soil samples were exhaustively extracted firstly with acetone, followed by water saturated n-butanol in a Soxhlet apparatus, while plant samples were extracted sequentially with methanol, followed by three times with methanol/water (8:2). Conjugates present in the extracts were hydrolysed overnight with 2 mol/L HCl. For the characterization and identification of the radioactivity, TLC against reference standards was applied.

Radioactive residues in soils samples decreased from 1.1 mg eq/kg at 0 DAT to 0.28 mg eq/kg over 97 days and remained fairly constant up to the last sampling time point (Table 24).

Table 24 TRR and extractability of radioactive residues in soil samples (soil layer 0–10 cm)

Soil sample (Event)	Total	Extracted		Unextracted	
	mg eq/kg	mg eq/kg	% TRR	mg eq/kg	% TRR
0 DAT (date of application)	1.1	1.1	99	<0.02	1
28 DAT (1st PBI)	0.92	0.65	71	0.27	29
56 DAT (2nd PBI)	0.52	0.25	48	0.27	52
97 DAT (harvest lettuce, 1st PBI)[1]	0.28	0.06	22	0.22	78
133 DAT (harvest carrots, 1st PBI)	0.42	0.075	18	0.35	82
156 DAT (harvest lettuce, 2nd PBI)	0.33	0.058	18	0.27	82
163 DAT (harvest barley, 1st PBI)	0.40	0.075	19	0.33	81
169 DAT (harvest carrots, 2nd PBI)	0.31	0.054	18	0.25	82
209 DAT (harvest barley, 2nd PBI)	0.39	0.068	18	0.32	82

Notes:

[1] Soil layer 0-21 cm.

Radioactivity in plant samples was generally low, peaking in barely straw at 0.1 mg eq/kg. In edible plant matrices, the TRR was highest in barley grain at up to 0.030 mg eq/kg, while in lettuce and carrot root radioactivity remained at <0.01 mg eq/kg (Table 25). Samples from the later PBIs showed similar levels of radioactivity, often slightly higher compared to the earlier PBI. Extractability of samples from the 28 day PBI ranged between 57–83 percent TRR, except for wheat grain where the extractability was significantly lower. Extractability of the 56 day PBI samples was generally lower as well (Table 25). Due to low levels of radioactivity, lettuce, carrot and barley grain samples were not analysed further.

Table 25 TRR and extractability of radioactive residues from rotational crops after application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate to bare soil at 1.8 kg ai/ha

Plant back interval	TRR	Extracted		Unextracted	
	mg eq/kg	mg eq/kg	% TRR	mg eq/kg	% TRR
Lettuce					
28 DAT	<0.01	<0.01	62	<0.01	38
56 DAT	<0.01	<0.01	23	<0.01	77

Plant back interval	TRR mg eq/kg	Extracted		Unextracted	
		mg eq/kg	% TRR	mg eq/kg	% TRR
Carrot top					
28 DAT	0.018	0.011	60	<0.01	40
56 DAT	0.021	<0.01	47	0.011	53
Carrot root					
28 DAT	<0.01	<0.01	83	<0.01	17
56 DAT	<0.01	<0.01	59	<0.01	41
Barley grain					
28 DAT	0.025	<0.01	25	0.019	75
56 DAT	0.030	<0.01	19	0.024	81
Barley straw/stalks					
28 DAT	0.077	0.047	61	0.030	39
56 DAT	0.087	0.053	61	0.034	39
Barley straw (1-10cm)					
28 DAT	0.10	0.058	57	0.045	43
56 DAT	0.090	0.053	59	0.037	41

Characterization and identification of the radioactivity was only performed in the extracts of barley straw. While levels of parent pyridate or any other metabolite were constantly <0.01 mg eq/kg, the main portion of the extracted radioactivity was allocated to saccharides. No characterization of the unextracted straw residue was performed. Rather a comparison to primary crop metabolism studies was done, stating that in those studies the large fraction of unextracted radioactivity could be allocated to natural constituents such as cellulose, starch, proteins, lignin etc.

Animal metabolism

Metabolism studies were provided for ruminants (lactating cow and goat) and poultry (laying hens and broiler chicken) using [4,5-pyridazine-^{14}C]-radiolabelled pyridate. Metabolism in laboratory animals was evaluated by the WHO Panel of the current Meeting.

Ruminants

A metabolism study with one lactating cow was performed with [4,5-pyridazine-^{14}C]-radiolabelled pyridate (Cameron, *et al*., 1989, PYRIDATE_018). The compound was administered once at a dose of 0.3 mg/kg bw (equivalent in feed not stated) at day 1 and 14 by intrarumenal injection. After administration of the first dose, urine and faeces were collected once daily for 7 consecutive days (except urine which was collected three times within the first 24 hours). Milk was collected twice daily throughout the study. Blood samples were taken in increasing intervals within 7 days after the first administration. The animal was sacrificed approximately 6 hours after administration of the second dose and organs, tissues and body fluids were collected.

Total radioactivity in liquid samples such as urine, plasma, milk and various extracts were directly measured by LSC. Faecal, whole blood and tissue samples were subjected to combustion prior to the determination of total radioactivity by LSC. Liver and kidney samples were homogenized with methanol. Liver samples were further cleaned up on C18 cartridges. Characterization of the extracts was carried out by TLC against reference standards.

After three days, the elimination of administered radioactivity was complete. The main route occurred via urine (92 percent AR), followed by faeces (8.6 percent AR). In all other organs, tissues and body fluids, radioactivity was significantly lower. A summary of the recovered radioactivity is presented in Table 26 and Table 27.

Table 26 Cumulative excretion of radioactive residues after the first intrarumenal injection of [4,5-pyridazine-^{14}C]-radiolabelled pyridate to a lactating cow

Portion Analysed	% AR								
	Sampling Periods After First Dosing (hours)								
	0-6	0-12	0-24	0-48	0-72	0-96	0-120	0-144	0-168
Urine	24	70	82	91	92	92	92	92	92
Faces	-	-	3.2	6.3	7.6	8.1	8.3	8.5	8.6
Milk	-	-	0.15	0.16	-	-	-	-	0.16
Total	-	-	86	98	100	100	101	101	101

Table 27 Recovered radioactive residues after the second intrarumenal injection of [4,5-pyridazine-^{14}C]-radiolabelled pyridate to a lactating cow

Matrix	% AR	mg eq/kg
Liver	0.757	0.138
Kidney	1.977	1.957
Heart	0.199	0.098
Lung	0.265	0.111
Brain	0.007	0.025
Ovaries	0.003	0.080
Skeletal muscle (shoulder)	<0.0001	0.034
Skeletal muscle (rump)	<0.0001	0.034
Subcutaneous fat	<0.0001	0.021
Perirenal fat	<0.0001	0.019
Skin	<0.0001	0.035
Sciatic nerve	<0.0001	0.051
Bile	0.063	0.464
Plasma	n.r.	0.269
Whole blood	n.r.	0.180
Milk	0.067	0.021
Bladder urine	2.261	49.638

In milk the total radioactivity was very low, reaching a maximum of 0.03 mg eq/kg after 7 hours. After 31 hours no radioactivity was detected in any milk samples. The results are summarized in Table 28.

Table 28 Recovered radioactive residues in milk after the first intrarumenal injection of [4,5-pyridazine-^{14}C]-radiolabelled pyridate to a lactating cow

Days	Time (hours)	mg eq/kg	% TRR
1 (morning)	0	0.0	0.0
1 (afternoon)	7	0.03	0.1
2 (morning)	23	0.01	0.05
2 (afternoon)	31	0.0	0.01
3 (morning) – 8 (morning)	47-168	0.0	0.0

Characterization of the radioactivity in edible tissues was only done for liver and kidney. While no information was given on the extractability of the residue, co-chromatography of the extracts with reference standards identified pyridafol in kidney (level not given), as well as pyridafol-N- and O-glucoside (0.0–0.1 mg eq/kg) in liver.

A second metabolism study with one lactating goat was performed with [4,5-pyridazine-^{14}C]-radiolabelled pyridate (Ellgehausen, 1987, PYRIDATE_019). The compound was administered orally once

daily to one lactating goat at 2.8 ppm (0.38 mg/kg bw) for 10 consecutive days. Urine and feces were collected once daily (also more frequently during the depuration phase), while milk was collected twice daily. The animal was sacrificed approximately 24 hours after the last dose and samples of organs, tissues and body fluids were collected.

Total radioactivity in liquid samples such as urine, bile, cage wash and various extracts were directly measured by LSC. Faecal, blood, milk and tissue samples were subjected to combustion or digestion prior to the determination of total radioactivity by LSC.

After homogenization, liver and kidney samples were extracted four times with acetone/water (8:2), followed by one acetone Soxhlet extraction. The extracts were evaporated to the aqueous remainder and partitioned against dichloromethane. The radioactivity in unextracted residue was determined by combustion followed y LSC. Milk samples were deproteinated with acetone, followed by liquid-liquid partitioning against n-hexane/dichloromethane (1:1). Only urine samples were subjected to hydrolysis with 1 mol/L hydrochloric acid. Characterization of the extracts was carried out by TLC, as well as by GC-FID and GC-MS.

The total recovery of the administered radioactivity was equal to 103 percent. The majority of the radioactivity was found in urine (95 percent AR) followed by faeces (6.5 percent AR). Radioactive residues in the edible tissues were low at 0.019 mg eg/kg and 0.033 mg eq/kg in liver and kidney, respectively. In milk, residues were low as well at 0.04 mg eq/kg. A summary of the recovered radioactivity is presented in Table 29 and Table 30.

Table 29 Recovered radioactive residues after oral administration of 2.8 ppm [4,5-pyridazine-^{14}C]-radiolabelled pyridate for 10 consecutive days to a lactating goat

Matrix	% AR
Urine	95
Faeces	6.5
Cage wash	1.3
Milk	0.04
Tissues/organs	0.04
Total	103

Table 30 Total radioactive residues in organs and tissues after oral administration of 2.8 ppm [4,5-pyridazine-^{14}C]-radiolabelled pyridate for 10 consecutive days to a lactating goat

Matrix	mg eq/kg	% AR
Liver	0.019	0.01
Kidney	0.033	<0.01
Muscle	0.003	0.01
Spleen	0.004	<0.01
Heart	0.004	<0.01
Mammary	0.005	<0.01
Brain	0.010	<0.01
Fat	0.009	0.01
Bile	0.047	<0.01

In milk, the detected radioactivity was very low, ranging between 0.015–0.048 mg eq/kg. The results are summarized in the following Table 31. A plateau was reached after 3 days of consecutive administration of the compound Figure 3.

Table 31 Recovered radioactive residues in milk after oral administration of 2.8 ppm [4,5-pyridazine-^{14}C]-radiolabelled pyridate for 10 consecutive days to a lactating goat

Day	TRR (mg eq/kg)
1 (morning)	0.015
1 (afternoon)	0.027
2 (morning)	0.024
2 (afternoon)	0.039
3 (morning)	0.025
3 (afternoon)	0.048
4 (morning)	0.020
4 (afternoon)	0.041
5 (morning)	0.019
5 (afternoon)	0.040
6 (morning)	0.019
6 (afternoon)	0.037
7 (morning)	0.017
7 (afternoon)	0.039
8 (morning)	0.019
8 (afternoon)	0.038
9 (morning)	0.017
9 (afternoon)	0.029
10 (1 h after last dose)	0.016
10 (8 h after last dose)	0.030
10 (23 h after last dose)	0.004

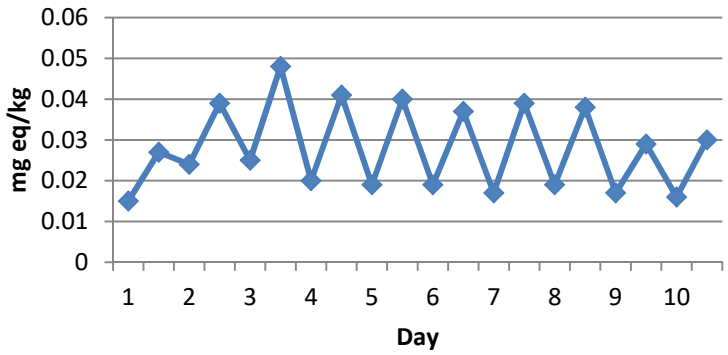


Figure 3 Time course of the concentrations of pyridate in milk

Extraction with acetone/water (8:2) followed by acetone released 63 percent TRR from liver and 87 percent TRR from kidney. Since radioactivity was low, identification was only successful in kidney, where metabolites pyridafol and pyridafol-OMe were assumed to be present at 32 percent TRR (0.010 mg eq/kg) and 48 percent TRR (0.015 mg eq/kg), respectively (Table 32). In milk only CL-9673 was identified at 49–71 percent TRR (0.012–0.015 mg eq/kg) (Table 33).

Table 32 Extractability and identification/characterization of residues from liver and kidney after oral administration of 2.8 ppm [4,5-pyridazine-^{14}C]-radiolabelled pyridate for 10 consecutive days to a lactating goat

Fraction	Liver		Kidney	
	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR	100	0.016	100	0.031

Fraction	Liver		Kidney	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Solvent extracts	63	0.010	87	0.027
Water soluble	10	0.002	6.8	0.002
Dichloromethane soluble	53	0.008	80	0.025
Pyridafol (assumed)	-	-	32	0.010
Pyridafol-OMe (assumed)	-	-	48	0.015
Unextracted	37	0.006	13	0.004
Total recovered radioactivity	100	0.016	100	0.031

Table 33 Identification/characterization of residues from milk after oral administration of 2.8 ppm [4,5-pyridazine-^{14}C]-radiolabelled pyridate for 10 consecutive days

Fraction	Milk (1 h after last dose)		Milk (8 h after last dose)	
	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR	100	0.016	100	0.030
Whey (after protein precipitation)	91	0.015	87	0.026
CL-9673	71	0.012	49	0.015
Unknown M1	20	0.003	29	0.008
Unknown M2	n/d		2.8	0.001
Unknown M3	n/d		6.1	0.002
Precipitate (proteins)	9.2	0.001	13	0.004
Total recovered radioactivity	100	0.016	100	0.030

Poultry

A metabolism study was performed with 6 laying hens and 6 broiler chickens receiving a single oral dose of [4,5-pyridazine-^{14}C]-radiolabelled pyridate at 2.5–4.7 ppm (0.2 mg/kg bw) (Cameron, *et al*., 1989, PYRIDATE_020). Excreta were collected once daily up to 96 hours post dose, while eggs were collected twice daily if possible and separated in to egg yolk and white. All animals were sacrificed at 96 hours post dose, washed, plucked and rewashed prior to analysis for total radioactivity. No individual organs or tissues were collected.

After dilution with water if necessary, the total radioactivity in liquid samples such as egg yolk and white, cage wash and solutions was directly measured by LSC. Excreta samples were subjected to combustion prior to the determination of total radioactivity by LSC.

Within 24 hours post dose, the majority of the radioactivity was found in excreta at 93–96 percent, increasing to 97–99 percent after 96 hours, demonstrating fast elimination of the compound after cessation of the dosing (Table 34).

Table 34 Mean recovered radioactive residues after a single oral administration of [4,5-pyridazine-^{14}C]-radiolabelled pyridate to laying hens and broiler chickens

Sample	Mean recovered, in %AR	
	Laying hens (n=6)	Broiler (n=6)
Excreta (0-96 h)	99	97
Cage wash (0-96 h)	3.2	5.9
Cage debris (0-96 h)	2.3	0.82
1st bird wash	0.05	0.05
2nd bird wash	0.09	0.07
Carcass	0.43	0.20
Total (0-96h)	105	104

Detected radioactivity in egg yolks and egg whites was consistently <LOQ for throughout the sampling time of 0–96 hours post-dose, with the exception of egg whites collected at 24–48 hours post dose with a mean of 0.03 percent AR (0.004 mg eq/kg).

No further identification or characterization of the radioactive residues in organs or tissues was performed. However, in excreta metabolites pyridafol and hydroxylated pyridafol accounted for up to 74 percent TRR and 44 percent TRR, respectively.

A second metabolism study was performed with 9 laying hens receiving [4,5–pyridazine-^{14}C]-radiolabelled pyridate orally once daily for 5 consecutive days at ~3 ppm (0.19 mg/kg bw) (Ellgehausen, 1987, PYRIDATE_021). Excreta and eggs were collected once daily and during the depuration phase additionally at 4, 8, and 24 hours after the last treatment. Eggs were separated in yolk and white. Three hens each were sacrificed at 8 hours, 3 days and 7 day after the final dose and liver, kidney, stomach, heart, muscle, brain, skin, blood, ovaries and spleen were collected.

Samples of blood/plasma, tissues, organs, egg yolk and white were treated with tissue solubiliser followed by determination of the total radioactivity by LSC. Excreta and cage wash were lyophilized and subjected to combustion prior to the determination of total radioactivity by LSC.

The majority of the radioactivity was found in excreta, at 93–96 percent AR, indicating rapid elimination (Table 35). Radioactive residues in the edible matrices were generally low, peaking at 0.04 mg eq/kg in kidney after 8 hours depuration. An even lower radioactivity was observed in egg samples with maximum residues in yolks and whites at 0.007 mg eq/kg and 0.01 mg eq/kg, respectively (Table 36). No further identification or characterization of the radioactive residues in organs or tissues was performed.

Table 35 Mean recovered radioactive residues after oral administration of 3 ppm [4,5-pyridazine-^{14}C]-radiolabelled pyridate to laying hens for five consecutive days

Sample	Mean recovered, in %AR Time sacrificed after last administration		
	8 hours	72 hours	168 hours
Excreta	93	95	96
Cage wash	3.5	3.1	4.6
Eggs	<0.1	<0.1	<0.1
Tissues/organs/blood	0.3	<0.1	<0.1
Total	97	98	101

Table 36 Total radioactive residues in tissues, organs, blood and eggs after oral administration of 3 ppm [4,5-pyridazine-^{14}C]-radiolabelled pyridate to laying hens for five consecutive days

Sample	TRR (mg eq/kg) Time sacrificed after last administration		
	8 hours	72 hours	168 hours
Liver	0.020	<0.008	<0.008
Kidney	0.040	<0.008	<0.008
Muscle (chest)	<0.006	<0.006	<0.006
Muscle (leg)	<0.008	<0.008	<0.008
Stomach	<0.008	<0.008	<0.008
Heart	n.d.	<0.008	<0.008
Brain	<0.006	<0.006	<0.006
Fat (stomach)	<0.020	<0.020	<0.020
Fat (kidney)	<0.020	<0.020	<0.020
Skin (and adjacent fat)	0.009	<0.006-0.008	<0.006

Sample	TRR (mg eq/kg) Time sacrificed after last administration		
	8 hours	72 hours	168 hours
Ovaries	0.009	<0.008	<0.008
Spleen	<0.010	<0.010	<0.010-0.011
Blood	0.023	<0.016	<0.016
Blood plasma	0.027	<0.018	<0.018
Egg whites (24-96 h)	<0.006-0.007	0.006-0.007	<0.006-0.007
Egg whites (depuration)	0.008	<0.006-0.007	<0.006-0.010
Egg yolk (24-96 h)	<0.006-0.006	<0.006-0.006	<0.006-0.006
Egg yolk (depuration)	0.006	<0.006-0.007	<0.006-0.007

A metabolic pathway of pyridate in animals is proposed in Figure 4.

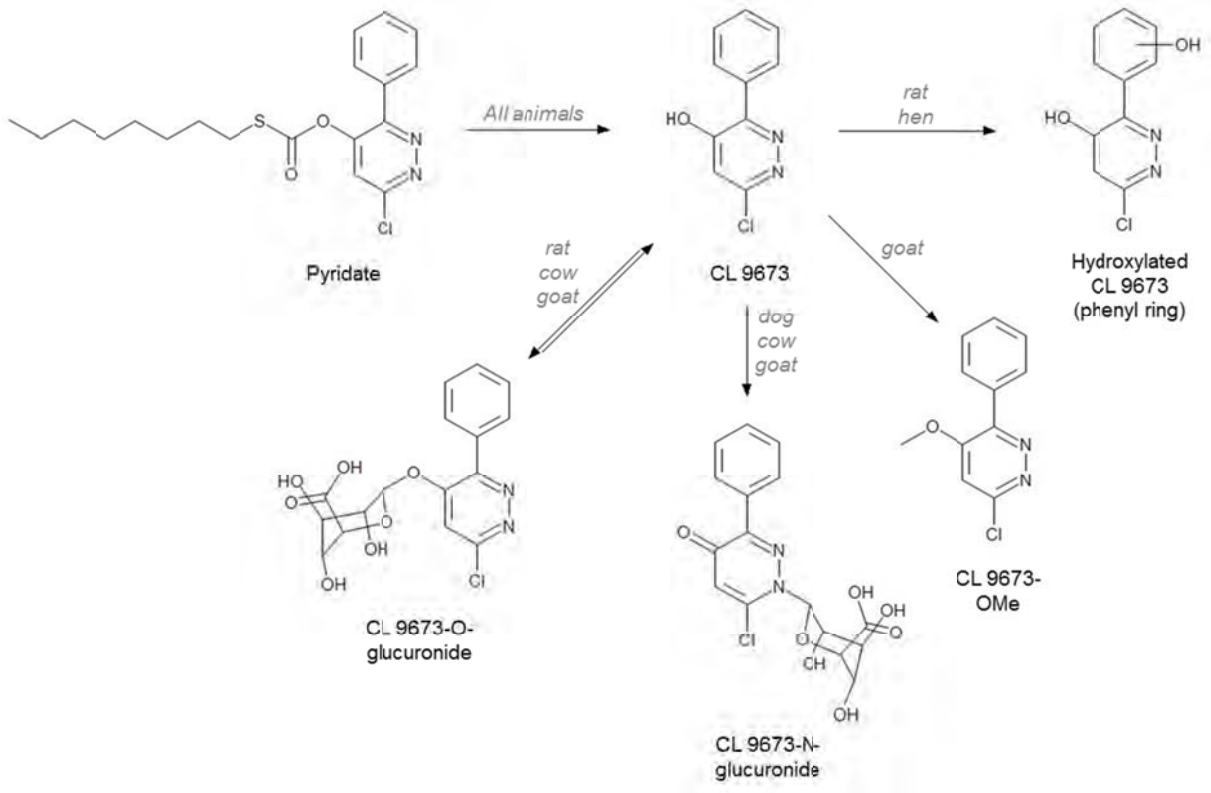


Figure 4 Proposed metabolic pathway of pyridate in animals

RESIDUE ANALYSIS

Analytical methods

For the analysis of pyridate and metabolites in various plant and animal matrices, analytical methods suitable for enforcement and data generation purposes were submitted. An overview of these methods is presented in Table 37.

Table 37 Overview of analytical methods for pyridate and metabolites

Method	Matrix	Extraction	Clean-Up	Analyte, Detection, LOQ
No. 758 a (Data generated with	Maize grain and plant, cereal grain,	Alkaline solution of ammonium	Hydrolyzation of conjugates with 0.05	LC-LC-UV LOQ: 0.05-0.1 mg/kg

Method	Matrix	Extraction	Clean-Up	Analyte, Detection, LOQ
this method cannot be considered as reliable)	straw and plant, rape leaves and seeds, ryegrass, turnip leaves and roots, poppy seed, rice, carrot leaves and root	acetate, acetone and morpholine (100+20+0.5/v)	mol/L sulfuric acid in methanol (pH 4-5); partitioning between ammonium acetate and dichloromethane	(pyridafol); 0.5-1 mg/kg (pyridate as pyridafol); 0.1-0.85 mg/kg (pyridafol-O-glucoside as pyridafol)
No. 758 d &758 e	Maize grain, straw and plant, rape plant, pod, stem and seeds, Field pea stem, pod and seeds, brassicas, Leek, onion, grapes. peppermint	Alkaline solution of ammonium acetate, acetone and morpholine (100+20+0.5/v)	Hydrolysation of conjugates with 1 mol/L sulfuric acid (pH 4); partitioning between ammonium acetate and dichloromethane, clean up on a silica cartridge	LC-LC-UV LOQ: 0.05 mg/kg (pyridafol); 0.5-50 mg/kg (pyridate as pyridafol)
No. 1005 (Data generated with this method cannot be considered as reliable)	Peanut hulls and nutmeat	Acetone/water (4:1)	Hydrolysation of conjugates with 10 mol/L sulfuric acid; partitioning between ammonium acetate and dichloromethane, clean up on a silica cartridge	LC-LC-UV LOQ: 0.05 mg/kg (pyridafol); 0.5 mg/kg (pyridate as pyridafol); 0.15 mg/kg (pyridafol-O-glucoside as pyridafol)
REM 191.01	Maize plant and grain, grass, kale	Acetone/ammonium acetate (5:1) + morpholine	Hydrolysation of conjugates with concentrated hydrochloric acid (pH 4), clean up on a C18 cartridge, solid-supported liquid-liquid partition with n-hexane/tert-butyl methyl ether (1:1)	LC-LC-UV LOQ: 0.02 mg/kg (pyridate as pyridafol)
S11-03700	Sweet corn grain, leek, cauliflower, broccoli, rape seed	Alkaline solution of acetone/ammonium acetate (5:1) + morpholine	Hydrolysation of conjugates with 1 mol/L sulfuric acid (pH 4-4.5); partitioning with dichloromethane	LC-MS/MS, ESI+, m/z 207→68, 207→104 LOQ: 0.05 mg/kg as pyridate (validated with pyridafol, pyridate as pyridafol, pyridafol-O-glucoside as pyridafol) ILV available
No. 1211	Milk, eggs, muscle, kidney, fat, liver	Alkaline solution of ammonium acetate, acetone and morpholine (100+20+0.5)	Partitioning between ammonium acetate and dichloromethane, liver only: clean up on a silica cartridge	LC-LC-UV LOQ: 0.03 mg/kg (pyridafol & (pyridate as pyridafol)
S11-01578	Milk, eggs, muscle, kidney, fat, liver	Acetonitrile/water (5:1) + morpholine	Clean up on a C18 SPE cartridge	LC-MS/MS, ESI+, m/z 207→104, 207→77 LOQ: 0.03 mg/kg (as pyridafol); 0.05 mg/kg (as pyridate) ILV available

Plant materials

Method No. 758 a (Bayzer & Heegemann, 1983, PYRIDATE_027)

The sample material is homogenized with an alkaline solution of ammonium acetate, acetone and morpholine (100+20+0.5) (pH 9 with NH_3), thereby converting pyridate to pyridafol. The extract is evaporated to the aqueous remainder, reconstituted in an alkaline solution of ammonium acetate and washed with dichloromethane. The conjugates in the alkaline phase are hydrolysed by refluxing with 0.05 mol/L sulfuric acid in methanol (pH 4–5). The hydrolysed phase is adjusted to pH 8–9 with ammonia, evaporated to dryness and partitioned between ammonium acetate and dichloromethane, firstly at pH 9, then at pH 4.5 and once re-extracted at pH 9.

Final determination of pyridafol was done by HPLC with column switching technology using a Polygosil-$N(CH_3)_2$ column and a Spherisorb ODS and detection at 280/300 nm (LC-LC-UV). Quantitation was done with external standards in solvent.

Partially recoveries were outside of the acceptable range, especially for pyridate as pyridafol and pyridafol-O-glucoside as pyridafol (Table 38). Also, recoveries were corrected for blank values, which not common practice in pesticide analysis. In conclusion, data generated with this method cannot be considered as reliable.

Table 38 Recovery data for the common moiety method No. 758 a measuring pyridate and its metabolites pyridafol and pyridafol-O/N-glucosides in various plant matrices using LC-LC-UV

Analyte	Matrix	Fortification Levels (mg/kg)	No of Samples	Recovery* (%)	Mean (%)	RSD (%)	Reference
Pyridafol	Maize (plant)	0.05	1	66	-	-	Bayzer & Heegemann, 1983, PYRIDATE_027
	Maize (grain)	0.05	1	66	-	-	
	Winter wheat (grain)	0.05	1	84	-	-	
	Winter wheat (plant)	0.1	1	73	-	-	
	Winter wheat (straw)	0.05	1	67	-	-	
	Oilseed rape (leaves)	0.05	1	70	-	-	
	Oilseed rape (seeds)	0.05	1	65	-	-	
	Raygrass (grass)	0.05	1	70	-	-	
	Turnip (leaves)	0.05	1	76	-	-	
	Turnip (beet)	0.05	1	70	-	-	
	Poppy	0.05	1	87	-	-	
	Rice	0.05	1	74	-	-	
	Carrot (leaves)	0.05	1	64	-	-	
	Carrot (root)	0.05	1	64	-	-	
Pyridate (as pyridafol)	Maize (plant)	0.5	1	61	-	-	
		1.0	1	80	-	-	
	Maize (grain)	0.1	1	50	-	-	
	Winter wheat (grain)	0.1	1	74	-	-	
	Winter wheat (plant)	1.02	1	72	-	-	
	Rice	0.1	1	80	-	-	
Pyridafol-O-glucoside (as pyridafol)	Maize (plant)	0.1	1	54	-	-	
	Maize (grain)	0.85	1	29	-	-	
	Winter wheat (grain)	0.1	1	78	-	-	
	Rice	0.1	1	71	-	-	

Notes:

*Corrected for blank values.

Method No. 758 d (Pfarl, 1989, PYRIDATE_028)

The sample material is homogenized with acetone/ammonium acetate (5:1) + 0.5 mL morpholine, thereby converting pyridate to pyridafol. The extract is evaporated to a "syrup-like" remainder, reconstituted in an alkaline ammonium acetate solution (pH 9) and washed with dichloromethane. The conjugates in the alkaline phase are hydrolysed by adding 1 mol/L sulfuric acid (pH 4) and letting the mixture react at 60 °C for 40 minutes. Following the hydrolysis, the mixture is partitioned against dichloromethane, the aqueous phase is discarded and the organic phase is cleaned up on a silica cartridge. CL9673 is eluted with methanol/dichloromethane (5:100), the eluate is evaporated to dryness, and the remainder is reconstituted in ammonium acetate solution (pH 9).

Final determination of pyridafol is done by HPLC with column switching technology using a Nucleosil 10-N$(CH_3)_2$ column and a Ultrasphere ODS and detection at 280/300 nm (LC-LC-UV). Quantitation is done with external standards in solvent.

Although no validation data was included in the study, the method is considered acceptable since sufficient validation data is provided in analytical reports of the respective field trials using method 758 d.

Method No. 758 e (Pfarl, 1992, PYRIDATE_029)

The extraction, clean-up and quantitation of method 758 e is identical to method No. 758 d. Presented recovery data seems to support the suitability of the method for the determination of pyridafol in high water content, high acid content, high oil content and dry matrices with an LOQ of 0.05 mg/kg (Table 39). However, validation data to demonstrate the conversion of pyridate and pyridafol-O-glucoside to pyridafol was insufficient or not available at all. Since hydrolytic conditions were comparable to method S11-01578 (see below), it can be assumed that method 758 e is also suitable for the detection of pyridate and pyridafol-O-glucosides as pyridafol. Although, chromatograms of fortified samples and matrix blanks were not included in the report, the method is considered acceptable since sufficient validation data including chromatograms are provided in the analytical reports of the respective field trials using method 758 e.

Table 39 Recovery data for the common moiety method No. 758 e measuring pyridate and its metabolite pyridafol in various plant matrices using LC-LC-UV

Analyte	Matrix	Fortification Levels (mg/kg)	No of Samples	Recovery (%)	Mean (%)	RSD (%)	Reference
Pyridafol	Maize (plant)	0.05	3	71-95	80	7.5	Pfarl, 1992, PYRIDATE_029
	Maize (stem)	0.05	3	81-95	88	8.0	
	Maize (grain)	0.05	3	69-83	76	9.2	
	Oilseed rape (plant)	0.05	3	72-106	89	19	
	Oilseed rape (pod)	0.05	3	63-83	73	14	
	Oilseed rape (stem)	0.05	3	86-90	88	2.3	
	Oilseed rape (seeds)	0.05	3	63-81	72	13	
	Field pea (stem)	0.05	4	62-90	76	18	
	Field pea (pod)	0.05	5	64-84	74	14	
	Field pea (seeds)	0.05	6	71-83	77	7.8	
	Brassicas (edible parts)	0.05	19	73-91	82*	11	
	Leek	0.05	2	75	75	n.a.	
	Onion (whole plant)	0.05	2	83	83	n.a.	
	Onion (bulb)	0.05	2	84	84	n.a.	
	Grapes (bunches)	0.05	9	74-92	83	11	
	Peppermint (dried, tea)	0.05	4	68-100	84*	19	
		0.5	4	58-74	66*	12	
Pyridate	Maize (plant)	50	7	74-86	83	15	

Analyte	Matrix	Fortification Levels (mg/kg)	No of Samples	Recovery (%)	Mean (%)	RSD (%)	Reference
(as pyridafol)	Oilseed rape (plant)	50	3	73-87	80	8.8	
	Onion (whole plant)	50	2	79	79	n.a.	
	Peppermint (dried, tea)	0.5	4	62-72	67*	7.5	
		1.0	4	61-75	68*	10	

Notes:

*Corrected for blank values.

Method No. 1005 (Pfarl, 1990, PYRIDATE_030)

The sample material is homogenized with acetone/water (4:1), followed by filtration. The extract is evaporated to a "syrup-like" remainder, reconstituted in water/dichloromethane (1:1) and the pH set to 3–3.5 with acetic acid. After centrifugation, the conjugates of pyridafol in the aqueous phase are hydrolysed by adding 10 mol/L hydrochloric acid and acetone and letting the mixture react at 70 °C for 45 minutes. Residues of pyridate and pyridafol in the dichloromethane phase are treated morpholine (to convert pyridate to pyridafol) und alkaline conditions (pH >8) and partitioned again ammonium acetate buffer (pH 9). The aqueous layers are combined and partitioned against dichloromethane. While the aqueous phase is discarded, the organic phase containing pyridafol is further cleaned up on a silica cartridge. Pyridafol is eluted with methanol/dichloromethane (5:100), the eluate is evaporated to dryness, and the remainder is reconstituted in ammonium acetate solution (pH 9).

Final determination of pyridafol is done by HPLC with column switching technology using a Nucleosil 10-N$(CH_3)_2$ column and a Ultrasphere ODS and detection at 280/300 nm (LC-LC-UV). Quantitation is done with external standards in solvent.

Generally, one fortification per level is considered insufficient and recoveries of pyridafol-O-glucoside as pyridafol are just outside the acceptable range (Table 40). Also, recoveries were corrected for blank values, which not common practice in pesticide analysis. In conclusion, data generated with this method cannot be considered as reliable.

Table 40 Recovery data for the common moiety method No. 1005 measuring pyridate and its metabolites pyridafol, pyridafol-O/N-glucosides in peanut matrices using LC-LC-UV

Analyte	Matrix	Fortification Levels (mg/kg)	No of Samples	Recovery* (%)	Mean (%)	RSD (%)	Reference
Pyridafol	Peanut (hulls)	0.05	1	74	-	-	Pfarl, 1990, PYRIDATE_030
	Peanut (nutmeat)	0.05	1	74	-	-	
Pyridate (as pyridafol)	Peanut (hulls)	0.50	1	88	-	-	
	Peanut (nutmeat)	0.50	1	88	-	-	
Pyridafol-O-glucoside (as pyridafol)	Peanut (hulls)	0.15	1	67	-	-	
	Peanut (nutmeat)	0.15	1	67	-	-	

Notes:

Corrected for blank values.

Method REM 191.01 (Gasser, 1998, PYRIDATE_031, Gasser, 1998, PYRIDATE_032)

The homogenized sample material is sequentially shaken with acetone/ammonium acetate (5:1) + 0.5 mL morpholine, followed by acetone/ammonium acetate (5:1) + 0.2 mL ammonia with centrifugation in between extractions. The extracts are combined and are evaporated to the aqueous remainder. The alkaline extract (after addition of sodium hydroxide solution) is washed with ethyl acetate, the pH

adjusted to pH 4–4.5 by acidification with concentrated hydrochloric acid and conjugates hydrolysed at 60 °C for 40 minutes. After cool down to room temperature, the mixture is cleaned up on a C18 SPE cartridge and analytes are eluted with methanol. The eluate is evaporated to near dryness and the remainder acidified with hydrochloric acid, diluted with 1 mol/L aqueous sodium chloride solution, before further cleaned up by solid-supported liquid-liquid partition using against n-hexane/tert-butyl methyl ether (1:1).

Final determination of pyridafol is done by HPLC with column switching technology using a Zorbax SB-CN column and a Multospher 100 RP 18 FBS and detection at 220 nm (LC-LC-UV). Quantitation is done with external standards in solvent.

Mean recovery and precision were within acceptable limits, as well as linearity and selectivity was sufficiently demonstrated. However, the method was only tested with parent pyridate as pyridafol, but not with the O-glucoside. Since hydrolytic conditions were comparable to method S11-01578 (see below), it is assumed that method REM 191.01 is also suitable for the detection of pyridafol-O-glucosides. The method is considered acceptable for the determination of pyridate and its metabolites pyridafol and pyridafol-O-glucosides in high water content and dry matrices with an LOQ of 0.02 mg/kg per analyte (Table 41).

Table 41 Recovery data for the common moiety method REM 191.01 measuring pyridate as pyridafol in various plant matrices using LC-LC-UV

Analyte	Matrix	Fortification Levels (mg/kg)	No of Samples	Recovery (%)	Mean (%)	RSD (%)	Reference
Pyridate (as pyridafol)	Maize (whole plant)	0.02	5	92-95	94	1	Gasser, 1998, PYRIDATE_031, Gasser, 1998, PYRIDATE_032
		0.2	5	90-92	90	1	
	Maize (grain)	0.02	5	79-90	83	6	
		0.2	5	83-90	85	3	
	Grass	0.02	5	88-92	90	2	
		0.2	5	86-93	89	4	
	Kale	0.02	5	88-95	95	4	
		0.2	5	79-92	87	6	

Method S11-03700 (Weber, 2012, PYRIDATE_033); Independent laboratory validation (Mewis, 2012, PYRIDATE_034)

Pyridate, metabolite pyridafol and its O- and N-glucoside conjugates are extracted from plant material by maceration with an alkaline solution of ammonium acetate, acetone and morpholine (pH 9). The extract is evaporated at 60 °C until the aqueous phase remains (at this step pyridate is converted to pyridafol), and thereafter partitioned between an alkaline solution of ammonium acetate and dichloromethane. The aqueous fraction undergoes an acidic hydrolysis using sulphuric acid at pH 4 (60 minutes, 95 °C) for cleavage of pyridafol conjugates and the pyridafol residue is subsequently extracted into dichloromethane. The organic phase is evaporated to dryness, and the pyridafol residue is re-dissolved in a mixture of methanol/0.05 percent acetic acid.

Samples were analysed by LC-MS/MS in positive ionization mode using an Ascentis express C18 column; and monitoring the ion transitions m/z 207→68 and 207→104. Quantitation is done with external standards in solvent.

Mean recovery and precision were within acceptable limits, as well as linearity and selectivity was sufficiently demonstrated. Matrix effects were below 20 percent. The method is considered acceptable for monitoring of pyridate and its metabolites pyridafol and pyridafol-O-glucosides in high water content,

high oil content and dry matrices with an LOQ of 0.05 mg/kg per analyte (Tables 42 and 43). However, in the primary method the pyridafol-N-glucoside does not hydrolyse to pyridafol under acidic conditions. The method was not validated for acidic matrices, but since the extraction was done with an alkaline buffer solution, the matrix group can be considered as covered by high water content matrices. In conclusion, the method is considered to be suitable for monitoring, as well as for risk assessment purposes.

Table 42 Recovery data (n=5) for the common moiety method S11-03700 measuring pyridate, metabolites pyridafol and its O- and N-glucosides in various plant matrices using LC-MS/MS (Weber, 2012, PYRIDATE_033)

Analyte	Matrix	Fortification Levels (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Recovery (%)	Mean (%)	RSD (%)
			Primary transition: m/z 207→68			Confirmatory transition: m/z 207→104		
Pyridafol	Sweet corn, grain	0.05	69-81	77	6.0	73-85	78	5.9
		0.5	72-79	74	3.9	72-79	75	3.6
	Leek	0.05	72-82	77	5.7	70-84	76	7.0
		0.5	72-80	76	4.8	71-80	76	4.5
	Cauliflower	0.05	69-78	74	4.4	73-81	76	5.5
		0.5	80-84	82	1.8	81-86	84	2.3
	Broccoli	0.05	77-82	79	2.6	75-83	80	3.9
		0.5	75-82	78	3.2	77-81	80	1.9
	Rape seeds	0.05	80-88	84	4.0	81-82	81	0.6
		0.5	78-89	84	5.3	83-95	88	5.2
Pyridate (as pyridafol)	Sweet corn, grain	0.05	85-96	89	4.6	81-94	86	6.2
		0.5	87-96	92	3.7	88-94	91	2.4
	Leek	0.05	87-99	95	5.2	85-98	91	5.8
		0.5	85-99	91	6.3	87-101	91	6.5
	Cauliflower	0.05	89-99	93	4.6	86-97	91	4.4
		0.5	82-96	90	5.9	87-90	89	1.3
	Broccoli	0.05	91-97	94	3.1	95-102	98	3.1
		0.5	85-91	88	3.1	92-97	94	2.3
	Rape seeds	0.05	79-99	86	10	76-90	82	6.3
		0.5	82-109	97	10	85-106	98	8.7
Pyridafol-O-glucoside (as pyridafol)	Sweet corn, grain	0.05	87-91	90	1.8	85-98	91	5.1
		0.5	73-84	80	5.3	82-85	82	3.7
	Leek	0.05	71-104	86	15	74-107	89	14
		0.5	69-88	82	9.5	66-93	85	13
	Cauliflower	0.05	84-102	93	6.9	77-96	89	8.6
		0.5	81-92	85	5.6	80-87	84	3.3
	Broccoli	0.05	84-95	88	5.1	84-95	88	5.1
		0.5	76-85	81	5.4	76-85	81	5.4
	Rape seeds	0.05	84-105	93	8.8	82-90	84	4.0
		0.5	91-98	94	2.9	86-96	93	4.4
Pyridafol-N-glucoside (as pyridafol)	Sweet corn, grain	0.05	-	10	-	-	9.8	-
		0.5	-	7	-	-	7.8	-
	Leek	0.05	-	3.6	-	-	3.2	-
		0.5	-	2.6	-	-	2.4	-
	Cauliflower	0.05	-	4.0	-	-	3.6	-
		0.5	-	1.0	-	-	1.0	-
	Broccoli	0.05	-	5.4	-	-	3.8	-
		0.5	-	2.8	-	-	2.4	-
	Rape seeds	0.05	-	2.6	-	-	2.6	-
		0.5	-	1.0	-	-	1.0	-

Table 43 Recovery data (n=5) for the independent laboratory validation of the method by Weber (2012), measuring pyridate, metabolites pyridafol and its O-glucoside in various plant matrices using LC-MS/MS (Mewis, 2012, PYRIDATE_034)

Analyte	Matrix	Fortification Levels (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Recovery (%)	Mean (%)	RSD (%)
			Primary transition: m/z 207→68			Confirmatory transition: m/z 207→104		
Pyridafol	Sweet corn, grain	0.05	79-90	83	5.1	75-90	82	6.5
		0.5	75-79	78	2.3	75-80	78	3.1
	Broccoli	0.05	101-107	104	2.1	97-103	100	2.5
		0.5	81-90	86	6.0	82-92	88	5.9
	Rape seeds	0.05	84-92	88	3.1	85-93	91	3.6
		0.5	81-87	83	3.8	79-88	83	4.5
Pyridate (as pyridafol)	Sweet corn, grain	0.05	66-78	73	6.6	65-79	74	7.3
		0.5	76-89	83	7.6	74-89	83	8.0
	Broccoli	0.05	62-77	71	8.6	63-76	70	7.2
		0.5	71-90	77	9.7	73-90	78	9.2
	Rape seeds	0.05	82-89	85	3.7	85-92	89	3.6
		0.5	86-98	93	6.0	87-97	93	5.6
Pyridafol-O-glucoside (as pyridafol)	Sweet corn, grain	0.05	71-77	73	3.0	70-76	72	3.6
		0.5	66-74	71	6.0	66-77	73	7.2
	Broccoli	0.05	86-97	92	5.4	85-92	88	3.9
		0.5	79-83	81	1.9	81-84	83	1.2
	Rape seeds	0.05	78-84	81	3.5	78-86	82	4.0
		0.5	79-81	80	0.9	79-82	81	1.4

Animal matrices

Method 1211 (Pfarl, 1995, PYRIDATE_035)

The sample material is homogenized with alkaline acetone/ammonium acetate (5:1) + 0.5 mL morpholine, thereby converting pyridate to pyridafol. The extract is evaporated to a “syrup-like” remainder, reconstituted in an alkaline ammonium acetate solution (pH 9) and washed with dichloromethane. The aqueous phase is acidified with sulphuric acid to pH 3–3.5 and partitioned against dichloromethane. For liver only, an additional clean up step is performed on a silica SPE cartridge. The organic extracts of all matrices are evaporated to dryness, and the remainder is reconstituted in ammonium acetate solution (pH 9).

Final determination of pyridafol is done by HPLC with column switching technology using a Nucleosil 10-N$(CH_3)_2$ column and a Ultrasphere ODS and detection at 280/300 nm (LC-LC-UV). Quantitation is done with external standards in solvent.

Mean recovery and precision were within acceptable limits, as well as linearity and selectivity was sufficiently demonstrated (Table 44). The method is considered acceptable for risk assessment purposes for the determination of pyridate and its metabolite pyridafol in animal matrices with an LOQ of 0.03 mg/kg per analyte.

Table 44 Recovery data for method No. 1211 measuring pyridate and its metabolite pyridafol, in animal matrices using LC-LC-UV

Analyte	Matrix	Fortification Levels (mg/kg)	No of Samples	Recovery (%)	Mean (%)	RSD (%)	Reference
Pyridate (as pyridafol)	Meat	0.03	4	73-81	78	3.6	
		0.27	4	87-92	90	2.6	

Analyte	Matrix	Fortification Levels (mg/kg)	No of Samples	Recovery (%)	Mean (%)	RSD (%)	Reference
		0.55	4	83-88	85	2.0	
	Liver	0.03	4	77-88	82	3.6	
		0.27	4	71-80	76	3.7	
		0.55	4	70-79	76	4.1	
	Kidney	0.03	4	88-114	104	8.7	
		0.27	4	81-91	87	4.3	
		0.55	4	84-89	87	2.2	
	Fat	0.03	4	66-88	79	8.6	
		0.27	4	87-98	93	5.7	
		0.55	4	82-92	88	4.4	
	Milk	0.03	4	81-88	84	2.6	
		0.27	4	89-91	90	1.2	
		0.55	4	86-87	87	0.8	
	Egg	0.03	4	73-95	83	7.9	
		0.27	4	74-87	82	6.0	
		0.55	4	78-85	82	3.2	
Pyridafol	Meat	0.03	4	80-90	84	3.8	
		0.30	4	84-86	85	0.9	
		1.0	4	78-81	79	1.5	
	Liver	0.03	4	67-77	72	3.8	
		0.30	4	68-74	71	2.5	
		1.0	4	68-73	71	2.2	
	Kidney	0.03	4	87-107	98	7.1	
		0.30	4	91-92	92	0.8	
		1.0	4	68-78	74	4.4	
	Fat	0.03	4	83-100	92	6.5	
		0.30	4	84-89	87	2.3	
		1.0	4	77-84	81	3.2	
	Milk	0.03	4	73-90	82	5.0	
		0.30	4	83-92	87	3.6	
		1.0	4	88-91	90	1.3	
	Egg	0.03	4	77-103	92	9.4	
		0.30	4	82-87	84	2.5	
		1.0	4	49-75	62	14	

Method S11-01578 (Keller, 2012, PYRIDATE_036); Independent laboratory validation (Wiesner & Breyer, 2012, PYRIDATE_037)

Pyridate and its metabolite pyridafol are extracted from animal matrices by shaking with acetonitrile/water (5:1) in the presence of morpholine. The extract was acidified with 0.1 mol/L sulphuric acid and a salt mixture of magnesium sulphate, sodium chloride, trisodium citrate, disodium hydrogen citrate sesquihydrate (QuEChERS Citrate Mix) was added. After centrifugation, an aliquot of the extract was cleaned by transfer on a C18 SPE cartridge and rinsed dropwise. An aliquot of the eluate was then diluted with methanol/water (1:1 with 0.1 percent acetic acid) and analysed for residues of pyridate and pyridafol as pyridafol.

Samples were analysed by LC-MS/MS in positive ionization mode using a Phenomenex Synergi Hydro RP 80A column and monitoring the ion transitions m/z 207→104 and 207→77. Quantitation is done with external standards in blank matrix.

Mean recovery and precision were within acceptable limits, as well as linearity and selectivity was sufficiently demonstrated (Table 45). The method is considered acceptable for monitoring of pyridate and

its metabolite pyridafol in animal matrices with an LOQ of 0.05 mg/kg as pyridate and 0.03 mg/kg for pyridafol. Compared to the method for food of plant origin, the hydrolysis of conjugates at pH 4 at 95 ˚C is not conducted. In addition, no validation with glucose conjugates was done. The method was independently validated (Table 46).

Table 45 Recovery data (n=5) for method S11-01578 measuring pyridate and its metabolite pyridafol in animal matrices using LC-MS/MS (Keller, 2012, PYRIDATE_036)

Analyte	Matrix	Fortification level (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Recovery (%)	Mean (%)	RSD (%)
			Primary transition: m/z 207→104			Confirmatory transition: m/z 207→77		
Pyridate	Milk	0.05	104-117	109	4.3	98-109	103	5.0
		0.5	94-105	102	4.6	99-107	101	3.3
	Eggs	0.05	83-91	88	4.2	86-95	91	4.8
		0.5	83-98	90	6.1	86-96	90	4.3
	Meat	0.05	87-101	95	5.2	84-110	101	10.4
		0.5	88-100	94	5.5	89-104	98	6.3
	Kidney	0.05	90-103	97	5.3	90-95	92	2.5
		0.5	83-91	89	3.6	83-92	89	4.1
	Fat	0.05	102-114	110	4.7	84-111	98	11
		0.5	93-116	106	7.9	98-115	104	6.4
	Liver	0.05	89-101	93	5.4	89-119	104	11
		0.5	82-96	88	6.8	75-85	81	4.9
Pyridafol	Milk	0.03	83-98	91	7.5	83-98	91	7.8
		0.3	83-101	87	10	83-93	89	5.8
	Eggs	0.03	74-86	82	5.9	80-90	87	4.2
		0.3	78-90	82	5.7	73-83	79	5.0
	Meat	0.03	72-96	88	10	84-95	91	5.0
		0.3	82-94	87	5.6	89-92	91	1.5
	Kidney	0.03	72-80	76	4.4	70-84	79	6.5
		0.3	65-89	79	12	69-86	77	8.2
	Fat	0.03	70-86	79	7.1	65-83	73	9.8
		0.3	72-100	83	14	70-97	83	13.0
	Liver	0.03	62-77	71	7.8	67-79	74	6.5
		0.3	64-76	72	6.8	66-76	72	5.1

Table 46 Recovery data (n=5) for the independent laboratory validation of the method by Keller (2012), measuring pyridate and its metabolite pyridafol in animal matrices using LC-MS/MS (Wiesner & Breyer, 2012, PYRIDATE_036)

Analyte	Matrix	Fortification level (mg/kg)	Recovery (%)	Mean (%)	RSD (%)	Recovery (%)	Mean (%)	RSD (%)
			Primary transition: m/z 207→104			Confirmatory transition: m/z 207→77		
Pyridate	Milk	0.05	116-123	118	2.3	109-119	115	3.5
		0.5	92-99	95	3.5	95-102	98	2.8
	Meat	0.05	109-118	114	2.8	110-122	116	3.7
		0.5	102-106	104	1.7	102-108	104	2.6
Pyridafol as pyridate	Milk	0.05	98-100	99	0.7	92-98	95	3.0
		0.5	77-84	80	3.2	80-87	83	3.1
	Meat	0.05	90-95	92	2.2	88-98	94	4.1
		0.5	87-90	88	1.5	87-91	88	1.9

STABILITY OF PESTICIDES RESIDUES IN STORED ANALYTICAL SAMPLES

Plant matrices

The storage stability of pyridate under frozen conditions was investigated in incurred radiolabelled residues originating from plant metabolism studies with maize, peanuts, broccoli and alfalfa (Zohner, 1988, PYRIDATE_039).

Leaves of maize, peanuts, broccoli and alfalfa were taken at 0 DAT and kept deep frozen for 4.2 months (maize), 4.5 months (peanuts), 9.4 month (broccoli) and 14 month (alfalfa) prior to analysis. Samples were homogenized and extracted sequentially with acetone and acetone/water at various ratios. The combined extracts were analysed by LSC and TLC and the residue expressed as the sum of pyridate, pyridafol and hydrolysable pyridafol conjugates. After periods of 10 to 28 months, re-analysis was conducted and the metabolic profile compared to the first analysis was considered. The recovered amount was based on all compounds analysed by the common moiety methodology for pyridate, CL 6972 and its hydrolysable conjugates (Table 47).

Table 47 Storage stability of pyridate in incurred residues from whole plant samples of maize, rape, field pea and onion (greens only)

Crop	DAT (h)	Storage period (months)	Residues		Subsequent storage (months)	Residues
			Extracted (% TRR)	Sum of pyridate, pyridafol and hydrolysable pyridafol conjugates (% TRR)		Sum of pyridate, pyridafol and hydrolysable pyridafol conjugates (% TRR)
Maize leaves	2.75	4.2	99.7	97.7	26	86.0
Peanuts leaves	3.5	4.5	99.8	95.7	28	69.5
Broccoli leaves	1.5	9.4	99.8	99.8	12	85.7
Alfalfa	1	14	99.8	99.8	10	90.0

The storage stability of pyridate under frozen conditions was investigated in incurred residues from whole plant samples of maize, rape, field pea and onion (greens only) (Pfarl, 1996, PYRIDATE_038).

Samples were stored deep frozen at -20 °C and analysed for the first time after about 3 months (onion) to about one year (maize, rape, field pea) after sampling. The storage time between first and second analyses was about two years (rape, field pea), three years (onion) or five years (maize). Samples were analysed in duplicates according to method 758 d or 758 e as the sum of pyridate, pyridafol and hydrolysable pyridafol conjugates. (Table 48)

Table 48 Storage stability of pyridate in incurred residues from whole plant samples of maize, rape, field pea and onion (greens only)

Commodity	Mean pyridafol (mg/kg)[1]	Storage time (months)	% remaining	Mean procedural recovery (%)
Maize plant	24	14	-	76
	19	59	81	77
Rape plant	7.1	11	-	71
	11	25	155	84
Field pea plant	14	13	-	76
	13	24	93	85
Onion greens	5.2	3	-	80
	4.6	35	89	76

Notes:

[1] Results not corrected for recovery.

Animal matrices

The storage stability of pyridate under frozen conditions in milk, muscle, fat, liver and kidney fortified at 0.5 mg/kg was determined over a period of 7 months (Gasser, 2002, PYRIDATE_040). Samples were stored deep frozen at -18 °C and analysed in duplicate after 0, 3/4, and 7 months according to method 1211, as the sum of pyridate and pyridafol (Table 49).

Table 49 Storage stability of pyridate in animal matrices fortified at 0.5 mg/kg

Storage time (months)	Milk		Muscle		Fat		Liver		Kidney	
	% remaining	Mean concurrent recovery (%)[1]	% remaining	Mean concurrent recovery (%)[1]	% remaining	Mean concurrent recovery (%)[1]	% remaining	Mean concurrent recovery (%)[1]	% remaining	Mean concurrent recovery (%)[1]
0	90	89	79	83	79	84	73	76	78	76
3 (4 liver)	89		80		72		78		81	
7	76	83	67	77	70	80	76	80	74	84

Notes:

[1] Samples from 0 and 3 months were analysed in the same analytical sequence.

USE PATTERN

Pyridate is an herbicide used to control annual broad-leaved weeds. The GAP information taken from the submitted current labels for crops supported with residue data is summarized in Table 50.

Table 50 List of uses of pyridate (450 g/kg WP formulation, foliar spray)

Crop/ Commodity	Country	Application			PHI (days)
		Rate (g ai/ha)	Water volume (L/ha)	No or Seasonal max. (interval)	
Alfalfa	Austria	900	200-400	1	28
Cabbage, kale, Brussels sprouts, broccoli, cauliflower	Austria	450	200-400	2 (7-14)	42
Cabbage, kale, Brussels sprouts, broccoli, cauliflower	Austria	900	200-400	1	42
Chick-pea	Italy	900	200-600	1	45
Clover	Austria	900	200-400	1	28
Leek	Austria	450	200-400	2 (10-14)	28
Leek	Austria	900	200-400	1	28
Kohlrabi	Austria	900	200-400	1	42
Maize	Netherlands	900	150-400	1	Not stated (treatment from BBCH 14-16)
Onion, bulb	Austria	450	200-400	2 (10-14)	56
Onion, bulb	Austria	900	200-400	1	56
Onion, bulb	Italy	900	200-600	1	21
Spring onion	Austria	900	200-400	1	35
Spring onion	Netherlands	900	150-400	1	28
Sweet corn (Corn-on–the-cob)	Austria	450	200-600	2 (7-14)	42
Sweet corn (Corn-on–the-cob)	Austria	900	200-600	1	42
Sweet corn (Corn-on–the-cob)	Netherlands	900	150-400	1	Not stated (treatment from BBCH 14-16)

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as pyridate equivalents. When residues were not detected they are shown as below the LOQ, e.g., < 0.01 mg/kg. Application rates, spray concentrations and mean residue results have generally been rounded to two significant figures. Values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for percent recovery. Residues were generally determined as pyridafol and the pyridate equivalent calculated by multiplication with 1.834 (Pyridate: 378.9 g/mol and pyridafol: 206.6 g/mol).

A summary of the submitted trials is shown in Table 51.

Table 53 Pyridate – supervised residue trials (outdoor, foliar spray)

Commodity	Countries	Table
Onion, bulb	France, Italy, Austria	Table 52
Leek	Netherlands, France, Switzerland, Spain, Italy,	Table 53
Spring onion	Spain, Germany, United Kingdom, Greece	Table 54
Broccoli, cauliflower, Brussels sprouts	Austria, United Kingdom	Table 55
Cabbage	Germany, United Kingdom, France, Greece, Austria, Spain	Table 56
Kohlrabi	Austria	Table 57
Kale	France, Switzerland, United Kingdom	Table 58
Chickpea	France, Greece, Italy, Spain United States	Table 59 Table 60
Maize	Austria, France, Germany	Table 61
Sweet corn (Corn-on –the-cob)	France, Germany	Table 62
Alfalfa	Austria, France, Hungary	Table 63
Clover	Austria, France, United Kingdom	Table 64
Maize forage	Austria, France, Germany	Table 65
Sweet corn forage	France, Germany	Table 66
Maize straw	Austria, France, Germany	Table 67

Bulb vegetables

Onion, bulb

A total of nine field trials were conducted with bulb onion in France and Italy during the 2006 growing season (Partington, 2007, PYRIDATE_041) and in France, Greece, Spain and the UK during the 2009 growing season (Semrau, 2012, PYRIDATE_108). The trials received one applications (at BBCH 15–17 or 41–47) at a rate of 900 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined

using an Agrisearch in-house method, identical to method S11-03700, with a limit of quantification of 0.01 mg/kg as pyridafol (0.02 mg/kg expressed as pyridate). It was noted that this is in contradiction to the results table of the study where the LOQ as pyridate equivalents was set at 0.01 mg/kg. However, the latter LOQ seems acceptable since also procedural recoveries were performed with pyridate fortified at 0.01 mg/kg.

Another set of nine field trials were conducted with bulb onion in Austria during the 1986–1992 growing seasons (Heegemann, 1986, PYRIDATE_042; Pfarl, 1991, PYRIDATE_043; Pfarl, 1992, PYRIDATE_044; Pfarl, 1993, PYRIDATE_045) and in the UK during the 1990 growing season (Pfarl, 1991, PYRIDATE_097. The trials received one application (at BBCH 13–16) at a rate ranging between 900–1800 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using methods 758 c, 758 d or 758 e with a limit of quantification of 0.03 mg/kg as pyridafol (0.05 mg/kg expressed as pyridate). Mean procedural recoveries were generally acceptable, except for pyridafol in report No. 1095 were recoveries were at 61 percent, unacceptably low. The results are shown in Table 52.

Table 52 Residues of pyridate and pyridafol in bulb onion in supervised trials conducted in Europe using one foliar application

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)[1]		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Cumont, France, 2006, AF/10897/BC/1 (Spirit)	912	304	45-47	21	Bulb	NA	<0.01	AF/10897/BC Partington, 2007, PYRIDATE_041 Storage time: 4.9-5.3 months Method S11-03700 Procedural recoveries: 91±14 % (n=4) at 0.01-0.1 mg/kg
Villefranche, France, 2006, AF/10897/BC/2 (Rebouillon)	887	394	41	21	Bulb	NA	0.02	
Budrio, Bologna, Italy, 2006, AF/10897/BC/3 (Bianca)	898	599	45	21	Bulb	NA	<0.01	
Elne, Pyrenees-Orientales, France, 2009, S09-02296-01 (Guasman)	900	300	15-16	35	Bulb	NA	0.11[2]	S09-02296 Semrau, 2012, PYRIDATE_108 Storage time: 3-8 months Method S11-03700 Procedural recoveries: 95 % (n=2) at 0.01-0.1 mg/kg pyridate 77 % (n=2) at 0.01-0.1 mg/kg pyridafol
Arnissa, Pella, Greece, 2009, S09-02296-02 (Dorata Di Parma)	910	303	17	33	Bulb	NA	<0.01	
Anhydro, Pella, Greece, 2009 S09-02296-03 (Redwing)	850	283	17	33	Bulb	NA	<0.01	
Conil De La Frontera, Spain, 2009 S09-02296-04 (Spring star)	863	383	17	35	Bulb	NA	0.02	
Pozohondo, Albacete, Spain, 2009 S09-02296-05 (Castillo)	870	290	45	35	Bulb	NA	<0.01	
Cottam, Nottinghamshire, United Kingdom, 2009 S09-02296-06 (Hyfort F1)	810	270	45	35	Bulb	NA	<0.01	

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)[1]		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Leonding, Austria, 1986 (Gelber Wiener)	900	300	13-14	65	Bulb Greens	<0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	<0.05, <0.05 (<u>0.05</u>) <0.05, <0.05 (<0.05)	Report No. 874 Heegemann, 1986, PYRIDATE_042 Storage time: ~2 months. Method 758 c Procedural recoveries: 79 % (n=2) at 0.05 mg/kg pyridafol
	1800	600	13-14	65	Bulb Greens	<0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	<0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05)	
Raasdorf, Austria, 1988 (not reported)	1350	300	14-16	0 89	Whole plant Bulb	4.4, 4.5 (4.4) <0.03, <0.03 (<0.03)	8.1, 8.3 (8.2) <0.05, <0.05 (<0.05)	Report No. 1095 Pfarl, 1991, PYRIDATE_043 Storage time: ~24 months. Method 758 d Procedural recoveries: 75±1.5 % (n=4) at 50 mg/kg pyridate 61±25 % (n=3) at 0.05 mg/kg pyridafol
Leopoldsdorf, Austria, 1988 (Lagergold Rinsburger)	1350	300	13-14	0 77	Whole plant Bulb	6.9, 11 (8.8) <0.03, <0.03 (<0.03)	13, 19 (16) <0.05, <0.05 (<0.05)	
Schönau, Austria, 1991 (Wiener Brantkugel)	910	300	13-14	0 21 93	Whole plant Bulb	5.4, 6.6 (6.0) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	9.9, 12 (11) <0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05)	Report No. 1127 Pfarl, 1992, PYRIDATE_044 Storage time: max. 6 months. Method 758d Procedural recoveries: 79 % (n=2) at 50 mg/kg pyridate 85±6.6 % (n=4) at 0.01-0.05 mg/kg pyridafol
Markgrafneusiedl, Austria, 1992 (Wiener Bronzekugel)	900	300	13-14	0 20 82	Whole plant Bulb	5.9, 5.9 (5.9) 0.042, <0.03 (0.036) <0.03, <0.03 (<0.03)	11, 11 (11) 0.077, <0.05 (0.064) <0.05, <0.05 (<0.05)	Report No. 1147 Pfarl, 1993, PYRIDATE_045 Storage time: max. 3 months. Method 758 e Procedural recoveries: 81 % (n=4) at 25 mg/kg pyridate 72±15 % (n=8) at 0.05 mg/kg pyridafol
Breitstetten I, Austria, 1992 (Cobra)	900	300	13-14	0 20 82	Whole plant Bulb	4.1, 4.0 (4.0) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	7.5, 7.3 (7.4) <0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05)	
Breitstetten II, Austria, 1992 (Spirit)	900	300	13-14	0 20 82	Whole plant Bulb	5.1, 5.1 (5.1) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	9.4, 9.4 (9.4) <0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05)	
Schönau, Austria, 1992 (Wiener Bronzekugel)	900	300	13-14	0 20 82	Whole plant Bulb	6.8, 5.1 (6.0) 0.038, 0.043 (0.040) <0.03, <0.03 (<0.03)	12, 9.4 (11) 0.070, 0.079 (0.073) <0.05, <0.05 (<0.05)	

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)[1]		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Whittlesford, Cambridge, UK, 1990 (Balstora)	1800	200	Semi mature	29	Bulb	<0.03	<0.05	Report No. 1102 Pfarl, 1991, PYRIDATE_097 Storage time: max. 11 months Method 758 d 87 % (n=2) at 0.05 mg/kg pyridafol

Notes:

[1] Results from two replicate field samples are presented and values in parentheses represent mean values.

[2] The bulbs of trial S09-02296-01 were harvested at an immature stage (i.e. BBCH 42) and not at normal commercial harvest (BBCH 49), as was done in the other trials, because the sampling timing was set at a PHI of 35 days. The BBCH-stage 42 corresponds to "20 % of the expected bulb or shaft diameter reached" and is therefore clearly an immature stage that is not representative for normal commercial harvest.

NA: Not analysed.

Leek

A set of 14 field trials were conducted with leek in France (in 1996 & 2006), Italy (in 2006), the Netherlands (in 1987 &1989), Spain (in 2006), Switzerland (in 2001) and in the UK (in 1990). The trials received one application (at BBCH 17 to early harvest) at a rate of 900g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using methods 758 d and 758 e with a LOQ of 0.03 mg/kg as pyridafol (0.05 mg/kg as pyridate), method REM 191.01 with a LOQ of 0.02 mg/kg as pyridafol (0.04 mg/kg as pyridate) or a method identical to method S11-03700 with a LOQ of 0.01 mg/kg as pyridafol (0.02 mg/kg as pyridate). It was noted that the LOQ for pyridate in the method equivalent to S11-03700 is in contradiction to the results table of the study, where the LOQ as pyridate equivalents was set at 0.01 mg/kg. However, the latter LOQ seems acceptable since also procedural recoveries were performed with pyridate fortified at 0.01 mg/kg. The results are shown in Table 53.

Table 53 Residues of pyridate and pyridafol in leek in supervised trials conducted in Europe using one foliar application

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Groningen, The Netherlands, 1989 (Certina)	900	300	Early harvest	23	0.034, 0.034, 0.038, <0.03, <0.03 (0.033)	0.062, 0.062, 0.070, <0.05, <0.05 (0.060)	Report No. 1055 Pfarl, 1990, PYRIDATE_046 Storage time: max. 7 months Method: 758 d; Procedural recoveries: 82±4 % (n=4) at 0.05 mg/kg
Hoorn, The Netherlands, 1989 (Winterreus)	900	300	Early harvest	101	<0.03, <0.03, <0.03, <0.03 (<0.03)	<0.05, <0.05, <0.05, <0.05 (<0.05)	
Thorée-les-Pins, Northern France, 1996 (Davina)	956	425	45	0 7 14 21 28	7.5, 6.5 (7.0) ctrl: 0.01 3.5, 2.1 (2.8) 1.5, 1.5 (1.5) 0.66, 0.86 (0.76) 0.35, 0.34 (0.35) ctrl: 0.014	14, 12 (13) ctrl: 0.02 6.4, 3.9 (5.1) 2.8, 2.8 (2.8) 1.2, 1.6 (1.4) 0.64, 0.62 (0.63) ctrl: 0.026	Study No. R96-031 Report No. 1289 Pfarl, 1997, PYRIDATE_047 Storage time: max. 2.6 months Method: 758 e Procedural recoveries: 96±9.2 % (n=4) at 0.05-

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Cheviré-le-Rouge, Northern France, 1996 (Portura)	911	405	45	0 7 14 21 28	4.9, 5.5 (5.2) 2.3, 1.5 (1.9) 1.0, 1.5 (1.2) 0.68, 0.79 (0.74) 0.46, 0.41 (0.44)	9.0, 10 (9.5) 4.2, 2.8 (3.5) 1.8, 2.8 (2.3) 1.3, 1.5 (1.4) 0.84, 0.75 (0.81)	25 mg/kg pyridate 96±3.9 % (n=4) at 0.03-3.0 mg/kg pyridafol
Appily, Northern France, 1996 (Portura)	873	388	17	0 7 14 21 28	4.1, 2.3 (3.2) 1.4, 3.0 (2.2) 0.81, 0.41 (0.61) 0.10, 0.22 (0.16) 0.098, 0.049 (0.074)	7.5, 4.2 (5.9) 2.6, 5.5 (4.0) 1.5, 0.75 (1.1) 0.18, 0.40 (0.29) 0.18, 0.090 (0.14)	
Labergement les Auxonne, Northern France, 1996 (Profina)	872	387	41	0 7 14 21 28	3.9, 4.1 (4.0) 0.50, 0.92 (0.71) 0.38, 0.30 (0.34) 0.11, 0.12 (0.12) 0.032, <0.03 (0.031)	7.2, 7.5 (7.3) 0.92, 1.7 (1.3) 0.70, 0.55 (0.62) 0.20, 0.22 (0.21) 0.059, 0.044 (0.051)	
Chessel, Switzerland, 2001 (Prelina)	900	500	41	0 28	4.2 <0.02, 0.02 (0.02)	7.7 <0.04, 0.04 (0.04)	Study No.: 3036/01 Gasser, 2002, PYRIDATE_048 Storage time: max. 5.6 months Method REM 191.01 Procedural recoveries: 66 % (n=1) at 20 mg/kg pyridate; 87±11 % (n=4) at 0.02-20 mg/kg pyridafol
Kerkdriel, the Netherlands, 1987 (Winter leek)	900	400	Early harvest	28	0.037, <0.03, <0.03, <0.03, <0.03 (0.031)	0.068, <0.05, <0.05, <0.05, <0.05 (0.057)	Study No.: R 87-05 Anonymous, 1987, PYRIDATE_049 Storage time: max. 9 months Method: 758 c; Procedural recoveries: 90±11 % (n=3) level not reported
Kruningen, the Netherlands, 1987 (Cortina)	900	400	Early harvest	0 28	0.30, 0.45, 0.72, 0.17 (0.41) 0.096, 0.14, 0.093 (0.11)	0.54, 0.82, 1.3, 0.31 (0.75) 0.18, 0.25, 0.17 (0.20)	
Cavazzana, Italy, 2006 AF/10899/BC/3 (Armor)	914	508	43	0 14 21 28 42	NA -	11 0.82 0.25 0.08 0.02	AF/10899/BC Harrison, 2007, PYRIDATE_050 Storage time: 2.3-4.9 months Method identical to S11-03700 Procedural recoveries: 83±13 % (n=5) at 0.01-20 mg/kg pyridate 77±5.0 % (n=5) at 0.01-10 mg/kg pyridafol
Conil de la, Frontera, Spain, 2006 AF/10899/BC/4 (Arial)	891	396	43-45	14 21 28	NA	0.26 0.17 0.05	
Gagnac, Southern France, 2006 AF/10899/BC/5 (Helios)	984	298	41	0 14 21 28 42	NA	13 0.55 0.16 0.11 0.04	
Manziat, Southern France, 2006 AF/10899/BC/6 (Numhens)	900	400	42	14 21 28	NA	0.38 0.40 0.22	

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Whittlesford, Cambridge, UK, 1990 (Verina)	1800	200	Semi mature	29	0.13 (corrected for recovery) Mature bulb	NA	Report No. 1102 Pfarl, 1991, PYRIDATE_097 Storage time: max. 11 months Method 758 d; 75 % (n=2) at 0.05 mg/kg pyridafol

Spring onion

A total of six field trials was conducted with green onion in Spain during the 2006 growing season (Partington, 2007, PYRIDATE_041) and in Germany, Greece, Spain and the UK, during the 2009 growing season (Semrau, 2012, PYRIDATE_051). The trials received one application (at BBCH 13–14 or 41–43) at a rate of 900 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using a method identical to method S11-03700, with a limit of quantification of 0.01 mg/kg as pyridafol (0.02 mg/kg as pyridate). The results are shown in Table 54.

Table 54 Residues of pyridate and pyridafol in green onion in supervised trials conducted in Europe using one foliar application

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Conil de la Frontera, Spain, 2006 AF/10897/BC/4 (Elody)	941	418	41	21	NA	0.02	AF/10897/BC Partington, 2007, PYRIDATE_041 Storage time: 1.5 months Method identical to S11-03700;Procedural recoveries: 91±14 % (n=4) at 0.01-0.1 mg/kg
Herxheim, Rheinland-Pfalz, Germany, 2009 S09-02297-01 (Totem)	925	308	40	0	1.1	2.0	S09-02297 Semrau, 2012, PYRIDATE_051 Storage time: 7.5-12 months Method identical to S11-03700 Procedural recoveries: 89±15 % (n=12) at 0.01-20 mg/kg pyridate 78±9.8 % (n=12) at 0.01-10 mg/kg pyridafol
				14	0.093	0.17	
				27	0.049	0.09	
				34	0.022	0.04	
				40	<0.01	<0.01	
Clifford Chambers, Warwickshire, United Kingdom, 2009 S09-02297-02 (Green Banner)	885	295	13-14	0	1.8	3.3	
				14	0.13	0.23	
				27	0.022	0.04	
				34	0.038	0.07	
				41	0.027	0.05	
Ionia, Thessaloniki, Greece 2009 S09-02297-03 (Nea Magnisia)	880	293	13	0	3.8	6.9	
				14	0.18	0.33	
				28	0.022	0.04	
				35	0.011	0.02	
				42	<0.01	<0.01	
Profitis, Thessaloniki, Greece 2009 S09-02297-04 (Lagada)	896	299	13	0	3.3	6.0	
				14	0.22	0.40	
				28	0.027	0.05	
				35	<0.01	0.01	
				42	<0.01	<0.01	
Casas de Guijarro, Cuenca, Spain, 2009 S09-02297-05 (Red Bull)	973	324	43	0	1.5	2.7	
				14	0.011	0.02	
				29	<0.01	<0.01	
				34	<0.01	<0.01	
				40	<0.01	<0.01	

Brassica vegetables (except brassica leafy vegetables)

Flowerhead brassicas (broccoli, cauliflower)

A total of four field trials were conducted with broccoli in Austria during the 1986 growing season (Heegemann, 1987, PYRIDATE_052) and in the UK during the 1989-90 growing season (Pfarl, 1991, PYRIDATE_097; Pfarl, 1991, PYRIDATE_097). Additionally, trials were conducted with cauliflower and Brussels sprouts in the UK during the 1990 growing season (Pfarl, 1991, PYRIDATE_097). The trials received one application (at different growth stages) at a rate ranging between 900-2000 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using methods 758 c or d with a limit of quantification of 0.03 mg/kg as pyridafol (0.05 mg/kg as pyridate). The results are in Table 55.

Table 55 Residues of pyridate and pyridafol in broccoli, cauliflower and Brussels sprout in supervised trials conducted in Europe using one foliar application

Location, Year, Trial No., Crop (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Leonding, Austria, 1986 Broccoli (asparagus broccoli, variety not reported)	900	300	17-18	0 34	Green plants	34 <0.03	62 <0.05	Report No. 891 Heegemann, 1987, PYRIDATE_052 Storage time: 4 months Method 758 c; Procedural recoveries: 82 % (n=2) at 25-50 mg/kg pyridate, 93±13 % (n=4) at 0.03-0.05 mg/kg pyridafol
	1800	300	17-18	0 34	Green plants	51 <0.03	93 <0.05	
Southfleet, Kent, UK, 1989 Broccoli (calabrese, Cruser)	2000	200	Starting to head	24	Head	<0.03	NA	Report No. 1056c Pfarl, 1991, PYRIDATE_099 Storage time: not reported Method 758 d,, 74 % (n=2) at 0.05 mg/kg pyridafol
Wellsborn, Warwick, UK, 1989 Broccoli (Late Purple Sprouting)	2000	200	Sprouts developing	41	Mature plants	<0.03	NA	Report No. 1102 Pfarl, 1991, PYRIDATE_097 Storage time: max. 11 months. Method 758 d 91±8.8 % (n=5) at 0.05 mg/kg pyridafol
Whittlesford, Broccoli Cambridge, UK, 1990 (calabrese)	1800	200	Not reported	28	Mature plant	0.14[1]	NA	
Friskney, Boston, Lincolnshire, UK, 1990 Cauliflower (Revito)	1800	200	Curds forming	21	Mature	<0.03	NA	
Wrangle, Lincolnshire, UK, 1990 Brussels sprout (Titurel)	1800	200	0.5-3 cm buttons	21	Buttons	<0.03	NA	Report No. 1102 Pfarl, 1991, PYRIDATE_097 Storage time: max. 11 months Method 758 d 76±2.1 % (n=4) at 0.05 mg/kg pyridafol
Wellsborn Warwick, UK, 1989 Brussels sprout (Late Purple Sprouting)	2000	200	Buttons forming	41	Buttons	<0.03	NA	

Notes:

[1]corrected for recovery

Cabbages

A total of 36 field trials were conducted with cabbage in France, Germany, Greece, Spain and the UK during the 2009 growing season (Semrau, 2012, PYRIDATE_053), in the UK during the 1988, 1990, 1995 & 1996/97 growing seasons (Carrier & Pfarl, 1996, PYRIDATE_055; Carrier, 1997, PYRIDATE_056; Pfarl, 1989, PYRIDATE_096; Pfarl, 1991, PYRIDATE_097) and Austria during the 1986 & 1988 growing seasons

(Pfarl, 1989, PYRIDATE_057; Heegemann, 1986, PYRIDATE_091; Heegemann, 1986, PYRIDATE_092; Heegemann, 1986, PYRIDATE_093; Pfarl, 1989, PYRIDATE_094; Pfarl, 1989, PYRIDATE_095). The trials received one application (at BBCH 14-19 or 41-43 or not stated) at a rates ranging between 900-1800 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using methods 758 c & 758 e with a LOQ of 0.03 mg/kg as pyridafol (0.05 mg/kg as pyridate), a Novartis method with a LOQ of 0.03 mg/kg as pyridafol (0.05 mg/kg as pyridate) or a method identical to method S11-03700 with a LOQ of 0.01 mg/kg for both, pyridafol and pyridate.

Additionally, three field trials were conducted with fodder cabbage in France during the 1982 growing season (Bosio, 1982, PYRIDATE_054). The trials received one application (at 4F-5F) at a rate of 900 g ai/ha. The method was not stated, but the description is similar to method 758 a, with an LOQ of 0.2 mg/kg as pyridafol. The results are shown in Table 56.

Table 56 Residues of pyridate and pyridafol in cabbage in supervised trials conducted in Europe using one foliar application

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Lambsheim, Rheinland-Pfalz, Germany, 2009 S09-02290-01 (Mozart)	920	307	19	0	Plant	NA	8.3	S09-02290 Semrau, 2012, PYRIDATE_053 Storage time: 6.8-10 months Method identical to S11-03700 Procedural recoveries: 85 % (n=7) at 0.01-20 mg/kg pyridate 75 % (n=7) at 0.01-10 mg/kg pyridafol Validation: 93±8.6 % (n=10) at 0.01-0.1 mg/kg
				13			0.23	
				28			0.03	
				43	Head		<0.01	
				56			<0.01	
				70			<0.01	
Hesketh Bank, Southport, United Kingdom, 2009 S09-02290-02 (Savoy)	911	200	42-43	0	Plant	NA	4.0	
				15			0.84	
				26			0.32	
				40	Head		0.02	
				54			<0.01	
				67			<0.01	
Saint Laurent de la Salanque, Pyrênées Orientales, Southern France, 2009, S09-02290-03 (Melissa)	940	313	41	0	Plant	NA	3.8	
				14			0.04	
				28			0.02	
				41	Head		<0.01	
				62			<0.01	
				70			<0.01	
Iona, Thessaloniki, Greece, 2009 S09-02290-04 (Banner)	967	322	18	0	Plant	NA	6.3	
				14			0.49	
				28			0.29	
				42	Head		<0.01	
				57			<0.01	
				70			<0.01	
Xativa, Valencia, Spain, 2009 S09-02290-05 (Savoy)	902	401	19	0	Plant	NA	16	
				14			1.9	
				28			0.8	
				42	Head		<0.01	
				56			<0.01	
				71			<0.01	
Bolbaite, Valencia, Spain, 2009 S09-02290-06 (Milano)	923	410	18	0	Plant	NA	4.5	
				14			0.60	
				28			0.65	
				41	Head		<0.01	
				55			0.01	
				68			<0.01	

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Beeston Green, Bedfordshire, UK, 1995, R95-039-01 (Wirosa)	913	251	Not reported	0 42	Head	4.2 0.067	7.7 0.12	Study No. R95-039 Carrier & Pfarl, 1996, PYRIDATE_055 Storage time: 1.9-4.7 months Method: 758 e Procedural recoveries: 92 % (n=2) at 50 mg/kg pyridate 94±9.5 % (n=7) at 0.03-1.0 mg/kg pyridafol
Charingworth, Gloucestershire, UK, R95-039-02 (Tundra)	913	251	Not reported	0 7 14 28 42	Head	6.5 0.56 1.2 0.18 0.21	12 1.0 2.2 0.33 0.38	
Old Leake, Boston, Lincolnshire, UK R95-039-03 (Colt)	902	246	Not reported	0 42	Head	11 <0.03	20 <0.05	
Ebrington, Gloucestershire, UK; R95-039-04 (Stonehead)	902	246	Not reported	0 7 14 28 42	Head	14 1.9 0.14 <0.03 <0.03	26 3.5 0.26 <0.05 <0.05	
Wrangle, Lincolnshire, UK, 1996, R96-082-01 (Tundra)	930	256	Not reported	0 42	Head	5.5, 6.0 (5.7) 0.28, 0.29 (0.28) Ctrl: 0.038	9.9, 11 (10) 0.50, 0.52 (0.50)	Study No. R96-082 Carrier, 1997, PYRIDATE_056 Storage time: 1.8-3.5 months Method: Novartis Agro Europe method Procedural recoveries: 106±28 % (n=5) at 0.05-25 mg/kg pyridate 109±29 % (n=4) at 0.03 mg/kg pyridafol
Leake Commonside, Lincolnshire, UK, 1996, R96-082-02	930	256	Not reported	0 42	Head	6.7, 7.4 (7.0) Ctrl: 0.031 0.34, 0.29 (0.32)	12, 13 (13) Ctrl: 0.056 0.61, 0.52 (0.57)	
Weston on Avon, Warwickshire, UK, 1996, R96-082-03 (Offenham Winter Green)	915	252	Not reported	0 7 14 28 42	Head	17, 19 (18) 6.0, 9.0 (7.5) 2.6, 2.9 (2.8) 1.0, 1.3 (1.2) 0.53, 0.52 (0.53)	31, 34 (33) 11, 16 (14) 4.7, 5.2 (5.0) 1.8, 2.3 (2.2) 0.95, 0.94 (0.95)	
Badsey, Worcestershire, UK, 1996 R96-082-04 (Celtic)	915	252	Not reported	0 7 14 28 42	Head	6.5, 6.8 (6.7) 3.7, 4.1 (3.9) 2.1, 1.7 (1.9) 0.88, 0.79 (0.83) 0.47, 0.47 (0.47)	12, 12 (12) 6.7, 7.4 (7.0) 3.8, 3.1 (3.4) 1.6, 1.4 (1.5) 0.85, 0.85 (0.85)	
Whittlesford, Cambridge, UK, 1989 (not reported)	1800	200	16-18	37	Head	<0.03	NA	Study No. 1056a Pfarl, 1989, PYRIDATE_096 Storage time: max. 10 months Method: 758 d Procedural recoveries: 87±15 % (n=6) at 0.05 & 0.1 mg/kg pyridafol
Southfleet, Kent, United Kingdom, 1989 (Castello)	2000	200	Not reported	23	Head	0.077	NA	
I.H.R., United Kingdom, 1989 (Golden Acre)	2000	200	41	22	Head	0.033	NA	
Eferding, Austria, 1988 (White - Mana-Frühkopf)	1350	300	14-15	0 18 45	Whole plants	37 Ctrl: max 0.068 <0.03 <0.03	NA	Study No. 1015c Pfarl, 1989, PYRIDATE_057 Storage time: max. 9 months Method: 758 c Procedural recoveries: 85 % (n=1) at 50 mg/kg pyridate 111 % (n=1) at 0.1 mg/kg pyridafol
Linz/Haag, Austria, 1988 (White - Mana-Frühkopf)	1350	300	14-15	0 17 43	Whole plants	37 0.078 <0.03	NA	
Marchtrenk, Austria, 1988 (White - Mana-Frühkopf)	1350	300	14	0 17 52	Whole plants	40 Ctrl: max 0.13 0.074 Ctrl: max 0.076 <0.03	NA	

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Eferding, Austria, 1988 (Red - Septemberrot)	1350	300	14-15	0 18 85	Whole plants	19 Ctrl: max 0.053 0.044 Ctrl: max 0.017 <0.03	NA	Study No. 1015d Pfarl, 1989, PYRIDATE_094 Storage time: max. 10 months Method: 758 c Procedural recoveries: 89±17 % (n=3) at 50 mg/kg pyridate 78±7.6 % (n=4) at 0.05 & 0.1 mg/kg pyridafol
Linz/Haag, Austria, 1988 (Red - Septemberrot)	1350	300	14-15	0 17 78	Whole plants	25 Ctrl: max 0.07 0.076 <0.03	NA	
Marchtrenk, Austria, 1988 (Red - Septemberrot)	1350	300	14	0 17 79	Whole plants	17 Ctrl: max 0.12 0.28 <0.03	NA	
Eferding, Austria, 1988 (September)	1350	300	14-15	0 18 85	Whole plants	30 Ctrl: max 0.052 0.072 Ctrl: max 0.027 <0.03	NA	Study No. 1015e Pfarl, 1989, PYRIDATE_095 Storage time: max. 10 months Method: 758 c Procedural recoveries: 100±7.7 % (n=4) at 50 mg/kg pyridate 88±6.8 % (n=5) at 0.05 & 0.1 mg/kg pyridafol
Linz/Haag, Austria, 1988 (September)	1350	300	14-15	0 17 78	Whole plants	25 Ctrl: max 0.048 0.11 <0.03	NA	
Marchtrenk, Austria, 1988 (September)	1350	300	14	0 17 79	Whole plants	33 Ctrl: max 0.062 0.11 <0.03	NA	
Leonding, Austria, 1986 (White – not reported)	900	300	17-18	1 62	Whole green plants Flesh Leaves	16 <0.03 <0.03	NA	Study No.: 890 Heegemann, 1986, PYRIDATE_091 Storage time: 4.6 month Method 758 c Procedural recoveries: 73 % (n=2) at 25-50 mg/kg pyridate 76±4.7 % (n=4) at 0.05 & 0.03 mg/kg pyridafol
	1800	300	17-18	1 62	Whole green plants Flesh Leaves	34 <0.03 <0.03	NA	
Leonding, Austria, 1986 (Red - not reported)	900	300	17-18	1 70	Whole green plants Flesh Leaves	8.0 <0.03 <0.03	NA	Study No.: 890a Heegemann, 1986, PYRIDATE_092 Storage time: 4.6 month Method 758 c Procedural recoveries: 88 % (n=2) at 25-50 mg/kg pyridate 91±1.3 % (n=3) at 0.05 & 0.03 mg/kg pyridafol
	1800	300	17-18	1 70	Whole green plants Flesh Leaves	18 <0.03 <0.03	NA	
Leonding, Austria, 1986 (Savoy - not reported)	900	300	16-17	1 61	Whole green plants Flesh Leaves	30 <0.03 <0.03	NA	Study No.: 890b Heegemann, 1986, PYRIDATE_093 Storage time: 4.6 month Method 758 c

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
	1800	300	16-17	1 61	Whole green plants Flesh Leaves	54 <0.03 <0.03	NA	Procedural recoveries: 84 % (n=2) at 25-50 mg/kg pyridate 98±14 % (n=3) at 0.05 & 0.03 mg/kg pyridafol
Freistone, Boston, Lincolnshire, UK, 1990 (Stonehead)	1800	200	41	16	Whole heads	<0.03	NA	Report No. 1102 Pfarl, 1991, PYRIDATE_097 Storage time: max. 11 months Method 758 d 80±9.4 % (n=10) at 0.05 mg/kg pyridafol
Wrangle, Boston, Lincolnshire, UK, 1990 (Tundra)	1800	200	Heads formed	21	Whole heads	0.11[1]	NA	
Cottenham, Cambridge, UK, 1990 (Horizon)	1800	200	47	27	Whole heads	0.45[1]	NA	
Cottenham, Cambridge, UK, 1990 (Costelle)	1800	200	Heads formed	29	Whole heads	<0.03	NA	
Cottenham, Cambridge, UK, 1990 (Bison)	1800	200	Heads formed	47	Whole heads	<0.03	NA	
Grenoux, Northern France, 1982 H683 (Proteor)	900	500	5F	56	Fodder cabbage	<0.03	<0.05	BEER.82.012 Bosio, 1982, PYRIDATE_054 Storage time: <1 month Method not stated, but similar to 758 a Procedural recoveries: 75 % (n=2) at 0.5 mg/kg pyridate 70 % (n=1) at 0.1 mg/kg pyridafol
Village-Viley, France, 1982 H685 (Proteor)	900	500	4F	35	Fodder cabbage	<0.03	<u><0.05</u>	
Rennes, Northern France, 1982 Trial No.: H687 (Sarbo)	900	500	4F	49	Fodder cabbage	<0.03	<u><0.05</u>	

Notes:

[1] Corrected for recovery.

Kohlrabi

Four field trials were conducted with kohlrabi in Austria during the 1986 and 1988 growing seasons (Pfarl, 1989, PYRIDATE_058; Heegemann, 1986, PYRIDATE_098). The trials received one application (at BBCH 14-17) at rates ranging between 900-1800 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using method 758 c with a LOQ of 0.03 mg/kg as pyridafol (0.05 mg/kg as pyridate). The results are shown in Table 57.

Table 57 Residues of pyridate and pyridafol in kohlrabi in supervised trials conducted in Austria using one foliar application

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Eferding, Austria, 1988 (Lanro)	1350	300	14-15	0 18 38	Whole plants	46, 22 (34) Ctrl: max 0.089 <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	NA	Study No. 1015b Pfarl, 1989, PYRIDATE_058 Storage time: max. 9 months Method: 758 c Procedural recoveries: 98±1.0 % (n=3) at 50 mg/kg pyridate 91±6.8 % (n=4) at 0.05-0.1 mg/kg pyridafol (corrected for blank values)
Linz/Haag, Austria, 1988 (Lanro)	1350	300	14-15	0 17 39	Whole plants	17, 21 (19) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	NA	
Marchtrenk, Austria, 1988 (Lanro)	1350	300	14	0 17 39	Whole plants	24, 31 (27) Ctrl: max 0.14 <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	NA	
Leonding, Upper Austria, 1986 (Roggli, white)	900	300	15-17	1 34	Whole plants Leaves and stalks Fruit	18 0.058 <0.03	32 0.1 <u>≤0.05</u>	Study No.: 892 Heegemann, 1986, PYRIDATE_098 Storage time: 4.6 months Method 758 c Procedural recoveries: 81 % (n=2) at 25 & 50 mg/kg pyridate 86±14 % (n=4) at 0.05 & 0.03 mg/kg pyridafol
	1800	300	15-17	1 34	Whole plants Leaves and stalks Fruit	62 0.17 0.036	NA	

Notes:

NA: Not analysed.

Leafy vegetables (including brassica leafy vegetables)

Kale

Eight field trials were conducted with kale in France, Switzerland and the UK during the 1998 growing season (Gasser, 1999, PYRIDATE_059, Gasser, 1999, PYRIDATE_060, Gasser, 1999, PYRIDATE_061, Gasser, 1999, PYRIDATE_062, Gasser, 1999, PYRIDATE_063, Gasser, 1999, PYRIDATE_064, Gasser, 1999, PYRIDATE_065, Gasser, 1999, PYRIDATE_066). The trials received one application (at BBCH 13–15) at a rate of 900 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using method REM 191.01 with a LOQ of 0.02 mg/kg as pyridafol (0.04 mg/kg as pyridate). The results are shown in Table 58

Table 58 Residues of pyridate and pyridafol in kale (whole plant) in supervised trials conducted in Europe using one foliar application

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Vouvry, Gartenacker, Switzerland, 1998 (Grüner Angeliter)	900	500	14	0 7 14 28 42	17 0.72 0.14 <0.02 <0.02	31 1.3 0.26 <0.05 <u><0.05</u>	Study No. 3043/98 Gasser, 1999, PYRIDATE_059 Storage time: 8 months Method: REM 191.01 Procedural recoveries: 92 % (n=1) at 20 mg/kg pyridate, 100 % (n=2) at 0.02-0.2 mg/kg pyridafol
Vouvry, Monthey II, Switzerland, 1998 (Grüner Angeliter)	900	500	15	42	<0.02, <0.02 (<0.02)	<0.05, <0.05 (<u><0.05</u>)	Study No. 3044/98 Gasser, 1999, PYRIDATE_060 Storage time: 8 months Method: REM 191.01 Procedural recoveries: 90 % (n=1) at 0.5 mg/kg pyridate 89 % (n=1) at 0.02 mg/kg pyridafol
Station Road, Chipping Campden, Gloucestershire, UK, 1998 (Marrow Stem)	922	400	13-14	0 3 7 20 40	30 6.4 2.0 0.43 0.05	55 12 3.7 0.79 <u>0.092</u>	Study No. 3005/98 Gasser, 1999, PYRIDATE_061 Storage time: 8 months Method: REM 191.01 Procedural recoveries: 86 % (n=1) at 40 mg/kg pyridate 108 % (n=2) at 0.02-0.2 mg/kg pyridafol
Hidcote Boyce Chipping Campden, Gloucestershire, UK, 1998 (Bitern)	922	400	14-15	0 28 42	24 0.03 <0.02	44 0.055 <u><0.05</u>	Study No. 3006/98 Gasser, 1999, PYRIDATE_062 Storage time: 7 months Method: REM 191.01 Procedural recoveries: 81 % (n=1) at 20 mg/kg pyridate 98 % (n=2) at 0.02-0.2 mg/kg pyridafol
Capendu, Southern France, 1998 (Protéor)	900	400	41	0 42	8.7, 7.9 (8.3) 0.04, 0.05 (0.05)	16, 15 (15) 0.072, 0.090 (<u>0.081</u>)	Study No. 3083/98[1] Gasser, 1999, PYRIDATE_063 Storage time: 5 months Method: REM 191.01 Procedural recoveries: 82 % (n=2) at 0.5 & 20 mg/kg pyridate 121 % (n=1) at 0.02 mg/kg pyridafol
Capendu, Southern France, 1998 (Melino)	919	408	41	0 7 14 28 42	6.2 1.7 0.48 0.04 0.04	11 3.1 0.88 0.074 0.074	Study No. 3084/98[1] Gasser, 1999, PYRIDATE_064 Storage time: 5 months Method: REM 191.01 Procedural recoveries: 79 % (n=1) at 20 mg/kg pyridate 89 % (n=2) at 0.02 & 0.2 mg/kg pyridafol
Mauguio, Southern France, 1998 (Métino)	951	423	42	0 42	5.9, 5.3 (5.6) <0.02, <0.02 (<0.02)	11, 9.8 (10) <0.05, <0.05 (<u><0.05</u>)	Study No. 3085/98[2] Gasser, 1999, PYRIDATE_065 Storage time: 5 months Method: REM 191.01 82 % (n=2) at 0.5 & 20 mg/kg pyridate 121 % (n=1) at 0.02 mg/kg pyridafol

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Mauguio, Southern France, 1998 (Protéor)	955	425	44	0 7 14 28 42	9.0 2.1 0.82 0.13 0.09	17 3.9 1.5 0.24 0.17	Study No. 3086/98[2] Gasser, 1999, PYRIDATE_066 Storage time: 5 months Method: REM 191.01 Procedural recoveries: 75 % (n=1) at 20 mg/kg pyridate 86 % (n=2) at 0.02 & 0.2 mg/kg pyridafol

Notes:

[1] It was noted that trial 3083/98 and 3084/98 were performed at the same location/time and differed only in the cultivated kale variety. Therefore the trials could not be considered as independent.

[2] It was noted that trial 3085/98 and 3086/98 were performed at the same location. However, treatment and harvest time each differed by 7 days. Therefore the trials are considered as independent.

Legume vegetables

Chickpea

Six field trials were conducted with chickpeas in France, Greece, Italy and Spain during the 2015 growing season (Grall, 2016, PYRIDATE_067). The trials received one application (at BBCH 19) at a rate of 900 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using method S11-03700 with a LOQ of 0.05 mg/kg as pyridate (Table 59).

Table 59 Residues of pyridate and pyridafol in chick-peas from field trials in Europe with a single foliar application

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Mouries, Southern France, 2015 Trial FR01 (Flamenco)	958	329	19	0 7 14 27 35 35 69 69	Whole plant Green seed Rest of plant Seed Straw	NA	64 9.1 3.3 0.89 <0.05 0.07 <0.05 0.09	Study No. EGL-15-22375 Grall, 2016, PYRIDATE_067 Storage time: 4.4-7.0 months Method: S11-03700 Additional validation with pyridate and pyridafol at 0.05 & 0.5 mg/kg in whole plant, green seed, dry seed and straw for two ion transitions. All recoveries (n=80) were betwee 70-89 % with RSDs <12 % Procedural recoveries: 88±11 % (n=12) at 0.05-100 mg/kg as pyridate
Belcassel, Southern France, 2015 Trial FR02 (Vulcano)	894	307	19	59 59 72 72	Green seed Rest of plant Seed Straw	NA	<0.05 <0.05 <0.05 <0.05	

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Nistal, Spain, 2015 Trial ES03 (Pico Pardal)	884	303	19	0 6 14 28 49 49 84 84	Whole plant Green seed Rest of plant Seed Straw	NA	41 9.4 0.29 <0.05 <u><0.05</u> <0.05 <0.05 <0.05	
Bienvenida, Spain, 2015 Trial ES04 (Crema)	888	305	19	37 37 86 86	Green seed Rest of plant Seed Straw	NA	<0.05 <u><0.05</u> <0.05 <0.05	
Roccabianca, Italy, 2015 Trial IT05 (Local variety)	894	307	19	0 7 14 28 49 49 73 73	Whole plant Green seed Rest of plant Seed Straw	NA	70 1.2 0.11 <0.05 <u><0.05</u> <0.05 <0.05 <0.05	
Larissa, Thessalyl, Greece, 2015 Trial GR06 (Quarpanzo)	877	301	19	0 7 14 27 40 40 70 70	Whole plant Green seed Rest of plant Seed Straw	NA	71 3.3 0.26 0.05 <u><0.05</u> <0.05 <0.05 <0.05	

Another four field trials were conducted with chick-peas in the United States (growing season not reported) (Anonymous, year not stated, PYRIDATE_068). The trials received two applications (at BBCH 12–16 and 20 ± 2 days later) at a rate of 1000 and 2000 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using method 758 e with a LOQ of 0.03 mg/kg as pyridafol (0.05 mg/kg as pyridate). However, no analytical report, including chromatograms, linearity etc. was included. The results are shown in Table 60.

Table 60 Residues of pyridate and pyridafol in chickpeas from field trials in the United States conducted with 2 foliar applications (20 days interval)

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH at final appl.			Pyridafol	Pyridate equivalents	
Idaho, year not specified	1000	Not reported	20 ± 2 days after BBCH 12-16	60	Seed	0.067, 0.030 (0.049)	NA	Study No. 03866 Anonymous, year

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH at final appl.			Pyridafol	Pyridate equivalents	
ID03 (not reported)	2000	Not reported	20 ± 2 days after BBCH 12-16	60	Seed	<0.03, <0.03 (<0.03)	NA	not stated, PYRIDATE_068 Storage time: max. 2.8 months Method: 758 e Procedural recoveries: 76 % (n=5) at 0.05 mg/kg pyridafol
Oregon, year not specified OR28 (not reported)	1000	Not reported	20 ± 2 days after BBCH 12-16	64	Seeds with hulls Seeds	<0.03, <0.03, <0.03, <0.03 (<0.03) <0.03, <0.03, <0.03, <0.03 (<0.03)	NA	
	2000	Not reported	20 ± 2 days after BBCH 12-16	64	Seeds with hulls Seeds	<0.03, <0.03, <0.03, <0.03 (<0.03) <0.03, <0.03, <0.03, <0.03 (<0.03)	NA	
Washington, year not specified WA31 (not reported)	2000	Not reported	20 ± 2 days after BBCH 12-16	61	Seed	<0.03, <0.03 (<0.03)	NA	
California, year not specified CA88 (not reported)	1000	Not reported	20 ± 2 days after BBCH 12-16	64	Seed	<0.03, <0.03, <0.03, <0.03 (<0.03)	NA	
	2000	Not reported	20 ± 2 days after BBCH 12-16	64	Seed	<0.03, <0.03, <0.03, <0.03 (<0.03)	NA	

Cereal grains

Maize

Fifteen field trials were conducted with maize in France during the 1992, 1996 and 2000 growing seasons (Gasser, 2002, PYRIDATE_101; Krennhuber, 1997, PYRIDATE_102, Pfarl, 1992, PYRIDATE_104), in Austria in the 1990 & 1991 growing seasons (Pfarl, 1991, PYRIDATE_103; Pfarl, 1994, PYRIDATE_104; Pfarl, 1992, PYRIDATE_105; Pfarl, 1992, PYRIDATE_106) and in Germany during the 1991 growing season (Pfarl, 1992, PYRIDATE_107). The trials received one application (at BBCH 14-17) at rates ranging from 900 to 1800 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using method REM 191.01 with a LOQ of 0.02 mg/kg as pyridafol (0.04 mg/kg as pyridate), or method 758 d & e with a limit of quantification of 0.03 mg/kg as pyridafol (0.05 mg/kg expressed as pyridate). It was noted that no storage stability study for dry crops was available. Hence, the storage period of the field samples could not be validated. The results are shown in Table 61.

Table 61 Residues of pyridate and pyridafol in maize grain from field trials in Europe with a single foliar application

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Marsillagues, France, 2000 (Cecilia)	900	400	16-17	117	<0.02, <0.02 (<0.02)	<0.04, <0.04 (<0.04)	Study No. 3013/00 Gasser, 2002, PYRIDATE_101 Storage time: max. 16 months Method: REM 191.01 Procedural recoveries: 96 % (n=2) at 0.02 & 0.2 mg/kg pyridafol
Peyrens, France, 1996 (Calis)	894	397	16-17	134	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<0.05)	Study No. 1294 Krennhuber, 1997, PYRIDATE_102 Storage time: max. 3 months (not validated) Method: 758 e Procedural recoveries: 90 % (n=2) at 0.05 mg/kg pyridate 87 % (n=2) at 0.03 mg/kg pyridafol
Garrevaques, France, 1996 (Cecilia)	907	403	16-17	142	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<0.05)	
Montluel, France, 1996 (Perseval)	885	393	16	144	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<0.05)	
Montanay, France, 1996 (Occitan)	899	399	16	138	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<0.05)	
Leonding, Austria, 1990 (Dea)	1370	300	14	139	<0.03	NA	Report No. 1092 Pfarl, 1991, PYRIDATE_103 Storage time: max. 2 months Method 758 d 71 % (n=2) at 0.05 mg/kg pyridafol
Marchtrenk, Austria, 1990 (Dea)	1370	300	15	131	<0.03	NA	
Linz, Austria, 1990 (LG5)	1370	300	16	121	<0.03	NA	
Ansfelden, Austria, 1991 (Dea)	1360	300	16	116	<0.03	NA	Study No. 1123 Pfarl, 1992, PYRIDATE_105 Storage time: max. 2 months Method: 758 d Procedural recoveries: 76±9.1 % (n=3) at 0.05 mg/kg pyridafol
Leonding, Austria, 1991 (Dea)	1360	300	16-17	110	<0.03	NA	
Leonding/Biolabor, Austria, 1991 (LG5)	1360	300	15	112	<0.03	NA	
Ansfelden, Austria, 1991 (Dea)	1360	300	16	116	<0.03	NA	Study No. 1125 Pfarl, 1992, PYRIDATE_106 Storage time: max. 3 months Method: 758 d Procedural recoveries: 74±4.7 % (n=3) at 0.05 mg/kg pyridafol
Leonding, Austria, 1991 (Dea)	1360	300	16-17	110	<0.03	NA	
Leonding/Biolabor, Austria, 1991 (LG5)	1360	300	15	112	<0.03	NA	
France, 1992 (not reported)	900	Not reported	Not reported	63 75	<0.03 <0.03	NA	Report No. 1207 Pfarl, 1994, PYRIDATE_104 Storage time: not reported Method 758 e 85±4.1 % (n=6) at 0.05 mg/kg pyridafol
France, year not reported (not reported)	900	Not reported	Not reported	Not reported	<0.03	NA	
	1800	Not reported	Not reported	Not reported	<0.03	NA	
Frankfurt, Germany, 1991 Trial 9149-01 (Mona)	900	400	33	92	<0.03	<0.05	Report No. 1129 Pfarl, 1992, PYRIDATE_107 Storage time: max. 6 months Method: 758 d Procedural recoveries: 82 % (n=2) at 0.05 mg/kg pyridafol
Gerolsbach, Germany, 1991 Trial 9149-04 (Julia)	900	400	33	77	<0.03	<0.05	

Sweet corn (Corn-on-the-cob)

Three field trials were conducted with sweet corn in France during the 1998 growing season (Gasser, 1999, PYRIDATE_100; Gasser, 2002). The trials received one application (at BBCH 16–18) at a rate of 900 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using method REM 191.01 with a LOQ of 0.02 mg/kg as pyridafol (0.04 mg/kg as pyridate).

Additional nine field trials with maize were considered if at an intermediate stage samples of maize cob (at BBCH 71-85) were taken. These trials were performed in France during the 1996 & 2000 growing seasons (Gasser, 2002, PYRIDATE_101; Krennhuber, 1997, PYRIDATE_102) and in Germany during the 1991 growing season. The trials received one application (at BBCH 16–33) at a rate of 900 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using method REM 191.01 with a LOQ of 0.02 mg/kg as pyridafol (0.04 mg/kg as pyridate) or method 758 d & e with a limit of quantification of 0.03 mg/kg as pyridafol (0.05 mg/kg expressed as pyridate). The results are shown in Table 62.

Table 62 Residues of pyridate and pyridafol in sweet corn from field trials in Europe with a single foliar application

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Centre Nord, France, 1998 RE98062 (Challenger)	900	250	16-18	45 59 69	Grains	<0.02 <0.02 <0.02	<0.05 <0.05 <0.05	Study No. 3116/98 Gasser, 1999, PYRIDATE_101 Storage time: max. 11 months Method: REM 191.01 Procedural recoveries: 79±8.5 % (n=6) at 0.02 & 0.2 mg/kg pyridafol
Aquitaine Sud, France, 1998 RE98063[1] (C40)	900	300	16-17	43 60 71	Cobs Grains	<0.02 <0.02 <0.02	<u><0.05</u> <0.05 <0.05	
Aquitaine Sud, France, 1998 RE98064[1] (C40)	900	300	17	43 60	Grains	<0.02 <0.02	<0.05 <0.05	
Marsillagues, France, 2000 (Cecilia)	900	400	16-17	60	Cobs (BBCH 71)	<0.02, <0.02 (<0.02)	<0.04, <0.04 (<u><0.04</u>)	Study No. 3013/00 Gasser, 2002, PYRIDATE_101 Storage time: max. 16 months Method: REM 191.01 Procedural recoveries: 75 % (n=2) at 0.02 & 0.2 mg/kg pyridafol
Peyrens, France, 1996 (Calis)	894	397	16-17	92	Cobs w/o husks (BBCH 83-85)	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<u><0.05</u>)	Study No. 1294 Krennhuber, 1997, PYRIDATE_102 Storage time: max. 3 months Method: 758 e Procedural recoveries: 91 % (n=2) at 0.05 mg/kg pyridate 91 % (n=2) at 0.03 mg/kg pyridafol
Garrevaques, France, 1996 (Cecilia)	907	403	16-17	86	Cobs w/o husks (BBCH 83-85)	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<u><0.05</u>)	
Montluel, France, 1996 (Perseval)	885	393	16	98	Cobs w/o husks (BBCH 83-85)	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<u><0.05</u>)	
Montanay, France, 1996 (Occitan)	899	399	16	96	Cobs w/o husks (BBCH 83-85)	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<u><0.05</u>)	

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Frankfurt, Germany, 1991 Trial 9149-01 (Mona)	900	400	33	82	Cobs	<0.03	<u><0.05</u>	Report No. 1129 Pfarl, 1992, PYRIDATE_107 Storage time: max. 6 months Method: 758 d Procedural recoveries: 80±4.1 % (n=4) at 0.05 mg/kg pyridafol
Seligenstadt, Germany, 1991 Trial 9149-02 (Dea)	900	400	33	77	Cobs	<0.03	<u><0.05</u>	
Groß Rönnau, Germany, 1991 Trial 9149-03 (Bonny)	900	400	33	76	Cobs	<0.03	<u><0.05</u>	
Gerolsbach, Germany, 1991 Trial 9149-04 (Julia)	900	400	33	63	Cobs	<0.03	<u><0.05</u>	

Notes:

[1] It was noted that trial RE98063and RE98064 were performed at the same location and year. However, sowing time differed by 2 weeks. Therefore the trials are considered as independent.

Feeding commodities

Alfalfa

A total of 12 field trials were conducted with alfalfa in Hungary during the 1989 growing season (Anonymous, 1989, PYRIDATE_069), in France during the 1992 & 1998 growing seasons; in Austria during the 1986 & 1987 growing seasons (Heegemann, 1986, PYRIDATE_076, Pfarl, 1990, PYRIDATE_077). The trials received one application (mostly at BBCH 14–16 or not stated) at a rate of 900, 1350 or 1800 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using methods REM 191.01 with a LOQ of 0.02 mg/kg as pyridafol (0.04 mg/kg as pyridate); 758 c, 758 d or 758 e with a LOQ of 0.03 mg/kg as pyridafol (0.05 mg/kg as pyridate). The results are in Table 63.

Table 63 Residues of pyridate and pyridafol in alfalfa (whole plant) from field trials in Europe using a single foliar application

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Vörös csillag, Hungary, 1989 (Lucerne)	1280	300	5-10 cm	90	NA-	<0.05	Study No.: 198.9 Anonymous, 1989, PYRIDATE_069 Storage time: 6.7 months Method: GC-ECD Procedural recoveries: 80 % (n=1) at 0.05 mg/kg pyridate
Thivilli, Northern France, 1998 (Alizé)[1]	885	393	30-35 cm	28	0.15, 0.13 (0.14)	0.28, 0.24 (<u>0.26</u>)	Study No. 3087/98 Gasser, 1999, PYRIDATE_070 Storage time: 8.1 months; Method: REM 191.01 Procedural recoveries: 101 % (n=1) at 0.5 mg/kg pyridate 74 % (n=1) at 0.02 mg/kg pyridafol

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Thivilli, Northern France, 1998 (Alizé)[1]	870	870	30-35 cm	28	0.05, 0.04 (0.045)	0.05, 0.07 (0.06)	Study No. 3062/98 Gasser, 1999, PYRIDATE_073 Storage time: 8.1 months; Method: REM 191.01 Procedural recoveries: 101 % (n=1) at 0.5 mg/kg pyridate 74 % (n=1) at 0.02 mg/kg pyridafol
Capendu, Southern France, 1998 (Provence)[1]	887	394	14-15	28	0.07, 0.08 (0.075)	0.13, 0.15 (0.14)	Study No. 3088/98 Gasser, 1999, PYRIDATE_071 Storage time: 8.9 months; Method: REM 191.01 Procedural recoveries: 101 % (n=1) at 0.5 mg/kg pyridate 74 % (n=1) at 0.02 mg/kg pyridafol
Capendu, Southern France, 1998 (Provence)[1]	887	394	14-15	28	0.13, 0.12 (0.12)	0.24, 0.22 (0.23)	Study No. 3063/98 Gasser, 1999, PYRIDATE_075 Storage time: 8.9 months; Method: REM 191.01 101 % (n=1) at 0.5 mg/kg pyridate 74 % (n=1) at 0.02 mg/kg pyridafol
Le Cres, Southern France, 1998 (Magali)[2]	880	391	15	27	0.03, 0.03 (0.03)	0.05, 0.05 (0.05)	Study No. 3089/98 Gasser, 1999, PYRIDATE_072 Storage time: 7.6 months; Method: REM 191.01 Procedural recoveries: 90 % (n=1) at 0.5 mg/kg pyridate 92 % (n=1) at 0.02 mg/kg pyridafol
Le Cres, Southern France, 1998 (Magali)[2]	910	404	15	29	<0.02, <0.02 (<0.02)	<0.05, <0.05 (<0.05)	Study No. 3064/98 Gasser, 1999, PYRIDATE_074 Storage time: 9.1 months; Method: REM 191.01 Procedural recoveries: 90 % (n=1) at 0.5 mg/kg pyridate 92 % (n=1) at 0.02 mg/kg pyridafol
Leonding, Austria, 1986 (not reported)	900	300	15-16	0 32 53 124	153, 147 (150) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	282, 271 (276) <0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05)	Study No. 872 Heegemann, 1986, PYRIDATE_076 Storage time: 5.4 months; Method: 758 c 81 % (n=2) at 25 & 50 mg/kg pyridate 85±4.1 % (n=6) at 0.03 & 0.05 mg/kg pyridafol
	1800	300	15-16	0 32 53 124	280, 245 (260) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	516, 452 (484) <0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05)	
Marchtrenk, Austria, 1987 (Suere)	1350	Not reported	Not reported	21 40 53	0.33, 0.29 (0.31) 0.036, 0.03 (0.033) <0.03, <0.03 (<0.03)	0.61, 0.53 (0.57) 0.066, 0.05 (0.033) <0.05, <0.05 (<0.05)	Study No. 1059 Pfarl, 1990, PYRIDATE_077 Storage time: max. 37 months Method: 758 d 85±1.5 % (n=4) at 50 mg/kg pyridate 77±8.5 % (n=6) at 0.05 & 0.1 mg/kg pyridafol

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Lungitz, Austria, 1987 (n.r.)	1350	Not reported	Not reported	0 18 41 64	78 0.20 <0.03 <0.03	144 0.37 <0.05 <0.05	(corrected for blank values)
Bourgogne, France, 1993 Trial: SFR R 92 590 (Alligio)	900	320	Not reported	49 56 63	<0.03 <0.03 <0.03	<0.05 <0.05 <0.05	Study No. 1181 Pfarl, 1995, PYRIDATE_078 Storage time: max. 15 months Method: 758 e 71±4.2 % (n=3) at 0.05 mg/kg pyridafol
Bourgogne, France, 1993 Trial: SFR R 92 591 (Alligro and Risy)	900	320	Not reported	41 49 56	<0.03 <0.03 <0.03	<0.05 <0.05 <0.05	

Notes:

[1] Trials performed at the same location and time. Therefore the trials could not be considered as independent.

[2] Trials performed at the same location. However, treatment and harvest time each differed by 6 weeks. Therefore the trials are considered as independent.

Clover

A total of 11 field trials were conducted with clover in France during the 1998 and 2000 growing seasons, in the United Kingdom during the 1998 growing season (Gasser, 1999, PYRIDATE_081) and in Austria during the 1989 growing season (Pfarl, 1989, PYRIDATE_087). The trials received one application (at various BBCH stages) at a rate of 900 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using methods REM 191.01 with a LOQ of 0.02 mg/kg as pyridafol (0.04 mg/kg as pyridate) or 758 d with a LOQ of 0.03 mg/kg as pyridafol (0.05 mg/kg as pyridate). The results are shown in Table 64.

Table 64 Residues of pyridate and pyridafol in clover (whole plant) from field trials in Europe conducted with a single foliar application

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Thiville, Northern France 2000 (Almirat)[1]	880	391	65-67	28	0.47, 0.68 (0.58)	0.86, 1.2 (1.0)	Study No.: 0021302 Pointurier, 2001, PYRIDATE_079 Storage time: 11 months Method: REM 191.01 Procedural recoveries: 75 % (n=2) at 0.02 & 0.2 mg/kg pyridate
Thiville, Northern France 2000 (Trincat)[1]	920	409	65-67	28	1.0, 0.65 (0.83)	1.8, 1.2 (1.5)	Study No.: 0021303 Pointurier, 2001, PYRIDATE_080 Storage time: 10 months Method: REM 191.01 Procedural recoveries: 75 % (n=2) at 0.02 & 0.2 mg/kg pyridate

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Thiville, Northern France 2000 (Heusers ostaat)[1]	981	436	65-67	28	0.08, 0.05 (0.065) ctrl: peak area 2x larger than in treated samples	0.14, 0.09 (0.12)	Study No.: 0021301 Pointurier, 2001, PYRIDATE_082 Storage time: 12 months Method: REM 191.01 Procedural recoveries: 75 % (n=2) at 0.02 & 0.2 mg/kg pyridate
Chipping Campden, Gloucestershire, United Kingdom 1998 (Britta)	922	400	14	0 28	23 <0.02	42 <0.04	Study No.: 3007/98 Gasser, 1999, PYRIDATE_081 Storage time: 5.7 months Method: REM 191.01 84 % (n=1) at 20 mg/kg pyridate 81 % (n=1) at 0.02 mg/kg pyridafol
Castelnau d'Aude, Southern France 1998 (Loras)[2]	875	389	40	0 14 28 43 60	11 ctrl: 0.07 1.6 0.43 0.19 0.21 ctrl: 0.05	20 2.9 0.79 0.35 0.39	Study No. 3118/97 Gasser, 1999, PYRIDATE_083 Storage time: 9.6 months Method: REM 191.01 Procedural recoveries: 79 % (n=1) at 20 mg/kg pyridate 146 % (n=1) at 0.02 mg/kg; 74 % (n=1) at 0.2 mg/kg pyridafol
Castelnau d'Aude, Southern France 1998 (Loras)[2]	862	383	40	0 14 28 43 60	19 2.4 0.39 0.44 0.85 ctrl: 0.02	35 4.3 0.70 0.79 1.5	Study No. 3120/97 Gasser, 1999, PYRIDATE_085 Storage time: 12 months Method: REM 191.01 Procedural recoveries: 75 % (n=1) at 20 mg/kg pyridate 87±19 % (n=3) at 0.02 & 0.2 mg/kg pyridafol
Mauguio, Southern France 1998 (Poppelsdorfer)[2]	947	421	33	0 15 29 43 60	12 0.75 0.06 0.04 0.14	22 1.4 0.11 0.07 0.26	Study No. 3119/97 Gasser, 1999, PYRIDATE_084 Storage time: 9.8 months Method: REM 191.01 81 % (n=1) at 20 mg/kg pyridate 94 % (n=2) at 0.02 & 0.2 mg/kg pyridafol
Mauguio, Southern France 1998 (Poppelsdorfer)[2]	910	404	33	0 15 29 43 60	24 0.95 0.13 0.06 0.13 ctrl: 0.02	44 1.8 0.24 0.11 0.24	Study No. 3121/97 Gasser, 1999, PYRIDATE_086 Storage time: 11 months Method: REM 191.01 81 % (n=1) at 20 mg/kg pyridate 81 % (n=2) at 0.02 & 0.2 mg/kg pyridafol
Hargelsberg, Austria, 1989 (Reichersberger neu)	900	300	15 cm	0 19 46	37 0.033 <0.03	68 0.061 <0.05	Study No. R 89-05 Pfarl, 1989, PYRIDATE_087 Storage time: max. 22 months Method: 758 d 74±16 % (n=?) at 50 mg/kg pyridate 70±4 % (n=?) at 0.05-0.1 mg/kg pyridafol
Neumarkt, Austria, 1989 (Reichersberger neu)	900	300	8 cm	0 20 48	58 0.097 0.060	107 0.18 0.11	
Niederneukirchen, Austria, 1989 (Reichersberger neu)	900	300	15 cm	0 15 43	51 <0.03 <0.03	94 <0.05 <0.05	

Notes:

[1] It was noted that trial 0021301, 0021302 and 0021303 were performed at the same location and year. However, treatment and harvest time each differed by 2-6 weeks. Therefore the trials are considered as independent.

[2] It was noted that trial 3118/97 and 3120/97 as well as trial 3119/97 and 3121/97 were performed at the same location and time. Therefore the trials could not be considered as independent.

Maize forage

18 field trials were conducted with maize in France during the 1996 & 2000 growing season and in Germany during the 1991 growing season (Pfarl, 1992, PYRIDATE_107). The trials received one application (at BBCH 14-17) at rates ranging between 900 and 1370 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using method REM 191.01 with a LOQ of 0.02 mg/kg as pyridafol (0.04 mg/kg as pyridate) or method 758 d & e with a limit of quantification of 0.03 mg/kg as pyridafol (0.05 mg/kg expressed as pyridate). The results are shown in Table 65.

Table 65 Residues of pyridate and pyridafol in maize forage from field trials in Europe conducted with a single foliar application

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues found [mg/kg][1]		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Marsillagues, France, 2000 (Cecilia)	900	400	16-17	0 29 60 77	Whole plant Forage (without cobs) Whole plant	21 <0.02 <0.02, <0.02 (<0.02) <0.02	38 <0.04 <0.04, <0.04 (<0.04) <u>≤0.04</u>	Study No. 3013/00 Gasser, 2002, PYRIDATE_101 Storage time: max. 16 months Method: REM 191.01 Procedural recoveries: 79 % (n=1) at 20 mg/kg pyridate 71±6.0 % (n=7) at 0.02-20 mg/kg pyridafol
Peyrens, France, 1996 (Calis)	894	397	16-17	0 30 61 92	Whole plant Rest of plant	59, 50 (55) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	106, 90 (99) <0.05, <0.05 (<0.05) <0.05, <0.05 (<u>≤0.05</u>) <0.05, <0.05 (<0.05)	Study No. 1294 Krennhuber, 1997, PYRIDATE_102 Storage time: max. 3 months Method: 758 e Procedural recoveries: 92±14 % (n=5) at 0.05 & 50 mg/kg pyridate 95±11 % (n=5) at 0.03 & 0.3 mg/kg pyridafol
Garrevaques, France, 1996 (Cecilia)	907	403	16-17	0 29 59 86	Whole plant Rest of plant	35, 25 (30) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	64, 45 (54) <0.05, <0.05 (<0.05) <0.05, <0.05 (<u>≤0.05</u>) <0.05, <0.05 (<0.05)	
Montluel, France, 1996 (Perseval)	885	393	16	0 28 60 98	Whole plant Rest of plant	16, 27 (22) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	29, 49 (40) <0.05, <0.05 (<0.05) <0.05, <0.05 (<u>≤0.05</u>) <0.05, <0.05 (<0.05)	

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues found [mg/kg][1]		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Montanay, France, 1996 (Occitan)	899	399	16	0 28 60 96	Whole plant Rest of plant	33, 40 (36) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03) <0.03, <0.03 (<0.03)	59, 72 (65) <0.05, <0.05 (<0.05) <0.05, <0.05 <u>(<0.05)</u> <0.05, <0.05 (<0.05)	
Leonding, Austria, 1990 (Dea)	1370	300	14	0 43 99	Whole plant	57 <0.03 <0.03	NA	Report No. 1092 Pfarl, 1991, PYRIDATE_103 Storage time: max. 6 months
Marchtrenk, Austria, 1990 (Dea)	1370	300	15	0 35 97	Whole plant	57 <0.03 <0.03	NA	Method 758 d 64±27 % (n=3) at 50 mg/kg pyridate 81±8.9 % (n=4) at 0.05 mg/kg pyridafol
Linz, Austria, 1990 (LG5)	1370	300	16	0 21 85	Whole plant	25 <0.03 <0.03	NA	
Ansfelden, Austria, 1991 (Dea)	1360	300	16	0 18 25 88	Whole plant	32 <0.03 <0.03 <0.03	NA	Study No. 1123 Pfarl, 1992, PYRIDATE_105 Storage time: max. 5 months
Leonding, Austria, 1991 (Dea)	1360	300	16-17	0 15 21 82	Whole plant	38 <0.03 <0.03 <0.03	NA	Method: 758 d Procedural recoveries: 80±6.9 % (n=3) at 50 mg/kg pyridate 83±14 % (n=7) at 0.05 mg/kg pyridafol
Leonding/Biolabor, Austria, 1991 (LG5)	1360	300	15	0 26 83	Whole plant	75 <0.03 <0.03	NA	
Ansfelden, Austria, 1991 (Dea)	1360	300	16	0 18 25 88	Whole plant	62 <0.03 <0.03 <0.03	NA	Study No. 1125 Pfarl, 1992, PYRIDATE_106 Storage time: max. 5 months Method: 758 d Procedural recoveries: 80±5.0 % (n=3) at 50 mg/kg pyridate 76±16 % (n=7) at 0.05 mg/kg pyridafol
Leonding, Austria, 1991 (Dea)	1360	300	16-17	0 15 21 82	Whole plant	37 <0.03 <0.03 <0.03	NA	
Leonding/Biolabor, Austria, 1991 (LG5)	1360	300	15	0 26 83	Whole plant	61 <0.03 <0.03	NA	
Frankfurt, Germany, 1991 Trial 9149-01 (Mona)	900	400	33	1 19 49 82	Whole plant Plant (leaf + stem)	15 0.31 0.038 0.038	27 0.56 0.068 <u>0.068</u>	Report No. 1129 Pfarl, 1992, PYRIDATE_107 Storage time: max. 9 months Method: 758 d Procedural recoveries: 75±5.4 % (n=5) at 50 mg/kg pyridate 73±11 % (n=7) at 0.05 mg/kg pyridafol
Seligenstadt, Germany, 1991 Trial 9149-02 (Dea)	900	400	33	1 17 49 77	Whole plant Plant (leaf + stem)	12 0.065 <0.03 <0.03	22 0.12 <0.05 <u><0.05</u>	
Groß Rönnau, Germany, 1991 Trial 9149-03 (Bonny)	900	400	33	1 14 45 76	Whole plant Plant (leaf + stem)	16 0.059 <0.03 <0.03	29 0.11 <0.05 <u><0.05</u>	

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues found [mg/kg][1]		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Gerolsbach, Germany, 1991 Trial 9149-04 (Julia)	900	400	33	1	Whole plant	12	22	
				4		2.0	3.6	
				30		0.074	0.13	
				63	Plant (leaf + stem)	<0.03	<0.05	

Additionally, three field trials were conducted with sweet corn forage in France during the 1998 growing season (Gasser, 1999, PYRIDATE_100). The trials received one application (at BBCH 16–18) at a rate of 900 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using method REM 191.01 with a LOQ of 0.02 mg/kg as pyridafol (0.04 mg/kg as pyridate) (Table 66).

Table 66 Residues of pyridate and pyridafol in sweet corn forage from field trials in France conducted with a single foliar application

Location, Year, Trial No., (variety)	Application			DAA	Sample	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH			Pyridafol	Pyridate equivalents	
Centre Nord, France, 1998 RE98062 (Challenger)	900	250	16-18	0	Whole plant	18	32	Study No. 3116/98 Gasser, 1999, PYRIDATE_101 Storage time: max. 11 months Method: REM 191.01 Procedural recoveries: 82±9.9 % (n=3) at 20 mg/kg pyridate 76±4.2 % (n=8) at 0.02-2 mg/kg pyridafol
				45		<0.02	<0.04	
				59		<0.02	<0.04	
				69		<0.02	<0.04	
Aquitaine Sud, France, 1998 RE98063 (C40)[1]	900	300	16-17	0	Whole plant	19	34	
				43	Forage (without cobs)	<0.02	<0.04	
				60		<0.02	<0.04	
				71		<0.02	<0.04	
Aquitaine Sud, France, 1998 RE98064 (C40)[1]	900	300	17	0	Whole plant	11	20	
				43	Forage (without cobs)	<0.02	<0.04	
				60		<0.02	<0.04	

Notes:

[1] It was noted that trial RE98063 and RE98064 were performed at the same location and year. However, sowing dates differed by 2 weeks. Therefore the trials are considered as independent.

Maize straw

Eleven field trials were conducted with maize in France during the 1996 & 2000 growing seasons (Gasser, 2002, PYRIDATE_101; Krennhuber, 1997, PYRIDATE_102) and in Austria during the 1990 & 1991 growing seasons (Pfarl, 1991, PYRIDATE_103; Pfarl, 1994, PYRIDATE_105; Pfarl, 1994, PYRIDATE_106). The trials received one application (at BBCH 16–18) at rates ranging between 900 to 1370 g ai/ha. Residues of pyridate and pyridafol, including conjugates were determined using method REM 191.01 with a LOQ of 0.02 mg/kg as pyridafol (0.04 mg/kg as pyridate) or method 758 d & e with a limit of quantification of 0.03 mg/kg as pyridafol (0.05 mg/kg expressed as pyridate). Results are shown in Table 67.

Table 67 Residues of pyridate and pyridafol in maize straw from field trials in Europe conducted with a single foliar application

Location, Year, Trial No., (variety)	Application			DAA	Residues (mg/kg)		Report, Reference, Storage period
	g ai/ha	L/ha	BBCH		Pyridafol	Pyridate equivalents	
Marsillagues, France, 2000 (Cecilia)	900	400	16-17	117	<0.02, <0.02 (<0.02)	<0.04, <0.04 (<u>0.04</u>)	Study No. 3013/00 Gasser, 2002, PYRIDATE_101 Storage time: max. 16 months Method: REM 191.01 Procedural recoveries: 81 % (n=2) at 0.02 & 0.2 mg/kg pyridafol
Peyrens, France, 1996 (Calis)	894	397	16-17	134	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<u>0.05</u>)	Study No. 1294 Krennhuber, 1997, PYRIDATE_102 Storage time: max. 3 months Method: 758 e Procedural recoveries: 105 % (n=2) at 0.05 mg/kg pyridate 96 % (n=2) at 0.03 mg/kg pyridafol
Garrevaques, France, 1996 (Cecilia)	907	403	16-17	142	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<u>0.05</u>)	
Montluel, France, 1996 (Perseval)	885	393	16	144	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<u>0.05</u>)	
Montanay, France, 1996 (Occitan)	899	399	16	138	<0.03, <0.03 (<0.03)	<0.05, <0.05 (<u>0.05</u>)	
Leonding, Austria, 1990 (Dea)	1370	300	14	139	<0.03	NA	Report No. 1092 Pfarl, 1991, PYRIDATE_103 Storage time: max. 2 months Method 758 d 92±26 % (n=3) at 0.05 mg/kg pyridafol
Marchtrenk, Austria, 1990 (Dea)	1370	300	15	131	<0.03	NA	
Linz, Austria, 1990 (LG5)	1370	300	16	121	<0.03	NA	
Ansfelden, Austria, 1991 (Dea)	1360	300	16	116	<0.03	NA	Study No. 1123 Pfarl, 1992, PYRIDATE_105 Storage time: max. 2 months Method: 758 d Procedural recoveries: 88±7.9 % (n=3) at 0.05 mg/kg pyridafol
Leonding, Austria, 1991 (Dea)	1360	300	16-17	110	<0.03	NA	
Leonding/Biolabor, Austria, 1991 (LG5)	1360	300	15	112	<0.03	NA	
Ansfelden, Austria, 1991 (Dea)	1360	300	16	116	<0.03	NA	Study No. 1125 Pfarl, 1992, PYRIDATE_106 Storage time: max. 3 months Method: 758 d Procedural recoveries: 85±12 % (n=3) at 0.05 mg/kg pyridafol
Leonding, Austria, 1991 (Dea)	1360	300	16-17	110	<0.03	NA	
Leonding/Biolabor, Austria, 1991 (LG5)	1360	300	15	112	<0.03	NA	

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of residue during processing

No study was provided to simulate pasteurization, boiling, baking and brewing and sterilization

Residues after processing

Peanut and maize

The transfer of the total pyridate residue into maize and peanut oil was investigated with radiolabelled incurred residues from nature of residue studies (Zohner, 1988, PYRIDATE_088). The nature of residue

studies were performed with exaggerated rates of 1×3600 g ai/ha (4×) for peanut and 1×1800 g ai/ha (2×) in maize. Samples of maize grain were harvested at 118 DAT, while peanuts were harvested at 91 and 223 DAT. For processing both commodities were extracted with petroleum ether in a Soxhlet apparatus, followed by evaporation of the solvent. The total radioactivity the RAC was determined by combustion, followed by LSC. The oil samples were analysed by TLC and gel permeation chromatography. Additionally an alkaline hydrolysis was performed to determine distribution of the radioactivity in the glycerol and fatty acid moieties (Table 68).

Table 68 Total radioactivity residues (TRR) found in peanut and corn samples before and after oil processing and oil yields, calculated on a dry weight basis.

Crop	Project No.	Oil Yield* (%)	TRR (mg/kg)	
			Before oil processing	In oil
Maize grain	M8503M	3.6	0.016	0.019
		4.1	0.019	0.018
Peanut Nutmeat	M8503E	30.7	0.054	0.050
		30.7	0.054	0.050
	M8713	34.5	0.127	0.100
		32.8	0.136	0.106

Notes:

*Average of duplicate determinations.

Attempts to separate the radioactivity from the oil by GPC and to identify individual components were not successful. The following alkaline hydrolysis of the oil demonstrated no release of residues >LOD and a uniform distribution of radioactivity between fatty acids and glycerol.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Lactating cows

The transfer of residues of pyridate into animal matrices was investigated in a study with dairy cows (Cameron, *et al*., 1989, PYRIDATE_089). The study was conducted with [4,5-pyridazine-^{14}C]-radiolabelled pyridate at treatment rates of 1 (1×), 3.3 (3×), and 10 (10×) ppm (0.03, 0.1 and 0.3 mg/kg bw) for 28 days.

The cows in the treatment groups (three animals per group) were treated twice daily via an implanted cannula directly into the rumen. Milk samples were collected twice daily. Additionally, selected milk samples (day 2, 9, 16, 23) were separated into fat, curds and whey samples. Whole blood samples were taken immediately before each treatment and after treatment on day 1 and day 27. Plasma was separated from blood samples by centrifugation. All cows were sacrificed within 6 hours of administration of the last dose. Samples of liver, kidney, heart, lung, brain, skeletal muscle (shoulder, rump, dorsal), subcutaneous fat, perirenal fat, whole blood, plasma, milk (collected immediately before sacrifice), bladder urine and bile were collected and taken for analysis.

Total radioactivity in liquid samples such as urine, plasma, milk and various extracts were directly measured by LSC. Whole blood and tissue samples were subjected to combustion prior to the determination of total radioactivity by LSC.

Muscle, liver and kidney samples were homogenized with methanol. Fat samples were ground with acid washed sand and mixed with methanol. Plasma and bile samples were mixed with methanol and ethanol, respectively. Urine was cleaned up after dilution with citrate buffer on a C18 cartridge.

Conjugates in kidney, liver and urine were cleaved with 2 mol/L hydrochloric acid. Characterization of the extracts was carried out by HPLC against reference standards.

Residues in milk plateaued after one day in all dosing groups. In edible tissues residues were highest in kidney ranging from 0.19 mg/kg (1×) to 1.9 mg/kg (10×). The findings in tissues are summarised in Table 86. An individual component was only identified in muscle, kidney, liver and fat of the 10× dosing group with a retention time close to pyridafol. Additionally, in urine and bile pyridafol and its glucuronides were identified (Table 69).

Table 69 Average total radioactive residues (TRR) found in animal samples following twice daily intrarumenal administration of [^{14}C]-pyridate at concentrations of 1, 3.3 and 10 ppm

Portion analysed	Sampling point		TRR (mg/kg or mg/L)			
			Control	1 ppm	3.3 ppm	10 ppm
Milk	Day 1	a.m.	n/d	n/d	n/d	n/d
		p.m.	n/d	0.002	0.009	0.018
	Day 2	a.m.	n/d	0.003	0.009	0.020
		p.m.	n/d	0.003	0.014	0.026
	Day 3	a.m.	n/d	0.002	0.011	0.023
		p.m.	n/d	0.003	0.015	0.026
	Day 4	a.m.	n/d	0.003	0.011	0.024
		p.m.	n/d	0.003	0.014	0.033
	Day 5	a.m.	n/d	0.003	0.011	0.022
		p.m.	n/d	0.003	0.013	0.027
	Day 6	a.m.	n/d	0.003	0.010	0.023
		p.m.	n/d	0.003	0.012	0.030
	Day 7	a.m.	n/d	0.003	0.010	0.022
		p.m.	n/d	0.003	0.014	0.026
	Day 8	a.m.	n/d	0.003	0.011	0.022
		p.m.	n/d	0.003	0.014	0.031
	Day 9	a.m.	n/d	0.003	0.011	0.025
		p.m.	n/d	0.003	0.013	0.029
	Day 10	a.m.	n/d	0.003	0.012	0.026
		p.m.	n/d	0.003	0.014	0.029
	Day 11	a.m.	n/d	0.003	0.011	0.023
		p.m.	n/d	0.003	0.011	0.029
	Day 12	a.m.	n/d	0.003	0.012	0.021
		p.m.	n/d	0.003	0.014	0.031
	Day 13	a.m.	n/d	0.003	0.011	0.022
		p.m.	n/d	0.003	0.014	0.029
	Day 14	a.m.	n/d	0.003	0.011	0.021
		p.m.	n/d	0.004	0.010	0.029
	Day 15	a.m.	n/d	0.003	0.011	0.022
		p.m.	n/d	0.003	0.016	0.027
	Day 16	a.m.	n/d	0.002	0.012	0.021
		p.m.	n/d	0.003	0.016	0.027
	Day 17	a.m.	n/d	0.003	0.011	0.024
		p.m.	n/d	0.003	0.015	0.031
	Day 18	a.m.	n/d	0.003	0.013	0.023
		p.m.	n/d	0.003	0.016	0.030
	Day 19	a.m.	n/d	0.003	0.012	0.023
		p.m.	n/d	0.003	0.015	0.029
	Day 20	a.m.	n/d	0.003	0.011	0.023
		p.m.	n/d	0.004	0.016	0.026
	Day 21	a.m.	n/d	0.003	0.012	0.023
		p.m.	n/d	0.004	0.016	0.021

Portion analysed	Sampling point		TRR (mg/kg or mg/L)			
			Control	1 ppm	3.3 ppm	10 ppm
	Day 22	a.m.	n/d	0.003	0.013	0.024
		p.m.	n/d	0.003	0.016	0.031
	Day 23	a.m.	n/d	0.003	0.013	0.022
		p.m.	n/d	0.003	0.016	0.032
	Day 24	a.m.	n/d	0.002	0.013	0.024
		p.m.	n/d	0.003	0.016	0.029
	Day 25	a.m.	n/d	0.002	0.013	0.023
		p.m.	n/d	0.003	0.017	0.031
	Day 26	a.m.	n/d	0.003	0.012	0.023
		p.m.	n/d	0.003	0.015	0.032
	Day 27	a.m.	n/d	0.003	0.012	0.022
		p.m.	n/d	0.003	0.013	0.030
	Day 28	a.m.	n/d	0.003	0.013	0.024
		p.m.	n/d	0.003	0.015	0.031
Fat	Day 23		n/d	0.004	0.013	0.01
Curds	Day 23		n/d	0.009	0.036	0.07
Whey	Day 23		n/d	0.002	0.006	0.01
Plasma	Day 1	6 h post 1st dose	n/d	0.014	0.056	0.14
	Day 27	6 h post 53rd dose	n/d	0.016	0.054	0.18
Blood	Day 1	6 h post 1st dose	n/d	0.008	0.040	0.10
	Day 27	6 h post 53rd dose	n/d	0.010	0.038	0.12
Liver	Post-sacrifice		n/d	0.019	0.118	0.20
Kidney	Post-sacrifice		n/d	0.194	0.575	1.88
Heart	Post-sacrifice		n/d	0.009	0.033	0.08
Lung	Post-sacrifice		n/d	0.009	0.031	0.08
Brain	Post-sacrifice		n/d	0.001	0.005	0.01
Skeletal muscle (dorsal)	Post-sacrifice		n/d	0.004	0.008	0.04
Skeletal muscle (rump)	Post-sacrifice		n/d	0.003	0.009	0.02
Skeletal muscle (shoulder)	Post-sacrifice		n/d	0.002	0.008	0.02
Subcutaneous fat	Post-sacrifice		n/d	0.003	0.017	0.02
Perirenal fat	Post-sacrifice		n/d	0.007	0.012	0.01
Bile	Post-sacrifice		n/d	0.054	0.196	0.68
Whole blood	Post-sacrifice		n/d	0.012	0.047	0.12
Plasma	Post-sacrifice		n/d	0.017	0.068	0.18
Bladder urine	Post-sacrifice		n/d	1.976	6.034	20.33
Milk	Post-sacrifice		n/d	0.003	0.015	0.03

Notes:

Data are expressed as mg of pyridate equivalents per sample material and are the mean of triplicate analyses.

n/d = Not detected.

Laying hens

The transfer of residues of pyridate into animal matrices was investigated in a study with laying hens (Johnston*, et al*., 1989, PYRIDATE_090). The study was conducted with [4,5-pyridazine-^{14}C]-radiolabelled pyridate at treatment rates of 1.3 (1×), 4 (3×), and 13 (10×) ppm (0.1, 0.3 and 1 mg/kg bw) for 28 days.

The hens in the treatment groups (10 animals per group) were treated with pyridate by gavage twice daily. Eggs were collected twice daily throughout the study period and separated into yolks and whites. All hens were sacrificed within 6 hours of administration of the last dose and samples of muscle (leg, breast), fat pad, skin and fat, liver, whole blood, plasma, kidney and heart were collected and taken for analysis.

Total radioactivity in liquid samples such as plasma, egg yolk and white and various extracts were directly measured by LSC. Whole blood and tissue samples were subjected to combustion prior to the determination of total radioactivity by LSC.

Ground kidney samples were suspended in water, followed by precipitation of protein with trichloroacetic acid. Muscle and fat samples were mixed with quartz sand, followed by extraction with methanol. Liver, plasma, excreta, egg white and yolk were extracted with methanol. Conjugates in excreta, kidney, liver and plasma were cleaved with 2 mol/L hydrochloric acid. Characterization of the extracts was carried out by HPLC against reference standards.

Residues in eggs whites plateaued after two days in all dosing groups, while in egg yolks a plateau was reached after 5–7 days. In edible tissues residues were highest in kidney ranging from 0.05 mg/kg (1×) to 0.23 mg/kg (10×). The findings in tissues are summarised in Table 87. An individual component was identified in egg yolk, kidney, liver and muscle of the 10x dosing group only, with a retention time close to pyridafol (Table 70).

Table 70 Average total radioactive residues (TRR) found in various animal samples following twice daily administration of [^{14}C]-pyridate at concentrations of 0 (Group 1), 1.3 (Group 2), 4 (Group 3) and 13 ppm (Group 4)

Portion analysed	Sampling point	TRR (mg/kg or mg/L)			
		Control	1.3 ppm	4 ppm	13 ppm
Egg white	Day 1	n/d	0.002	n/d	0.004
	Day 2-28	n/d	0.003-0.005	0.008-0.010	0.024-0.032
Egg yolk	Day 1	n/d	n/d	0.001	0.004
	Day 2-28	n/d	0.002-0.003	0.002-0.008	0.005-0.023
Liver	Post-sacrifice	n/d	0.023	0.062	0.090
Kidney	Post-sacrifice	n/d	0.050	0.136	0.228
Heart	Post-sacrifice	n/d	0.011	0.026	0.035
Leg muscle	Post-sacrifice	n/d	0.004	0.008	0.014
Breast muscle	Post-sacrifice	n/d	0.003	0.007	0.009
Fat pad	Post-sacrifice	0.001	0.002	0.003	0.008
Skin and fat	Post-sacrifice	n/d	0.008	0.021	0.054
Plasma	Post-sacrifice	0.001	0.023	0.062	0.130
Whole blood	Post-sacrifice	0.001	0.017	0.053	0.071

Note:

Data are expressed as the average mg of pyridate equivalents per sample material and are averages of ten analyses; n/d = Not detected.

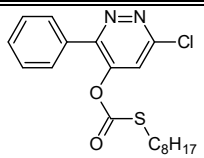
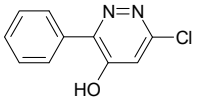
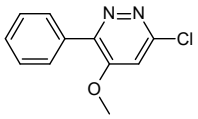
APPRAISAL

Pyridate is an herbicide of the phenylpyridazine class used to control annual broad-leaved weeds. It acts by inhibiting the photosynthetic electron transport at the photosystem II. The IUPAC name of pyridate is *O*-6-chloro-3-phenylpyridazine-4-yl *S*-octyl thiocarbonate

Pyridate was scheduled at the Fiftieth Session of the CCPR for evaluation as a new compound by the 2020 JMPR, reviewed by the 2019 JMPR for toxicology, where an ADI of 0–0.2 mg/kg bw and an ARfD of 2 mg/kg bw were established. The residue assessment was rescheduled to the current JMPR.

The Meeting received information on identity, physicochemical properties, metabolism (plant, confined rotational crops and animals), environmental fate, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials, fate of residues in processing, and livestock feeding studies.

Table 71 Abbreviations used for relevant compounds referred to in the appraisal

Code	Name	Structure
Pyridate (CL 11344)	6-chloro-3-phenylpyridazine-4-yl-S-octyl-thiocarbonate	 378.9 g/mol
Pyridafol (CL 9673)	6-chloro-4-hydroxy-3-phenylpridazine	 206.6 g/mol
Pyridafol-O-methyl (CL 9673-OMe; CL 9869)	6-chloro-4-methoxy-3-phenylpridazine	 220.7 g/mol

Physical and chemical properties

Pyridate and pyridafol (CL 9673) are not volatile. Pyridate is hydrolytically and photolytically unstable, resulting in the cleavage of the ester bond leading to pyridafol. The n-octanol water partition coefficient (log P_{ow}) of pyridate is 4.0.

Plant metabolism

The Meeting received plant metabolism studies in broccoli, maize and peanuts following application of [4,5-pyridazine-^{14}C]-radiolabelled pyridate.

Broccoli

[4,5-pyridazine-^{14}C]-radiolabelled pyridate was foliar applied once at the 2–3 leaf stage using a rate of 1.8 kg ai/ha under combined greenhouse and outdoor conditions. Plant samples were taken at 0 (immediately after the treatment), 14, 45, 73, 94 and 108 DAT.

The TRR was highest at 20 mg eq/kg in leaves immediately after treatment and declined to 0.011 mg eq/kg in samples taken at 108 DAT. In the edible parts (flower heads), TRR was significantly lower at 0.009 mg eq/kg (DAT 75, 96, 108 combined).

Broccoli samples were extracted with acetone and acetone/water (8:2). Conjugates were hydrolyzed with 2 mol/L hydrochloric acid and β-glucosidase in extracts. Extracted radioactivity in the leaves and edible parts (flowers) was high, ranging between 75-100 percent TRR.

Post-extracted solids (PES) in leaf samples (14 or 45 DAT) was further subjected to 2 mol/L acetonic HCl and enzymatic hydrolysis for characterisation of radioactivity in natural products, such as starch, proteins, pectin and lignin.

Characterization of the radioactivity in broccoli flowers was not performed due to their low radioactivity in the extract. Parent pyridate was a major identified residue in leaf samples from 0 and 14 DAT, accounting for 42–60 percent TRR (2.6–12 mg eq/kg), while in leaves from 45 DAT parent pyridate was not detected at all. As a major metabolite, pyridafol was detected in leaf samples from 0 and 14 DAT, accounting for 7.4–18 percent TRR (0.45–3.5 mg eq/kg). In addition, N- and O-glucoside conjugates of pyridafol were found as the predominant residue at 14 and 45 DAT (19–25 percent TRR, 0.06–1.5 mg eq/kg). Two unknown components were detected at significant levels in leaf samples from 14 and 45 DAT, in combination accounting for 7.1 percent (0.43 mg eq/kg) and 23 percent TRR (0.07 mg eq/kg), respectively. One of these was characterized as very polar and resistant to hydrolysis treatment. The other was also characterized as being highly polar and susceptible to hydrolysis with beta-glucosidase (no corresponding aglycon identified).

Maize

The Meeting received two studies with maize (study 1: indoors at nights and outdoors during daytime; study 2: only outdoors), both performed with [4,5-pyridazine-^{14}C]-radiolabelled pyridate. Maize plants at BBCH 14–15 (study 1) or BBCH 16–17 (study 2) were treated once with pyridate at 1.8 (study 1) or 1.73 (study 2) kg ai/ha. Plant samples were taken at 0 (immediately after the treatment), 14, 45, 90 and 118 (study 1) or 148 (study 2) DAT.

Generally, the detected radioactivity was higher in study 2. In both studies the TRR was highest in leaves ranging between 17 mg eq/kg (study 1) and 168 mg eq/kg (study 2) at 0 DAT and declined to 0.5 mg eq/kg (study 1) at 108 DAT and 0.27 mg eq/kg (study 2) at 148 DAT. The radioactivity in newly grown plant parts was at least one order in magnitude lower in both studies at the respective sampling time points compared to the treated leaves, indicating limited translocation in the plant. In maize grain, the TRR was low in both studies ranging between 0.01-0.014 mg eq/kg.

In both studies, samples were solvent extracted with acetone and with acetone/water (8:2), followed by a harsher acidic or enzymatic hydrolysis of the PES. Solvent extracted radioactivity was high in the treated leaves from 0–14 DAT, ranging between 82–100 percent, but was lower in treated leaves from 45–108 DAT and in newly grown leaves, ranging between 24–75 percent TRR.

For maize grain, the radioactivity in the extract was < 0.01 mg eq/kg (study 1, 118 DAT) and no further analysis of the radioactivity was performed. No extraction was performed in grain samples from study 2 (148 DAT).

In the treated leaves from study 1 taken at 0 and 14 DAT, parent pyridate was a major identified residue, accounting for 82 percent TRR (14 mg eq/kg) and 11 percent TRR (0.31 mg eq/kg), respectively. In leaves taken at later DAT, parent pyridate was at or below the LOD.

As a major metabolite, pyridafol was detected in leaf samples taken at 0 and 14 DAT, accounting for 15–16 percent TRR (0.44–2.6 mg eq/kg), decreasing to levels of around or below the LOD in leaf samples taken at later DAT, its N- and O-glucosides accounting for 5.4–7.0 percent TRR (0.07–0.15 mg eq/kg).

Two unknown components were detected at significant levels in leaf samples taken at 14–90 DAT, accounting for a total of 15–38 percent TRR (0.07-0.69 mg eq/kg). One compound was characterized as being highly polar and was susceptible to hydrolysis with beta-glucosidase (no corresponding aglycon identified). The other decomposed into many unknown compounds following acid hydrolysis, however none could be attributed to pyridafol.

In the treated leaves of study 2 (14 DAT only), neither parent pyridate (3.0 percent TRR; 0.80 mg eq/kg), nor pyridafol (5.3 percent TRR; 1.4 mg eq/kg) were identified as major residues. Instead, three unknown components accounted for 9.5 percent TRR (2.5 mg eq/kg), 20 percent TRR (5.3 mg eq/kg) and 41 percent TRR (11 mg eq/kg).

Peanut

[4,5-pyridazine-^{14}C]-radiolabelled pyridate was foliar applied once to peanut plants (12.7 cm high) using a rate of 3.6 kg ai/ha under combined greenhouse and outdoor conditions. Plant samples were taken at 0 (immediately after the treatment), 14, 45 and 219 DAT.

The TRR was highest at in peanut forage taken immediately after treatment at 59 mg eq/kg and in hay from 45 DAT at 38 mg eq/kg. However, levels in both matrices declined to 0.22 mg eq/kg in forage and 1.5 mg eq/kg in hay taken at 219 DAT. In peanut hulls and nutmeat from 219 DAT, TRR levels were 0.36 mg eq/kg and 0.04 mg eq/kg, respectively.

The samples were extracted with acetone and acetone/water (8:2). Conjugates were hydrolyzed with 2 mol/L hydrochloric acid and β-glucosidase. Extracted radioactivity was high from peanut forage taken at 0 and 14 DAT, ranging between 78–100 percent TRR, but lower for forage samples taken at later DAT, as well as for peanut hay and hulls ranging between 38–64 percent TRR. For nutmeat, the extracted radioactivity was even lower at 33 percent TRR.

The PES was further subjected to harsher treatments using acid and enzymatic hydrolysis as well as characterisation of radioactivity into natural products for forage samples (45 DAT), demonstrating assignment to natural constituents such as starch, proteins, pectin and lignin.

Characterization of the residue in the nutmeat was not performed due to its low radioactivity in the extract.

Parent pyridate and pyridafol were major identified residues in forage samples from 0 DAT only, accounting for 86 percent TRR (51 mg eq/kg) and 10 percent TRR (6.2 mg eq/kg), respectively. In forage from later DAT, as well as in hay or hulls levels of pyridate and pyridafol were much lower (up to 4.6 percent TRR, 0.83 mg eq/kg) or non-detectable and also pyridafol-N or O-glucoside was found at low levels not exceeding 5.6 percent of the TRR (max. 1.1 mg eq/kg in hay). Two unknown components were detected at significant levels in forage (14-45 DAT), hay and hulls accounting for 14–19 percent TRR (1.4–5.4 mg eq/kg), 10–18 percent TRR (0.15–6.9 mg eq/kg) and 11 percent (0.04 mg eq/kg), respectively. One minor identified metabolite was pyridafol-OMe at up to 4.7 percent TRR (0.07 mg eq/kg) in hay (215 DAT).

Alfalfa

Reference to an additional plant metabolism study for the use of pyridate on alfalfa was made in the storage stability study on incurred radioactive residues. The study was not provided to the Meeting.

In summary, the metabolic pathway of pyridate in broccoli, maize and peanut was similar. In all studies, pyridate did undergo rapid hydrolytic cleavage into pyridafol. Biotransformation of pyridafol occurred mainly by glucosidic conjugation, yielding in the pyridafol-N-glucoside and pyridafol-O-glucoside. Further degradation led to highly polar metabolites, before the radioactivity was incorporated into the carbon pool of natural plant constituents. Only limited translocation in the plant was observed.

Environmental fate

For the investigation of the environmental fate of pyridate, the Meeting received studies on hydrolysis, aerobic soil degradation, soil photolysis and on the behaviour in confined rotational crops.

Hydrolysis

Pyridate was shown to be susceptible to hydrolysis, by cleavage of the ester bond leading to pyridafol. DT_{50} values at 25 °C ranged between 117 hours at pH 4 to 6.2 hours at pH 9. The Meeting concluded that hydrolysis of pyridate will be a significant route of degradation in the aquatic environment.

Aerobic soil metabolism

The rate of degradation of pyridate and its metabolite pyridafol was studied in various aerobic soils using [4,5-pyridazine-^{14}C]-radiolabelled pyridate, pyridafol and pyridafol-OMe. Rapid degradation of pyridate was observed at 20°C with estimated half-lives ranging from 0.3 to 3.3 days. Metabolite pyridafol peaked at day 1 or 2 at a maximum of 72–91 percent AR and showed moderate degradation with half-lives ranging from 17–43 days. Additionally, metabolite, pyridafol-OMe was detected at up to 3.5–16 percent AR on day 7–70 and showed moderate degradation with half-lives ranging from 12–25 days. The Meeting concluded that pyridate and its metabolites are not persistent in soil.

Soil photolysis

The soil surface photolytic behaviour of pyridate and its metabolite pyridafol was studied in an aerobic soil using [4,5-pyridazine-^{14}C]-radiolabelled pyridate. Under the assumption of average daylight of 12 hours, half-lives were estimated for pyridate and pyridafol at 1.8 days and 19 days respectively. Further degradation products were not identified. The Meeting concluded that photolysis is a significant degradation pathway for pyridate and moderately affects its metabolite pyridafol.

Rotational crop metabolism

Confined rotational crop studies

The Meeting received one confined rotational crop study under mixed outdoor and indoor conditions with lettuce, carrots and spring barley grown in rotation.

The study was conducted with [4,5-pyridazine-^{14}C]-radiolabelled pyridate applied at a rate equivalent to 1.8 kg ai/ha to a silty loam soil and plant back intervals (PBIs) of 28 and 56 days. The nature and level of radioactive residues from the 28 day PBI were investigated in lettuce at 97 DAT, in carrots at 133 DAT and in barley at 163 DAT. In crops from the 56 day PBI residues were investigated in lettuce at 156 DAT, in carrots at 169 DAT and in barley at 209 DAT.

Radioactivity in plant samples was generally low (< 0.01 mg eq/kg), except for barely straw (up to 0.1 mg eq/kg) and barley grain (up to 0.030 mg eq/kg).

Samples were extracted with methanol and methanol/water (8:2). Conjugates present in the extracts were hydrolysed overnight with 2 mol/L HCl. Extractability for all samples was between 19–83 percent TRR. No characterization of the unextracted residue was performed.

Characterization and identification of the radioactivity was only performed in the extracts of barley straw. While no pyridate or metabolites could be identified, the main portion of the extracted radioactivity was allocated to saccharides.

The Meeting concluded that uptake from soil is low and pyridate in metabolized into highly polar metabolites, before the radioactivity was incorporated into the carbon pool.

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating cows and goats and laying hens where animals were dosed with [4,5-pyridazine-^{14}C]-radiolabelled pyridate.

Rats

The metabolism of pyridate in rats was reviewed in the framework of the toxicological evaluation by the WHO Core Assessment Group of the 2019 JMPR.

Cattle

[4,5-pyridazine-14C]-radiolabelled pyridate was administered once as a single dose of 0.3 mg/kg bw (equivalent in feed not stated) at day 1 and 14 by intra-ruminal injection to one cow. After administration of the first dose, urine and faeces were collected once daily for 7 consecutive days (except urine which was collected three times within the first 24 h). Milk was collected twice daily throughout the study. The animal was sacrificed approximately 6 hours after administration of the second dose and tissues were collected.

After three days, the elimination of administered radioactivity was complete. The main route occurred via urine (92 percent AR), followed by faeces (8.6 percent AR).

In organs and tissues, radioactivity was significantly lower with 1.957 mg eq/kg in the kidney, 0.138 mg eq/kg in the liver and < 0.0034 mg eq/kg in the muscle. In milk the total radioactivity was low too, reaching a maximum of 0.03 mg eq/kg after 7 hours. After 31 hours, no radioactivity was detected in any milk samples.

Characterization of the radioactivity in edible tissues was only done for liver and kidney. While no information was given on the extractability of the residue, co-chromatography of the extracts with reference standards identified pyridafol in kidney (level not given), as well as pyridafol-N- and O-glucoside (up to 0.1 mg eq/kg) in liver.

Goats

[4,5-pyridazine-^{14}C]-radiolabelled pyridate was administered orally once daily to one lactating goat at 2.8 ppm (0.38 mg/kg bw and d) for 10 consecutive days. Urine and faeces were collected once daily, while milk was collected twice daily. The animal was sacrificed approximately 24 hours after the last dose and samples of organs, tissues and body fluids were collected.

The majority of the radioactivity was found in urine (95 percent AR) followed by faeces (6.5 percent AR). Radioactive residues in the edible tissues were low at 0.019 mg eg/kg and 0.033 mg eq/kg in

liver and kidney, respectively. Similar levels were found in milk, ranging between 0.015–0.048 mg eq/kg. A plateau was reached in milk after 3 days of consecutive administration. Residues in muscle and fat were 0.003 mg eq/kg and 0.009 mg eq/kg, respectively.

Extraction with acetone/water (8:2) followed by acetone released 63 percent TRR (0.01 mg eq/kg) from liver and 87 percent TRR (0.027 mg eq/kg) from kidney. Since radioactivity was low, identification was only successful in kidney, where metabolites pyridafol and pyridafol-OMe were tentatively identified, present at 32 percent TRR (0.010 mg eq/kg) and 48 percent TRR (0.015 mg eq/kg), respectively. In milk only pyridafol was identified at 49–71 percent TRR (0.012–0.015 mg eq/kg).

Poultry

A metabolism study was performed with 6 laying hens and 6 broiler chickens receiving a single oral dose of [4,5-pyridazine-^{14}C]-radiolabelled pyridate at 2.5–4.7 ppm (0.2 mg/kg bw and d). Excreta were collected once daily up to 96 h post dose, while eggs were collected twice daily if possible and separated in to egg yolk and white. All animals were sacrificed at 96 hours post dose. No individual organs or tissues were collected.

Within 24 hours post-dose, the majority of the radioactivity was found in excreta at 93-96 percent, increasing to 97–99 percent after 96 hours, demonstrating fast elimination.

Detected radioactivity in egg yolks and egg whites was consistently <LOQ for throughout the sampling time of 0–96 h post dose, with the exception of egg whites collected at 24–48 h post dose with a mean of 0.004 mg eq/kg.

Since no organs or tissue were collected, no further identification or characterization of the radioactive residues was performed. However, in excreta, metabolites pyridafol and hydroxylated pyridafol accounted for up to 74 percent TRR and 44 percent TRR, respectively.

A second metabolism study was performed with 9 laying hens receiving [4,5-pyridazine-^{14}C]-radiolabelled pyridate orally once daily for 5 consecutive days at ~3 ppm (0.19 mg/kg bw and d). Excreta and eggs were collected once daily and during the depuration phase additionally at 4, 8, and 24 hours after the last treatment. Eggs were separated in yolk and white. Three hens each were sacrificed at 8 hours, 3 days and 7 days after the final dose and organs, tissues and body fluids were collected.

Within 8–168 hours post-dose, the majority of the radioactivity was found in excreta at 93-96 percent AR, indicating rapid elimination. Radioactive residues in the edible matrices after 8 hours depuration were generally low, at 0.04 mg eq/kg in kidney, 0.02 mg eq/kg in liver and < 0.01 mg eq/kg in all other edible tissues. Similar levels were found in egg samples with maximum residues in yolks and whites at 0.007 mg eq/kg and 0.01 mg eq/kg, respectively.

No further identification or characterization of the radioactive residues in organs or tissues was performed.

In summary, the Meeting concluded that in all species investigated (cows, goats, hens and rats), the total administered radioactivity was predominantly eliminated in excreta. Information on the metabolic pathway was scarce, mostly due to the low levels of radioactivity in various organs and tissues, but seems comparable between species. The only metabolites identified in edible matrices were pyridafol, found in goat kidney and milk, and pyridafol-OMe in goat kidney.

Methods of analysis

The Meeting received information on analytical methods for pyridate in plant and animal matrices.

For matrices of plant origin, the basic principle of most methods employed extraction with alkaline solution of acetone/ammonium acetate (5:1) + morpholine, thereby converting pyridate to pyridafol. Conjugates were hydrolysed with sulfuric or hydrochloric acid. Clean up involved one or a combination of the following: partitioning between ammonium acetate and dichloromethane, clean up on a silica or C18 cartridge, solid-supported liquid-liquid partition with n-hexane/tert-butyl methyl ether (1:1). Pyridafol was determined by either HPLC with column switching technology and UV detection (LC-LC-UV) with an LOQ of 0.02 or 0.05 mg/kg (as pyridafol), or by LC-MS/MS in positive ESI mode with an LOQ of 0.05 mg/kg (as pyridate).

For matrices of animal origin, two methods were provided. The first method employed extraction with alkaline solution of acetone/ammonium acetate (5:1) + morpholine, hydrolysation of conjugates with sulfuric acid, followed by clean up employing partitioning with dichloromethane and SPE on a silica cartridge. Quantitation was done by LC-LC-UV with an LOQ of 0.03 mg/kg (as pyridafol). The second method involved extraction with acetonitrile/water (5:1) in the presence of morpholine and clean up by SPE on a C18 cartridge. Quantitation of pyridafol was done by LC-MS/MS in positive ESI mode with an LOQ of 0.03 mg/kg (spiked and expressed as pyridafol) or 0.05 mg/kg (spiked and expressed as pyridate).

The Meeting concluded that the presented methods were sufficiently validated and are suitable to measure the total residue of pyridate as pyridafol in plant and animal matrices commodities.

Stability of pesticide residues in stored analytical samples

The Meeting received information on storage stability of pyridate (expressed as the sum of pyridate, pyridafol and hydrolysable pyridafol conjugates) in incurred residues (radiolabelled and non-radiolabelled) from various raw plant commodities.

In a study with radiolabelled residues performed in high water commodities (maize leaves, peanut leaves, broccoli leaves and alfalfa), samples were analysed for total pyridate initially after 4–14 months and re-analysed after an additional 10–28 months. After the first sampling, total pyridate residues covered >99 percent of the TRR analysed by the total residue method. In the second analysis, 86–90 percent TRR were recovered, suggesting that total pyridate was stable for at least 30 month under deep freezer conditions for maize, broccoli and alfalfa leaves. In peanut forage, 69.5 percent of the residue were recovered after 32 months in the second analysis, which is also the maximum storage period tested in this study.

A second study was performed with non-radiolabelled incurred residues in high water commodities (maize plant, rape plant, field pea plant and onion greens). Within the study the samples were analysed for the first time after about 3 to 14 months after sampling, followed by a second analysis after 24 to 59 months. No significant degradation of residues was observed in the respective time interval (> 81 percent of the first analysis).

The Meeting noted that the first analysis did not occur within a short period after harvest to quantify the time zero residue levels. However, since stability of high water commodities was demonstrated in the radiolabelled study, the Meeting concluded that storage up to 14 months is covered by the radiolabelled storage stability study and subsequent months by the non-radiolabelled study.

The Meeting agreed that the demonstrated storage stability on high water plant commodities was at least 59 months and covered the residue sample storage intervals used in the field trials considered by the current Meeting. However, no storage stability data was provided for other plant commodities or animal commodities.

Definition of the residue

Plant commodities

In the plant metabolism studies conducted on broccoli, maize and peanut, no identification of the total radioactivity in broccoli flowers, maize grain or peanut nutmeat was performed.

In other matrices, parent pyridate was a major residue only in samples taken at 0/14 DAT (broccoli leaves 60/42 percent TRR; maize leaves 82/11 percent TRR; peanut forage 86 percent TRR). Significant levels of pyridafol were mainly found in samples taken at 0 DAT (peanut forage 10 percent TRR) and declined rapidly at later DATs. At higher sampling intervals, the N- and O-glucoside of pyridafol were quantified in all matrices, but in major amounts only in broccoli leaves at 19–25 percent TRR.

The Meeting noted that no suitable single maker compound is present at harvest to measure pyridate residues in plant commodities. However, analytical methodology is available capable to quantify the total residue of parent pyridate, its metabolite pyridafol and conjugates thereof.

The Meeting decided to set the residue definition for compliance with the MRL as the sum of pyridate, its hydrolysis product pyridafol and hydrolysable conjugates of pyridafol, expressed as pyridate.

In all matrices investigated, unknown components (two in broccoli leaves, two in maize leaves and three in peanut forage, hay and hull) were found in major amounts, representing up to 41 percent TRR (up to 11 mg eq/kg). Characterisation of these unknown components indicated that they are not structurally related to pyridafol and are not analysed by the total residue method. The application of pyridate as a herbicide happens early in the growing season. Since the peak occurrence of these unknown components was observed primarily at 14 to 90 DAT, which corresponds to PHIs in the use patterns provided, the Meeting concluded that they are likely to be present in harvested commodities and therefore demand identification and consideration in the dietary risk assessment, depending on their toxicological properties. Although identification in metabolism studies was only performed in inedible matrices, major levels of unknown components were found in all crop matrices, suggesting general occurrence in plants. In addition, the Meeting noted that a metabolism study on alfalfa exists, which was not made available to the current Meeting.

Without identification of these unknown components found at significant levels and information on the metabolism of pyridate in alfalfa, the Meeting decided not to establish a residue definition for dietary exposure purposes for pyridate in plant matrices.

Animal commodities

In ruminant metabolism studies, performed in a lactating cow and a goat, parent pyridate was not detected. The hydrolysis product pyridafol was tentatively identified in the goat at 32 percent TRR in kidney and 49-71 percent TRR in milk as well as pyridafol-OMe in kidney at 48 percent TRR. In the cow, the presence of pyridafol in the kidney and the N- and O-glucosides of pyridafol in the liver were postulated.

In poultry metabolism studies, no identification or characterization of the radioactive residues in organs or tissues was performed. However, in excreta metabolites pyridafol and hydroxylated pyridafol accounted for up to 74 percent TRR and 44 percent TRR, respectively.

In radiolabelled farm animal feeding studies performed with lactating cows and laying hens, one individual component was potentially identified in edible tissues of the 10x dosing group with a retention time close to pyridafol. Additionally, pyridafol and its glucuronides were identified in urine and bile from the cow feeding study.

The Meeting noted that the rate of identification in animal metabolism studies was generally low and no suitable single maker compound is present to measure pyridate residues in animal commodities. However, analytical methodology is available capable to quantify the total residue of parent pyridate, its metabolite pyridafol and conjugates thereof, which is suitable for enforcement purposes.

Parent pyridate has a octanol-water partition coefficient of 4.0, suggesting potential accumulation in fat. However, in farm animal feeding studies, total residues in liver, kidney and muscle were generally higher than in fat. Also, no accumulation of residues in egg yolk compared to egg white was observed. Consequently, the Meeting decided that residues of total pyridate in animal commodities are not fat-soluble.

Pending information on the nature of unknown components in plant matrices, exposure of livestock animals via feed cannot be characterized. The Meeting decided not to establish a residue definition for dietary exposure purposes for pyridate in animal matrices.

The Meeting recommended the following residue definitions for pyridate:

Definition of the residue for compliance with the MRL for plant and animal commodities: *Sum of pyridate and 6-chloro-4-hydroxy-3-phenylpridazine (*pyridafol*) (incl. conjugates), expressed as pyridate*.

Definition of the residue for dietary exposure purposes for plant and animal commodities: *Not established.*

The Meeting considers the residue not to be fat soluble.

Results of supervised residue trials on crops

Supervised trials were available for the use of pyridate on onion, bulb, leek, spring onion, broccoli, cabbage, kohlrabi, kale, chickpea, maize and sweet corn (corn-on–the-cob), as well as on alfalfa and clover.

Product labels were available from Germany, Austria, Italy and the Netherlands

In some field trials, the residue was expressed as equivalents of metabolite pyridafol. In order to convert these residues into pyridate equivalents, a molar weight factor of 1.8 was applied ($M_{Pyridate}/M_{pyridafol}$ = 378.9 g/mol^{-1}/206.6 g/mol^{-1} = 1.8).

The Meeting decided not to establish a residue definition for dietary exposure purposes for plants. Consequently, only maximum residue levels according to the residue definition for enforcement purposes are estimated, but no STMR or HR value.

Bulb vegetables

Onion, bulb

The critical GAP for bulb onions in Italy allows one foliar application of pyridate at 900 g ai/ha with a PHI of 21 days.

Field trials with bulb onion following GAP treatment (±25 percent) were conducted in France and Italy. The ranked order of residues for estimating maximum residue levels and dietary risk assessment was (n=3): < 0.01(2), 0.02 mg/kg.

Based on the lack of data matching the GAP, the Meeting concluded that no maximum residue level could be estimated for pyridate in onion based on the Italian GAP.

A GAP for onions in Austria was provided allowing a maximum of one foliar application of pyridate at 900 g ai/hL and a PHI of 56 days. One field trial, with bulb onion following GAP treatment (±25 percent), was conducted in Austria. The ranked order of residues for estimating maximum residue levels was (n=1): < 0.05 mg/kg.

Due to an insufficient number of supervised field trials matching the GAP, the Meeting concluded that no maximum residue level could be estimated for pyridate in bulb onion.

Leek

The critical GAP for leeks in Austria allows one foliar application of pyridate at 900 g ai/ha with a PHI of 28 days.

Field trials with leek following GAP treatment (±25 percent) were conducted in the Netherlands, France, Switzerland, Italy and Spain. The ranked order of residues for estimating maximum residue levels was (n=12): 0.04, 0.05, 0.051, 0.057, 0.06, 0.08, 0.11, 0.14, 0.20, 0.22, 0.63, 0.81 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg for pyridate in leek.

Spring onion

The critical GAP for spring onion in the Netherlands allows one foliar application of pyridate at 900 g ai/ha with and a PHI of 28 days.

Field trials with spring onion following GAP treatment (±25 percent) were conducted in France, Germany, the UK, Greece and Spain. The ranked order of residues for estimating maximum residue levels was (n=6): < 0.01, 0.02, 0.04, 0.05, 0.07, 0.09 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg for pyridate in spring onion.

Brassica vegetables (except brassica leafy vegetables)

The critical GAP for brassica vegetables (cabbage, Brussels sprouts, broccoli, cauliflower, kohlrabi) in Austria allows one foliar application of pyridate at 900 g ai/ha with and a PHI of 42 days.

Broccoli

Field trials with broccoli following GAP treatment (±25 percent) were conducted in Austria. The ranked order of residues for estimating maximum residue levels was (n=1): < 0.05 mg/kg.

Due to an insufficient number of supervised field trials matching the GAP, the Meeting concluded that no maximum residue level could be estimated for pyridate in broccoli.

Brussel spouts

No field trials with Brussel sprouts approximated GAP treatment (±25 percent) and residues were only determined as pyridafol *per se*.

Due to an insufficient number of supervised field trials matching the GAP, the Meeting concluded that no maximum residue level could be estimated for pyridate in Brussel sprouts.

Cabbage, head

Field trials with cabbages following GAP treatment (±25 percent) were conducted in France, Germany, Greece, Spain and the United Kingdom. The ranked order of residues for estimating maximum residue levels was (n=16): < 0.01(4), 0.01, 0.02, < 0.05(4), 0.12, 0.38, 0.50, 0.57, 0.85, 0.95 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg for pyridate in cabbage, head.

Cauliflower

No field trials with cauliflower approximated GAP treatment (±25 percent) and residues were only determined as pyridafol per se.

Due to an insufficient number of supervised field trials matching the GAP, the Meeting concluded that no maximum residue level could be estimated for pyridate in cauliflower.

Kohlrabi

Field trials conducted with kohlrabi following GAP treatment (±25 percent) were conducted in Austria. The ranked order of residues for estimating maximum residue levels was (n=1): < 0.05 mg/kg.

Based on the lack of data matching the GAP, the Meeting concluded that no maximum residue level could be estimated for pyridate in kohlrabi.

Kale

The critical GAP for kale in Austria allows one foliar application of pyridate at 900 g ai/ha with and a PHI of 42 days.

Field trials with kale following GAP treatment (±25 percent) were conducted in Switzerland, the UK and France. The ranked order of residues for estimating maximum residue levels was (n=7): < 0.05(4), 0.081, 0.092, 0.17 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for pyridate in kale.

Chick-pea (succulent seeds)

The critical GAP for chick-peas in Italy allows one foliar application of pyridate at 900 g ai/ha up to BBCH 19 with and a PHI of 45 days.

Field trials with chick-peas were conducted in France, Greece, Italy and Spain. The ranked order of residues in legume chick-pea seeds for estimating maximum residue levels was (n=6): < 0.05(6) mg/kg.

The Meeting estimated a maximum residue level of 0.05* mg/kg for pyridate in chickpeas (succulent seeds).

Pulses

Chick-pea (dry)

The critical GAP for chick-peas in Italy allows one foliar application of pyridate at 900 g ai/ha up to BBCH 19 with and a PHI of 45 days.

Field trials with chick-peas were conducted in France, Greece, Italy and Spain. The Meeting noted that storage intervals in all trials were longer than one months. No information on storage stability in high protein matrices was provided and therefore the Meeting could not assess the validity of the measured results.

Due to an insufficient number of supervised field trials matching the GAP, the Meeting concluded that no maximum residue level could be estimated for pyridate in chickpeas (dry).

Cereal grains

Maize

The critical GAP for maize in the Netherlands allows one foliar application of pyridate at 900 g ai/ha at BBCH 16 and with the PHI covered by the conditions of use.

Field trials with maize following GAP treatment (±25 percent) were conducted in France and Germany. The ranked order of residues for estimating maximum residue levels was (n=7): < 0.04, < 0.05(6) mg/kg.

The Meeting noted that no data on storage stability in high starch content matrices was available although field trial samples were stored for 3–16 months (4×3 months, 2×6 months and 1×16 months). Based on the provided metabolism studies on maize dosed at 2× GAP rate, the total radioactive residue in maize grain was low at 0.01–0.014 mg eq/kg. No further identification was undertaken in the samples and therefore the fraction of the TRR covered by the common moiety method remains unknown. However, given the rapid metabolism of pyridate, including the incorporation of the radioactivity into natural constitutes, the Meeting concluded that the levels of the components covered by the residue definition are expected to be even lower. Also, field trial samples treated at 150 percent GAP rate also showed no residues in maize grain for pyridafol after 2 months of storage.

In view of the low radioactivity found in maize metabolism study and the analysis of trial samples according to the common moiety method, the Meeting decided to accept the trials without demonstration of storage stability and estimated a maximum residue level of 0.05(*) mg/kg for pyridate in maize grain.

Sweet corn (Corn-on-the-cob)

The critical GAP for sweet corn in Austria allows one foliar application of pyridate at 900 g ai/ha with and a PHI of 42 days.

Field trials with sweet corn following GAP treatment (±25 percent) were conducted in France. The ranked order of residues in sweet corn (corn-on-the-cob) for estimating maximum residue levels was (n=1): < 0.05(1) mg/kg. The ranked order of residues in sweet corn (kernels) for estimating maximum residue levels was (n=2): < 0.05(2) mg/kg.

Alternatively, a GAP for sweet corn in the Netherlands was provided, allowing one foliar application of pyridate at 900 g ai /ha at BBCH 16 and with the PHI covered by the conditions of use.

Field trials with sweet corn following GAP treatment (±25 percent) were conducted in France. The ranked order of residues for estimating maximum residue levels was (n=10): < 0.04, < 0.05(9) mg/kg. The ranked order of residues in sweet corn (kernels) for estimating maximum residue levels was (n=2): < 0.05(2) mg/kg.

The Meeting noted that only the dataset for sweet corn (corn-on-the-cob) according to the GAP from the Netherlands was sufficiently large and estimated a maximum residue level of 0.05(*) mg/kg for pyridate in sweet corn (corn-on-the-cob)

Residues in animal feeds

Alfalfa forage

The critical GAP for alfalfa in Austria allows one foliar application of pyridate at 900 g ai/ha with and a PHI of 28 days.

Field trials with alfalfa following GAP treatment (±25 percent) were conducted in Austria and France. The ranked order of residues was (n=5): < 0.05(2), 0.05, 0.23, 0.26 mg/kg.

Since alfalfa forage is utilised only as a feed items and insufficient data on the nature of residues relevant for calculating the livestock animal dietary burden are available, the Meeting did not estimate median or highest residues for pyridate.

Clover forage

The critical GAP for clover in Austria allows one foliar application of pyridate at 900 g ai /ha with and a PHI of 28 days.

Field trials with clover following GAP treatment (±25 percent) were conducted in France and the United Kingdom. The ranked order of residues was (n=6): < 0.04, 0.12, 0.26, 1.0, 1.5(2) mg/kg.

Since clover forage is utilised only as a feed items and insufficient data on the nature of residues relevant for calculating the livestock animal dietary burden are available, the Meeting did not estimate median or highest residues for pyridate.

Maize forage

The critical GAP for maize in the Netherlands allows one foliar application of pyridate at 900 g ai/ha at BBCH 16 and with the PHI covered by the conditions of use.

Field trials with maize following GAP treatment (±25 percent) were conducted in France and Germany. The ranked order of residues in maize forage collected at growth stages typical for commercial harvest was (n=12): < 0.04(4), < 0.05(7), 0.068 mg/kg.

Since maize forage is utilised only as a feed items and insufficient data on the nature of residues relevant for calculating the livestock animal dietary burden are available, the Meeting did not estimate median or highest residues for pyridate.

Maize stover

The critical GAP for maize in the Netherlands allows one foliar application of pyridate at 900 g ai/ha at BBCH 16 and with the PHI covered by the conditions of use.

Field trials with maize following GAP treatment (±25 percent) were conducted in France. The ranked order of residues in maize straw for estimating maximum residue levels was (n=5): < 0.04, < 0.05(4) mg/kg.

The Meeting estimated a maximum residue level of 0.05* mg/kg (DM) for pyridate in maize stover.

Fate of residues during processing

The Meeting received no information on the hydrolysis of pyridate, simulating typical processing conditions.

The fate of pyridate residues has been examined simulating commercial processing of maize and peanuts using radiolabelled pyridate. The TRRs in the RAC and in processed commodities (maize and peanut oil) were comparable. However, no components according to the residue definition were identified in the processed commodities and the nature of radioactivity remained unknown. Therefore, the Meeting decided that no processing factors addressing the residue definition for pyridate could be derived.

Residues in animal commodities

Farm animal feeding studies

The Meeting received feeding studies involving pyridate on lactating cows and laying hens using radiolabelled pyridate.

The study with lactating cows was conducted at treatment rates of 1, 3.3 and 10 ppm. Total radioactive residues in milk plateaued after one day in all dosing groups (0.003, 0.015 and 0.033 mg eq/kg, respectively). In edible tissues residues were highest in kidney ranging from 0.19 mg eq/kg (1×) to 1.9 mg eq/kg (10×). An individual component was only identified in muscle, kidney, liver and fat of the 10x dosing group with a retention time close to pyridafol. Additionally, pyridafol and its glucuronides were identified in urine and bile.

The study with laying hens was conducted at treatment rates of 1.3, 4 and 13 ppm. Total radioactive residues in eggs whites plateaued after two days in all dosing groups (0.005, 0.01 and 0.032 mg eq/kg respectively), while in egg yolks a plateau was reached after 5–7 days (0.003, 0.008 and 0.023 mg eq/kg, respectively). In edible tissues residues were highest in kidney ranging from 0.05 mg eq/kg (1×) to 0.23 mg eq kg (10×). An individual component was identified in egg yolk, kidney, liver and muscle of the 10x dosing group only, with a retention time close to pyridafol.

Farm animal dietary burden

Due to insufficient data on the nature of residues relevant for the livestock animal dietary burden, the Meeting decided that no calculation was possible.

Animal commodity maximum residue levels

The Meeting noted that the provided farm animal feeding studies lacked detailed residue data on components covered by the residue definition. Generally, only TRR levels were provided for milk, egg and various tissues, while the presence of metabolite pyridafol was only qualitatively described.

Therefore, the Meeting decided that no recommendations for animal commodities could be given based on the available information from farm animal feeding studies.

RECOMMENDATIONS

Definition of the residue for compliance with the MRL for plant and animal commodities: *Sum of pyridate and 6-chloro-4-hydroxy-3-phenylpridazine (pyridafol) (incl. conjugates), expressed as pyridate*.

Definition of the residue for dietary exposure purposes for plant and animal commodities: *Not established.*

The residue is not fat-soluble.

DIETARY RISK ASSESSMENT

As the Meeting was unable to recommend residue definitions for dietary risk assessment for plants and animal commodities, chronic and acute dietary risk assessments could not be conducted.

FURTHER WORK OR INFORMATION

Desirable information

- Submission of existing metabolism data for pyridate in alfalfa.

- Identification of major unknown components in all plant matrices from broccoli, maize and peanut metabolism studies.
- Data on the transfer of pyridate residues to farm animals (ruminants and poultry) are required, as the information provided was not sufficient to give recommendation for maximum residue levels in animal matrices.
- Storage stability information on commodities of high protein and starch content are required.

  Information on pyridate analytical methods targeting single compounds.
- Information on processed commodities.

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PYRIDATE_097	Pfarl, D.C.	1991	Residues of pyridate and its main metabolites free CL-9673 and hydrolysable CL-9673 conjugates in broccoli, Brussels sprouts, cabbage (white), calabrese, cauliflower, leek and onion treated with Lentagran 45 WP corresponding to 1.8 kg and 2.0 kg pyridate a.i./ha. Report No. 1102, Agrolinz Agrarchemikalien GmbH. GLP: yes. Unpublished
PYRIDATE_098	Heegemann, W.	1986	Residues of pyridate and its main metabolites free CL 9673 and hydrolysable CL 9673 conjugates in kohlrabi treated with 2 and 4 kg Lentagran WP/ha. Report No. 892, Chemie Linz, Austria. GLP: no. Unpublished
PYRIDATE_099	Pfarl, D.C.	1990	Residues of pyridate and its main metabolites free CL 9673 and hydrolysable CL 9673 conjugates in calabrese treated with LENTAGRAN WP. Report No. 1056c, Agrolinz Agrarchemikalien GmbH. GLP: no. Unpublished
PYRIDATE_100	Gasser, A.	1999	Determination of pyridate (SAN 319) in or on sweet corn after application of formulation "LENTAGRAN" in France/south. Report No. 3116/98, Novartis Crop Protection AG. GLP: yes. Unpublished

Pyridate

Code	Author	Year	Title, Institute, Report reference
PYRIDATE_101	Gasser, A.	2002	Residue study with pyridate (SAN 319) in or on maize in France (south) Report No. 3013/00, Novartis Crop Protection AG. GLP: yes. Unpublished
PYRIDATE_102	Krennhuber, K., Pfarl, D.C.	1997	Determination of residues of pyridate in corn (zea mays) matrices after application of Lentagran 600 EC under field conditions in France (SEU), 1996. Report No. 1294 Agrolinz Melamin GmbH, GLP: yes. Unpublished
PYRIDATE_103	Pfarl, D.C.	1991	Residues of pyridate and its main metabolites free CL-9673 and hydrolysable CL-9673 conjugates in maize treated with 3.0 kg LENTAGRAN WP/ha. Report No. 1092, Agrolinz Agrarchemikalien GmbH. GLP: yes. Unpublished
PYRIDATE_104	Pfarl, D.C.	1994	Residues of pyridate and its main metabolites free CL-9673 and hydrolysable CL-9673 conjugates in maize treated with Lentagran and Bropyr. Report No. 1207, Agrolinz Agrarchemikalien GmbH. GLP: yes. Unpublished
PYRIDATE_105	Pfarl, D.C.	1992	Residues of pyridate and its main metabolites free CL-9673 and hydrolysable CL-9673 conjugates in maize treated with 3.0 kg LENTAGRAN WP/ha. Report No. 1123, Agrolinz Agrarchemikalien GmbH. GLP: yes. Unpublished
PYRIDATE_106	Pfarl, D.C.	1992	Residues of pyridate and its main metabolites free CL-9673 and hydrolysable CL-9673 conjugates in maize treated with 3.0 kg LENTAGRAN WP/ha. Report No. 1125, Agrolinz Agrarchemikalien GmbH. GLP: yes. Unpublished
PYRIDATE_107	Pfarl, D.C.	1992	Residues of pyridate and its main metabolites free CL-9673 and hydrolysable CL-9673 conjugates in maize treated with 2.0 l LENTAGRAN EC/ha. Report No. 1129, Agrolinz Agrarchemikalien GmbH. GLP: yes. Unpublished
PYRIDATE_108	Semrau, J.	2012	Determination of residues of Pyridate after one application of Lentagran 45 WP in bulb onions at 6 sites in Northern and Southern Europe 2009 S09-02296. Eurofins Agroscience, Stade, Germany. GLP: yes. Unpublished

QUINCLORAC (287)

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Presentation at the 2022 JMPR was by Mr C Sieke, Federal Institute for Risk Assessment, DE

EXPLANATION

Quinclorac is a systemic herbicide used with uptake through roots and foliage and used to control annual grass and broadleaf weeds. It was evaluated by the 2015 JMPR for the first time for toxicology and for residues and re-evaluated in 2017 (R) for additional uses. The 2015 JMPR established an ADI of 0–0.4 mg/kg bw, and an ARfD of 2 mg/kg bw.

For plant commodities, the residue definition for compliance with MRLs is quinclorac plus quinclorac conjugates and the residue definition for the estimation of dietary intakes is quinclorac plus quinclorac conjugates plus quinclorac methyl ester expressed as quinclorac. The 2015 JMPR noted that quinclorac methyl ester has a toxicological potency up to 10 times that of quinclorac and agreed to multiply the quinclorac methyl ester residues with a factor of 10 to express it as quinclorac equivalents.

For animal commodities the residue definition for compliance with MRLs and for estimation of dietary intakes is quinclorac plus quinclorac conjugates. The residue is fat-soluble.

Quinclorac was scheduled at the Fifty-first Session of the CCPR for the re-assessment of residue trials for oil seed rape after re-analysis using different analytical methods and the consideration of commercial demonstration trials for rapeseed.

The current Meeting received information on a use pattern, the re-analysis of residue trial samples and commercial demonstration trials for rapeseed. A use pattern, new residue trials and additional validation data for cranberries were also received by the current Meeting.

RESIDUE ANALYSIS

Cranberries and rape seed

Residues of quinclorac and quinclorac methyl ester were determined using method D1607/0. The total residues of quinclorac were determined in three consecutive extraction procedures. For the 1st extraction, samples were extracted with acetonitrile/water (1/1). Parent quinclorac and quinclorac methyl ester were determined using LC-MS/MS. For the 2nd extraction, the seed and forage marc with aqueous phase from the 1st extraction were extracted with acetone/10 mM phosphate buffer at pH 7 (1/1) and with acetone, respectively. Parent quinclorac and quinclorac methyl ester were determined using LC-MS/MS. For the 3rd extraction, the marc from the 2nd extraction was treated with 1N NaOH at 100 °C for 1 hour. This harsh hydrolysis to release quinclorac conjugates was used in the plant metabolism study for rape seed.

Quinclorac and quinclorac conjugates were determined as quinclorac using LC-MS/MS. After each sample extraction and clean-up, residues are determined by LC-MS/MS, monitoring ion transitions at m/z 242→224 (quantitation) and m/z 242→161 (confirmation) for quinclorac, and m/z 256→224 (quantitation) and m/z 256→161 (confirmation) for quinclorac methyl ester. Total residues of quinclorac were reported as the sum from each extraction procedure. Similarly, the total residues of quinclorac methyl ester were determined in two consecutive extraction procedures and reported as the sum from each extraction procedure.

The 2017 JMPR concluded that this method was suitable for the separate determination of quinclorac, including its conjugates, and quinclorac methyl ester residues in rapeseed and forage. The current Meeting received procedural recovery data for cranberries. The procedural recovery data are summarised in table 1.

Table 1 Summary of method procedural recovery data for the determination of quinclorac and quinclorac methyl ester residues in cranberries

Analyte	Fortification level (mg/kg)	Number of samples	Recoveries (%)	Repeatability (% RSD)
Quinclorac	0.02	3	95, 100, 105	5
	0.2	2	101, 104	-
	2	3	91, 94, 95	2.2
Quinclorac methyl ester	0.02	3	82, 93, 95	7.8
	0.2	3	89, 93, 102	7
	2	3	87, 89, 91	2.3

Rapeseed

Multi-residue analytical method (Based on QuEChERS method for acidic herbicides)

The method is similar to the multi-residue analytical method D1502/1 considered by the 2017 JMPR. However, the initial extraction employed is different.

Samples were prepared by homogenizing with liquid nitrogen/dry ice. A subsample was hydrated with water and the pH was adjusted to >9 with NaOH. After shaking the sample for 30 minutes and centrifuging, an aliquot was removed and shaken with dichloromethane (DCM). The DCM was then discarded, the pH adjusted to ≤2 with sulfuric acid.

Samples were then shaken with acetonitrile and cleaned-up by a mixture of "QuEChERS" salts ($MgSO_4$, NaCl, trisodium citrate dihydrate and disodium hydrogen citrate sesquihydrate). The residues in the organic phase were diluted with acetonitrile/water (10/90, v/v) for quinclorac analyses. Final determination was achieved using LC-MS/MS. The ion transitions monitored were m/z 242 → m/z 224 and m/z 242 → m/z 161.

Recovery data were generated during the analysis of the samples from the monitoring trials. A summary of the recovery data are outlined in Table 2.

Table 2 Summary of procedural recovery data for LC-MS/MS method

Analyte	Trial Year	Fortification level (mg/kg)	Number of samples	Recoveries (%)
Quinclorac	2016	0.01	2	84, 110
		1	2	120, 117
	2017	0.01	3	136, 120, 107
		1	3	113, 99, 112

Methods employed in the re-analysis of rape seed samples

The methods employed in the re-analysis of samples were methods D9708/1 and D9806, considered by the 2015 JMPR, and method D1607/01, considered by the 2017 JMPR.

Method D9708/1

Residues of quinclorac were extracted from rape seeds using hexane/0.1 NaOH solution followed be partitioning with acetonitrile. Final determination was by HPLC-MS/MS using the ion transition m/z 240-196 for quantification. The LOQ validated for quinclorac in/on oil seed rape was 0.05 mg/kg.

As the method does not contain a hydrolysis step, then any conjugates of quinclorac present will not be sufficiently released. However, as the metabolism data showed that parent conjugates are not expected for rape seed this is of no concern. A radio-validation study considered by the 2015 JMPR showed that extraction with acetone/0.1 M NaOH converts the quinclorac methyl ester (if present) partly back into the parent compound. This means the parent levels in rapeseed may be overestimated and hence the JMPR in 2015 concluded that this method was not suitable for the estimation of MRLs.

Method D9806

Quinclorac methyl is extracted from rape seed using acetonitrile/hexane and then partitioned with acetonitrile/water and methanol. Final determination is by HPLC-MS/MS using the ion transition m/z 255 – 224 for quantification. The LOQ validated was 0.05 mg/kg. This method is suitable for the determination of quinclorac methyl ester residues in rape seed.

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

No additional information was received by the current Meeting on the stability of quinclorac and quinclorac methyl ester residues in stored samples.

USE PATTERN

The use patterns for cranberries and rape seed are outlined in table 3. The GAP for cranberries was considered by the 2015 JMPR and the GAP for rapeseed was considered by the 2017 JMPR. The current Meeting received labels for these GAPs as they remain valid.

Table 3 Use pattern for cranberries and rapeseed

Crop	Country	Indoor/ outdoor	Type	Timing of application	Rate (g ai/ha)	No. of appl (interval)	PHI (days)
Cranberries	United States	Outdoor	Ground spray	See PHI	280	2 (30 days)	60
Rape seed	Canada	Outdoor	Foliar spray	See PHI	100	1	60

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Cranberries

Four residue trials conducted in the United States in 2008 were considered by the 2015 JMPR to support the GAP. These trials were conducted at an application rate of 2 × 280 g ai/ha with a RTI of 30 days. These trials are summarized in Table 4.

Table 4 Residue trials data, generated by foliar treatment, for cranberries conducted in the United States (evaluated by the 2015 JMPR)

Location	Application			Residues			Trial
Year, (variety)	Total Rate, (kg ai/ha)	Growth stage	PHI (days)	Matrix	Total quinclorac (mg/kg)	mean (mg/kg)	Trial comment
United States Plymouth County, MA, 2008 (Stevens) Stevens 1	2 x 0.27	Bloom July 5, Fruit set July 31	59	Mature cranberries	0.50, 0.60	0.55[a]	08000.08-MA01 2010/7018348
United States Wareham, MA 2008 (Early Blacks) Early blacks 1	2 x 0.28	Bloom July 5, Fruit set July 31	59	Mature cranberries	0.16, 0.20	0.18	08000.08-MA03 2010/7018348
United States Warrens, WI 2008 (Stevens) 5	2 x 0.28	Bloom, July 7 Fruiting August 4	57	Mature cranberries	0.17, 0.16	0.17	08000.08-WI01 2010/7018348
United States Warrens, WI 2008 (Ben Lear) 5	2 x 0.28	Bloom, July 7 Fruiting August 4	57	Mature cranberries	0.16, 0.15	0.16[b]	08000.08-WI02 2010/7018348
United States Langlois, OR 2008 (Pilgrims) 12	2 x 0.29	End of bloom, July 1 Green fruit August 6	62	Mature cranberries	0.66, 0.68	0.67	08000.08-OR10 2010/7018348

Notes:

[a:] Not considered in 2015 Report, but considered by current JMPR.

[b:] Considered in 2015 Report, but not considered by current JMPR.

Five new residue trials conducted in the United States in 2019 were provided to the current Meeting. At each trial site two applications of quinclorac at a rate of 280 g ai/ha were made with a RTI of 30 days.

Cranberries were harvested between 49–66 days after the last application. Cranberries were stored frozen at ≤ -18 °C. All samples were analysed within 30 days.

Samples of cranberries were analysed separately for quinclorac including conjugates and quinclorac methyl ester using method D 1607/01. Procedural recoveries were conducted at 0.02 mg/kg

and 0.2 mg/kg. The recoveries ranged from 97–134 percent for quinclorac and 86–100 percent for quinclorac methyl.

The residue trials are summarized in Table 5.

Table 5 Residue trials data, generated by foliar treatment, for cranberries conducted in the United States

Location, Country Year, Crop/Variety	Rate (g ai/ha)	RTI (days)	Growth stage at last treatment	DALA	Quinclorac residues (mg/kg)	Methyl ester residues (mg/kg)
GAP United States	280 × 2	30	See DALA	60		
East Wareham, MA, United States, 2019 Cranberry/ Stevens	291 280	- 30	Fruiting	49	0.24, 0.26 (0.25)	<0.02, <0.02 (<0.02)
				54	0.16, 0.17 (0.17)	<0.02, <0.02 (<0.02)
				59	0.20, 0.28 (0.24)	<0.02, <0.02 (<0.02)
				64	0.32, 0.19 (0.26)	<0.02, <0.02 (<0.02)
				69	0.16, 0.17 (0.17)	<0.02, <0.02 (<0.02)
Langlois, OR, United States, 2019 Cranberry/ Stevens	291 280	- 30	Immature fruit	61	0.081, 0.078 (0.08)	<0.02, <0.02 (<0.02)
Langlois, OR, United States, 2019 Cranberry/ Stevens	280 280	- 30	Fruiting - yellow	66	0.11, 0.10 (0.11)	<0.02, <0.02 (<0.02)
Junction City, WI, United States, 2019 Cranberry/ 6H1	280 280	- 30	Fruiting	59	0.56, 0.55 (0.56)	<0.02, <0.02 (<0.02)
Warrens, WI, United States, 2019 Cranberry/ Stevens	291 280	- 30	Fruiting	59	0.074, 0.11 (0.09)	<0.02, <0.02 (<0.02)

Note:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

Rape seed

No new residue trials were received by this Meeting. Information has been provided to re-consider the residue trials evaluated by the JMPR in 2015; data have been provided to compare the different analytical methods used with the potential to establish analytical correction factors.

In 2015, the JMPR considered sixteen residue trials conducted in Canada and one trial conducted in the United States. These trials are summarized in Table 6.

The trials were conducted at an application rate of 1 × 100 g ai/ha applied at various growth stages with samples taken for analysis 60 days after the application.

Samples were collected and frozen prior to analysis. Samples were stored for up to 6 months prior to quinclorac analysis and for up to 12 months prior to quinclorac methyl ester analysis.

Samples were analysed using method D9708/1 for quinclorac (including quinclorac conjugates using acetone/0.1 M NaOH and LC-MS/MS) and method D9806 for quinclorac methyl ester (using acetone and LC-MS/MS). Procedural recoveries were generated at fortification levels of 0.05–0.5 mg/kg with recoveries ranging from 61–88 percent for quinclorac and 77–120 percent for quinclorac methyl ester.

The 2015 JMPR concluded that the method used in the trials overestimated the residues of quinclorac. A radio-validation study considered by the 2015 JMPR showed that extraction with acetone/0.1 M NaOH converts the quinclorac methyl ester (if present) partly back into the parent compound. This means the parent levels in rapeseed may be overestimated and hence the method is not suitable for the estimation of MRLs.

Table 6 Residue trials data, generating via a foliar application, for rape seed conducted in Canada and the United States in 1997 (evaluated by the JMPR in 2015)

Location, Country Year, Crop/Variety	Rate (g ai/ha)	Growth stage	DALA	Matrix	Quinclorac residues (method D9708/1) (mg/kg)	Methyl ester residues(method D9806) (mg/kg)	Reference
GAP Canada	100	See PHI	60				
Hines Creek, Alberta, Canada 1997 Canol[a/]Reward	100	6-leaf stage	60	seed	<0.05, <0.05	<0.05, <0.05	Guirguis, M., 1998
Fairview, Alberta, Canada 1997 Canol[a/]Reward	100	6-leaf stage	53 60 67 74	seed	<0.05 <0.05 <0.05 <0.05	<0.05 <0.05 <0.05 <0.05	
Lacombe, Alberta, Canada 1997 Canol[a/]Quantum	100	7 leaves and bolting	60	seed	0.10, 0.09 (0.10)	0.19, 0.17 (0.18)	
Stettler, Alberta Canada 1997 Canol[a/]Quantum	100	7-8 leaf stage	60	seed	0.18, 0.22 (0.2)	0.09, 0.08 (0.09)	
Red Deer, Alberta Canada 1997 Canol[a/]Hyson 110	100	5 leaf stage	60	seed	<0.05, <0.05	0.12, 0.13 (0.13)	
Aberdeen, Saskatchewan, Canada 1997 Canol[a/]Quantum	100	3-8 leave stage	60	seed	0.14, 0.12 (0.13)	0.06, <0.05 (0.06)	
Rosthern, Saskatchewan Canada 1997 Canol[a/]Quantum	100	3 leaves	60	seed	0.30, 0.18 (0.24)	0.09, 0.09 (0.09)	
Wakaw, Saskatchewan Canada 1997 Canol[a/]Ebony	100	5 – 10 leaves	60	seed	<0.05, <0.05	<0.05, <0.05	
Melfort, Saskatchewan	100	5-10 leaves	60	seed	0.09, 0.08	0.08 0.06	

Location, Country Year, Crop/Variety	Rate (g ai/ha)	Growth stage	DALA	Matrix	Quinclorac residues (method D9708/1) (mg/kg)	Methyl ester residues(method D9806) (mg/kg)	Reference
Canada 1997 Canol[a/]Quantum					(0.09)	(0.07)	
Duck Lake, Saskatchewan Canada 1997 Canol[a/]Garrison	100	6-8 leaves and flowering	60	seed	0.21, 0.25 (0.23)	0.12, 0.10 (0.13)	
Hague, Saskatchewan Canada 1997 Canol[a/]Garrison	100	5-7 leaves and flowering	60	seed	0.63, 0.57 (0.6)	0.14, 0.11 (0.13)	
Saskatoon, Saskatchewan Canada 1997 Canol[a/] Garrison	100	4-6 leaves	60	seed	0.85 0.86 (0.86)	0.15 0.12 (0.14)	
Boussevain, Manitoba, Canada 1997 Canol[a/] 46A05	100	11 leaves	60	seed	0.24 0.21 (0.23)	0.23, 0.07 (0.15)	
Minto, Manitoba, Canada 1997 Canol[a/] A5471	100	11 leaves	60	Seed	0.15 0.17 (0.16)	<0.05 <0.05	
Portage La Prairie, Manitoba, Canada 1997 Canol[a/] 46A72	100	22 leaves and flowering	60	seed	<0.05 0.05 (0.05)	0.10 0.10 (0.01)	
Bagot, Manitoba, Canada 1997 Canol[a/] Quantum	100	8- 10 leaves and mid flowering	60	seed	0.21 0.21 (0.21)	0.23 0.13 (0.18)	
New Rockford/ United States 1997 Canol[a/] Hyola 308	100	22 leaves and early bloom	53 60 67 74	Seed	0.07 0.05 (0.06) <0.05 0.06 (0.06) 0.06 0.05 (0.06) <0.05 <0.05	<0.05 <0.05 (0.05) <0.05 <0.05 (<0.05) <0.05 <0.05 (0.05) <0.05 <0.05 (<0.05)	

Note:

Values in parentheses = mean of two independent representative treated samples taken at the trial site.

Re-analysis of previously submitted supervised field trials with other analytical methods

To support the use of the above trials in a reconsideration of the maximum residue level for rape seed oil, information has been provided to demonstrate the extent of the overestimation of the residue levels of quinclorac.

The extent of overestimation has been determined by re-analysing the rape seed samples from the trials considered by the 2017 JMPR using several different analytical methods.

In 2017, the JMPR considered a total of nine residue trials conducted in Canada and the United States. The trials were conducted in 2016 with an application rate of 0.1–0.105 kg ai/ha applied at the 2–6 leaf stage 60 days prior to harvest. The samples were stored frozen for up to 145 days prior to analysis.

Residues of quinclorac, quinclorac conjugates and quinclorac methyl ester were determined using method D 1607/01 (three extractions: a) acetonitrile/water, b) acetone/phosphate buffer pH 7, c) 0.1 M NaOH at 100 ˚C; separate analysis of each extract for parent and methyl-ester using LC-MS/MS). Procedural recoveries of quinclorac and quinclorac methyl ester fortified in control canola seed samples were 74 ± 11 percent (quinclorac) and 96 ± 11 percent (quinclorac methyl ester) at 0.01 mg/kg, and 79 ± 20 percent (quinclorac) and 107 percent (quinclorac methyl ester) at 1.0 mg/kg. The LOQ for both analytes was 0.01 mg/kg.

The current Meeting received information on the re-analysis of the samples from the 2016 trials. The samples were re-analysed using the following methods:

Method D1607/01 determines total quinclorac (quinclorac plus quinclorac conjugates) and quinclorac methyl ester separately Samples are extracted in sequence with a) acetonitrile/water, b) acetone/phosphate buffer pH 7, c) 1 M NaOH at 100 ˚C for 1 hour. Each extract is analysed separately for parent and the methyl-ester using LC-MS/MS.

Method D9708/01 determines total quinclorac (quinclorac plus quinclorac conjugates). It overestimates quinclorac by converting some of the quinclorac methyl ester (if present) to quinclorac; it underestimates quinclorac as release of quinclorac conjugates (if present) is incomplete. Rape seed is not expected to contain conjugates (according to the metabolism studies).

Method D9806 is suitable for the determination of quinclorac methyl ester. The samples were stored frozen prior to the re-analysis for up to 670 days. A summary of the residue trials along with the results of the original analysis and the re-analysis are summarised in Tables 7 and 8 for quinclorac and quinclorac methyl ester respectively.

Table 7 Quinclorac residues in rape seed from supervised trials conducted in Canada and the United States in 2016 (trials evaluated by the 2017 JMPR with the re-analysis considered by the current Meeting)

Rape seed country, year (variety)	Application					DALA Days	Original analysis: method D1607/01 (mg/kg)	Quinclorac residue following re-analysis (mg/kg)	
	Form	g ai/ha	water, L/ha	Growth Stage	no.			Method D1607/01	Method D9708/1
GAP, Canada	***SL***	***100***	**100**	***2-6 leaf stage***	***1***	***60***	-	-	-
United States, 2016 Northwood/ ND (L252) **Trial 1**	SL	101	101	BBCH 15-16	1	60	0.021	0.021	< 0.05 (0.03)
							0.023	0.021	< 0.05 (0.03)

Rape seed country, year (variety)	Application					DALA Days	Original analysis: method D1607/01 (mg/kg)	Quinclorac residue following re-analysis (mg/kg)	
	Form	g ai/ha	water, L/ha	Growth Stage	no.			Method D1607/01	Method D9708/1
United States, 2016 Carrington/ ND (L252) **Trial 2**	SL	101	100	BBCH 15	1	60	0.033	0.03	<0.05 (0.035)
							0.033	0.03	<0.05 (0.04)
Canada, 2016 Branchton/ ON (Pioneer 46H75) **Trial 3**	SL	100	100	BBCH 16	1	60	0.054	0.051	0.11
							0.055	0.049	0.074
Canada, 2016 Portage la Prairie/MB (Dekalb 74-44 BL) **Trial 4**	SL	104	104	BBCH 14	1	60	<0.01	<0.01 (0.0096)	<0.05 (0.014)
							0.010	0.011	<0.05 (<0.01)
Canada, 2016 Hanley/SK (Liberty Link L130 Invigor) **Trial 5**	SL	101	100	BBCH 14-15	1	60	0.014	0.014	<0.05 (0.012)
							0.015	0.011	<0.05 (0.019)
Canada, 2016 Okanagan Falls/BC (Liberty Link Invigor) **Trial 6**	SL	103	102	BBCH 15-16	1	60	0.099	0.088	0.17
							0.11	0.096	0.17
Canada, 2016 Neepaw[a/] MB (Dekalb 74-44 BL) **Trial 7**	SL	103	101	BBCH 14	1	60	0.012	0.016	<0.05 (0.022)
							0.017	0.012	<0.05 (0.014)
Canada, 2016 Brandon/MB (L252) **Trial 8**	SL	100	105	BBCH 12	1	60	<0.01	<0.01 (0.0038)	<0.05 (<0.01)
							<0.01	<0.01 (0.0045)	<0.05 (<0.01)
United States, 2016 American Falls/ID (Hyclass 930) **Trial 9**	SL	105	104	BBCH 13	1	60	0.016	0.014	<0.05 (0.028)
							0.017	0.014	<0.05 (0.025)

Notes:

Values in parentheses are the residue levels determined below the limit of quantification (LOQ).

Method D1607/01: LOQ = 0.01 mg/kg, LOD = 0.002 mg/kg.

Method D9708/1: LOQ = 0.05 mg/kg, LOD = 0.01 mg/kg.

Table 8 Quinclorac methyl ester residues in rape seed from supervised trials in Canada and the United States (trials evaluated by the 2017 JMPR with the re-analysis considered by the current Meeting)

Rape seed country, year (variety)	Application					DALA Days	Original residue of quinclorac methyl ester reported in study (method D1607/01 (mg/kg)	Quinclorac methyl ester residue following re-analysis (mg/kg)	
	Form	g ai/ha	water, L/ha	Growth Stage	no.			Method D1607/01	Method D9806
GAP, Canada	***SL***	***100***	**100**	***2-6 leaf stage***	***1***	***60***	-	-	-
United States, 2016 Northwood/ ND (L252) **Trial 1**	SL	101	101	BBCH 15-16	1	60	0.08	0.074	<0.05 (<0.01)
							0.094	0.073	<0.05 (0.018)
United States, 2016 Carrington/ ND (L252) **Trial 2**	SL	101	100	BBCH 15	1	60	0.14	0.10	<0.05 (0.02)
							0.14	0.11	< 0.05 (0.028)
Canada, 2016 Branchton/ ON (Pioneer 46H75) **Trial 3**	SL	100	100	BBCH 16	1	60	0.20	0.15	<0.05 (0.025)
							0.20	0.13	<0.05 (0.017)
Canada, 2016 Portage la Prairie/MB (Dekalb 74-44 BL) **Trial 4**	SL	104	104	BBCH 14	1	60	0.023	0.019	<0.05 (<0.01)
							0.024	0.023	<0.05 (<0.01)
Canada, 2016 Hanley/SK (Liberty Link L130 Invigor) **Trial 5**	SL	101	100	BBCH 14-15	1	60	0.056	0.044	<0.05 (0.02)
							0.05	0.038	<0.05 (0.017)
Canada, 2016 Okanagan Falls/BC (Liberty Link Invigor) **Trial 6**	SL	103	102	BBCH 15-16	1	60	0.12	0.077	<0.05 (0.033)
							0.12	0.09	<0.05 (0.031)
Canada, 2016 Neepaw[a/]MB (Dekalb 74-44 BL) **Trial 7**	SL	103	101	BBCH 14	1	60	0.024	0.034	<0.05 (<0.01)
							0.038	0.026	<0.05 (0.011)
Canada, 2016 Brandon/MB (L252) **Trial 8**	SL	100	105	BBCH 12	1	60	<0.01	<0.01 (0.0077)	<0.05 (<0.01)
							0.01	<0.01 (0.0097)	<0.05 (<0.01)
United States,	SL	105	104	BBCH	1	60	0.062	0.052	<0.05 (0.034)

Rape seed country, year (variety)	Application					DALA Days	Original residue of quinclorac methyl ester reported in study (method D1607/01 (mg/kg)	Quinclorac methyl ester residue following re-analysis (mg/kg)	
	Form	g ai/ha	water, L/ha	Growth Stage	no.			Method D1607/01	Method D9806
2016 American Falls/I (Hyclass 930) **Trial 9**				13			0.069	0.050	<0.05 (0.034)

Notes:

Values in parentheses are the residue levels determined below the limit of quantification (LOQ).

Method D1607/01: LOQ = 0.01 mg/kg, LOD = 0.002 mg/kg.

Method D9806: LOQ = 0.05 mg/kg, LOD = 0.01 mg/kg.

Along with the re-analysis summarized in Tables 7 and 8, the analytical correction factors outlined in tables 9 and 10 for quinclorac and quinclorac methyl ester were presented.

Table 9 Analytical correction factors for quinclorac

Rape seed country, year (variety)	Quinclorac residue following re-analysis (mg/kg)		% difference of methods (Analytical correction factor)
	Method D1607/01	Method D9708/1	
GAP, Canada	-	-	
United States, 2016 Northwood/ ND (L252) **Trial 1**	0.021	< 0.05 (0.03)	143**
	0.021	< 0.05 (0.03)	143
United States, 2016 Carrington/ ND (L252) **Trial 2**	0.03	<0.05 (0.035)	117
	0.03	<0.05 (0.04)	133
Canada, 2016 Branchton/ ON (Pioneer 46H75) **Trial 3**	0.051	0.11	216
	0.049	0.074	151
Canada, 2016 Okanagan Falls/BC (Liberty Link Invigor) **Trial 6**	0.088	0.17	193
	0.096	0.17	177
United States, 2016 American Falls/ID (Hyclass 930) **Trial 9**	0.014	<0.05 (0.028)	200

Notes:

** (Residue value determined by method D9708//residue value determined by D1607/01) * 100.

= (0.03/0.021)* 100.

= 142 percent.

Monitoring trials (also referred to as demonstration trials)

The current Meeting also received commercial demonstration trials. These trials were conducted to verify the residue levels of quinclorac in rape seed oil when treated at the Canadian GAP under commercial conditions. The trials were not generated following standard criteria and the report did not contain the standard field and analytical phases.

A total of 7 demonstration trials were conducted in 2016 and 2017 in Canada. At each trial site two use patterns were investigated. One plot was treated at a rate of 1 × 125 g ai/ha applied pre-sowing. A second plot was treated at a rate of 1 × 75 g ai/ha with the application applied at the 2–6 leaf stage.

It is stated that samples were collected at normal commercial harvest and followed the approved label conditions, although the dates of harvest are not given and hence the time between the application and harvest is not known.

The samples from the trials conducted in 2016 were stored at ambient temperature for up to 4 months prior to shipment to the laboratory. The storage conditions prior to analysis following shipment are not reported. The samples from the trials conducted in 2017 were stored frozen prior to analysis; the dates of analysis and therefore the length of storage are not reported.

Residues of quinclorac were determined using the LC-MS/MS method based on the QuEChERS method for acidic herbicides. This included extraction using NaOH which is therefore likely to result in a portion of the quinclorac methyl ester (if present) being converted back into quinclorac. Procedural recoveries were conducted at 0.01 mg/kg and 1 mg/kg with recoveries in the range of 84–136 percent.

As the demonstration trials were focused on the residue definition for compliance with MRLs, quinclorac methyl ester residues were not determined. A summary of the trials is outlined in Table 10.

Table 10 Residue trials data for oilseed rape conducted in Canada in 2016 and 2017 (monitoring trials)

Location, Country Year, Crop/Variety	Rate (g ai/ha)	Growth stage at last application	DALA (days)†	Crop part	Quinclorac (mg/kg)	Reference
GAP Canada	125	Pre-emergency	Not stated	Seeds	0.039	Cleveland, C. 2019
Lethbridge, Alberta, Canada 2016					0.046	
					0.039	
					0.062	
	75	2-6 leaf stage	Not stated	Seeds	0.064	
					0.063	
					0.074	
					1.3	
Estlin, Saskatchewa, Canada 2016	125	Pre-emergency	Not stated	Seeds	<0.01	
					<0.01	
					<0.01	
					<0.01	
	75	2-6 leaf stage	Not stated	Seeds	<0.01	
					<0.01	
					<0.01	
					<0.01	
Estlin, Saskatchewa,	125	Pre-emergency	Not stated	Seeds	<0.01	
					<0.01	

Location, Country Year, Crop/Variety	Rate (g ai/ha)	Growth stage at last application	DALA (days)†	Crop part	Quinclorac (mg/kg)	Reference
Canada 2016					<0.01	
					<0.01	
	75	2-6 leaf stage	Not stated	Seeds	<0.01	
					<0.01	
					<0.01	
					<0.01	
Winkler, Manitoba, Canada 2016	125	Pre-emergency	Not stated	Seeds	0.022	
					0.029	
					<0.01	
					<0.01	
	75	2-6 leaf stage	Not stated	Seeds	0.038	
					0.031	
					0.046	
					0.063	
Estlin, Saskatchewa, Canada 2017	125	Pre-emergency	Not stated	seeds	<0.01	
					<0.01	
					<0.01	
					<0.01	
	75	2-6 leaf stage	Not stated	Seeds	0.051	
					0.043	
					0.027	
					0.018	
Saskatoon, Saskatchewan, Canada 2017	125	Pre-emergency	Not stated	Seeds	0.040	
					0.031	
					0.076	
					0.055	
	75	2-6 leaf stage	Not stated	Seeds	0.029	
					0.011	
					0.011	
					0.012	
Lethbridge, Alberta, Canada 2016	125	Pre-emergency	Not stated	Seeds	0.19	
					0.22	
					0.26	
					0.23	
	75	2-6 leaf stage	Not stated	Seeds	0.10	
					0.10	
					0.13	
					0.14	

Notes:

† Individual results represent independent representative treated samples taken at commercial harvest at the trial site.

FATE OF RESIDUES DURING PROCESSING

No additional information was received by the current Meeting.

RESIDUES IN ANIMAL COMMODITIES

No additional information was received by the current Meeting.

APPRAISAL

Quinclorac is a systemic herbicide used with uptake through roots and foliage and used to control annual grass and broadleaf weeds. It was evaluated by the 2015 JMPR for the first time for toxicology and for residues and re-evaluated in 2017 (R) for additional uses. The 2015 JMPR allocated an ADI of 0–0.4 mg/kg bw, and an ARfD of 2 mg/kg bw.

For plant commodities, the residue definition for compliance with MRLs is quinclorac plus quinclorac conjugates and the residue definition for the estimation of dietary intakes is quinclorac plus quinclorac conjugates plus quinclorac methyl ester expressed as quinclorac. The 2015 JMPR noted that quinclorac methyl ester has a toxicological potency up to 10 times that of quinclorac and agreed to multiply the quinclorac methyl ester residues with a factor of 10 to express it as quinclorac equivalents.

For animal commodities the residue definition for compliance with MRLs and for estimation of dietary intakes is quinclorac plus quinclorac conjugates. The residue is fat-soluble.

Quinclorac was scheduled at the Fifty-first Session of the CCPR for the re-assessment of residue trials for oil seed rape previously considered by the JMPR, which had used an unsuitable analytical method and could not be used for estimating a maximum residue level.

The current Meeting received information on a use pattern, the re-analysis of residue trial samples and commercial demonstration trials for rapeseed at the request of the CCPR51. A use pattern, new residue trials and additional validation data for cranberries were also received by the current Meeting.

Methods of analysis

The current Meeting considered additional procedural recovery data to support the analytical methods previously considered by the JMPR in 2015 and 2017.

Method D1607/01

Residues in cranberries and in oil seed rape were determined using method D1607/01 which was considered by the 2017 JMPR. This method employs three consecutive extraction procedures that allows the separate determination of quinclorac, quinclorac conjugates and quinclorac methyl ester. Final determination was by LC-MS/MS.

The 2017 JMPR concluded that this method was suitable for the analysis of quinclorac and quinclorac methyl ester residues in rape seed and forage. The LOQ for the sum of quinclorac and quinclorac conjugates residues was 0.01 mg/kg, and the LOQ for residues of quinclorac methyl ester was 0.01 mg/kg.

The current Meeting considered procedural recovery data for cranberries. The meeting concluded that method D1607/01 was suitable for the determination of quinclorac and quinclorac methyl ester residues in cranberries. The LOQ for residues for the sum of quinclorac and quinclorac conjugates is 0.02 mg/kg, and the LOQ for residues of quinclorac methyl ester is 0.01 mg/kg.

Multi-residue analytical method (Based on QuEChERS method for acidic herbicides)

The method was employed in the analysis of the rape seed samples from the commercial demonstration trials (=monitoring trials) provided to the current Meeting. The method is similar to the multi-residue analytical method D1502/1 considered by the 2017 JMPR, although the initial extraction employed is different.

Samples were prepared by homogenizing with liquid nitrogen/dry ice. A subsample was hydrated with water and the pH was adjusted to >9 with NaOH. After shaking the sample for 30 minutes and centrifuging, an aliquot was removed and shaken with dichloromethane (DCM). The DCM was then discarded, the pH adjusted to ≤2 with sulfuric acid and extraction was achieved using acetonitrile and 'QuEChERS' salts. Final determination was by LC-MS/MS.

A radio-validation study considered by the 2015 JMPR showed that extraction with acetone/0.1 M NaOH converts the quinclorac methyl ester (if present) partly back into the parent compound. As this may result in an over estimation of parent levels in rapeseed the Meeting considered the method used in the monitoring trials as unsuitable for the estimation of maximum residue levels.

Stability of pesticide residues in stored analytical samples

The JMPR 2015 concluded that for quinclorac residues were stable in cranberries for at least 14 months of storage, covering storage intervals in the newly provided supervised field trials.

For rape seed samples, the 2015 JMPR concluded that quinclorac residues in oil seed rape are stable for at least 22 months. In the re-analysis of the rape seed samples from trials provided in 2017, maximum storage intervals after sampling were 670 days (=22 months).

Results of supervised residue trials on crops

Cranberries

The critical GAP is for the United States, which consists of two applications at 280 g ai/ha, a re-treatment interval (RTI) of 30 days and pre-harvest interval (PHI) of 60 days.

In 2015, four independent trials matching the GAP were assessed by the JMPR. The residues of quinclorac (including conjugates) were reported as (n=4): 0.16, 0.17, 0.18 and 0.67 mg/kg. The highest individual residue measured in cranberries was 0.68 mg/kg. Residues of quinclorac methyl ester were not determined.

The current Meeting noted that the selection of trial data in the 2015 Report does not correspond to the field trial results underlined correctly in the evaluation and instead of a value of 0.55 mg/kg a lower concentration of 0.16 mg/kg from a replicate trial was considered. Consequently, the corrected ranking of residues of quinclorac (including conjugates) was (n=4): 0.17, 0.18, 0.55 and 0.67 mg/kg. The highest individual residue measured in cranberries remains at 0.68 mg/kg.

The current Meeting received five additional new independent residue trials conducted in the United States in 2019 matching the cGAP. Residues of quinclorac (including conjugates) were (n= 5): 0.08, 0.09, 0.11, 0.26 and 0.56 mg/kg. Residues of quinclorac methyl ester were < 0.02(5) mg/kg.

Based on all trials, residues of quinclorac and its conjugates in cranberry for MRL estimation were (n=9): 0.08, 0.09, 0.11, 0.17, 0.18, 0.26, 0.55, 0.56 and 0.67 mg/kg.

The highest individual residue measured in cranberries was 0.68 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg in cranberries confirming the previous recommendation.

Residues of quinclorac methyl ester were < 0.02 mg/kg in all five of the new trials considered by the current Meeting. In the supporting plant metabolism study on strawberries reviewed by the 2015 Meeting, quinclorac methyl ester residues were below the limit of detection at DAT 60, corresponding to the pre-harvest interval of the critical GAP. Therefore, the Meeting decided that to estimate residues for dietary exposure, a residue of 0.02 mg/kg for quinclorac methyl ester represents a conservative estimate and should be taken into account for the calculation for the total quinclorac residue in all supervised field trials without measurement of the analyte.

The total residue was calculated based on the formula: (quinclorac + quinclorac conjugate) + 10 × quinclorac methyl ester, all expressed as quinclorac.

Residues for dietary intake estimation in cranberries were (n=9): 0.28, 0.29, 0.31, *0.37*, *0.38*, 0.46, *0.75*, 0.76 and *0.87* mg/kg (values in italics involve no measurement of quinclorac methyl ester). The highest individual residue in cranberries is 0.88 mg/kg.

Based on this data set the Meeting estimated an STMR and HR value of 0.375 mg/kg and 0.88 mg/kg respectively.

Rape seed

The critical GAP is from Canada which allows one application of 100 g ai/ha with a PHI of 60 days.

No new supervised field trials were provided to the current Meeting.

The 2015 JMPR considered 17 residue trials conducted in 1997. As residues were determined using method D9708/1, which may overestimate the quinclorac level due to partial hydrolysis of the quinclorac methyl ester, the trials were not suitable for the estimation of a maximum residue level.

The 2017 JMPR considered nine additional residue trials conducted in 2016, that matched the cGAP for Canada, involving analysis with method D1607/01, measuring quinclorac methyl ester and quinclorac (incl. conjugates) separately, and estimated a maximum residue level of 0.15 mg/kg and an STMR of 0.64 mg/kg.

The current Meeting received information on the re-analysis of the samples from the 2016 trials with the intention to derive correction factors, allowing consideration of trials conducted in 1997. The samples were re-analysed using the following methods:

- Method D1607/01 involves three extractions 1) acetonitrile/water, 2) acetone/phosphate buffer pH 7, 3) 1 M NaOH at 100 °C for 1 hr, each extract analysed separately for parent (including parent released from conjugates) and the methyl-ester (considered by the 2017 JMPR);
- Method D9708/1 involves extraction with acetone/0.1 M NaOH for quinclorac and may convert the methyl ester partly to parent. Hence, it may overestimate the quinclorac residue level (considered by the 2015 JMPR);
- Method D9806 involves extraction with acetone for quinclorac methyl ester (considered by the 2015 JMPR).

The Meeting noted that re-analysis of quinclorac residues using method D9708/1 gave less than the validated LOQ of 0.05 mg/kg in seven of the nine trials. In the other two trials, method D9708/1 recovered 151–216percent of the residue initially measured with method D 1607/01.

The Meeting noted a high variability in the results from both methods in combination with a validated LOQ for method D9708/1, too high for quantification of residues in most samples. Therefore, the Meeting decided not to derive analytical correction factors, which would introduce significant uncertainty to the estimations and does not follow its common assessment practice. The Meeting confirmed its previous conclusion that the residue trials from 1997 are not suitable for estimating a maximum residue level.

In addition, the Meeting considered seven commercial demonstration trials (=targeted monitoring data) provided by the request of the CCPR51, conducted according to the Canadian GAP. Each trial consisted of two sub-plots at rates of 75 or 125 g ai/ha and involved analysis of four field samples for each sub-plot.

Residues of quinclorac were determined using the QuEChERS method for acidic herbicides. This included extraction using NaOH which is therefore likely to result in a portion of the quinclorac methyl ester (if present) being converted back into quinclorac.

Noting the potential overestimation of residues according to the residue definition for enforcement purposes, only five individual results (from two sub-plots) out of 56 total measurements exceeded the maximum residue level of 0.15 mg/kg recommended by the 2017 JMPR. In addition, lack of information on important trial parameters such as the dates of harvest and storage periods prior to analysis were noted. The Meeting decided the provided information was unsuitable for maximum residue level estimation.

The Meeting confirmed its previous recommendations of an MRL of 0.15 mg/kg, a median residues value for livestock feed of 0.017 mg/kg for rapeseed and an STMR of 0.64 mg/kg for the estimation of dietary intake, as well as for rape seed oil edible of 0.70 mg/kg

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Table 11 Recommendations for residues of quinclorac from the 2022 JMPR

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New	Previous		
FB 0265	Cranberries	1.5	1.5	0.375	0.88
SO 0495	Rape seeds	0.15	0.15	0.64 (median: 0.017 for feed calc.)	-
OR 0495	Rape seed oil, edible			0.70	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for quinclorac is 0–0.4 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for quinclorac were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 0–1 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of quinclorac from uses considered by the JMPR is unlikely to present a public health concern

Acute dietary exposure

The ARfD for quinclorac is 2 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for quinclorac were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR report.

The IESTIs were 0percent of the ARfD for children and 0percent of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of quinclorac from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Author	Report No./Trial ID	Year	Title, Institute
Lennon, G	IR-4 PR No 12639	2020	Quinclorac: magnitude of the residue in cranberry
Cleveland, C., Lutz, K., Headon, C.	2019/ 2046959	2019	Magnitude of Quinclorac (BAS 514 H) residues in replicated demonstration plot research trials in Canada BASF Corp., Research Triangle Park NC, United States of America
Saha, M.	2018/ 7005740	2018	Analysis of canola seed samples from study 805114 using analytical methods, D9708/1 for quinclorac, D9806 for quinclorac methyl ester and D1607/01 for quinclorac and quinclorac methyl ester for comparison of residue values EPL Bio-Analytical Services Inc., Niantic IL, United States of America

QUINTOZENE (064)

First draft prepared by Dr Yukiko Yamada, International Food Safety Consultant, National Institute on Health Sciences, Ministry of Health, Labour & Welfare, Japan

EXPLANATION

Quintozene, pentachloronitrobenzene (IUPAC name), is an aromatic fungicide, used as soil fungicide or for seed treatment of various vegetables, cereal grains, and oil seeds. It is also used as a slime inhibitor in industrial waters and soil treatment of lawns and ornamentals.

Quintozene was first evaluated by the JMPR in 1969 as a new compound for toxicology and residues. It was reviewed under the CCPR periodic re-evaluation by the 1995 JMPR for toxicology and residues.

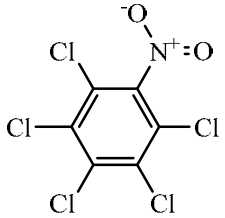
The 1998 JMPR received data on environmental fate, residues in rotational crops, additional supervised trials and processing on certain commodities. The 1998 JMPR confirmed the residue definitions and estimated maximum residue levels and STMRs for a number of commodities. The 2004 JMPR estimated maximum residue levels for spices on a basis of monitoring data.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the 2021 Meeting, including periodic re-evaluation of quintozene. As a result of postponement of the evaluation, quintozene is evaluated by the current Meeting.

No specification has been established for quintozene by the Joint FAO/WHO Meeting on Pesticide Specifications.

The Meeting received information on identity, chemical and physical properties, plant and animal metabolism, rotational crop studies, environmental fate, residue analysis and storage stability, use pattern, supervised trials on various crop, and processing studies.

IDENTITY

ISO common name:	Quintozene
Chemical name	
IUPAC:	Pentachloronitrobenzene (IUPAC PIN, 1,2,3,4,5-pentachloro-6-nitrobenzene)
CAS:	1,2,3,4,5-pentachloro-6-nitrobenzene
CAS Registry No.:	82-68-8
CIPAC No.:	78
Synonym	quintozene (pentachloronitrobenzene)
Structural formula:	
Molecular formula:	$C_6Cl_5NO_2$
Molecular mass:	295.33 g/mol

PHYSICAL AND CHEMICAL PROPERTIES

Property	Results	Reference
Appearance	Off white to dark tan coloured crystalline solid with slight musty odour (TGAI)	Suppl. MRID 406686602
Density (specific gravity)	1.72 g/mL (TGAI)	Suppl. MRID 406686602
Dissociation constant	No dissociation	Suppl. MRID 406686602
Melting point	141–146 °C (TGAI)	Suppl. MRID 406686602
Boiling point	Not required (solid at room temperature)	Suppl. MRID 406686602
Vapour pressure	1.27×10^{-2} Pa at 25 °C (Purity, 99 percent)	Thomson, 1989, 900-PCH-305
Solubility in water	1×10^{-4} g/L at 25 °C (purity, 99.2 percent)	Batorewicz & Bakker, 1988, 900-PCH-252
Solubility in organic solvents at 20°C	Acetone 134 g/L Acetonitrile, 46 g/kg Dichloromethane 345 g/kg Ethyl acetate 161 g/L Hexane 33 g/kg Methanol 12 g/kg n-Octanol 8.6 g/L Toluene 618 g/kg (purity, 99.9 percent)	Donnelly, 1998, 900-PCH-175
Octanol/water partition coefficient (Log K_{ow})	5–6 (pH 7, 27 °C) (purity, 99.2 percent)	Polakoff B.M., 1987, 900-PCH-244
Hydrolysis in sterile buffer in the dark	Hydrolytically stable (after 30 days at 25 °C, at pH 5, 7 or 9, no significant degradation was observed) (purity, 99.2 percent)	Bowman B.R., 1988a, 900-PHO-008
Photolysis in sterile water under artificial light	Half-life of quintozene (0.075 mg/L) in sterilized acetate buffer at pH 5 at 25±1 °C under 32 h continuous irradiation with Xenon arc lamp (650 W/m^2): 13.4 h (r^2 =.0.99). Photodegradation products after 32 h: isomeric mixture of chlorinated hydroxybenzenes and/or chloronitrophenols (comprising 49.8 percent of the applied radioactivity).	Horree, D.J., 1992, 900-PHO-018

Formulations

Quintozene is available in the following formulations:

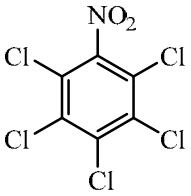
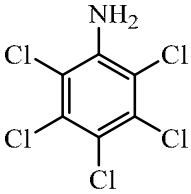
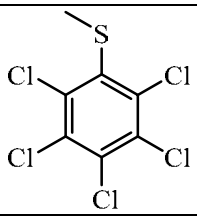
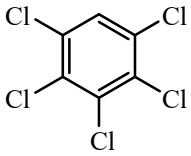
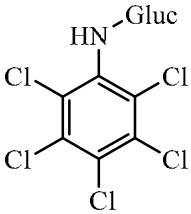
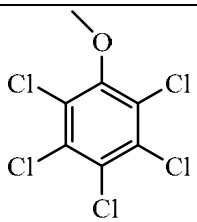
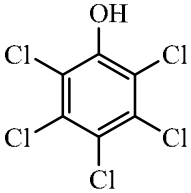
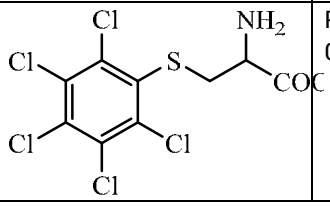
- Flowable (FL) formulation containing quintozene at 480 g/L (40 percent, w/w);
- Wettable powder (WP) formulation containing quintozene at 750 g/kg;
- Emulsifiable concentrate (EC) formulation containing quintozene at 240 g/L;
- Granule (GR) formulation containing quintozene at 100 g/kg.

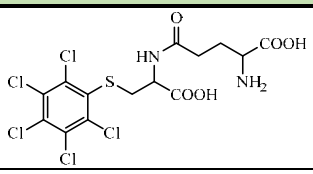
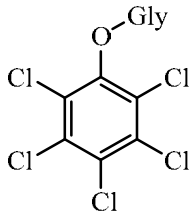
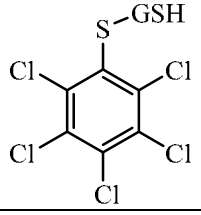
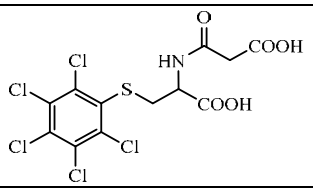
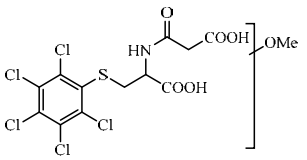
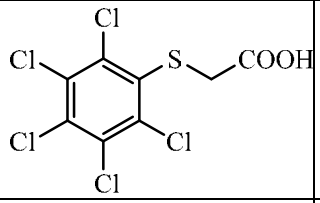
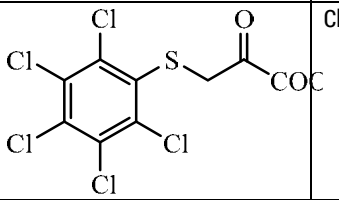
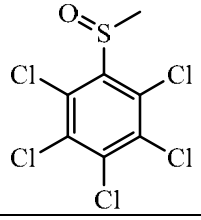
According to the manufacturer, hexachlorobenzene is allowed up to 0.03–0.05 percent as an impurity in TGAI in the countries where quintozene or PCNB (see Table 1) is registered (the 1995 JMPR reported the maximum to be 0.1 percent at the time of evaluation.).

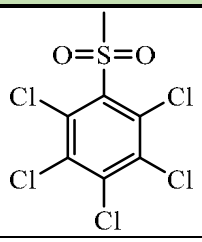
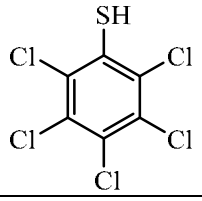
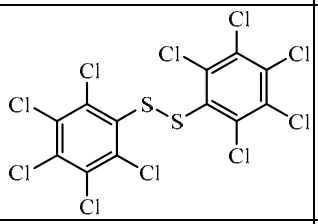
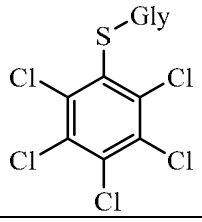
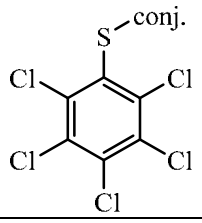
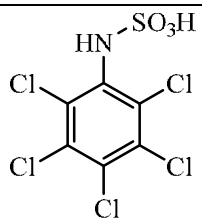
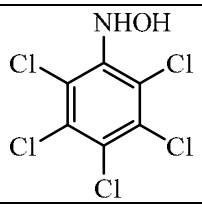
METABOLISM AND ENVIRONMENTAL FATE

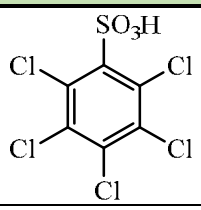
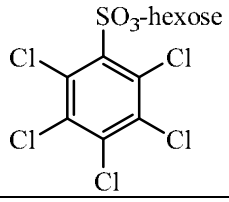
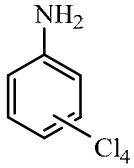
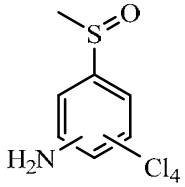
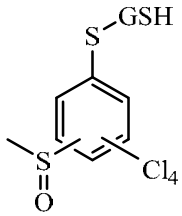
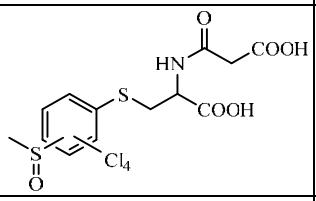
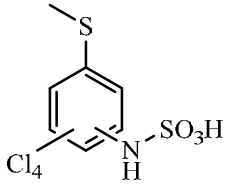
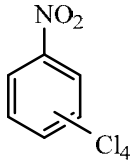
Table 1 below shows the code numbers, IUPAC names and structures of the compounds appearing in the various metabolism and environmental fate studies.

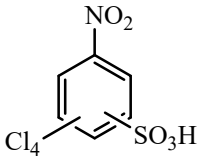
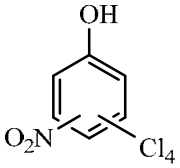
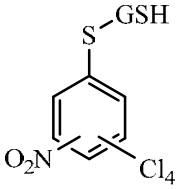
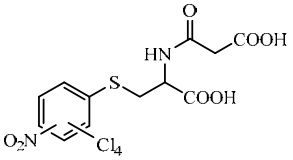
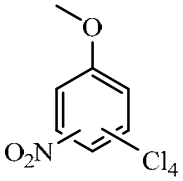
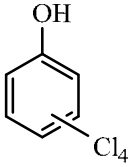
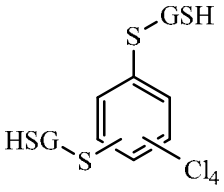
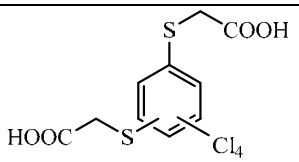
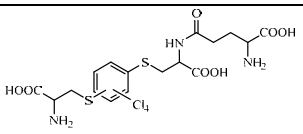
Table 1 Structure of compounds appearing in metabolism and environmental fate studies

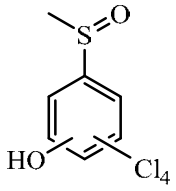
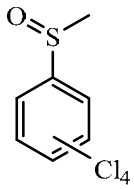
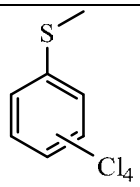
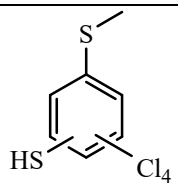
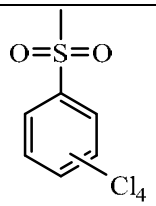
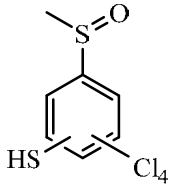
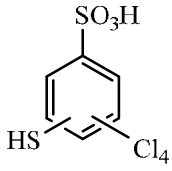
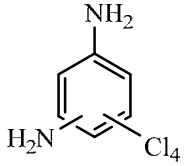
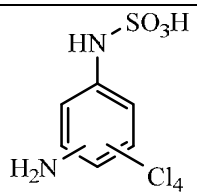
Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
5 chlorine atoms on the phenyl ring			
Quintozene Synonym: PCNB (295.32)	Pentachloronitrobenzene		Potato (whole, peel), peanut root, cabbage, Rotational crops (lettuce, wheat foliage), Chicken fat
PCA (265.34)	Pentachloroaniline		Potato (whole, peel), peanut, Rotational crops (turnip top & root, lettuce, wheat foliage), Goat liver, kidney, fat, milk, Chicken fat, egg
PCTA (296.41)	Methyl pentachlorophenyl sulfide (pentachlorothioanisole)		Potato (whole, peel), cabbage, Rotational crops (turnip root, lettuce, wheat foliage), Chicken muscle, egg
PB (250.32)	Pentachlorobenzene		Potato, Rotational crops (lettuce, wheat foliage), Chicken egg
PCA-Gluc (455.49)	*N*-(pentachloroaniline) glucuronide		Goat liver, kidney
PCAN (280.35)	Pentachloroanisole		Potato
PCP (266.32)	Pentachlorophenol		Rotational crops (turnip top & root)
PCP Cys (369.46)	*S*-(pentachlorophenyl) cysteine		Potato (whole, peel), Chicken kidney

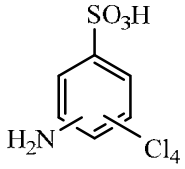
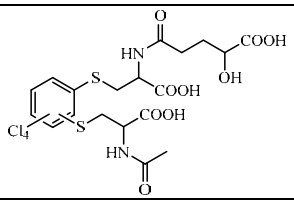
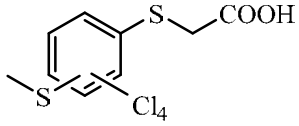
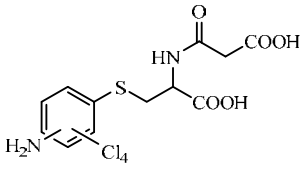
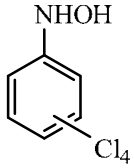
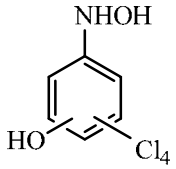
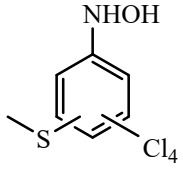
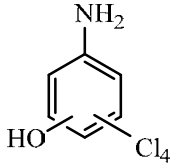
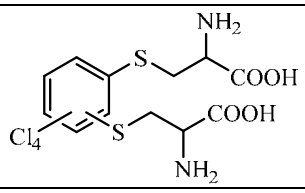
Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
PCP-GluCys (498.58)	S-(Pentachlorophenyl)-γ-glutamylcysteine		Potato (whole, peel)
PCP Gly (428.46)	Pentachlorophenol glycoside		Potato peel
PCP-GSH (555.63)	S-(Pentachlorophenyl) glutathione		Potato, Rotational crops (wheat foliage)
PCP-MalCys (455.51)	*S*-(Pentachlorophenyl) malonylcysteine or Pentachlorothiophenyl malonylcysteine (PCTP-MalCys)		Potato, peanut, Rotational crops (lettuce)
PCP-MalCys ester (469.54)	S-(Pentachlorophenyl) malonylcysteine monomethyl ester		Potato (whole, peel), peanut root Chicken excreta
PCP thioacetate (340.42)	*S*-(pentachlorophenyl) thioacetate		Chicken liver, kidney, muscle, excreta
PCP thiopyruvate (368.43)	*S*-(pentachlorophenyl) thiopyruvate		Chicken excreta
PCTASO (PCPMS) (C5MX) (312.41)	Pentachlorophenyl methyl sulfoxide (Pentachlorothioanisole sulfoxide)		Cabbage, Rotational crops (turnip top & root, wheat foliage), Soil (rotational crop study) Chicken liver

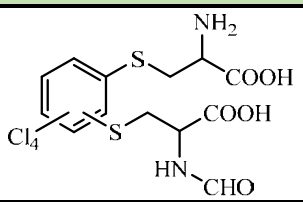
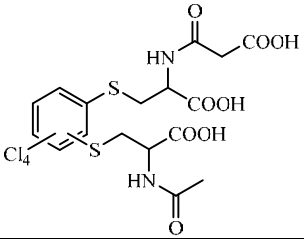
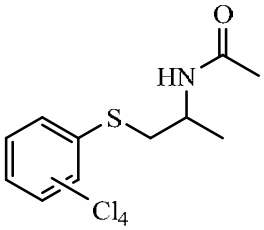
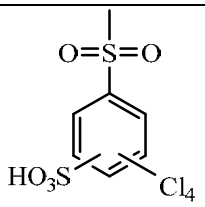
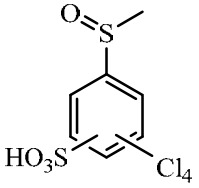
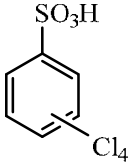
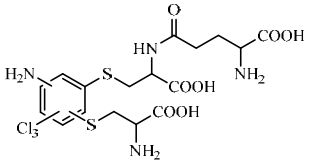
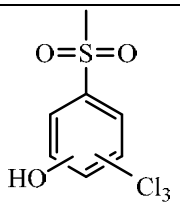
Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
PCTASOO (C5MS) (328.41)	Pentachlorothioanisole sulfone		Soil
PCTP (282.38)	Pentachlorothiophenol		Goat liver, kidney Chicken liver, egg, excreta
PCTP dimer (562.75)	Pentachlorothiophenol dimer		Goat liver
PCTP-Gly (444.53)	Pentachlorothiophenyl glycoside		Potato peel
PCTP-X (-)	Pentachlorothiophenyl conjugate		Potato peel, Goat urine
Pentachloroaniline sulfamate (345.40)	Pentachloroaniline sulfamate		Goat urine
NOHPCA (281.34)	*N*-Hydroxypentachloroaniline		Potato (whole, peel), peanut root, cabbage, Goat liver, Chicken kidney
NOHPCA-Gluc [a] (455.49)	Glucuronide of NOHPCA	--	Goat liver

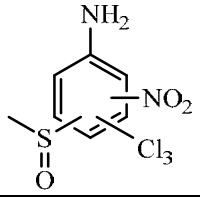
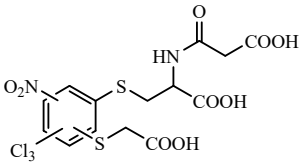
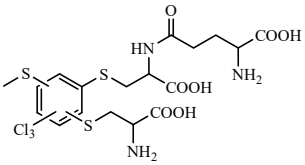
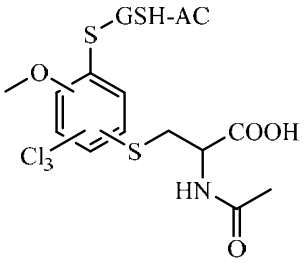
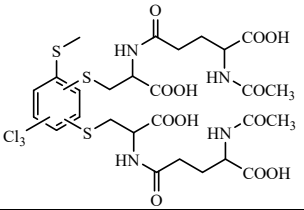
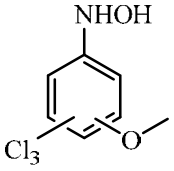
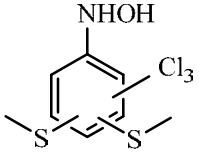
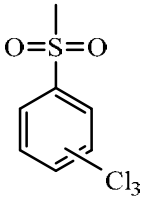
Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
C5SA (330.38)	Pentachlorobenzene-sulfonic acid		Rotational crops (turnip top & root, lettuce, wheat foliage)
C5SAHx (492.52)	Pentachlorobenzene-sulfonic acid hexose ester		Rotational crops (wheat foliage)
C5MX	See PCTASO		
C5MS	See PCTASOO.		
4 chlorine atoms on the phenyl ring			
TCA (230.90)	Tetrachloroaniline		Peanut root
TCA sulfoxide isomers (292.98)	Tetrachloroaniline methyl sulfoxide		Potato, Goat kidney, Chicken fat
TC-MES-P-GSH (583.27)	S-(Tetrachloro-methyl sulfoxy-phenyl)glutathione		Potato
TC-MES-P-MalCys (483.15)	S-(Tetrachloro-methyl sulfoxy-phenyl) malonylcysteine		Potato
TC-MET-A sulfamate (357.04)	Tetrachloro-methylthio-aniline sulfamate		Potato
TCNB isomers (260.88)	Tetrachloronitrobenzene		Potato (whole, peel)

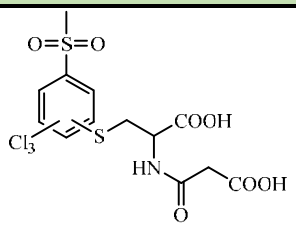
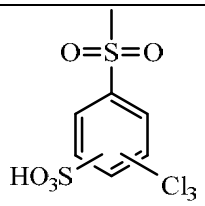
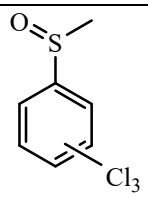
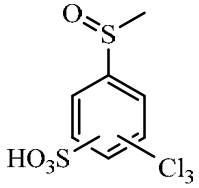
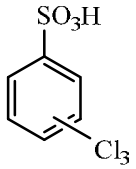
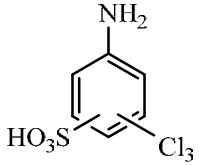
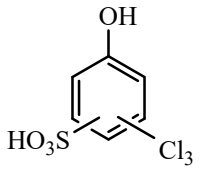
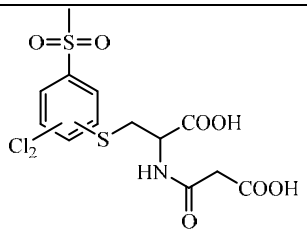
Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
TCNB sulfonic acid isomers (340.94)	Tetrachloronitrobenzene sulfonic acid		Potato
TCNP (276.88)	Tetrachloronitrophenol		Potato (peel)
TCNP-GSH (566.18)	S-(Tetrachloronitrophenyl) glutathione		Potato, peanut
TCNP-MalCys (466.06)	S-(Tetrachloronitrophenyl) malonylcysteine		Potato, peanut
TCNTA isomers (290.91)	Tetrachloronitrothioanisole		Potato
TCP (231.88)	Tetrachlorophenol		Potato (peel)
TCP-diGSH (826.49)	S,S'-(Tetrachlorophenyl) diglutathione		Potato
TCP-dithioacetate (396.07)	S,S'-(Tetrachlorophenyl) dithioacetate		Potato
TCP-GluCys-Cys (583.27)	S,S'-(Tetrachlorophenyl)-γ-glutamyl cysteine-cysteine		Potato

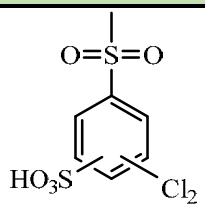
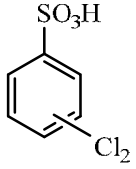
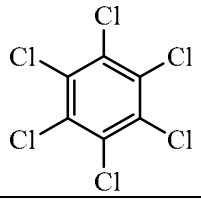
Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
TCP sulfoxide (293.97)	Tetrachlorohydroxyphenyl methyl sulfoxide		Potato
TCPM sulfoxide (C4MX) (277.97)	Tetrachlorophenyl methyl sulfoxide		Cabbage, Rotational crops (wheat foliage)
TCTA (261.97)	Tetrachlorothioanisole		Goat kidney, Chicken fat
TCTP S-Met (294.03)	Tetrachloro (methylthio) thiophenol		Goat liver, kidney, urine
TCTASOO (C4MS) (TCP methyl sulfone) (293.97)	Tetrachlorophenyl methyl sulfone		Cabbage, Rotational crops (turnip top & root, lettuce) Chicken kidney, muscle
TCTP sulfoxide (310.03)	Tetrachlorothiophenol methyl sulfoxide		Potato
TCTP sulfonic acid (328.00)	Tetrachlorothiophenol sulfonic acid		Potato
AM TCA isomers (245.91)	Aminotetrachloroaniline		Potato
AM TCA sulfamate (325.97)	Aminotetrachloroaniline sulfamate		Potato

Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
AM TCB sulfonic acid (310.95)	Aminotetrachlorobenzene sulfonic acid or (C4SANH) (tetrachlorosulfanilic acid)		Potato, peanut, Rotational crops (wheat foliage)
AC TCP-Cys-CysHOG (626.29)	Acetyl S,S'-(tetrachlorophenyl) cysteine-cysteinyl α-hydroxyglutarate		Potato
MTCP-TAA (352.07)	*S*-[(methylthio)tetrachloro-phenyl]-2-thioacetic acid		Peanut root
N-malonyl-S-(tetrachloroaminophenyl)-cysteine (436.08)	N-malonyl-S-(tetrachloroaminophenyl)-cysteine		Peanut
NOHAM TCA (246.90)	N-Hydroxyamino-tetrachloroaniline		Potato
NOHAM TCP (262.90)	N-Hydroxyamino-tetrachlorophenol		Potato
NOHAM TCTA isomers (292.98)	N-Hydroxyamino-tetrachlorophenol		Potato
OH TCA (246.90)	Hydroxytetrachloroaniline		Potato
C4CyCy (454.16)	*S,S'*-tetrachlorophenyl dicysteine		Rotational crops (wheat foliage)

Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
C4CyFCy (482.17)	*S*-(tetrachlorophenyl)-cysteine-*S'*-formylcysteine		Rotational crops (lettuce)
C4MaCyFCy (582.24)	*S*-(tetrachlorophenyl)-*N*-malonylcysteine-*S'*-formylcysteine		Rotational crops (lettuce, wheat foliage)
C4MeAcCy (347.08)	*N*-{1-methyl-2[(tetrachlorophenyl)-thio] ethyl}acetamide (tetrachlorophenyl methyl cysteine)		Rotational crops (lettuce, wheat foliage)
C4MSSA (374.02)	Tetrachlorosulfophenyl methyl sulfone (tetrachlorothioanisole sulfone, sulfonic acid)		Rotational crops (wheat foliage)
C4MXSA (358.03)	Tetrachlorosulfophenyl methylsulfoxide (tetrachlorothioanisole sulfoxide, sulfonic acid)		Rotational crops (wheat foliage)
C4SA (295.94)	Tetrachlorobenzene-sulfonic acid		Rotational crops (turnip top & root, lettuce, wheat foliage)
C4MX	See TCPM sulfoxide		
C4MS	See TCTASOO		
3 chlorine atoms on the phenyl ring			
RCA GluCys-Cys (563.85)	S,S'-(trichloroanilino)-γ-glutamylcysteine-cysteine		Potato
RCHM sulfone (275.52)	Trichlorophenol methyl sulfone		Cabbage

Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
RCNA sulfoxide (303.54)	Trichloronitroaniline methyl sulfoxide		Potato
RCNP-MalCys thioacetate (521.72)	S,S'-(trichloronitrophenyl) malonyl cysteine-thioacetate		Potato
RCTA-GluCys-Cys (594.92)	S,S'-(trichlorothioanisole)-γ-glutamylcysteine-cysteine		Potato
diAC RCAN GSH-Cys (719.98)	Diacetyl *S,S'*-(trichloro-anisole) glutathione-cysteine		Potato
diAC RCTA-diGluCys (808.11)	Diacetyl *S,S'*-(trichloro-thioanisole) diglutathione		Potato
NOHAM RC-OME-A (242.48)	N-hydroxyamino-trichloro-methoxy aniline		Potato
NOH RC-diMET-A (304.63)	N-hydroxy-trichloro-dimethylthioaniline		Potato
C3MS (259.53)	Trichlorophenyl methyl sulfone		Cabbage, Rotational crops (turnip top & root, lettuce, wheat foliage)

Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
C3MSMaCy (464.71)	*N*-malonyl-S-trichloro-(methylsulfonophenyl)-L-cysteine *S*-(trichlorophenyl methyl sulfone)-malonyl cysteine		Rotational crops (turnip root, lettuce)
C3MSSA (339.58)	Trichlorosulfophenyl methyl sulfone (trichlorothioanisole sulfone, sulfonic acid)		Rotational crops (turnip top & root, wheat foliage)
C3MX (243.53)	Trichlorophenyl methyl sulfoxide		Cabbage
C3MXSA (323.58)	Trichlorosulfophenyl methyl sulfoxide (trichlorothioanisole sulfoxide, sulfonic acid)		Rotational crops (wheat foliage)
C3SA (261.50)	Trichlorobenzenesulfonic acid		Rotational crops (turnip top & root, wheat foliage)
C3SANH (276.51)	Trichlorosulfanilic acid		Rotational crops (wheat foliage)
C3SAOH (277.50)	Hydroxy-trichlorobenzene sulfonic acid (trichloro-hydroxybenzene sulfonic acid)		Rotational crops (wheat foliage)
2 chlorine atoms on the phenyl ring			
C2MSMaCy (430.27)	*N*-malonyl-S-dichloro-(methylsulfonophenyl)-L-cysteine-*S*-(dichlorophenyl methyl sulfone)-malonyl cysteine		Rotational crops (turnip top & root)

Compound Name/Code (MW, g/mol)	IUPAC name	Structure	Found in (food and feed commodities):
C2MSSA (305.14)	Dichlorosulfophenyl methyl sulfone (dichlorothioanisole, sulfonic acid)		Rotational crops (wheat foliage)
C2SA (227.06)	Dichlorobenzenesulfonic acid		Rotational crops (turnip top & root, wheat foliage)
Impurity			
HCB (284.77)	Hexachlorobenzene		Impurity

Notes:

[a] Assumed to be equal to PCA-gluc from the proposed structure in the goat metabolism study.

Some code keys (not comprehensive).

First two letters of the code: PC, pentachloro-; TC, tetrachloro-; and RC, trichloro-.Cn, phenyl ring with n chlorine atoms attached to the ring.

SA, sulfonic acid; SO, sulfoxide; SOO, sulfone; MX, methyl sulfoxide; MS, methyl sulfone; Ma or Mal, malonyl; Cys or Cy, cysteinyl; F, formyl, Me, methyl.

The fate and behaviour of quintozene in plants, animal, and soils were investigated using the quintozene uniformly labelled with ^{14}C in the phenyl ring of the molecule. The positions of ^{14}C are shown in Figure 1.

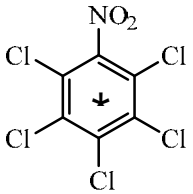


Figure 1 [U-^{14}C]-phenyl labelled quintozene used in the metabolism and environmental fate studies

Plant metabolism

The Meeting received information on the fate of [U-^{14}C]-phenyl labelled quintozene (hereafter abbreviated as ^{14}C-quintozene) in cabbage, potato and peanut after soil treatments. Residues in harvested seeds of maize, peas, sugar beet, wheat and soya bean were also investigated after seed treatment. In the following texts, TRR is expressed in mg-quintozene equivalents/kg.

Cabbage

***Study 1** (McManus & Maisonet, 1990, 900-RES-082)*

The metabolism of quintozene was studied on cabbage plants (variety Stonehead) grown in a greenhouse (temperature controlled) in galvanized steel containers (61 cm × 61 cm × 183 cm = 2 ft × 2 ft × 6 ft) containing soil treated with a single application of ^{14}C-quintozene at 53.8 kg ai/ha before transplanting of 28-day old cabbage plants. Whole plants of cabbage were harvested at one-quarter to one-third (49 DAT), one-half (70 DAT) and full (154 DAT) maturity. At the final harvest, each cabbage head was cut with a knife at the base. The remaining leaves from each of the cabbage plants were also collected. Samples from 49 DAT and 70 DAT were stored frozen immediately after harvest and kept frozen before and after analysis. At the final harvest, two samples of cabbage heads and surrounding leaves were stored in a refrigerator. All remaining samples from the final harvest were stored in a freezer at approximately -20 °C before and after analysis.

Samples were homogenized to a fine consistency with an aid of liquid nitrogen. Homogenized samples were analysed for total radioactive residues (TRR) by combustion / liquid scintillation counting (LSC).

For characterization/identification of residues, a sample aliquot (30 g) was extracted three times with methanol/water (80:20; v/v; 180 mL), followed by acetone (180 ml), then air dried. Liquid fractions were radio-assayed by LSC and unextracted residue in the solids determined by combustion/LSC. The unextracted radioactivity was hydrolysed with methanolic HCl (the ratio of methanol and HCl was not described) at 60 °C for 30 minutes.

The methanol/water extract of the leaf sample was evaporated under vacuum to a small amount and the remaining liquid was passed through a C-18 reverse phase cartridge. Metabolites were eluted with methanol. Radioactive residues were separated by reverse-phase HPLC using a gradient from 100 percent water to 100 percent methanol (both of them contained 1 percent formic acid) and analysed by mass spectrometry in the chemical ionization mode. The radioactive metabolites detected in the radio-chromatogram were collected by preparative HPLC. Collected metabolites were concentrated under nitrogen prior to mass spectral analysis.

The two samples of whole plants taken at the immature stages showed the TRR ranging from 4.1 to 7.9 mg eq/kg. At the maturity, the highest levels of radioactivity were found in the outer leaves (11–18 mg/kg), with lower levels in the heads of 0.70–2.5 mg/kg. (Table 2)

Table 2 Radioactive residues in immature cabbage plants and mature cabbage heads and outer leaves following soil treatment with quintozene at 53.8 kg ai/ha before planting

Sample	DAT	Maturity	TRR (mg eq/kg)	
			Individual values	Mean
Immature whole plant	48	1/4–1/3	7.72; 7.88	7.80
	70	1/2	4.09; 6.39	5.24
Mature head	154	Full	0.874, 0.704, 0.776, 0.757; 1.38, 2.49, 0.914, 1.22	1.14
Mature outer leaves	154	Full	13.0, 11.9, 14.9, 14.6; 16.1, 17.9, 11.7, 11.0	13.89

Table 3 shows that methanol/water extracted 57.5 percent of the total radioactivity in cabbage leaves and acetone 14.7 percent TRR. The post extraction solid (PES) contained 27.8 percent TRR. After refluxing the PES in methanolic HCL for 30 minutes at 60 °C, the radioactivity was soluble in methanol.

Table 3 Extraction of radioactive residues in mature cabbage outer leaves by the solvents and by reflux

Extraction Procedure	% TRR (in outer leaves)
3x MeOH/H_2O (80:20; v/v)	57.5
1x Acetone	14.7
PES	27.8
Heating PES in MeOH/HCl (30 min)	Solubilized

Seven metabolites were identified in MeOH/water extracts of leaf, all of which were more polar than quintozene. From the peak areas of HPLC radio-chromatograms, percent TRR were calculated. These metabolites were isolated and collected by preparative HPLC and analysed by mass spectrometry or by comparison with standard reagents if they were available.

The two main compounds were TCPM sulfoxide (36.1 percent TRR) and TCPM sulfone (41.9 percent TRR). Five minor components (all <10 percent TRR) were identified as a ring-hydroxylated C3MS, C3MS, NOHPCA, PCTASO and PCTA (Table 4).

Attempts were made to identify and quantify metabolites in the solubilized fraction. It was found that the HPLC of solubilized fraction showed similar metabolite profile as the methanol/water extract but the quantification was unsuccessful due to low level radioactivity of each peak and interference from the endogenous plant materials.

Table 4 Metabolites in the methanol/water extracts of cabbage outer leaves following soil application of quintozene at 53.8 kg ai/ha (in the order of high to low polarity)

Component	% TRR
Ring-hydroxylated C3MS	3.7
C3MS [a]	8.9
TCPM sulfoxide (C4MX) [a]	36.1
NOHPCA	4.0
TCPM sulfone [a]	41.9
PCTASO	4.9
PCTA	0.5
Total	100

Notes:

[a] Previously reported as a metabolite of quintozene in parsnip (Cairns *et al.,* 1987).

***Study 2** (Premkumar & Brown, 1992, 900-RES-003)*

Cabbage plants (variety, Copenhagen) were grown in outdoor test plots in California, United States, with sandy loamy soil treated with a single application of ^{14}C-quintozene at 33.7 kg ai/ha. Cabbage seeds were sown after the treatment. Cabbage was harvested at an immature stage (120 DAT) as whole plants, and at maturity (209 DAT) and separated into heads with wrapper leaves, heads without wrapper leaves, and wrapper leaves. Samples were stored frozen immediately after sampling and shipped on dry ice to the analytical laboratory.

Samples were homogenized together with dry ice to a powder-like consistency and stored at -20 °C until analysis. Homogenized samples were analysed for TRR by combustion/LSC.

Table 5 shows the TRR values of all the samples. The highest TRR was observed in immature cabbage plants. The mature cabbage contained significantly less radioactivity and most of the radioactivity was located in wrapper leaves. The combustion was repeated for wrapper leaves and whole cabbage after a long period of freezer storage. The second combustion resulted in higher TRR values,

possibly due to desiccation and repeated thawing and freezing. The TRR values in soil ranged from 2.33 to 5.45 mg eq/kg on a wet weight basis, which indicates that the distribution of quintozene was heterogenous.

Table 5 Radioactive residues in immature cabbage, mature cabbage and soil after soil treatment by quintozene at 33.7 kg ai/ha before planting

Sample	DAT	Mean TRR (mg eq/kg) [a]
Immature cabbage	120	3.37
Mature whole cabbage	209	0.28
Mature cabbage head	209	0.11
Mature wrapper leaves	209	2.03
Soil (0–15 cm)	2 h	5.06 (wet weight basis)

Notes:

[a] Corrected for matrix and oxidizer recovery.

The cabbage samples (whole plant, heads with and without wrapper leaves, and wrapper leaves) were first extracted with hexane followed by methanol. Portions of the mature whole cabbage and mature cabbage wrapper leaf samples were stored frozen. After a long period of storage, they were subjected to the same extraction procedures as above. In these later extractions, the methanol extracts were partitioned with iso-octane, and the iso-octane layers and aqueous layers were separately concentrated under nitrogen gas flow.

The soil samples were extracted with acetone, which extracted 100 percent of TRR.

Sequential extraction with hexane and then methanol extracted a total of about 40–70 percent TRR with about 30–60 percent TRR remaining in the PES. The extractability into hexane and methanol were comparable for each matrix (much higher extractability in methanol than in hexane) as shown in Table 6.

Table 6 Extraction of radioactive residues in immature and mature cabbage samples, after about 5 months and 20–32 months of frozen storage after harvest

Sample	Extraction timing, months after harvest	Hexane extract		Methanol extract		Total		PES	
		mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR
Immature cabbage	5.6	0.626	17.8	1.14	32.5	1.77	50.3	1.59	45.2
Mature cabbage head	5.0	0.004	3.6	0.041	40.0	0.045	43.6	0.057	56.4
Mature outer leaves	4.7 [a]	0.067	2.8	0.879	36.9	0.946	39.7	1.44	60.3
	32 [b]	0.173	6.1	1.40	49.2	1.57	55.3	1.21	42.7
Mature whole cabbage	20 [c]	0.008	2.3	0.161	45.0	0.169	47.3	0.189	52.6
	31 [d]	0.022	7.7	0.17	59.7	0.192	67.4	0.093	32.6

Notes:

[a] TRR, 2.38 mg eq/kg.

[b] TRR, 2.84 mg eq/kg.

[c] TRR, 0.359 mg eq/kg.

[d] TRR, 0.285 mg eq/kg.

The hexane and methanol extracts were profiled by TLC and reversed phase HPLC-UV (gradient with 1 percent acetic acid and acetonitrile). Extractions and HPLC analyses were conducted sometimes more than two years after the harvest due to a problem in developing the HPLC method. Due to the long duration between the harvest and extraction and between extraction and analysis, storage stability was tested for metabolites in the hexane extracts and those in the methanol extracts of wrapper leaves and whole cabbage by comparing the chromatograms before and after the storage. It was found that the metabolites in the hexane extracts were stable during the storage while those in methanol extracts were not. Initial analysis showed more complex profile than the later analysis. Polar metabolites in peaks E, F, G and H seemed to degrade to those in peak D while M1 and M2 in peak C were stable. Only the analytical results of non-polar metabolites and M1/M2 can be used.

In the immature cabbage sample, the level of identification was low at 18.1 percent and total recovery was also low at 65.5 percent TRR. Seven compounds were identified: parent and five metabolites, PCA, PCTA, 6-TCNB and the malonylcysteine conjugates of PCP and TCNP (contained in a single peak of C of HPLC), and impurity HCB. None of them exceeded 10 percent TRR. Metabolites in four polar peak regions (A, D, E and F) were not identified due to their low-level presence.

Table 7 Metabolites in the hexane and methanol extracts of immature cabbage following soil application with quintozene at 33.7 kg ai/ha before planting

Component/region	Hexane extract		Methanol extract		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Quintozene	0.316	9.0	0.011	0.3	0.327	9.3
PCA	0.190	5.4	--	--	0.190	5.4
PCTA	0.011	0.3	--	--	0.011	0.3
HCB	0.032	0.9	--	--	0.032	0.9
6-TCNB	--	--	0.004	0.1	0.004	0.1
M1 & M2 (C) [a]	0.060	1.7	0.014	0.4	0.074	2.1
				Total identified	0.638	18.1
A	--	--	0.004	0.1	0.004	0.1
D	0.021	0.6	0.032	0.9	0.053	1.5
E	--	--	0.004	0.1	0.004	0.1
F	--	--	0.021	0.6	0.021	0.6
				PES	1.590	45.2
				TOTAL	2.303	65.5

Notes:

[a] M1 = TCNP-MalCys; M2 = PCP-MalCys.

In the mature wrapper leaves, the level of identification was low (4.8 or 15.6 percent TRR). Six compounds were identified: parent, the metabolites PCA, PCTA, PB and the malonylcysteine conjugates of PCP and TCNP. None of them exceeded 10 percent TRR. Metabolites in six peak regions (B, D, E, F, G) could not be identified. The TRR of region D was 33 percent TRR (0.93 mg eq/kg) but D region contained a series of peaks suggesting the presence of several components. After freezer storage for about 2 years, metabolites in regions B, E–G seem to be converted to those in region D (less polar region).

The HPLC chromatogram of the last extraction conducted 35 months after harvest showed no peak in the hexane extract and only peaks D and H in the methanol extract, 36.9 and 6.0 percent TRR respectively corresponding to 0.709 and 0.115 mg eq/kg. The PES from methanol extraction contained 43.6 percent TRR (0.839 mg eq/kg).

Table 8 Metabolites in the hexane and methanol extracts of mature cabbage wrapper leaves following soil application with quintozene at 33.7 kg ai/ha before planting

Component /peak	Extraction 4.7 months after harvest						Extraction 32 months after harvest					
	Hexane ext.		Methanol ext.		Total		Hexane ext.		Methanol ext.		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Quintozene	0.023	1.0	--	--	0.023	1.0	0.047	1.6	--	--	0.047	1.6
PCA	0.014	0.6	--	--	0.014	0.6	0.030	1.1	--	--	0.030	1.1
PCTA	0.009	0.4	--	--	0.009	0.4	0.004	0.2	--	--	0.004	0.2
PB	--	--	--	--	--	--	0.017	0.6	--	--	0.017	0.6
M1 & M2 (C) [a]	0.007	0.3	0.063	2.6	0.069	2.8	0.014	0.5	0.329	11.6	0.343	12.1
Total identified					0.114	4.8	Total identified				0.424	15.6
B	--	--	0.012	0.5	0.012	0.5	--	--	--	--	--	--
D	0.022	0.9	0.194	8.1	0.216	9.1	0.059	2.1	0.871	30.7	0.930	32.8
E	--	--	0.082	3.4	0.082	3.4	--	--	--	--	--	--
F	--	--	0.195	8.2	0.089	8.2	--	--	--	--	--	--
G	--	--	0.089	3.7	0.195	3.7	--	--	--	--	--	--
H	--	--	0.245	10.3	0.245	10.3	--	--	0.221	7.8	0.221	7.8
PES					1.470	61.7	PES				1.211	42.7
TOTAL					2.388	100.2	TOTAL				2.802	98.8

Notes:

[a] M1 = TCNP-MalCys; M2 = PCP-MalCys.

The methanol extract and PES of the mature cabbage wrapper leaf sample was hydrolysed with 6 mol/L HCl and partitions with dichloromethane. However, only 12.7 percent TRR of methanol extract and 1.9 percent TRR of PES were solubilized.

In the mature whole cabbage, only PCTA and the malonylcysteine conjugates of PCP and TCNP could be identified at small levels up to 5.5 percent TRR (0.016 mg/kg) whereas six regions (up to 43 percent TRR (0.12 mg/kg)) could not be identified. Parent quintozene was not detected.

Table 9 Metabolites in the methanol extracts of mature whole cabbage following soil application with quintozene at 33.7 kg ai/ha before planting

Component/peak	Extraction 20 months after harvest		Extraction 31 months after harvest	
	mg/kg	% TRR	mg/kg	% TRR
PCTA	0.008	2.3	--	--
M1 & M2 (C) [a]	0.017	4.7	0.016	5.5
Total identified	0.025	7.0	0.016	5.5
D	0.070	19.6	0.122	42.7
E	0.023	6.5	--	--
F	0.012	3.3	--	--
G	0.015	4.2	--	--
H	0.016	4.5	--	--
PES	0.189	52.6	0.093	32.6
TOTAL	0.359	100.0	0.253	88.5

Notes:

[a] M1 = TCNP-MalCys; M2 = PCP-MalCys.

Potato

Study 1 *(Parkins, 1990, 900-RES-005)*

Potato seed pieces (variety unknown) were planted outdoor into the soil treated with ^{14}C-quintozene by pre-plant incorporation at a rate of 21.1 kg ai/ha.

Potato tubers were harvested at early maturity (11 weeks after planting) and brushed to remove adhering soil. Samples were immediately frozen for shipment and storage until analysis. Frozen samples were thawed to room temperature just prior to analysis. Subsamples were rinsed with warm tap water, dried with paper towels, and cut into quarters. Most of the quarters were separated into peel and flesh fractions while the remaining part was used as the whole potato fraction. The samples were blended with an equal weight of dry ice. The dry ice was allowed to sublime, and aliquots of blended sample were taken for combustion.

Samples were extracted with 80 percent methanol. The methanol was evaporated, and the remainder was adjusted to pH 5.5, and partitioned with chloroform. The spent aqueous phase was then adjusted to pH 2.0 and partitioned with ethyl ether.

Unextracted radioactivity in potato peels was released by heating in anhydrous methanolic HCl for methanolysis at 60 °C for up to two hours according to the method of Tang and Crone (Analytical Biochemistry, Vol. 182 (2), pages 289–294, 1989). The peel hydrolysates were dried by rotary evaporation at 30 °C. The residues were resolubilized in water, partially cleaned up by C18 solid phase extraction and eluted with acetonitrile and methanol. These eluates were analysed by HPLC by co-chromatography with reference standards.

The identification of metabolites focused on the peel, since the highest level of radioactivity was detected in the peel. The aqueous methanol, chloroform and ethyl ether fractions were cleaned up by TLC and HPLC (C18 reverse phase column) followed by mass spectrometric identification. The aqueous phase received additional clean-up on an XAD2 column. Samples were introduced into the mass spectrometer by direct exposure probe utilizing positive and negative chemical ionization with ammonia, methane and isobutane as reagent gases, by thermos-spray utilizing ammonium acetate ionization, or by GC.

TRR obtained by combustion were 2.39 mg/kg in whole potatoes, 11.26 mg/kg in potato peel, and 0.76 mg/kg in potato flesh. Analysis of the chloroform, ethyl ether and water fractions revealed that in potato peel, 38 percent of the total radioactivity (sum of TRRs in fractions and PES) was soluble in chloroform, 11 percent in ethyl ether and 10 percent in water with 40 percent remained in the PES. (Table 10). There is a marked difference in the TRR obtained by combustion versus that obtained by summing up the TRR values in the extracts and PES. This may be because of quenching during LSC. For further calculation of percent TRR, the values obtained as the sum of fractions and PES are used.

Table 10 Radioactive residues in combusted potato and in extracts of potato after soil treatment by quintozene at 21.1 kg ai/ha before planting

Sample	TRR [a] by combustion mg eq/kg	TRR in fractions and PES (mg eq/kg)				
		Chloroform	Ethyl ether	Water	PES	Total
Peel	11.26	6.39	1.83	1.80	6.79	16.81
Flesh	0.76	0.09	0.07	0.16	0.09	0.41
Whole potato	2.39	0.12	0.15	0.23	0.13	0.63

Notes:

[a] Before extraction.

TLC of the chloroform fraction from the peel sample separated into three radioactive bands, each of which was recovered from the plates and further fractionated by reversed phase HPLC. Chloroform soluble metabolites were identified by reconstructed ion chromatography (RIC) and mass spectrometry as PCTA, TCP, unchanged parent, PCA, TCNP and NOHPCA. The first five compounds were found only or mostly in the chloroform fractions while NOHPCA was found in chloroform, ethyl ether and water fractions at almost equal amounts.

TLC of the ethyl ether fraction from the peel sample separated five radioactive bands. The three bands comprising 91 percent of the radioactivity present on the TLC plate were further resolved by HPLC into eight distinct peaks. Potato callus tissue was treated with ^{14}C-quintozene to generate-sufficient material for identification. Ethyl ether soluble material from the callus tissue yielded metabolite peaks with retention times similar to those from the peel fractions. Based on mass spectrometric fragmentation patterns, the following compounds were tentatively identified: PCP-Cys, PCTP-X, PCTP-Gly, PCP-glycoside (PCP-Gly), PCTP-MalCys, a gamma-glutamylcysteine and a glycoside conjugate. The exogenous moieties were identified as PCTP and PCP but which exogenous and endogenous moieties were connected was not identified. However, the likely products are PCTP gamma-glutamylcysteine (PCTP-GluCys) and PCP-Gly (found in a different fraction from traction of the PCP-Gly mentioned above).

TLC of the water extract isolated three radioactive bands which were further separated by HPLC. Identification of radioactive material in the peaks was not confirmed by mass spectrometry, but identities were assumed to be the same as similar elution peaks from chloroform and ethyl ether soluble fractions.

The radioactivity in whole potato was extracted and fractionated as described for peel; the chloroform, ethyl ether, and aqueous extracts were examined by HPLC. The chloroform and ethyl ether soluble fractions each contained four peaks. Tentative identification, based on identifications in the peel, are: NOHPCA, PCA, PCTA, PCTP-MalCys, PCTP-Cys, an artefact and an unknown. The water-soluble fraction yielded two HPLC peaks: PCT-Cys (possibly misidentified) and NOHPCA.

Table 11 Metabolites in the chloroform, ethyl ether and water extracts of potato peel and whole potato following soil application with quintozene at 21.1 kg ai/ha before planting

Component/region	Potato peel		Whole potato	
	mg/kg [a]	% TRR [b]	mg/kg [c]	% TRR [d]
Quintozene	4.10	24.4	n.d.	n.d.
PCTA	0.49	2.9	0.06	9.5
TCP	1.00	5.9	n.d.	n.d.
PCA	3.07	18.3	0.04	6.3
TCNP	0.60	3.6	n.d.	n.d.
NOHPCA	0.30	1.8	0.01	1.6
PCTP-Cys	1.43	8.5	0.11	17.5
PCTPx	0.47	2.8	n.d.	n.d.
PCTP-Gly	0.61	3.6	n.d.	n.d.
PCP-Gly	0.07	0.4	n.d.	n.d.
PCTP-MalCys	0.62	3.7	0.20	31.7
PCTP-GluCys	0.09	0.5	n.d.	n.d.
Total identified	12.85	76.4	0.42	66.6
PES [c]	6.79	40.4	0.13	20.6
Unexplained [c,d]	-2.83	-16.8	0.08	12.8
Total	16.81	100	0.63	100

Notes:

n.d.: Not detected (LOD calculated to be 0.0062 mg eq/kg).

[a] Sum of the concentrations in chloroform, ethyl ether and water extracts.

[b] Based on the sum of TRR of extracts and PES as quintozene.

[c] Expressed as quintozene.

[d] Calculated by subtracting (radioactivity in PES) and (total identified radioactivity) from the total radioactivity.

Hydrolysis of the PES from potato peel (6.79 mg eq/kg) was reported to release 71 percent of the radioactivity and the released metabolites were purified by passing them through a reverse phase solid phase extraction cartridge and eluting them with acetonitrile and methanol. The eluates were analysed by HPLC with co-chromatography with known reference standards. The peaks matched those of PCA, PCP, and TCP (trace amount).

***Study 2**. (Premkumar and Brown, 1992, 900-RES-004)*

Potatoes (variety Centennial) were grown in outdoor plot in California, United States, in sandy loam soil treated pre-plant with a single application of ^{14}C-quintozene at 30.4 kg ai/ha and incorporated into a depth of 3 inches (7.6 cm). Duplicate samples were harvested from the treated plot. The aerial parts of potato (foliage) were harvested at a 92 DAT (early stage). Aerial parts and tubers were harvested separately at 122 DAT (early stage) and 154 DAT (mature stage). Samples were frozen immediately after sampling and shipped frozen to the analytical laboratory.

Samples were homogenized to a powder-like consistency with dry ice and stored at -20 °C until analysis. Homogenized samples were analysed for TRR by combustion/LSC. The TRR obtained by combustion/LSC of homogenized sample aliquots are shown in Table 10. The immature tuber sample (122 DAT) contained the highest TRR (11.3 mg eq/kg) while the TRR decreased drastically to 1.37 mg eq/kg at the maturity (154 DAT) as the tubers grew.

Table 12 Radioactive residues in potato foliage and tubers after soil treatment by quintozene at 30.4 kg ai/ha before planting

Sample	DAT	TRR (mg eq/kg) [a]	
		1st combustion	2nd combustion
Potato foliage (immature)	92	1.13	--
	122	5.74	7.07
Potato foliage (mature)	154	4.27	4.90
Potato tuber (immature)	122	11.25	--
Potato tuber (mature)	154	1.37	--
Soil (0–15 cm) [b]	2 h	3.74 (dw)	--
Soil (0–15 cm)	3	(dw)	--

Notes:

[a] Corrected for matrix and oxidizer recovery.

[b] Soil core was damaged during shipping, which was the reason for obtaining another soil sample at 3 DAT.

The potato matrices (foliage and tubers) were first extracted with hexane followed by methanol. The methanol extract and the PES from potato foliage and immature tubers were individually treated for 18 hours with 6 mol/L HCl at 120 °C in a closed tube in the presence of nitrogen. However, this attempt to release radioactivity was unsuccessful with only 21.7 percent of TRR in methanol extract and 5.4 percent of TRR in the PES were partitioned in dichloromethane with significant loss of radioactivity.

The extractabilities into the hexane and methanol extracts were comparable for each matrix. Extractability of the radioactivity in potato foliage and tubers with hexane and methanol was 53–77 percent TRR. The ratio of non-polar metabolites (radioactivity in hexane extracts) was low between 10–29 percent TRR. PES contained 23–48 percent TRR.

Table 13 Extraction of radioactive residues in immature and mature potato samples, after harvest and after about 2-years frozen storage

Potato sample	DAT	Extraction timing months after harvest	Hexane extract		Methanol extract		Total		PES	
			mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR
Foliage	122	4.8 [a]	1.16	14.6	3.32	41.8	4.48	56.4	3.46	43.6
Foliage	154	4.4 [b]	0.48	9.8	2.50	51.0	2.98	60.8	1.92	39.2
		24.0 [b]	1.01	20.6	1.68	34.3	2.69	54.9	2.21	45.1
		26.4 [b,f]	0.50	10.2	2.95	60.1	3.45	70.3	1.46	29.7
Tuber	122	2.9 [c]	2.07	11.5	7.37	41.0	9.44	52.5	8.54	47.5
		25.3 [d]	1.05	23.2	2.08	45.9	3.13	69.1	1.40	30.9
Tuber	154	24.1 [e]	0.27	27.5	0.27	28.0	0.54	55.5	0.28	28.9
		26.1 [e, f]	0.27	28.6	0.47	48.5	0.74	76.9	0.22	23.1

Notes:

[a] TRR, 7.95 mg eq/kg.

[b] TRR, 4.90 mg eq/kg.

[c] TRR, 17.97 mg eq/kg.

[d] TRR, 4.53 mg eq/kg.

[e] TRR, 0.962 mg eq/kg.

[f] Soxhlet extraction.

The hexane and methanol extracts were profiled by TLC and HPLC-UV.

In the 154 DAT potato foliage sample, only 6.0 percent or 12 percent TRR were identified and they comprised of four compounds: PCA, PCTA and the malonylcysteine conjugates of PCP and TCNP. None of these metabolites accounted for >10 percent TRR but found above 0.01 mg eq/kg. Three polar peak regions could not be identified while region D contained up to 48.1 percent TRR corresponding to 2.4 mg eq/kg. Parent quintozene was not detected.

Table 14 Metabolites in the hexane and methanol extracts of 154 DAT potato foliage sample following soil application with quintozene at 30.4 kg ai/ha before planting

Component /Region	Extraction 24.0 months after harvest [b]						Extraction 26.4 months after harvest [b]					
	Hexane		Methanol		Total		Hexane		Methanol		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
PCA	0.064	1.3	--	--	0.064	1.3	0.206	4.2	--	--	0.206	4.2
PCTA	--	--	0.020	0.4	0.020	0.4	--	--	--	--	--	--
M1 & M [a]	0.039	0.8	0.172	3.5	0.211	4.3	0.049	1.0	0.319	6.5	0.368	7.5
Total identified					0.295	6.0	Total identified				0.574	11.7
D	0.118	2.4	0.628	12.8	0.745	15.2	0.280	5.7	2.079	42.4	2.359	48.1
G	--	--	--	--	--	--	--	--	0.294	6.0	0.294	6.0
H	--	--	0.334	6.8	0.334	6.8	--	--	0.250	5.1	0.250	5.1
Unexplained	0.790	16.1	0.535	10.9	1.32	27.0	--	--	--	--	--	--
PES					2.212	45.1	PES				1.456	29.7
TOTAL					4.909	100.1	TOTAL				4.933	100.6

Notes:

[a] M1 = TCNP-MalCys; M2 = PCP-MalCys.

[b] TRR, 4.90 mg eq/kg.

As the 122 DAT potato tubers contained higher TRR than the 155 tubers, the hexane extract was used to validate the GC analytical method. Portions of the tuber samples were extracted 2.9 months after the harvest with ethyl acetate and the extract was cleaned up using Florisil column. The eluate from the Florisil column was concentrated and reconstituted with iso-octane for GC and HPLC. The results of analysis of iso-octane were shown for the earlier extraction in the following table.

In the 122 DAT potato tuber sample, nine compounds were identified: parent and the metabolites PCA, PCTA, PCTP, PB, 4-TCNB, 6-TCNB and the malonylcysteine conjugates of PCP and TCNP and the impurity HCB. Seven polar peak regions were not identified although region D accounted for up to 25.3 percent TRR and up to 2.44 mg eq/kg. Iso-Octane fraction showed similar metabolite pattern of polar metabolites as the hexane extract, but the Florisil clean-up apparently removed M1+M2 (region C) and region D. The levels of total identified were 23 percent and 38 percent of TRR in the two extractions.

Table 15 Metabolites in the iso-octane/hexane and methanol fractions of 122 DAT potato tuber sample following soil application with quintozene at 30.4 kg ai/ha before planting

Component /Region	Extraction 2.9 months after harvest (TRR: 17.970 mg eq/kg)						Extraction 25.3 months after harvest (TRR: 4.534 mg eq/kg)					
	iso-Octane		Methanol		Total		Hexane		Methanol		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Quintozene	0.061	0.3	--	--	0.061	0.3	0.023	0.5	--	--	0.023	0.5
PCA	0.689	3.8	0.395	2.2	1.084	6.0	0.630	13.9	0.045	1.0	0.676	14.9
PCTA	0.338	1.9	0.324	1.8	0.661	3.7	0.122	2.7	--	--	0.122	2.7
PB	0.086	0.5	0.395	2.2	0.481	2.7	0.086	1.9	0.512	11.3	0.599	13.2
HCB	0.010	0.1	--	--	0.010	0.1	--	--	--	--	--	--
4-TCNB	0.022	0.1	--	--	0.022	0.1	--	--	--	--	--	--
6-TCNB	0.003	0.0	--	--	0.003	0.0	--	--	--	--	--	--
M1&M2 [a]	--	--	1.815	10.1	1.815	10.1	0.064	1.4	0.254	5.6	0.317	7.0
Total identified					4.137	23.0	Total identified				1.737	38.3
B	--	--	0.288	1.6	0.288	1.6	--	--	--	--	--	--
D	--	--	2.444	13.6	2.444	13.6	0.127	2.8	1.020	22.5	1.147	25.3
E	--	--	0.341	1.9	0.341	1.9	--	--	--	--	--	--
F	--	--	0.073	0.4	0.072	0.4	--	--	--	--	--	--
G	--	--	0.108	0.6	0.108	0.6	--	--	--	--	--	--
H	--	--	1.186	6.6	1.186	6.6	--	--	0.240	5.3	0.240	5.3
Unexplained	--	--	0.665	3.7	0.665	3.7	--	--	--	--	--	--
PES					8.536	47.5	PES				1.400	30.9
TOTAL					17.776	98.9	TOTAL				4.524	99.8

Notes:

[a] M1=TCNP-MalCys, M2=PCP-MalCys

In the 154 DAT potato tuber sample, level of identification was low (21 percent TRR). Five compounds were identified: the metabolites PCA, PCTA, PB and the malonylcysteine conjugates of PCP and TCNP. Parent compound was not detected. Three peak regions with TRR up to 26.3 percent were not identified.

Table 16 Metabolites in the hexane and methanol extracts of 154 DAT potato tuber sample extracted 26.1 months after harvest [a], following soil application with quintozene at 30.4 kg ai/ha before planting

Component/region	Hexane ext.		Methanol ext.		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
PCA	0.091	9.5	--	--	0.091	9.5
PCTA	0.019	2.0	--	--	0.019	2.0

Component/region	Hexane ext.		Methanol ext.		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
PB	0.045	4.7	--	--	0.045	4.7
M1 & M2 (C)[b]	0.018	1.9	0.032	3.3	0.050	5.2
Total identified					0.205	21.4
D	0.042	4.4	0.162	16.8	0.204	21.2
H	--	--	0.253	26.3	0.253	26.3
Unknown	--	--	0.019	2.0	0.019	2.0
Unexplained	0.058	6.0	--	--	0.058	6.0
PES					0.222	23.1
TOTAL					0.962	100.0

Notes:

[a] TRR, 0.962 mg eq/kg.

[b] M1 =TCNP-MalCys, M2 = PCP-MalCys.

Study 3 *(Fang & Mertz, 1999, 900-RES-043)*

To better identify the metabolites of quintozene in potato, a new study was conducted. Potato plants (variety Superior) were grown outdoors in sandy loam soil. The soil was treated with ^{14}C-quintozene applied by pre-plant incorporation at a rate of 22.4 kg ai/ha or 67.3 kg ai/ha, and, on the same day, seed potatoes were planted in the treated soil in half wine barrels at a depth of 10 cm and covered by treated soil. As per agricultural practice, when the plants reached a height of 10–13 cm, they were "hilled" with treated soil, which was repeated when the plants had grown another 10–13 cm. The potato tuber, stem and foliage were harvested 95 days after treatment/planting when the plants had undergone renascence. The samples were stored frozen until analysis.

The frozen potatoes were washed with water to remove adhering soil. The samples were blended with an equal amount of dry ice and aliquots were taken for combustion. Potato tubers were extracted first with methanol followed by 80 percent aqueous methanol. The combined extracts were partitioned with hexane to obtain the hexane fraction. The residual solids from methanol and 80 percent methanol extraction were further extracted (exhaustive extraction) with 80 percent methanol, 50 percent aqueous methanol and then water. These extracts were combined with the aqueous layer from hexane partition. From the combined layer/extracts, methanol was removed and partitioned with ethyl ether to get ether-I fraction. The aqueous layer was adjusted to pH 3 and partitioned with ethyl ether to obtain ether-II fraction and final aqueous fraction. The solids remaining after the second set of extraction with aqueous methanol and water were regarded as PES.

The PES was subjected to enzymatic treatment with α-amylase, cellulase, iso-maltase, maltase, pectinase, and protease, each at their optimum pH. Then, the PES from the enzymatic treatment was subjected to acid treatment (1 mol/L acetyl chloride in methanol) or base hydrolysis (6 mol/L NaOH or KOH) for 2 hours at elevated temperature.

TRR in whole potatoes obtained by combustion were 1.12 mg eq/kg (22.4 kg ai/ha) and 3.54 mg eq/kg (67.3 kg ai/ha).

The majority of the radioactive residue (87–90 percent TRR) was extracted with methanol and methanol/water followed by exhaustive extraction with methanol/water mixtures and water. In the PES, low levels of radioactivity remained unextracted: 6.74 percent TRR (22.4 kg ai/ha) and 9.92 percent TRR (67.3 kg ai/ha).

The TRR present in whole potatoes and the fractions (hexane, ether-I, ether-II aqueous fractions and PES) obtained by extraction and partition are shown in Table 17.

Table 17 TRR and radioactivity in the hexane, ether-I, ether-II and aqueous fractions and PES of 95 DAT whole potato following soil application with quintozene before planting

Fraction	22.4 kg ai/ha		67.3 kg ai/ha	
	mg eq/kg	% TRR	mg eq/kg	% TRR
Whole potato tubers	1.12	100	3.54	100
Solvent extracted	1.05	93.5	3.27	92.3
Hexane fraction	0.18	16.4	0.40	11.4
Ether-I fraction	0.09	8.07	0.27	7.60
Ether-II fraction	0.29	25.5	1.03	29.1
Aqueous fraction	0.45	39.9	1.37	38.6
Total of fractions	1.01	89.8	3.07	86.7
PES	0.08	6.74	0.35	9.92

The metabolites in the fractions in the table above were identified by HPLC-MS, HPLC-MS/MS, GC-MS and GC-MS/MS. Parent and 48 metabolites were identified as shown in Table 18.

The metabolite profile was similar for both application rates, and the identified components together accounted for 60.1–64.1 percent of the TRR. Although no single component accounted for >10 percent of the TRR, except for PCP-MalCys from 67.3 kg ai/ha application, approximately half of the identified radioactivity (27–30 percent TRR) was comprised of five components. The two most abundant residues were parent (7.5–8.0 percent TRR) and PCP-MalCys (9.8–10 percent TRR). Another three metabolites were: PCA (2.4–4.3 percent TRR), PCTA (2.7–3.5 percent TRR), and AM TCB sulfonic acid (3.9–4.8 percent TRR). The rest 44 metabolites each accounted for ≤ 2.8 percent of the TRR. PB was detected at 1.0–1.9 percent of the TRR (≤0.014 mg/kg).

In the hexane fractions, the major component was parent quintozene. Eleven non-polar metabolites were identified with 12 other metabolites at levels too low to quantify. Impurity HCB was below the LOD of 0.001 mg/kg.

The ether-I fraction contained 11 non-polar metabolites. The ether-II fraction contained PCP-MalCys accounting for 10 percent TRR and eight structurally related compounds.

The aqueous fraction contained 32 metabolites. The most abundant metabolite was AMTCB Sulfonic acid accounting for about 9 percent and 6 percent TRR in this fraction.

The PES were treated with variety of enzymes. Protease released about 40 percent of the TRR in the PES, and the molecular sieves indicated that much of the radioactivity was associated with substances with molecular weight higher than 3000. The enzymes used to hydrolyse carbohydrate released minimal amount of radioactivity.

Table 18 Metabolites in the whole potato tuber following soil application with quintozene before planting

Compound	22.4 kg ai/ha		67.3 kg ai/ha	
	mg/kg	% TRR	mg/kg	% TRR
Quintozene	0.084	7.51	0.28	7.95
PCA	0.047	4.26	0.085	2.42
PCAN	0.008	0.70	0.014	0.40
PB	0.021	1.89	0.035	1.01
PCTA	0.039	3.48	0.096	2.71
NOH PCA	0.017	1.52	0.028	0.78
PCP-GluCys	0.009	0.84	0.042	1.17
PCP-GSH	0.014	1.22	0.056	1.58
PCP-MalCys	0.112	9.80	0.357	10.09
PCP-MalCys ester	0.006	0.54	0.087	2.46

Compound	22.4 kg ai/ha		67.3 kg ai/ha	
	mg/kg	% TRR	mg/kg	% TRR
TCA sulfoxide	0.001	0.12	0.003	0.07
TCA sulfoxide (isomer)	<0.001	0.01	0.001	0.02
TC-MET-A sulfamate	<0.001	0.02	0.004	0.12
2,3,5,6-TCNB	0.001	0.13	0.006	0.18
TCNB (isomer1)	Minor metabolite in the hexane fraction [a]			
TCNB (isomer2)	Minor metabolite in the hexane fraction [a]			
TCNB sulfonic acid	0.001	0.13	0.010	0.27
TCNB sulfonic acid (isomer)	Minor metabolite in the hexane fraction [a]			
TCNTA	<0.001	0.01	0.003	0.09
TCNTA (isomer1)	Minor metabolite in the hexane fraction [a]			
TCNTA (isomer2)	Minor metabolite in the hexane fraction [a]			
TCP sulfoxide	0.008	0.70	0.012	0.34
TCTP sulfoxide	0.009	0.78	0.029	0.81
TCTP sulfonic acid	Minor metabolite in the hexane fraction [a]			
AM TCA	0.023	2.07	0.050	1.40
AM TCA (isomer)	0.008	0.72	0.023	0.66
AM TCA sulfamate	0.005	0.48	0.016	0.44
AM TCB sulfonic acid	0.054	4.75	0.136	3.85
OH TCA	0.005	0.43	0.011	0.31
NOHAM TCA	Minor metabolite in the hexane fraction [a]			
NOHAM TCP	0.004	0.37	0.006	0.19
NOHAM TCTA	0.006	0.54	0.006	0.18
NOHAM TCTA (isomer)	0.001	0.12	0.001	0.04
TC-MES-P-GSH	0.023	2.06	0.044	1.25
TC-MES-P-MalCys	0.023	2.03	0.081	2.30
TCNP-GSH	0.011	0.95	0.069	1.94
TCNP-MalCys	0.014	1.19	0.055	1.55
TCP-diGSH	0.022	1.99	0.054	1.51
TCP-dithioacetate	0.017	1.52	0.071	2.00
TCP-GluCys-Cys	0.025	2.25	0.055	1.54
AC TCP-Cys-CysHOG	0.012	1.07	0.042	1.19
RCNA sulfoxide	Minor metabolite in the hexane fraction [a]			
NOH RC-diMET-A	Minor metabolite in the hexane fraction [a]			
NOHAM RC-OME-A	Minor metabolite in the hexane fraction [a]			
RCA-GluCys-Cys	0.010	0.87	0.032	0.89
RCNP-MalCys-thioacetate	0.023	2.04	0.061	1.72
RCTA-GluCys-Cys	0.019	1.69	0.057	1.60
diAC RCAN-GSH-Cys	0.017	1.47	0.041	1.15
diAC RCTA-diGluCys	0.021	1.87	0.071	2.00

Notes:

[a] 12 minor metabolites in the hexane fraction were identified by GC-MS accounting for 5.81 percent TRR (22.4 kg ai/ha) and 3.77 percent TRR (67.3 kg ai/ha) of the radioactivity in the hexane fraction.

Peanuts

***Study 1** (McManus, 1990, 900-RES-125)*

Peanut plants (variety unknown) were grown in soil that had been treated with ^{14}C-quintozene at a rate equivalent to 420 kg ai/ha for incorporation to a depth of 15 cm. Whether plants were grown outdoor or indoor was not described in the study report.

Peanut whole plants were harvested 21 weeks after planting/treatment and separated into vines, shells, nuts and roots. The vines were air dried. The roots and peanuts were washed with dilute detergent and rinsed with deionized water. The peanut shell and nutmeat were separated and blotted dry, along with the roots, with a paper towel and allowed to air-dry overnight. The samples were then homogenized in a blender with crushed dry ice and stored in plastic bags in the freezer. Subsamples were combusted for determination of TRR.

Sample aliquots of the ground roots, vines, shells and nutmeat were extracted by high-speed homogenization with methanol/water (80:20; v/v) followed by acetone. The extracts were concentrated under reduced pressure prior to clean-up by C-18 solid phase extraction. Radioactive residues in the cleaned-up extracts were analysed by reverse phase HPLC. The extracted plant materials were combusted for TRR analysis. The methanol/water extracts were applied to preparative TLC. The radioactive bands scraped from the plates were extracted with methanol and radioactive metabolites were isolated by preparative HPLC. Metabolites collected multiple times were combined and concentrated under nitrogen prior to mass spectrometry.

The PES were subjected to combustion/LSC. The PES was incubated at 37 °C for 48 hours in 0.5 mL methanolic-HCl (1 mol/L acetyl chloride in methanol).

The highest TRR levels were found in the roots at 1521 mg eq/kg. The vines, shells and nutmeat had lower residues ranging from 42 mg eq/kg in the vines to 5.2 mg eq/kg in the nutmeat. Extraction with methanol/water removed 64–88 percent of the radioactive residue. The PES retained radioactivity from 454 mg eq/kg in the roots to 0.94 mg eq/kg in the nutmeat. An average of more than 90 percent of these residues in the shells, vines and nutmeat was liberated by hydrolysis with methanolic HCl.

Table 19 Radioactive residues in peanut root, vine, shell and nutmeat samples following soil treatment with quintozene at 420 kg ai/ha before planting

Peanut sample	Total radioactivity		Extracted by solvents		PES	
	mg eq/kg	% TRR [a]	mg eq/kg	% TRR [b]	mg eq/kg	% TRR [b]
Roots	1521	28.0	1020	67.1	454	29.9
Vines	42.3	46.1	47.4	>100	14.8	35.0
Shells	128	23.9	121	94.1	4.46	3.5
Nutmeat	5.16	1.93	6.1	>100	0.94	18.3
	Total	99.9				

Notes:

[a] percent of TRR in whole plant.

[b] percent of TRR in respective sample.

The methanol/water extracts were analysed for characterization/identification of residues. The extracts contained seven metabolites. Two major metabolites were identified as PCP-MalCys and TCA, which were found in the roots (33 percent and 25 percent TRR, respectively), vines (20 percent and 14 percent TRR, respectively) and shells (42 percent and 22 percent TRR, respectively). PCP-MalCys was also found in nutmeat at 6.7 percent TRR (0.26 mg eq/kg). Of the five other metabolites, one was identified as MTCP-TAA (*S*-[(methylthio)tetrachlorophenyl]-2-thioacetic acid) which accounted for 14 percent TRR in vines but 8.7 percent TRR in roots and 7.4 percent TRR in shells. The other four minor metabolites (Ia, Ib, II and Vb) were also found in roots, vines, shells and nutmeat, but levels were too low for identification.

Table 20 Metabolites in the peanut samples following soil treatment with whole potato tuber following soil application with quintozene at 420 kg ai/ha before planting

Compound	Roots		Vines		Shells		Nutmeat	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Unknown Ia	n.d.	--	n.d.	--	n.d.	--	n.d.	--
Unknown Ib	48	5	1.1	3.3	n.d.	--	n.d.	--
Unknown II	n.d.	--	n.d.	--	n.d.	--	n.d.	--
PCP-MalCys	321.5	32.9	6.9	20.3	47.3	41.6	0.26	6.7
TCA	240	24.6	4.9	14.4	25.5	22.4	n.d.	--
MTCP-TAA	85.0	8.7	4.7	13.8	3.4	7.4	n.d.	--
Unknown Vb	135	13.8	7.5	22	12.6	11.1	0.74	19

Notes:

n.d.: Not detected.

Study 2 *(Premkumar & Brown, 1992; 900-RES-002)*

Peanuts (variety Jumbo Virginia) were grown in outdoor plots in California, United States. A single application of ^{14}C-quintozene at 37.9 kg ai/ha was made to sandy loam soil for incorporation into top 7.6 cm of soil. Peanut was planted into the treated soil on the same day of application.

The aerial parts of peanuts (foliage) were harvested at 92 DAT at an early stage. The mature sample was harvested at 185 DAT and separated into vines (foliage), hulls and nut meat. Samples were stored frozen immediately after sampling and shipped on dry ice to the analytical laboratory and stored frozen until analysis in two different freezers.

Samples were homogenized to a powder-like consistency, when possible, under assistance of dry ice and stored at -20 °C until analysis. Homogenized samples were analysed for TRR by combustion/LSC and the results are presented in Table 21.

Table 21 Radioactive residues in peanut foliage, hulls and nutmeat following soil treatment by quintozene at 37.9 kg ai/ha before planting

Peanut sample	DAT	TRR (mg eq/kg) [a]
Foliage	92	3.50
	154	3.97
Hulls	185	26.3
Nutmeat	185	2.00
Soil (0–15 cm)	3	5.94 (dw) [b]

Notes:

[a] Corrected for matrix and oxidizer recovery.

[b] Mean normalized value.

The peanut vines, hulls and nutmeat were first extracted with hexane followed by methanol. The extractions of vines, hulls and nutmeat were carried out more than once using the same extraction solvents at different timing. For peanut vine sample, the hexane extract was concentrated under nitrogen and the resulting hexane fraction was used for GC and HPLC analysis while the resulting aqueous fraction was combined with the methanol extract. The combined fraction was concentrated under nitrogen to obtain the methanol fraction. The remaining solids after methanol extraction were regarded as PES.

The extractabilities into hexane and then into methanol were comparable for each matrix. Extractability of the radioactivity in peanut foliage, hulls and nutmeat with hexane and methanol was 50–87 percent TRR and 13–50 percent TRR remained unextracted in the PES.

The soil was extracted with acetone which solubilized 96.7 percent TRR in soil.

Table 22 Extraction of radioactive residues in mature peanut vine, hulls and nutmeat following soil treatment by quintozene at 37.9 kg ai/ha before planting

Peanut sample	TRR	Hexane extract		Methanol ext.		Total		PES	
		mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR
Vines	4.10	0.594	14.5	1.64	40.1	2.24	54.6	1.86	45.4
Hulls	24.3	1.61	6.6	10.6	43.7	12.2	50.3	12.1	49.7
Nutmeat	2.10	1.19	56.6	0.643	30.6	1.83	87.2	0.269	12.8

The hexane and methanol extracts are profiled by TLC, HPLC-UV and GC-ECD.

In the 154 DAT vines, seven compounds were identified: parent and the metabolites PCA, PCTA, PB, 4-TCNB and the malonylcysteine conjugates of PCP and TCNP. Three peak regions (B, D and H), each of which accounted for less than 10 percent TRR, could not be identified.

Table 23 Metabolites in the hexane and methanol extracts of mature peanut vines following soil application with quintozene at 37.9 kg ai/ha before planting

Component/region	Hexane ext.		Methanol ext.		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Quintozene	0.080	2.0	0.030	0.7	0.110	2.7
PCA	0.074	1.8	--	--	0.074	1.8
PCTA	0.029	0.7	0.067	1.6	0.096	2.3
PB	0.036	0.9	--	--	0.036	0.9
4-TCNB	0.007	0.2	--	--	0.007	0.2
M1 & M2 (C) [a]	0.212	5.2	0.878	21.4	1.090	26.6
Total identified	0.357	8.8	0.945	23.0	1.303	31.8
B	--	--	0.039	1.0	0.039	1.0
D	--	--	0.402	9.8	0.402	9.8
H	--	--	0.270	6.6	0.270	6.6
				PES	1.861	45.4
				TOTAL	3.984	97.2

Notes:

[a] M1 = TCNP-MalCys and M2 = PCP-MalCys.

In the 185 DAT hulls, eight compounds were identified: parent and the metabolites PCA, PCTA, PB, 4-TCNB, 6-TCNB and the malonylcysteine conjugates of PCP and TCNP. One peak region D with TRR <10 percent was not identified.

Table 24 Metabolites in the hexane and methanol extracts of mature peanut hulls following soil application with quintozene at 37.9 kg ai/ha before planting

Component/region	Hexane ext.		Methanol ext.		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Quintozene	0.323	1.3	--	--	0.323	1.3
PCA	0.966	4.0	--	--	0.966	4.0
PCTA	0.290	1.2	--	--	0.290	1.2

Component/region	Hexane ext.		Methanol ext.		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
PB	0.109	0.4	--	--	0.109	0.4
4-TCNB	0.058	0.2	--	--	0.058	0.2
6-TCNB	0.007	0.03	--	--	0.007	0.03
M1 & M2 (C) [a]	0.413	1.7	7.924	32.6	8.337	34.3
Total identified	2.166	8.83	7.924	32.6	10.090	41.4
D		0.0	1.798	7.4	1.798	7.4
				PES	12.086	49.7
				TOTAL	23.973	98.6

Notes:

[a] M1 = TCNP-MalCys and M2 = PCP-MalCys.

The identification of 185 DAT nutmeat was not straightforward because it was based on three different extractions: third extraction was conducted to determine the distribution of radioactivity into hexane, methanol and PES (quantitative values in the hexane extract were based on the GC analysis from this extraction; fourth extraction and fifth extraction were conducted as the methanol extract from the third extraction did not provide satisfactory HPLC profiles due to low level radioactivity relative to natural products in this extract. The methanol extract from the fourth extraction was concentrated. Water was added to the concentrate which was then acidified and partitioned between diethyl ether and water. As in the process of concentration of the fourth extract 12 percent of the TRR was lost, in the fifth extraction, the methanol extract was partitioned into iso-octane without concentration. The loss of 12 percent was accounted for by the iso-octane fraction. Table 25 includes the values obtained from three different extractions.

In the 185 DAT nutmeat, eight compounds were identified: parent and the metabolites PCA, PCTA, PB, 4-TCNB, 6-TCNB and the malonylcysteine conjugates of PCP and TCNP. Three peak regions with TRR <10 percent were not identified.

Table 25 Metabolites in the hexane and methanol extracts of mature peanut meat following soil application with quintozene at 37.9 kg ai/ha before planting

Compoun[d] Region	**Hexane extract [b]**		**Methanol Extract**						**Total**	
			***iso*-Octane [c]**		**Ether [d]**		**Aqueous [d]**			
	mg/kg	**% TRR**	**mg/kg**	**percent TRR**	**mg/kg**	**percent TRR**	**mg/kg**	**percent TRR**	**mg/kg**	**percent TRR**
Quintozene	0.087	4.1	0.010	0.5	0.023	1.1	--	--	0.119	5.7
PCA	0.308	14.7	0.052	2.5	--	--	--	--	0.360	17.1
PCTA	0.095	4.5	0.104	4.9	--	--	--	--	0.199	9.5
PB	0.363	17.3	--	--	--	--	--	--	0.363	17.3
4-TCNB	0.004	0.21	--	--	--	--	--	--	0.004	0.21
6-TCNB	0.077	3.6	--	--	--	--	--	--	0.077	3.6
M1&M2(C) [a]	0.037	1.8	--	--	0.202	9.6	0.034	1.6	0.273	13.0
Total identified	0.971	46.2	0.166	7.9	0.225	10.7	0.034	1.6	1.296	66.4
D	--	--	--	--	0.102	4.8	0.071	3.4	0.172	8.2
F	--	--	--	--	0.004	0.18	--	--	0.004	0.18
H	--	--	--	--	0.004	0.17	0.039	1.8	0.042	2.0
PES									0.269	12.8
TOTAL									1.883	89.6

Notes:

[a] M1 = TCNP-MalCys and M2 = PCP-MalCys.

[b] From the third extraction.

[c] From the fifth extraction.

[d] From the fourth extraction.

The methanol extracts and the PES from peanuts foliage and nutmeat were hydrolysed by treatment for 18 hours (foliage) or 3 hours (nutmeat) with 6 mol/L HCl at 110 °C in a closed tube in the presence of nitrogen. The majority of radioactivity in the PES remained unextracted after HCl hydrolysis. It was not possible to solubilize significant amount of radioactivity contained in the PES. From methanol extract of nutmeat, after partitioning into iso-octane or methanol extract of vines, HCl hydrolysis released 60 percent or 42 percent, respectively, of the radioactivity in these extracts but it was not possible to identify the released radioactivity.

Study 3 *(McManus & DeMatteo, 1999; 900-RES-042)*

Peanut plants (variety Florunner) were grown in plastic buckets in a greenhouse, with artificial fluorescent lighting for a 14-h photo period, in Connecticut, United States, under simulated normal agronomic conditions. Two groups of treated plants were employed. One group (T1) was treated by pre-plant soil incorporation with ^{14}C-quintozene at a rate of 16.8 kg ai/ha and, after the treatment, peanut seeds were sown. The second group (T2) was treated with ^{14}C-quintozene in two band applications, each at 5.6 kg ai/ha, during and after pegging time, 68 days and 117 days after planting. Peanut seeds were sown on the same day in the first and second group.

Mature plants (193 DAT for pre-plant soil incorporation, and 76 DALA for band applications) were harvested by cutting the plants just above the soil line and vines, roots and peanuts (nutmeat and shells) were separated. The vines were air dried overnight at room temperature to obtain hay. The roots and peanuts were washed with deionized water. The peanut shells and nutmeat were air dried, along with the roots, overnight in a hood. The samples of hay, root, shell, and nutmeat were homogenized to a fine powder with dry ice. Levels of radioactivity in plant samples were determined by combustion/LSC.

The highest levels of radioactivity were found in the shells (166–211 mg eq/kg). The roots, hay and nutmeats had lower residues ranging from 49–122 mg eq/kg in the roots to 1.7–2.1 mg eq/kg in the nutmeat.

Table 26 Radioactive residues in mature peanut samples after soil treatment by quintozene

Peanut sample	TRR (mg eq/kg)	
	Pre-plant soil incorporation at 16.8 kg ai/ha (T1)	Two banded applications, each at 5.6 kg ai/ha (T2)
Hay (dried vines)	13.0	16.3
Nutmeat	2.14	1.72
Shells	166	211
Roots	122	49.0

Subsamples of homogenized hay, nutmeat, shell, and root were extracted by sonication with methanol/water (20:80; v/v) followed by acetone. After centrifugation, the supernatants were separated. The pellets were exhaustively extracted with each solvent system. All extracts were combined and concentrated by rotary evaporation prior to quantification by LSC. Additionally, hay extracts were partitioned with chloroform and two phases (organic and aqueous) were separately radio-assayed by LSC.

Alternatively, Soxhlet extraction was attempted. Aliquots of homogenized hay were extracted for 6 hours with methanol and the methanol extract was radio-assayed by LSC. Aliquots of homogenized

nutmeat were extracted for 4 hours with hexane to remove oils and then extracted with methanol. The hexane and methanol extracts were radio-assayed by LSC.

The remaining solids of nutmeat and hay were incubated at 60 °C for 24 hours in 5 mL methanolic HCl (1 mol/L acetyl chloride in methanol). Following centrifugation, the supernatant was analysed by LSC.

For extraction of radioactivity in nutmeat, Soxhlet extraction was more efficient than sonication. Soxhlet extraction removed 62–66 percent TRR in the nutmeat. For peanut hay, sonication was more efficient than Soxhlet extraction. Sonication removed 62–63 percent TRR in the hay.

Acid hydrolysis additionally removed 30–43 percent of respective TRR. The radioactivity remaining in the PES after acid hydrolysis ranged from 0.06 mg/kg in the nutmeat to 0.95 mg/kg in the hay.

Table 27 Extraction of radioactive residues in mature peanut nutmeat and hay following pre-plant soil incorporation at 16.8 kg ai/ha (T1) or two banded applications, each at 5.6 kg ai/ha (T2) with quintozene

Sample	Hexane ext.		Methanol ext.		MeOH/HCl hydrolysis		Total		PES [a]	
Treatment	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Nutmeat (extraction by Soxhlet system)										
T1	0.965	45.1	0.447	20.9	0.927	43.3	2.34	109	--	--
T2	0.673	39.1	0.401	23.3	0.588	34.2	1.66	96.6	0.058	3.40
Sample	MeOH/water ext.		Acetone ext.		MeOH/HCl hydrolysis		Total		PES [a]	
Treatment	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Hay (extraction by sonication)										
T1	8.14	62.6	--	--	4.89	37.6	13.0	100	--	--
T2	10.1	61.7	0.466	2.86	4.82	29.6	15.4	94.2	0.952	5.84

Notes:

[a] After acid hydrolysis

Quantitative HPLC analyses of nutmeats and hay extracts from the two band applications (post-emergence) identified 96.6 percent of the TRR in nutmeats and 91.7 percent of the TRR in hay. Metabolite identities were confirmed using GC/ECD, GC/MS and/or LC/MS.

In nutmeats, the major radioactive residue was quintozene, accounting for 96.6 percent of the TRR (1.7 mg/kg), along with trace levels of PCA.

Table 28 Metabolites in the hexane and methanol extracts and after methanolic HCl hydrolysis of mature peanut meat following 2 band applications with quintozene each at 5.6 kg ai/ha

Compound	Hexane ext. + Methanol Ext.		MeOH/HCl hydrolysate		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Quintozene	1.1	62.4	0.59	34.2	1.69	96.6
PCA	Trace		--	--	--	--
Total identified					1.69	96.6
PES					0.06	3.40
TRR					1.72	100

In hay, the main residues included: quintozene (18.8 percent TRR, 3.1 mg/kg), PCTP-MalCys (53.1 percent TRR, 8.65 mg eq/kg), and *N*-malonyl-*S*-(tetrachloroaminophenyl)-cysteine (19.8 percent TRR, 3.23 mg eq/kg). Trace amounts of TCNP-MalCys and TCNP-GSH were also identified.

Table 29 Metabolites in the hexane and methanol extracts and after methanolic HCl hydrolysis of mature peanut hay following 2 band applications with quintozene each at 5.6 kg ai/ha

Compound	MeOH/water ext. (Partitioned with chloroform/water)		MeOH/HCl hydrolysate		Total	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Quintozene	3.06	18.8	--	--	3.06	18.8
PCTP-MalCys	3.83	23.5	4.82	29.6	8.20	53.1
TCNP-MalCys	Trace		--	--		
N-malonyl-S-(tetrachloroaminophenyl)-cysteine	3.23	19.8	--	--	3.23	19.8
TCNP-GSH	Trace		--	--		
Total identified					14.5	91.7
Acetone extract (none identified)					0.47	2.86
PES					0.95	5.84
TRR					16.3	100

While no quantitative information was reported in the study report on metabolites in shell extracts, qualitative information was available. In the peanut shells following soil incorporation treatment, four radioactive metabolites were detected, two principal metabolites and two others in small amounts. Two principal metabolites were quintozene and PCTP-MalCys, and two at smaller amounts were TCNP-MalCys and tetrachloroaminophenyl sulfonic acid.

Treated Seeds (Selman, 1988; 900-RES-058)

Seeds of maize, peas, soya bean, sugar beet and wheat were treated with ^{14}C-quintozene at: 1.7 g ai/kg for sugar beet seeds, 0.72 g ai/kg for soya beans, and about 0.4 g ai/kg for maize, peas and wheat.

The treated seeds were sown and grown in an open-sided greenhouse which allowed exposure to natural sun and weather conditions while eliminating the potential for flooding from rain. Crop matrices were sampled at different growing stages to cover commercially available food and feed commodities. These samples were frozen on the same day of sampling and stored frozen until analysis. Each sample was analysed for radioactivity by combustion/LSC.

There was uptake of radioactivity by all the crops. The highest levels were found in the dry pea vines and soya bean stems at 1.8 and 1.5 mg eq/kg respectively at harvest. The levels in fresh pea vines, sugar beet roots, soya bean hay and wheat forage were 0.57, 0.46, 0.74 and 0.54 mg eq/kg, respectively, and in maize stover and wheat straw 0.02 and 0.06 mg eq/kg, respectively. None of the harvested seeds/grains from maize, wheat, soya bean or peas or other commodities than those mentioned above contained residues above the minimum quantifiable limits.

Table 30 Radioactive residues in commercial commodities following seed treatment of various crop seeds by quintozene

Crop	Commodity	DAT	Sampling to combustion (days)	Average minimum quantifiable limits (mg/kg)	TRR [a] (mg eq/kg)
Maize	Haylage [b]	48	13	0.0747	<0.0747
	Silage	90	15	0.0450	<0.0450
	Grain	125	27	0.0144	<0.0144
	Stover	125	27	0.0183	0.0242
Peas	Fresh Peas [b]	48	13	0.0703	<0.0703

Crop	Commodity	DAT	Sampling to combustion (days)	Average minimum quantifiable limits (mg/kg)	TRR [a] (mg eq/kg)
	Fresh Vines [b]	48	13	0.0815	0.567
	Dry Peas	76	19	0.0167	<0.0167
	Dry Vines	76	19	0.0161	1.81
Soya beans	Hay [b]	48	13	0.0545	0.735
	Beans	102	16	0.0143	<0.0143
	Stem	102	16	0.0151	1.47
Sugar beet	Roots	125	25	0.0551	0.457
	Tops	125	25	0.0636	<0.0636
Wheat	Forage [b]	34	27	0.0879	0.536
	Hay	63	28	0.0782	<0.0782
	Grain	125	27	0.0154	<0.0154
	Straw	125	27	0.0226	0.0558

Notes:

[a] Adjusted for percent dry weight.

[b] Mean of duplicate analysis; for all others, mean of triplicate analysis.

Summary of Plant Metabolism

Metabolism of quintozene in plants after pre-plant soil incorporation application was studied on cabbage, potato and peanut using quintozene uniformly labelled on phenyl ring. In the case of peanut, metabolism following two banded applications to soil at the pegging stage was also investigated. The studies were conducted either outdoor or in greenhouse (one of which allowed sunlight).

In the studies on these crops conducted in 1990 and 1992, intervals between the sample collection and extraction were long, some of them longer than 2 years, pending the development of appropriate analytical method. The storage stability tests conducted during these studies indicated that compounds extracted in non-polar fractions were relatively stable but those extracted in polar fractions might be degraded showing different profiles after long storage. In addition, the identified metabolites and their quantities of the same crop were not consistent between the 1990 study and 1992 study, and none of the compounds in the polar fractions was identified. Therefore, new studies were conducted in 1999 on potato and peanut.

In the newer studies on potato, close to 50 components were identified and quantified including those in the polar fractions showing similar profiles between 22.4 and 67.3 kg ai/ha application rates. About 60–64 percent TRR were identified and approximately about one half of the identified radioactivity (27–30 percent TRR) was comprised of five compounds. PCP-MalCys and its esters together accounted for 10–13 percent TRR. This compound was also found in the old studies on cabbage, potato and peanut. Parent quintozene accounted for 7.5–8.0 percent TRR. PCA and PCTA accounted for 2.4–4.3 percent and 2.7–3.5 percent TRR respectively. AM TCB sulfonic acid accounted for 3.9–4.8 percent TRR. No other metabolites accounted for more than 2.8 percent TRR while the concentrations of many metabolites were higher than 0.01 mg/kg. PB was detected up to 1.9 percent TRR (up to 0.014 mg/kg).

Newly identified metabolites have the common structure of phenyl ring with 2 to 5 chlorine atoms through dichlorination. The nitro group of quintozene was either reduced to NHOH, eliminated or displaced with sulfhydryl group of glutathione conjugate, which is metabolized or oxidized to become sulfoxide,

sulfone and sulfonic acid. They may be further metabolized to produce conjugates with glutathione, cysteine, malonylcysteine, glucose and others or incorporated into biomolecule.

In older studies on cabbage, potato and peanut, the most significant identified metabolites were the sum of TCNP-MalCys and PCP-MalCys (not separated) accounting for about 30 percent TRR. Quintozene, PCA and PCTA were also identified, sometimes more than 10 percent TRR. Therefore, in general, there may be common metabolism among crops.

The proposed metabolic pathway of quintozene in plants after soil treatment based on the new potato metabolism study is presented in Figure 2.

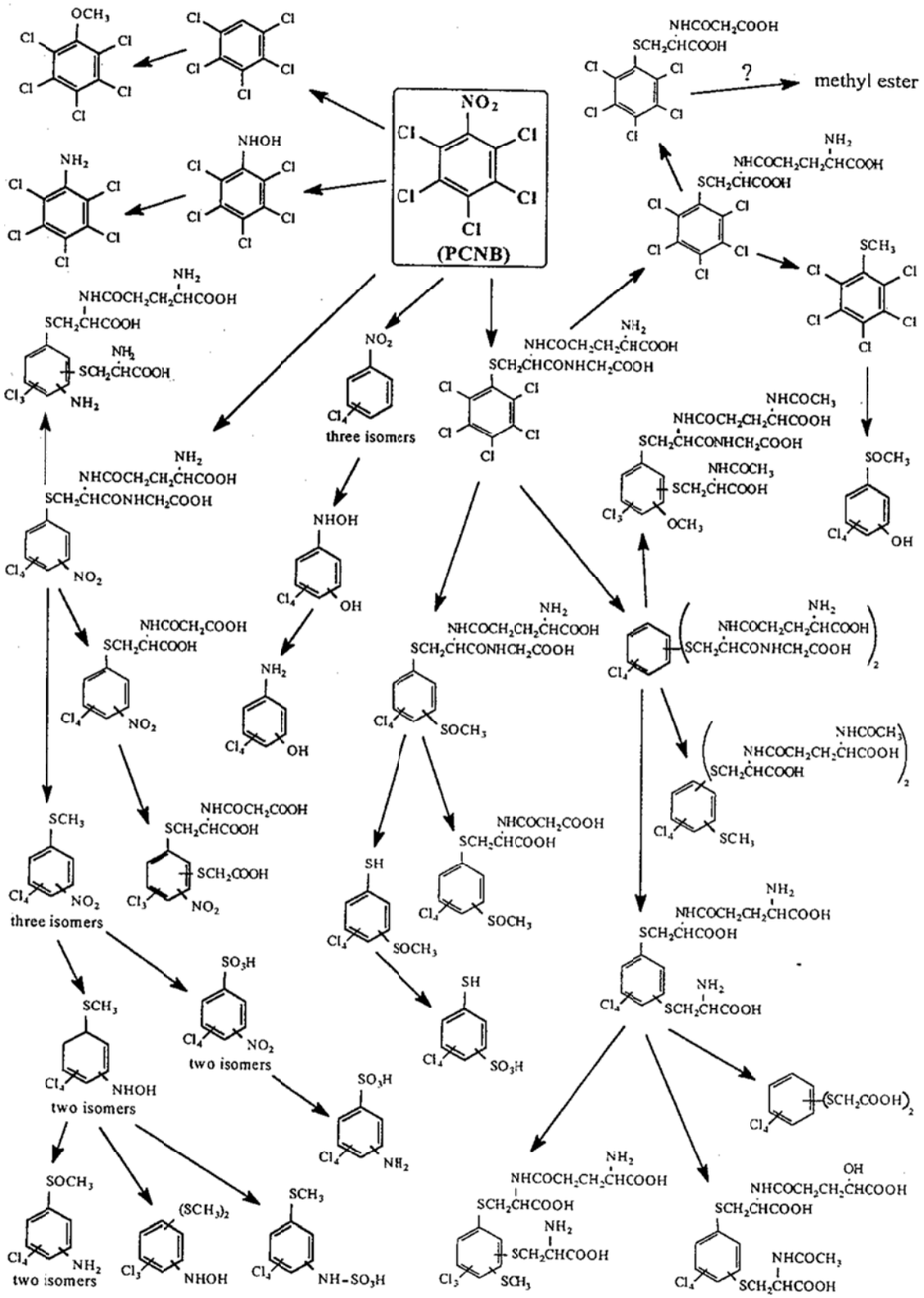


Figure 2 Proposed metabolic pathway of quintozene in plants after soil treatment (based on the studies on potato)

ENVIRONMENTAL FATE

The Meeting received information on environmental fate of quintozene: aerobic soil degradation, anaerobic soil degradation, soil photolysis, field dissipation in soil, adsorption and desorption in soil, and confined and field rotational crop studies. As quintozene has been registered for soil applications, and the use pattern included uses on potato and peanut, the results of studies on degradation in aerobic soil, soil photolysis, hydrolysis, rotational crop studies are relevant to this evaluation. According to the FAO Manual, field dissipation study is a conditional requirement depending on the results of confined rotational crop study. The relevant studies were evaluated and described below.

Aerobic degradation in soils

Study 1. *(Misra, 1993, 900-MET-012)*

Aerobic soil metabolism of ^{14}C-quintozene was studied for 365 days at quintozene concentration of 10 mg/kg on a microbially viable sandy loam soil (pH 6.3, organic matter 4.8 percent; cation exchange capacity 13.4 m eq/100 g, and soil moisture content 22 percent at 33 kPa). The soil temperature in dark was maintained at 25±1 °C and at or around 75 percent field moisture capacity (FMC) at 33 kPa. At the end of the incubation period, soil from the test vessel was transferred into a centrifuge bottle. The rinse of the test vessel with 10 percent 1/3 mol/L phosphoric acid in acetonitrile was added to the soil and the soil was extracted by shaking for about a minute followed by sonication for 10 minutes. Then the soil was centrifuged. The supernatant was collected and this procedure was repeated two more times and the combined soil extract was analysed by LSC and HPLC. GS-MS method was also used for samples from selected sampling periods to confirm the identity of metabolites.

Combustion analysis of the PES indicated that the extraction efficiency of the phosphoric acid decreased with increased incubation time. The soil samples from 30 to 365 days were additionally extracted with 20 percent 1/3 mol/L phosphoric acid in acetone and/or 20 percent 1/3 mol/L phosphoric acid in acetonitrile. The soil extracts were directly analysed by LSC and were concentrated under reduced pressure for HPLC analysis.

The major metabolites identified during the study were: PCA, PCTA, PB, PCTASO and PCTASOO with the maximum concentrations of 9 percent, 8 percent, 1 percent, 4 percent and 6 percent of applied quintozene, respectively. PCA and PCTA were the primary metabolites of quintozene during the study period. PCTASO and PCTASOO, the oxidized form of PCTA, were detected only in soil extracts.

The half-life of quintozene was estimated using a first order kinetics to be 189 days (coefficient of determination (R^2) = 0.79). However, the concentration of quintozene in soil reached less than half of the initially applied concentration in 60 days. The estimated half-life in first order approximation was higher because of the non-linearity of quintozene degradation. Independent first order regression analysis of the first five and last eight points of the data set improved correlation coefficients to 0.95 and 0.92, which gave estimated half-life values of 20 and 278 days, respectively.

Study 2. *(DeFelice, et al., 1977, 900-MOB-006)*

Laboratory leaching studies were conducted with ^{14}C-quintozene on four soil types: sandy loam (pH 4.8, organic matter 1.5 percent, moisture 13 percent at 33 kPa), silt loam (pH 5.0, organic matter 2.0 percent, moisture 30 percent at 33 kPa), sandy soil (pH 5.7, organic matter 0.9 percent, moisture 12 percent at 33kPa), and silt loam (pH 4.7, organic matter 2.5 percent, moisture not reported). ^{14}C-Quintozene was added uniformly to the top 5 cm of a column of 20 cm of untreated soil. Three column volumes of aqueous 0.01 mol/L $CaSO_4$ was then passed through the column. The profile of ^{14}C-quintozene was obtained in the $CaSO_4$ effluents and in 5 cm soil sections.

Quintozene was found to leach in small amounts (2 to 17 percent in four soil types) and only into the adjacent untreated soil zone. Less than 0.1 percent of the added radioactivity was found in the $CaSO_4$ effluent of all the soils tested. Leaching data on two soils aged for 30 days were similar to tests run immediately after treatment with ^{14}C-quintozene.

The recovery of radioactivity in each leaching experiment was in good agreement with the amount added to each column. Cold analyses of quintozene in the hexane phase after partitioning also agreed well with the radioactive count.

Photodegradation in soil

Study 1. *(Bowman B., 1988; 900-PHO-015)*

To samples of sandy loam soil (pH 6.2, organic matter 1.6 percent, moisture 15 percent at 33 kPa) in borosilicate vials, ^{14}C-quintozene in acetonitrile was added at 10.5 µg/g. The vials were then flame sealed to avoid volatilization and the soil samples were exposed to a xenon arc lamp (approximately ½ of the intensity of the sun; filtered to eliminate radiation wavelengths below 290 nm) for 30 days at 25±1 °C. Samples were removed at day 1, 1.02, 3.13, 7.10, 14.0 and 30.1, and extracted with acetone. The extracts were analysed by TLC for degradates by comparing with the standards (quintozene, 2,3,4,5-TCNB, PCA, PCTA, 2,3,5,6-TCNB, HCB and PB). The day 30 soil was extracted by acetone and analysed using GC-MS.

Quintozene was volatile on the surface of soil when irradiated without sealing the vessel. The mean overall total radioactivity recovered was 97.5 percent of the applied radioactivity when the sealed vessels were used. TLC analysis indicated that quintozene was photodegraded on the surface of soil: after 30 days of irradiation, PCA was the major photodegradation product (34.7 percent of the extracted radioactivity). There were no other major photodegradation products in 30 days. In the dark controls, the extracted radioactivity was essentially all quintozene. No degradation products were detected in these samples.

Based on the level of ^{14}C-quintozene in the extracts, a rate constant of 0.0313/day and a half-life of 22.2 days were calculated for the exposed soils. Quintozene was increasingly unextracted from the soil with time. A rate constant based on the loss of quintozene to soil binding was calculated to be 0.00696/day (half-life = 99.6 days). A corrected photolysis rate constant ($k_{exposed} - k_{dark}$) was calculated to be 0.0243/day (half-life = 28.5 days).

Study 2. *(Misra, 1993; 900-PHO-022)*

A thirty-day photolysis study of quintozene in a sandy loam soil (pH 6.3, organic matter 4.8 percent, moisture 22 percent at 33 kPa) was conducted by maintaining soils at around 75 percent FMC at 33 kPa and 25±1 °C, using a specially designed photolysis apparatus (jacketed stainless-steel box and a series of volatile traps with 1 mol/L NaOH and 1 mol/L H_2SO_4). To the soil, ^{14}C-quintozene was added at 9.6 µg/g and the soil was irradiated by a xenon arc light. A dark control study under similar conditions was carried out to compare the rates of degradation under artificial sunlight (12 hours light/12 hours dark cycle per day) with that of the dark control. At the end of irradiation period, the soil was extracted with 10 percent 1/3 mol/L phosphoric acid by shaking, sonication and centrifugation. Radioactivity in samples was measured using LSC. For analysing ^{14}C-quintozene and its degradates in the extracts, HPLC was primarily used. GC-MSD was used for confirmation purposes for the identification of quintozene and its degradates at selected sampling periods.

The major degradates of quintozene identified in sandy loam soil extracts after photo-irradiation were PCA and PCTA. These identities were confirmed by GC-MS. PCA and PCTA were formed in both dark control and under irradiation. PCTA formed faster in the dark control than under irradiation and PCA at

similar amount as under irradiation, which may indicate that photo irradiation does not play significant role in the formation of PCA and PCTA from quintozene. Most of the radioactive volatiles recovered in the traps were identified as quintozene.

Table 31 Distribution of quintozene and its photodegradation products in soil extracts, expressed in mg/kg-soil

Irradiated				Dark Control			
Days	Quintozene	PCA	PCTA	Days	Quintozene	PCA	PCTA
0	9.6	ND	ND	0	9.4	NA	NA
13	8.1	0.38	ND	1	9.2	ND	ND
25	6.3	0.41	0.04	2	8.2	0.16	0.04
49	4.8	0.52	ND	4	8.0	ND	ND
99	5.2	0.68	0.05	8	7.4	0.04	0.05
186	3.4	0.83	0.32	15	7.7	0.37	0.22
261	2.6	0.88	0.31	22	6.9	0.44	0.36
359	1..6	0.70	0.21	30	6.4	0.42	0.30

Notes:

NA, Not analysed.

ND, Below LOD.

The radioactivity not extracted from soil increased nearly two-fold under irradiation compared to the dark control. The half-life values estimated based on quintozene concentrations in soil under irradiation and dark control are 159 hours (13.2 days) and 62.1 days, respectively.

A significant amount of radioactive quintozene was found in the gasket holding the quartz glass plate on the open end of the test vessel for making the vessel air tight.

Field dissipation *(Rice et al., 1989, 900-DIS-015; 1989, 900-DIS-016; 1990, 900-DIS-001; Harned & Creeger, 1993, 900-DIS-012; Harned, 1997, 900-HUM-005; Lengen, 1989, 900-HUM-006)*

Field dissipation studies were conducted with a single pre-plant broadcast application of quintozene at various rates made to bare loamy sand or sandy loam soils in the United States. Immediately after the application, quintozene was incorporated to a depth of 10–15 cm. In some trials, immediately or one day after the application, broccoli or potato was planted. Additionally, a study was conducted on Bermuda grass turf to which quintozene was applied twice at 36.6 kg ai/ha four weeks apart.

Soil core samples to various depths (maximum 1.5 m) were taken on selected days after application. Each soil core sample was separated into increments of 15 cm long. Each increment was finely ground and extracted for analysis of quintozene, PCA, PCTA and PB and the impurity HCB.

The following table indicates the trial sites, types of the soil in 0–15 cm layers, conditions, application rates, maximum sampling periods, DT_{50} values of quintozene and total residues calculated using the first order model.

In general, quintozene and other 4 compounds were found at all sampling intervals in 0–15 cm layer till the end of the studies. It was not possible to calculate half-lives of these 4 compounds as they accumulated at rates greater than their dissipation. PCA was the most significant degradate of quintozene.

These data indicate that quintozene, its associated degradates and the impurity do not leach.

Table 32 Summary of field dissipation studies

Location in United States	Type of soil (0–15 cm)	Conditions	Application rate (kg ai/ha)	Sampling period (max. days)	DT_{50} (days) in 0-15 cm layer	
					Quintozene	Total [a]
Hawkinsville, GA	Loamy sand	Bare soil	11.2	535	12.9	-
Pattison, TX	Sandy loam	Bare soil	2.5	546	135	-
MN	Sandy loam	Bare soil + Potato [b]	28.0	546	193	444
Santa Cruz, CA	Sandy loam	Bare soil + Broccoli [b]	33.6	546	305	401
Madera, CA	Loamy sand	Bare soil + Broccoli [b]	33.6	546	128	263
Fresno, CA	Sandy loam	Bermuda grass turf	36.6 × 2	360	35.4	-

Notes:

[a] Sum of quintozene, PCA, PCTA, PB and HCB.

[b] Planted after the application of quintozene.

The geometric mean of DT_{50} of quintozene calculated from the above studies was 88 days (without the study on turfed soil, 106 days) and that of DT_{50} of total residues was 360 days, indicating that quintozene itself would be moderately persistent but the total residues were persistent.

Residues in rotational crops–Confined rotational crop study

Study 1. *(Halls, 1990, 900-RES-087; Putterman, 1993, 900-RES-104 Volume I Study Overview; Anonymous, 1993, 900-RES-103 Volume II In-Life Portion; Ford & Murty, 1993, 900-RES-105 Volume III Wheat; Ford & Murty, 1993, 900-RES-101 Volume IV Lettuce; Ford & Murty, 1993, 900-RES-102 Volume V Turnip)*

Six tanks (240 cm × 90 cm × 60 cm) were filled to 50 cm with sandy loam soil and located indoor in a greenhouse. Three tanks were used as control plots and the other three tanks as treated plots. Each treated plot was separated into three circular subplots, one for each crop type, which were then treated with ^{14}C-quintozene at 33.7 kg ai/ha.

Lettuce (var. Oakleaf and Black Seeded Simpson), turnip (var. Purple Top White Globe) and wheat (var. Marshall and Wheaton) were planted into the plots at plant back intervals (PBI) of 30, 120 and 365 days. A target cabbage plants (var. Copenhagen Market) was planted in the 120- and 365-day plots to represent cultivation of a target crop during the aging of the soil.

Crops were harvested at immature (33–55 days after planting) and mature (61–107 days after planting) growth stages. The aerial parts of the crops were taken as the immature samples of all crops and as the mature samples of lettuce and wheat. The whole turnip plant was taken at final harvest and separated into tops and roots. Mature wheat was separated into grain, hulls and straw.

Table 33 Harvest timing of immature and mature rotational crops at each plant back interval

PBI	Crop	Immature		Mature	
		Days after planting	Days after treatment	Days after planting	Days after treatment
30-Day	Lettuce	45	75	61	91
	Turnip	33	63	74	104
	Wheat	45	74	77	107
120-Day	Lettuce	34	154	67	187
	Turnip	34	154	67	187
	Wheat	34	154	92	212
365-Day	Lettuce	39	404	61	426
	Turnip	39	404	61	236

PBI	Crop	Immature		Mature	
		Days after planting	Days after treatment	Days after planting	Days after treatment
	Wheat	55	420	107	472

Combustion/LSC analysis were used for determination of radioactivity in each sample. Moisture content of each soil sample was determined and the radioactive residue levels in soil were expressed on a dry weight basis.

The TRR in 0–15 cm layer soil 2 hours after treatment with ^{14}C-quintozene were in the range of 6.56–14.3 mg eq/kg showing some tendency of decrease with aging of the soil. The TRR in 15–31 cm layer soil was significantly lower compared to 0–15 cm layer, but increased gradually from below the LOQ to the maximum 1.05 mg eq/kg.

Comparing with the treatment rate of 33.7 kg ai/ha, the TRR found in lettuce and turnip were not so high (1.40–5.67 mg eq/kg in immature samples). They showed the highest level at 30-day PBI. Significantly high levels of radioactivity were found in wheat straw regardless of PBI days, at 22.2–25.9 mg eq/kg, which were 3–5 times higher than the levels in the corresponding soil samples. The TRR in grain were low at 0.33–0.71 mg eq/kg but did not decrease at longer PBI.

Table 34 Total radioactive residue in soil (0–15 cm) and rotational crops at each plant back interval following a single application of quintozene at 33.7 kg ai/ha

PBI days	crop	Portion	TRR in 0–15 cm soil (mg eq/kg dw)				TRR in plant (mg eq/kg)	
			2 h [a]	Planting [b]	Immature [c]	Mature [c]	Immature [d]	Mature
30	Lettuce	-	12.2	12.6	8.36	10.0	3.31	1.62
	Turnip	Tops	9.72	8.41	6.30	7.14	4.61	3.63
		Roots						20.3
	Wheat	Straw	9.67	11.3	8.70	7.57	2.59	22.9
		Hulls						11.1
		Grain						0.332
120	Lettuce	-	10.3	8.02	7.19	8.72	1.40	0.147
	Turnip	Tops	6.56	5.89	5.75	5.53	2.39	1.73
		Roots						4.79
	Wheat	Straw	14.3	6.70	5.87	6.65	4.98	22.2
		Hulls						6.06
		Grain						0.710
365	Lettuce	-	13.0	4.84	5.93	5.36	5.67	0.608
	Turnip	Tops	11.4	6.52	6.42	5.61	1.60	0.73
		Roots						1.48
	Wheat	Straw	9.02	6.39	5.43	4.84	5.05	25.9
		Hulls						8.01
		Grain						0.376

Notes:

[a] Two hours after application.

[b] Same as the PBI.

[c] On the same day as immature samples and mature samples were obtained.

[d] The aerial parts of crops were obtained as samples.

For metabolite characterization, the crops were shipped frozen to the analytical laboratory. From homogenized lettuce and turnip top and root samples, endogenous water was removed by centrifugation

as “aqueous extract” and the remaining moist tissues were extracted with methanol. Water was added to the methanol fractions to make a methanol/water ratio to 60:40 (v/v) and the solutions were refrigerated to remove most of chlorophyll. The resulting solutions were partitioned with chloroform to extract the non-polar metabolites in the chloroform fraction. The “aqueous extract” and the aqueous methanol from the chloroform partition were pooled and processed for characterization of the polar metabolites. The solids remaining after methanol extraction was regarded as PES which were hydrolysed with 1 mol/L methanolic hydrochloric acid at 60 °C for 30 minutes. The solids were also treated with cellulase (see below). Fractions were analysed using LSC, HPLC and GC-MS. Mass spectra were obtained with a mass spectrometer using negative ion chemical ionization (NICI) with methane as the reagent gas.

Samples of wheat straw and hulls were extracted with methanol by soaking at 4 °C for 6 days and thereafter centrifuged. The pellets were re-extracted with methanol for one day and centrifuged. The resulting pellets were extracted twice with water. Methanol extracts were diluted with water to a methanol/water ratio of 60:40 (v/v) and partitioned with chloroform to obtain non-polar compounds. The combined aqueous and aqueous methanol portions were concentrated, freeze-dried, and reconstituted in water. The chloroform fractions were concentrated under nitrogen. The solids remaining after extraction with water was regarded as PES, which was acid-hydrolysed in the same way as for lettuce and turnip samples, and alkaline hydrolysed with 1 mol/L NaOH at 60 °C for 17 days. The PES from 30-day PBI wheat straw and post-hydrolysed residue were suspended in pH 4.5 acetate buffer with cellulase and incubated at 37 °C for 24 hours with shaking. Analyses were conducted in the same way as for lettuce and turnip samples.

The 120-day PBI wheat straw was subjected to a Bligh-Dyer extraction. The chloroform and aqueous methanol portions were separated from the solids. The chloroform portion was cleaned-up with a silica SPE cartridge, and the eluants were analysed by GC/NICI-MS. The aqueous methanol portion and the solids were separately subjected to alkaline hydrolysis (see above) and subsequently methylated with iodomethane in the presence of catalyst and toluene, resulted in extraction of radioactivity into toluene. The toluene fractions were cleaned-up with silica SPE cartridges, and the eluents were subjected to GC/NICI-MS.

The extractability by organic solvents were in the range of 36–67 percent TRR. Acid hydrolysis released additional 9.4–619 percent of TRR. In one example of wheat straw, it was demonstrated that the alkaline hydrolysis was slightly more efficient with 21–31 percent TRR released from the PES, compared to 14–16 percent released by the acid hydrolysis.

Table 35 Distribution of radioactivity in the organic solvent extracts and PES

PBI (days)	TRR (mg eq /kg)	Chloroform ext.		MeOH/water ext.		PES before hydrolysis		Acid hydrolysis		PES after hydrolysis		Extractability percent
		mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	
Lettuce												
30	1.24	0.260	21.0	0.186	15.0	0.675	54.4	0.229	18.5	0.445	35.9	36.0
120	0.16	0.029	18.1	0.050	31.5	0.070	44.0	0.015	9.4	0.055	34.6	49.6
365	0.44	0.085	19.3	0.152	34.5	0.196	44.5	0.046	10.5	0.150	34.0	53.8
Turnip roots												
365 [a]	1.44	0.664	46.1	0.301	20.9	0.480	33.3	0.230	16.0	0.249	17.3	67.0
Turnip tops												
365 [a]	0.76	0.032	4.2	0.440	57.9	0.293	38.5	0.090	11.8	0.203	26.7	62.1
PBI (days)	TRR mg eq /kg	Chloroform ext.		MeOH/water ext.		PES before hydrolysis		Acid hydrolysis		Alkaline hydrolysis		Extract ability
		mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	% TRR

PBI (days)	TRR (mg eq /kg)	Chloroform ext.		MeOH/water ext.		PES before hydrolysis		Acid hydrolysis		PES after hydrolysis		Extractability percent
		mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	
Wheat straw												
30	22.4	2.46	11.0	12.7	56.5	7.08	31.6	3.02	13.5	6.00	26.8	67.5
120	26.8	2.60	9.7	14.5	54.1	8.44	31.5	4.40	16.4	5.57	20.8	63.8
365	24.2	3.03	12.5	11.9	49.1	8.54	35.3	3.68	15.2	7.41	30.6	61.6
Wheat hulls												
30	9.61	0.423	4.4	4.76	49.5	3.99	41.5	1.62	16.9	not performed		53.9
365	7.31	0.270	3.7	3.34	45.7	3.07	42.0	1.29	17.6			49.4

Notes:

[a] As the US label requires "not to plant root crops in treated fields within 12 months of the last application unless quintozene is registered for use on those crops" (NB, potato), analysis was conducted only on the samples from the 365-day PBI.

Metabolite identification and quantification was performed by HPLC of the aqueous methanol extracts. Parent quintozene was not detected from any of the rotational crops with all PBIs, as in the methanol/water extracts of other studies. Identified metabolites were conjugated forms with either cysteine or glutathione. N,N'-Diacetyl-S,S'-(tetrachloro-p-phenylene)-L,L'-dicysteine / C4CyCy accounted for 12.2 percent TRR (0.093 mg/kg) in turnip tops, and N-acetyl-S-(pentachlorophenyl)-L-cysteine accounted for 12.9 percent TRR in wheat hulls. Other components accounted for <10 percent TRR but they were >0.05 mg/kg in wheat straw and hulls.

Table 36 Identification of radioactive metabolites in the methanol/water extracts of rotational crops at 30-, 120- and 365-day PBI

Component	30-day PBI		120-day PBI		365-day PBI	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Lettuce						
TRR	1.24	100	0.16	100	0.44	100
Methanol/water extract	0.186	15.0	0.050	31.5	0.152	34.5
N,N'-Diacetyl-*S,S'*-(tetrachloro-p-phenylene)-*L,L'*-dicysteine / C4CyCy	0.021	1.7	0.005	3.4	0.022	5.1
PCP-GSH	0.005	0.4	0.002	1.4	0.002	0.4
PCP-MalCys	0.041	3.3	0.000	0.3	0.004	0.8
Total identified		5.4		5.1		6.3
Turnip Roots						
TRR					1.44	100
Methanol/water extract					0.301	20.9
N,N'-Diacetyl-*S,S'*-(tetrachloro-p-phenylene)-*L,L'*-dicysteine / C4CyCy					0.060	4.2
S-(Pentachlorophenyl)-glutathionyl / N-malonyl-S-(pentachlorophenyl)-L-cysteine					0.001	0.1
Total identified						4.3
Turnip Tops						
TRR					0.76	100
Methanol/water extract					0.440	57.9
N,N'-Diacetyl-S,S'-(tetrachloro-p-phenylene)-L,L'-dicysteine / C4CyCy					0.093	12.2
S-(Pentachlorophenyl)-glutathionyl / N-malonyl-S-(pentachlorophenyl)-L-cysteine					0.001	0.1
Total identified						12.3
Wheat Straw						

Component	30-day PBI		120-day PBI		365-day PBI	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
TRR	22.4	100	26.8	100	24.2	100
Methanol/water extract	12.7	56.5	14.5	54.1	11.9	49.1
N,N'-Diacetyl-S,S'-(tetrachloro-p-phenylene)-L,L'-dicysteine	0.806	3.6	0.670	2.5	0.242	1.0
PCP-GSH	1.34	6.0	1.34	5.0	0.363	1.5
PCP-MalCys	0.426	1.9	--	--	1.694	7.0
TCP-diGSH	0.269	1.2	0.804	3	0.774	3.2
C4CyCy	--	--	0.429	1.6	0.750	3.1
N-acetyl-S-(pentachlorophenyl)-L-cysteine	--	--	--	--	0.266	1.1
Total identified		12.7		12.1		16.9
Wheat Hulls						
TRR	9.61	100			7.31	100
Methanol/water extract	4.76	49.5			3.34	45.7
N,N'-Diacetyl-S,S'-(tetrachloro-p-phenylene)-L,L'-dicysteine	0.490	5.1			0.110	1.5
PCP-GSH	0.538	5.6			0.526	7.2
PCP-MalCys	0.663	6.9			0.746	10.2
TCP-diGSH	0.250	2.6			0.175	2.4
C4CyCy	0.077	0.8			0.051	0.7
N-acetyl-S-(pentachlorophenyl)-L-cysteine	0.951	9.9			0.943	12.9
Total identified		33.6				34.9

The chloroform fractions of rotational crops were analysed by GC-MS. In addition to parent quintozene, PCA/pentachlorohydroxyaniline, PCTA, pentachlorophenylmethyl sulfoxide, and PB were identified with some others. However, no quantitative information was available for lettuce, turnip and wheat samples. Further examination revealed that while the levels of radioactivity remained similar for the wheat straw samples and hull samples, the character of the metabolites changed. With time, the metabolites became more polar and the extent of replacement of chlorine atoms on the aromatic ring increased (also seen in the plant metabolism studies).

Study 2. *(Heath, 1992, 900-RES-015)*

The trial location and setup of this study were the same as study 1 with the exception that the application was done 7 days after that in Study 1. Six plots (240 cm × 90 cm; 3 control and 3 treated) were filled to 45 cm with sandy loam soil and put in a greenhouse. Each "treated" plot was separated into three circular subplots (56 cm diameter), one for each crop type, which were then treated with ^{14}C-quintozene at 34.6 kg ai/ha.

The rotational crops, lettuce (Oakleaf for 31- and 121-day PBI and Black Seeded Simpson for 365-day PBI)), turnip (Purple Top White Globe for three PBIs) and wheat (Marshal spring wheat for 31- and 365-day PBI and Wheaton winter wheat for 121-day PBI) were planted into the plots. Cabbage plants (Copenhagen Market) was planted in the 121- and 365-day PBI plots to represent cultivation of a target crop during the aging of the soil between treatment of the soil and planting of the rotational crops, but the cabbage samples were not analysed. Crops were harvested at immature and mature (60–284 days after planting) growth stages.

Table 37 Harvest timing of rotational crops at each plant back interval

PBI	Crop	Days after planting
31-Day	Lettuce	60
	Turnip	75
	Wheat	75
121-Day	Lettuce	76

PBI	Crop	Days after planting
	Turnip	76
	Wheat	284
365-Day	Lettuce	61
	Turnip	61
	Wheat	90

Notes:

NB, Harvest dates of immature samples were not reported.

The harvested crop was divided into two and three subsamples of each half and were analysed by combustion/LSC.

Samples were extracted by macerating with a solvent (3× hexane followed by 3× methanol). Soil cores were extracted by shaking with acetone. Extracts were centrifuged to separate the supernatant from the residual PES. Extracts and PES were analysed for radioactivity using LSC or combustion/LSC.

The TRR in soil immediately after treatment with ^{14}C-quintozene at 34.6 kg ai/ha were within the range of 2.6–12.7 mg eq/kg and did not decrease with time. The acetone extracts of soil cores (0–15 cm) were analysed by HPLC. The chromatograms indicated the gradual degradation of quintozene during the study producing PCA and PCTASO.

The TRR in lettuce and turnip were not high compared to the rate of application, like in Study 1, and decreased over time as soil aged. Residues in wheat straw were high with 27.7 mg/kg at the 31-day PBI declining to 6.3 mg/kg for the 365-day PBI.

Table 38 Total radioactive residue in rotational crops at each plant back interval following a single application of quintozene at 34.6 kg ai/ha

PBI days	crop	Portion	TRR in plant (mg eq/kg)	
			Immature [a]	Mature
31	Lettuce	-	1.29	3.00
	Turnip	Tops	7.01	11.87
		Roots		11.37
	Wheat	Straw	5.02	27.71
		Grain		0.63
		Hulls		21.49
121	Lettuce	-	0.39	0.13
	Turnip	Tops	1.53	1.29
		Roots		5.79
	Wheat	Straw	3.44	13.36
		Grain		0.07
		Hulls		1.98
365	Lettuce	-	0.50	0.45
	Turnip	Tops	1.71	0.91
		Roots		1.91
	Wheat	Straw	1.89	6.28
		Grain		1.18
		Hulls		19.33

Notes:

[a] Immature crops were harvested as foliage only.

Metabolite identification and quantification was performed by HPLC of the hexane and methanol extracts.

Lettuce. Most of radioactivity in 121- and 365-day PBI lettuce was extracted in hexane and methanol (total of 77–80 percent TRR; (on average 28 percent in hexane and 50 percent in methanol). These extracts, however, were not analysed by HPLC. The methanol extract of the lettuce from the 365-day PBI was analysed by TLC, which showed radioactivity to be present over the length of the chromatogram, but no individual bands could be resolved, thus indicating a complex mixture of compounds. No further work was carried out on the PES of any samples.

Table 39 Distribution of radioactivity in the organic solvent extracts of mature lettuce

Extract	31-day PBI		121-day PBI		365-day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	13.41	100	0.15	100	0.43	100
Hexane extract	0.724	5.4	0.040	26.7	0.130	30.2
Methanol extract	0.724	5.4	0.080	53.3	0.200	46.5
Total extracted	1.45	10.8	0.120	80.0	0.330	76.7

Turnip roots. Analysis of the hexane extracts of 31-day PBI turnip roots (13 percent TRR) by HPLC showed the hexane soluble radioactivity consisted of quintozene, PCA, PCTA and PB. The hexane extracts of 121- and 365-day PBI were not analysed by HPLC. The radioactive profile in the methanol extract from the 31-day PBI sample was much more complex with the malonylcysteine conjugates of TCTP and PCTP (PCP) being the only compounds identified. The HPLC analysis of the methanol extract from the 121- and 365-day PBI could not identify radioactive compounds and therefore the methanol extract was hydrolysed with 6 mol/L HCl at 110 °C for 19 hours resulting in 63–66 percent of the radioactivity being associated with a fine black solid formed during hydrolysis.

The PES of roots retaining 28–50 percent TRR were hydrolysed with 6 mol/L HCL at 110 °C for 19 hours. However, acid hydrolysis at the severe conditions only solubilized small fractions of the radioactivity in PES (1.8–4.6 percent TRR).

Turnip tops. The hexane extracts contained 9.2–14 percent TRR, but, due to the low amount of radioactivity, they were not analysed. The methanol extracts contained 38–55 percent TRR and analysed by HPLC but it was not possible to identify the contained radioactivity. Hydrolysis of the methanol extract of 121-day PBI with 6 mol/L HCl as in a similar method as above resulted in 30 percent of the radioactivity being associated with a fine black solid formed during hydrolysis. No further characterization was attempted.

The PES containing 33–53 percent TRR were also hydrolysed with 6 mol/L HCl in the same way as above but only 5.6–7.9 percent TRR were solubilized. No further characterization was attempted.

As a conclusion, the hexane extracts contained identified compounds whereas methanol extracts showed a complex radioactive profile with the malonyl cysteine conjugates being the only compounds identified.

Table 40 Identification and characterization of radioactive metabolites in the hexane and methanol extracts of succeeding turnip at 31-, 121- and 365-day PBI

Extract / Fraction / Compound	31-day PBI		121-day PBI		365-day PBI	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Turnip Tops						
TRR	3.48	100	1.18	100	1.12	100
Hexane	0.320	9.2	0.170	14.4	0.140	12.5

Extract / Fraction / Compound	31-day PBI		121-day PBI		365-day PBI	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Methanol	1.32	37.9	0.610	51.7	0.610	54.5
Total extracted	1.64	47.1	0.78	66.1	0.75	67.0
PES	1.84	52.9	0.400	33.9	0.370	33.0
Acid hydrolysis	0.275	7.9	0.071	6.0	0.063	5.6
Turnip Roots						
TRR	17.10	100	4.59	100	2.91	100
Hexane	2.26	13.2	1.20	26.1	0.960	33.0
Quintozene	0.682	4.0	No analysis		No analysis	
PCA	0.693	4.1				
PCTA	0.140	0.8				
PB	0.494	2.9				
Total identified		11.8				
Polar metabolites	0.248	1.5				
Methanol	8.088	47.3	1.10	24.0	1.13	38.8
S-(tetrachlorothiophenol)-N-malonylcycteine; and PCP-MalCys [a]	2.661	15.6	--	--	--	--
Total extracted with hexane and methanol	10.348	60.5	2.3	50.1	2.09	71.8
PES I	6.737	39.4	2.29	49.9	0.821	28.2
Acid hydrolysis	0.593	3.5	0.083	1.8	0.134	4.6

Notes:

[a] Not appropriately quantified.

Wheat. Hexane did not extract a significant proportion of the radioactivity (<7 percent, except for 121-day PBI grain with 25 percent TRR extracted) so the hexane extract was not analysed. Methanol extracted 35–49 percent, 40–50 percent and 27–32 percent TRR respectively from grain, hull and straw. HPLC analysis of methanol extracts of hull and straw samples from all PBIs and grain sample from 365-day PBI showed a mixture of polar compounds, none of which corresponded to reference compounds. PES of straw from all PBIs and hull from 121- and 365-day PBI and grain from 365-day PBI contained, if reported, more than half of the TRR, except for the 121-day PBI grain. Hydrolysis of these PES samples with 6 mol/L HCl at 100–120 °C for 18–19h solubilized <1–30 percent TRR, which were not sufficient for identification.

Table 41 Identification and characterization of radioactive metabolites in the hexane and methanol extracts of succeeding wheat at 31-, 121- and 365-day PBI

Extract / Fraction	31-day PBI		121-day PBI		365-day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Wheat Grain						
TRR	0.76	100	0.08	100	1.25	100
Hexane	0.010	1.3	0.020	25.0	<0.013	<1.0
Methanol	0.370	48.7	0.03	37.5	0.440	35.2
Total extracted	0.380	50.0	0.05	62.5	0.44	35.2
PES I	not reported		0.03	~38	0.810	64.8
Acid hydrolysis	not performed				0.375	30.0
Wheat Hulls						
TRR	21.52	100	2.35	100	5.86	100
Hexane	0.323	1.5	0.031	1.3	<0.059	<1.0
Methanol	8.61	40.0	1.11	47.2	2.91	49.7
Total extracted	8.93	41.5	1.14	48.5	2.91	49.7
PES I	not reported		not reported		2.95	50.3

Extract / Fraction	31-day PBI		121-day PBI		365-day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Acid hydrolysis	not performed				0.469	8.0
Wheat Straw						
TRR	42.57	100	14.32	100	17.53	100
Hexane	1.11	2.6	0.931	6.5	0.254	1.5
Methanol	11.5	27.0	3.97	27.7	5.57	31.8
Total extracted	12.6	29.6	4.90	34.2	5.82	33.3
PES I	30.0	70.4	Not reported		11.7	66.7
Acid hydrolysis	5.62	13.2	not performed		<0.175	<1.0

The chloroform fractions of rotational crops were analysed by GC-MS. Parent quintozene and a couple of metabolites were identified as PCA/pentachlorohydroxyaniline, PCTA, PCTASO, and PB and many more. A quantification was not presented.

However, further examination revealed that while the levels of radioactivity remained similar for the different wheat crops, the character of the metabolites changed, and evidence was presented that with time the metabolites became more polar and the extent of replacement of chlorine atoms on the aromatic ring increased.

Study 3. *(Harned et al., 1998, 900-RES-121)*

Study 3 was conducted to better isolate and identify low-level metabolites in rotational crops using more modern and complex analytical techniques. ^{14}C-Quintozene was applied broadcast to a sandy loam soil at 2.2, 11.2, or 33.6 kg ai/ha and manually incorporated into the top 10 cm of soil. The trials were performed outdoors under typical growing conditions in Madera, CA. United States. After 35, 120 or 365 days of the application, lettuce was planted and after 30, 120 and 365 days, wheat was planted and grown in the treated soil. In the second re-trial, only lettuce was planted at the 30-day PBI. Turnips were planted 365 days after application at 2.2 and 11.2 kg ai/ha. Re-trials were conducted due to the heavy rain fall and weather conditions during the original trial.

Immature and mature lettuce leaves, wheat forage, grain and straw and turnip roots and tops were harvested for analysis as shown in the following table.

Table 42 Harvest timing of rotational crops at each plant back interval

Crop	Harvest (days after planting)					
	30-day PBI		120-day PBI		365-day PBI	
	Immature	Mature	Immature	Mature	Immature	Mature
Lettuce original trial [a]	161	184	71	122	114	140
Lettuce first retrial [a]	--	--	45	68	46	65
Lettuce second retrial	47	63	--	--	--	--
Turnip original trial	--	--	--	--	--	140
Turnip first retrial	--	--	--	--	65	73
Wheat original trial	119	204	57	125	114	188

Notes:

[a] The shortest PBI was 35 days.

TRR in the rotational crops and soil were determined by combustion/LSC. The TRR in soil immediately after treatment with quintozene at 33.6 kg ai/ha were within the range of 2.6–12.7 mg eq/kg.

TRR in rotational crops were >0.05 mg eq/kg in all samples except wheat grain planted 365 days after application at 2.2 kg ai/ha. The TRR in these crops were low considering the application rates, as in Study 1 and 2. Also the highest TRR were found in wheat straw while the wheat grain showed much lower TRR. Lettuce samples from 2.2 or 33.6 kg ai/ha were not analysed.

Table 43 Total radioactive residue in rotational crops at each plant back interval following a single application of quintozene at 2.2, 11.2 or 33.6 kg ai/ha

Plant, Portion/Growth stage	PBI (days)	Application rate (kg ai/ha)	TRR (mg eq/kg)
Lettuce, Immature	30 [a]	11.2	0.176
	120 [b]	11.2	0.145
	365	11.2	0.564
Lettuce, Mature	35 [a]	11.2	0.154
	120 [b]	11.2	0.100
	365	11.2	0.426
Turnip, Tops, Mature	365	2.2	0.050
	365	11.2	0.122
Turnip, Roots, Mature	365	2.2	0.051
	365	11.2	0.214
Turnip, Tops, Immature	365 [b]	11.2	0.748
Turnip, Tops, Mature	365 [b]	11.2	1.09
Turnip, Roots, Immature	365 [b]	11.2	1.11
Turnip, Roots, Mature	365 [b]	11.2	0.772
Wheat, Forage	30	2.2	0.384
	30	11.2	2.97
	30	33.6	4.06
	120	2.2	0.532
	120	11.2	3.35
	365	2.2	0.173
	365	11.2	0.637
Wheat, Grain	30	2.2	0.090
	30	11.2	0.792
	30	33.6	3.66
	120	2.2	0.119
	120	11.2	0.941
	365	2.2	0.031
	365	11.2	0.137
Wheat, Straw	30	2.2	2.23
	30	11.2	10.7
	30	33.6	24.1
	120	2.2	3.52
	120	11.2	16.8
	365	2.2	1.25
	365	11.2	4.61

Notes:

[a] 2nd re-trial.

[b] 1st re-trial.

Samples were finely ground with dry ice. Aliquots of homogenized samples were analysed by combustion/LSC. All remaining samples were extracted with solvents:

- Lettuce samples were extracted with methanol and a mixture of methanol/water (1:1) and the combined extracts were partitioned with chloroform;

- Turnip top samples (only mature samples) were extracted with methanol/water and the extract was partitioned with hexane followed by chloroform;
- Turnip root samples were extracted with methanol/water and the extract was partitioned with chloroform;
- Wheat forage and grain samples were extracted with methanol and methanol/water and the extract was partitioned with chloroform; and
- Wheat straw samples were extracted with methanol/water followed by methanol and the filtrate was partitioned with dichloromethane.

The PES after extraction of lettuce were treated with sequential digestions with three groups of enzymes, followed by hydrolysis with 0.5 mol/L KOH.:

- Cellulase, hemicellulase, sulfatase, β-glucosidase and pectinase (pH 5 compatible enzymes);
- α-amylase, β-glucuronidase and esterase (pH 7 compatible enzymes); and
- Trypsin, chymotrypsin and pronase-E (proteases).

The PES after extraction of turnip tops and roots were hydrolysed with Novozyme 249. The hydrolysate of turnip root PES was then hydrolysed by reflux with 1 mol/L HCl and then separated into hexane/ethyl acetate and aqueous fraction, which was partitioned with dichloromethane.

The PES of the wheat forage samples were composited by PBI and hydrolysed by several sequential techniques: enzymatic hydrolysis with pectinase, cellulase and hemicellulose at pH 5 at 37 °C; phosphate buffer at pH 6, followed by the enzymes pullulanase and α-amylase; then protease, α-chymotrypsin and trypsin at pH 7. The PES from the enzymatic hydrolyses was subsampled for combustion analysis and the remaining solids were subjected to acidic hydrolysis with 2 mol/L HCl/dioxane (1:9). Solids remaining after acidic hydrolysis were subjected to basic hydrolysis with 25 percent KOH at 25 °C.

The PES of the wheat straw samples were hydrolysed by several sequential techniques: incubation in phosphate buffer at pH 7 at 45 °C followed by α-amylase at 37°C; pronase at pH 7.2; pectinase at pH 4.0; acidic hydrolysis with 2 mol/L HCl/dioxane (1:9) at 70 °C. Solids remaining after acidic hydrolysis were subjected to basic hydrolysis with 25 percent KOH at 25 °C. The solids were further treated with 73 percent sulfuric acid at room temperature. Unhydrolysed solids from wheat forage and straw samples were analysed by combustion analysis.

The PES of the wheat grain samples were treated in the manner described for forage samples, except that the solids were treated for 66 hours at 37 °C at pH 5 with cell lysing enzymes, cellulase and hemicellulose prior to the pectinase, cellulase and hemicellulose treatment.

Lettuce: Methanol and methanol/water extracted a total of 56–77 percent of TRR: 27–44 percent in chloroform fraction (non-polar fraction), and 22–38 percent in aqueous fraction (polar fraction). There was no clear tendency according to the length of PBI. The nonpolar fraction contained 34 and 29 percent, 44 and 30 percent, and 29 percent, and 27 percent of the TRR in the immature and mature lettuce at 30-day, 120-day and 365-day PBI. The polar fraction contained 22 and 30 percent, 33 and 38 percent, and 38 and 36 percent of the TRR in the immature and mature lettuce at 30-day, 120-day and 365-day PBI.

The pH 5 and pH 7 compatible enzymes released an average of 2.3 percent and 3.3 percent of TRR, respectively, as a complex mixture of metabolites, none of which were of quantitative significance. The proteases released 9.1 and 9.9 percent, 8.4 and 10.1 percent, and 5.8 and 7.4 percent of the TRR in

the immature and mature lettuce at 30-day, 120-day and 365-day PBI. The KOH released 10.6 and 16.8 percent, 8.2 and 11.3 percent, and 8.5 and 7.9 percent of the TRR in the immature and mature lettuce at 30-day, 120-day and 365-day PBI.

Table 44 Distribution of radioactivity in lettuce in the organic solvent extracts, PES before and after various hydrolysis, and those released by hydrolysis (application rate, 11.2 kg ai/ha)

Extraction

PBI (days)	TRR (mg eq/kg)	Non-polar fraction		Polar fraction		PES before hydrolysis		Extract-ability [a] (percent)
		% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	
Lettuce, Immature								
30	0.154	34.4	0.0528	21.5	0.0330	30.9	0.0475	55.9
120	0.136	44.2	0.0600	32.6	0.0443	26.8	0.0363	76.8
365	0.579	29.0	0.1679	38.0	0.2196	29.2	0.1692	67.0
Lettuce, Mature								
30	0.108	29.0	0.0314	29.8	0.0323	40.5	0.0439	58.8
120	0.095	29.8	0.0284	38.1	0.0363	31.3	0.0297	67.9
365	0.427	27.2	0.1160	36.3	0.1549	29.1	0.1245	63.5

Notes:

[a] Compared with the radioactivity in the initial methanol and methanol/water extracts.

Hydrolysis of the PES shown above

PBI (days)	pH 5 compatible enzymes		pH 7 compatible enzymes		Proteases		KOH Hydrolysis		Final PES	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Lettuce, Immature										
30	2.1	0.0032	3.1	0.0046	9.1	0.0140	10.6	0.0163	4.5	0.0069
120	2.9	0.0039	5.0	0.0068	8.4	0.0114	8.2	0.0111	2.7	0.0037
365	2.1	0.0123	1.5	0.0086	5.8	0.0339	8.5	0.0491	3.2	0.0184
Lettuce, Mature										
30	1.5	0.0016	3.6	0.0039	9.9	0.0107	16.8	0.0182	10.3	0.0112
120	2.2	0.0021	3.9	0.0037	10.1	0.0096	11.3	0.0108	3.7	0.0035
365	2.3	0.0098	2.4	0.0102	7.4	0.0316	7.9	0.0337	2.4	0.0103

Notes:

[a] Compared with the radioactivity in the initial methanol and methanol/water extracts.

Wheat: From wheat forage (all PBI and application rates), methanol and methanol/water extracted a total of 52–76 percent of TRR: only 5.0–6.8 percent TRR in chloroform fraction (non-polar fraction), and much higher 45–70 percent TRR in aqueous fraction (polar fraction). In forage, quintozene was extensively metabolized to polar compounds.

Similar tendency was observed for wheat straw samples. From the wheat straw samples (from all PBI and application rates), methanol/water and methanol extracted a total of 56–70 percent TRR: dichloromethane fraction (nonpolar fraction) contained only 0.87–7.6 percent TRR while aqueous fraction (polar fraction) contained much higher 51–68 percent TRR.

Only 9.8–33 percent TRR in wheat grain were extracted with methanol and methanol/water showing higher extractability with longer PBI. The chloroform fraction contained 0.89–1.5 percent TRR while aqueous fraction contained 8.8–32 percent TRR (higher at longer PBI) with >57 percent TRR

unextracted. This indicated that the majority of non-polar and polar metabolites were either trapped, bound or incorporated into the grain matrix but at longer PBI they became more extractable.

Table 45 Distribution of radioactivity in wheat forage, grain and straw in the organic solvent extracts, and PES before hydrolysis (application rate, 2.2, 11.2 and 33.6 kg ai/ha)

PBI (days)	Appl. Rate (kg ai/ha)	TRR (mg eq/kg)	Non-polar fraction		Polar fraction		PES before hydrolysis		Extract-ability [a] (percent)
			% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	
Wheat forage									
30	2.2	0.384	6.27	0.0241	55.78	0.214	38.61	0.148	62.05
	33.6	4.06	6.77	0.275	45.26	1.84	39.83	1.62	52.03
120	2.2	0.532	4.99	0.0265	67.97	0.362	31.79	0.169	72.96
	11.2	3.35	5.50	0.184	53.97	1.81	34.76	1.16	59.47
365	2.2	0.173	6.62	0.0114	67.14	0.116	20.81	0.0359	73.76
	11.2	0.637	6.42	0.0409	69.62	0.443	20.32	0.129	76.04
Wheat grain									
30	33.6	3.66	1.01	0.0370	8.77	0.321	83.38	3.05	9.78
120	11.2	0.941	1.47	0.0138	14.95	0.141	77.51	0.729	16.42
365	11.2	0.137	0.89	0.0012	32.33	0.0443	57.49	0.0787	33.22
Wheat straw									
30	2.2	2.23	0.87	0.0194	60.60	1.35	36.35	0.809	61.47
	11.2	10.7	5.66	0.604	50.60	5.40	45.25	4.83	56.26
	33.6	24.1	7.59	1.83	54.96	13.3	34.56	8.34	62.55
120	2.2	3.5	2.38	0.0838	55.85	1.97	42.08	1.48	58.23
	11.2	16.8	4.45	0.749	54.57	9.18	37.17	6.25	59.02
365	2.2	1.25	2.76	0.0344	67.52	0.842	27.01	0.337	70.28
	11.6	4.61	4.65	0.214	62.62	2.89	34.41	1.59	67.27

The PES from wheat samples before hydrolysis were subjected to sequential hydrolysis. Only 4–8 percent of the TRR was released from forage by pH 5 compatible enzymes, indicating that most of the residues in the forage PES were bound, rather than trapped. The forage PES were also digested with pH 6 compatible enzymes and proteases, which did not release more than 4 percent of the TRR each. The three enzyme hydrolysates from each forage sample were composited and profiled by HPLC showing that the residues released were comprised of a complex mixture of metabolites.

The grain PES before hydrolysis were each digested with cell lysing enzymes (pH 5 compatible enzymes) to release residues trapped within the grain matrix. This treatment released 22–55 percent TRR, showing that a substantial amount of the residues in grain PES were physically trapped within the matrix. The grain PES were then digested with a variety of specific enzymes (pH 6 compatible enzymes and proteases). The pH 6 compatible enzymes (α-amylase and pullulanase) released an additional 11–16 percent of the TRR, indicating that the certain amounts of residues in grain were associated with polysaccharides. The proteases released less than 3 percent of the TRR from grain. The pH 5 and pH 6 compatible enzyme hydrolysates of grain (30-day and 120-say PBI) were composited and profiled by HPLC. The resultant reconstructed chromatograms showed a large non-retained peak which may be indicative of residues bound to large biomolecules which would not be retained by conventional HPLC. A complex mixture of retained metabolites were also observed.

Following enzymatic treatments, forage and grain PES samples were subjected to acid hydrolysis which released up to 16 percent of the TRR from forage PES, indicating that a significant amount of quintozene-related unextracted residues were incorporated into lignin in forage. Only a small amount (<5 percent) of the residues in grain were incorporated into ligninacious materials.

Residues still unreleased from forage and grain solids by the acid hydrolysis were subjected to alkaline hydrolysis. Less than 7 percent of TRR was released from forage PES, leaving 2 percent or less in the final PES. Up to 24 percent of the TRR was released from the grain PES by alkaline hydrolysis, leaving 3 percent or less of grain TRR remaining in the final PES.

Table 46 Distribution of radioactivity in wheat forage, grain and straw after enzymatic, acidic and alkaline hydrolysis and the PES after hydrolysis (application rate, 2.2, 11.2 and 33.6 kg ai/ha)

PBI (days)	Appl. Rate (kg ai/ha)	pH 5-compatible Enzymes		pH 6-compatible Enzymes		Proteases.		Acid hydrolysis		Base hydrolysis		Final PES	
		percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg	percent TRR	mg eq/kg
Wheat forage													
30	2.2	7.26	0.0279	3.85	0.0148	3.87	0.0149	15.58	0.0598	6.57	0.0252	2.20	0.0085
	33.6	7.49	0.304	3.98	0.162	3.99	0.162	16.07	0.6529	6.78	0.276	2.27	0.0923
120	2.2	5.58	0.0297	3.72	0.0198	2.85	0.0151	9.04	0.0481	4.86	0.0258	1.52	0.0081
	11.2	6.10	0.204	4.06	0.136	3.11	0.104	9.89	0.331	5.31	0.178	1.66	0.0555
365	2.2	4.46	0.0077	2.26	0.0039	2.34	0.0040	4.83	0.0083	3.87	0.0067	0.86	0.0015
	11.2	4.35	0.0277	2.21	0.0141	2.28	0.0145	4.71	0.0300	3.78	0.0241	0.84	0.0053
Wheat grain													
30	33.6	55.35	2.026	16.32	0.597	2.28	0.0836	2.54	0.0931	4.54	0.166	1.12	0.0409
120	11.2	42.01	0.395	13.68	0.129	1.70	0.0160	4.57	0.0429	17.27	0.163	2.62	0.0246
365	11.2	22.40	0.0307	10.88	0.0149	n.d.	n.d.	1.35	0.0018	23.63	0.0324	1.14	0.0016

Metabolites were characterized by RP-HPLC and/or normal-phase TLC. Metabolites of significance were isolated from the appropriate fractions by repetitive RP-HPLC and normal phase TLC. Isolated polar metabolites were further characterized and/or identified by chromatographic comparisons with authentic reference standards using LC- MS/MS or GC-MS.

Analyses of lettuce, turnip tops and roots, and wheat forage and straw identified up to 30 metabolites, the majority of which accounted for only low portions (<10 percent) of the radioactive residues at low concentrations.

Lettuce: Analysis of lettuce extracts was performed by GC-MS, HPLC-LSC and LC-MS/MS. Parent quintozene and twelve metabolites were isolated, identified and quantified in extracts of rotational lettuce. On average, 49 percent TRR was identified and quantified; the remainder was either unextracted (about 31 percent TRR) and hydrolysed with enzymes and KOH, or was present in polar extracts. Quintozene was a major metabolite in 30-day PBI immature and mature lettuce but in 120- and 365-day PBI samples, it was found <10 percent TRR and <0.01 mg/kg.

An average of 96 percent of the non-polar metabolites in lettuce extracts were identified and quantified. These metabolites represented 43–57 percent TRR (0.051–0.28 mg/kg). Quintozene accounted for more than 10 percent TRR (21 percent TRR) at 30-day PBI but both the percentage of TRR and concentrations decreased with longer PBI. PCA was detected at 11.5 percent TRR (0.011 mg/kg) in mature lettuce at 120-day PBI and C3MS at 10.9 percent TRR (0.063 mg.kg) in immature lettuce at 365-day PBI. Except for one unknown fraction, there was no other metabolite accounting for more than 10 percent TRR.

Table 47 Summary of identification/characterization of radioactivity in succeeding lettuce (application rate, 11.2 kg ai/ha)

Fraction/Compound	30 d PBI (immature)		30 d PBI (mature)		120 d PBI (immature)	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
TRR	100	0.154	100	0.108	100	0.136

Fraction/Compound	30 d PBI (immature)		30 d PBI (mature)		120 d PBI (immature)	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
$CHCl_3$/MeOH - extract (non-polar)	34.4	0.0528	29.0	0.0314	44.2	0.0600
PB	--	--	0.4	0.0003	--	--
Quintozene	21.0	0.0322	21.2	0.0230	2.9	0.0039
C3MS	2.5	0.0039	--	--	3.7	0.0050
PCA	9.8	0.0150	6.4	0.0069	5.2	0.0071
PCTA	2.0	0.0031	--	--	1.8	0.0025
C4MS 1 [b]	1.6	0.0025	--	--	13.6	0.0185
Conjugate 330 [a]	3.4	0.0052	2.6	0.0028	2.9	0.0039
Conjugates 247, 290, 313	--	--	4.6	0.0062	--	--
Unknowns	--	--	--	--	--	0.0062
Subtotal identified/characterized	40.3	0.619	35.2	0.0392	30.1	0.0409
MeOH/H_2O - extract (polar)	21.5	0.0330	29.8	0.0323	32.6	0.0443
C4CyFCy	1.8	0.0027	1.8	0.0019	4.6	0.0063
C5SA-conjugate	4.2	0.0065	2.0	0.0022	3.8	0.0052
C5SA			4.1	0.0044		
C5MaCy	4.2	0.0065	2.6	0.0029	--	--
Unknowns (each <0.005 mg/kg)	11.0	0.0169	14.0	0.0152	22.1	0.0300
Subtotal identified/characterized	10.2	0.0157	10.5	0.0114	8.4	0.0115
pH 5 enzymes hydrolysate	2.1	0.0032	1.5	0.0016	2.9	0.0039
pH 7 enzymes hydrolysate	3.1	0.0046	3.6	0.0039	5.0	0.0068
Protease hydrolysate	9.1	0.0140	9.9	0.0107	8.4	0.0114
PCA	1.4	0.0022	not analysed		not analysed	
Polar (non-retained)	2.3	0.0036				
Unknowns (each <0.0038)	2.8	0.0043				
KOH hydrolysate	10.6	0.0163	16.8	0.0182	8.2	0.0111
PCA	5.3	0.0082	8.5	0.0092	not analysed	
Polar (non-retained)	4.9	0.0076	2.5	0.0027		
Unknowns (each <0.0025)	1.2	0.0018	3.5	0.0038		
PES (final)	4.5	0.0069	10.3	0.0112	2.7	0.0037
Total identified	57.2	0.0880	49.6	0.0536	43.1	0.0586
Total characterized	79.4	0.1222	69.6	0.0753	65.2	0.0886

Fraction/Compound	120 d PBI (mature)		365 d PBI (immature)		365 d PBI (mature)	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
TRR	100	0.095	100	0.579	100	0.427
$CHCl_3$/MeOH - extract (non-polar)	49.9	0.0479	35.6	0.2058	35.7	0.1310
Quintozene	0.9	0.0008	0.8	0.0049	1.3	0.0056
C3MS	5.9	0.0056	10.9	0.0633	6.5	0.0276
PCA	11.5	0.0109	8.2	0.0475	9.2	0.0391
PCTA	0.4	0.0004	0.8	0.0046	1.2	0.0050
C4MS 1 [b]	8.8	0.0084	8.0	0.0464	4.8	0.0203
C4MS 2 [b]	6.8	0.0065	2.5	0.0145	1.6	0.0069
Conjugate 330 [a]	11.5	0.0109	2.5	0.0145	3.0	0.0128
Subtotal identified/characterized	45.8	0.0435	33.7	0.1957	27.6	0.1173
MeOH/H_2O - extract (polar)	38.1	0.0363	38.0	0.2196	36.3	0.1549
C3MS-MaCy	--	--	2.9	0.0168	2.8	0.0120
C4MaCyFCy	--	--	1.6	0.0094	1.7	0.0067
C4SA	--	--	2.3	0.0132	2.9	0.0123
C4CyFCy	2.8	0.0027	--	--	--	--
C5SA-conjugate	2.4	0.0022	--	--	--	--
C5SA	2.2	0.0021	5.9	0.0341	5.5	0.0235
Unknowns (each <0.005 mg/kg)	26.8	0.0254	26.4	0.1529 [c]	23.9	0.1022 [d]
Subtotal identified/characterized	7.4	0.007	12.7	0.0735	12.9	0.0545

Fraction/Compound	120 d PBI (mature)		365 d PBI (immature)		365 d PBI (mature)	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
pH 5 enzymes hydrolysate	2.2	0.0021	2.1	0.0123	2.3	0.0098
pH 7 enzymes hydrolysate	3.9	0.0037	1.5	0.0086	2.4	0.0102
Protease hydrolysate	10.1	0.0096	5.8	0.0339	7.4	0.0316
PCA	not analysed		0.7	0.0038	0.6	0.0025
Polar (non-retained)			1.6	0.0093	1.9	0.0079
Unknowns (each <0.0038)			2.3	0.0135	2.9	0.0124
KOH hydrolysate	11.3	0.0108	8.5	0.0491	7.9	0.0337
PCA	not analysed		2.3	0.0133	1.7	0.0074
Polar (non-retained)			4.7	0.0274	4.5	0.0193
Unknowns (each <0.0025)			2.5	0.0145	3.1	0.0131
PES (final)	3.7	0.0035	3.2	0.0184	2.4	0.0103
Total identified	53.2	0.0505	49.4	0.2863	42.7	0.1817
Total characterized	80.0	0.0759	90.5	0.5248	79.0	0.3366

Notes:

[a] Conjugate 330, cysteinyl conjugate but exact structure not confirmed.

[b] Two methyl tetrachlorophenyl sulfone isomers were found. Substitution positions not known because of insufficient material for NMR.

[c] One region with 0.0327 mg/kg consists of multiple compounds.

[d] One region with 0.0180 mg/kg consists of multiple compounds.

Turnip: Analysis of turnip extracts was performed by HPLC-LSC, GC-MS and LC-MS. Fourteen metabolites were isolated and identified. These metabolites represented 65–76 percent (0.039–0.094 mg/kg) and 78–81 percent (0.045 0.19 mg/kg) of the TRR in turnip top and root samples, respectively. Parent quintozene was not detected in any of the samples.

Table 48 Summary of identification/characterization of radioactivity in succeeding turnip tops at maturity (365-day PBI only)

Fraction/Compound	2.2 kg ai/ha		11.2 kg ai/ha	
	% TRR	mg/kg	% TRR	mg/kg
TRR	100	0.050	100	0.143
MeOH-4 – fraction	48.3	0.024	65.0	0.094
C3MSSA (M-A) & C2MSMaCy (M-D)	8.91	0.004	16.3	0.024
C2SA (M-B) & C3MSMaCy (M-C)	23.1	0.011	18.1	0.026
C3SA (M-E)	6.24	0.003	--	--
C4SA (M-F)	3.30	0.002	8.60	0.012
C5SA (M-G)	3.19	0.002	5.38	0.008
Unknown 1	1.96	0.001	5.87	0.008
Unknown 2	1.57	0.001	2.71	0.004
Unknown 3	--	--	3.82	0.006
Unknown 4	--	--	4.25	0.006
Subtotal identified/characterized	44.8	0.022	48.4	0.070
$CHCl_3$-2 – fraction	28.3	0.015	13.0	0.019
C3MSSA (M-A) & C2MSMaCy (M-D)	3.46	0.002	--	--
C3SA (M-E)	2.07	0.001	--	--
C4SA (M-F)	3.75	0.002	1.10	0.002
C5SA (M-G)	1.55	0.001	0.64	0.001
C3MS (M-I)	8.06	0.004	5.15	0.007
C3MS (M-J) & PCTASO (M-L)	3.95	0.002	2.14	0.003
C4MS (M-K)	--	--	0.77	0.001
PCP (M-M)	0.57	<0.001	0.43	0.001

Fraction/Compound	2.2 kg ai/ha		11.2 kg ai/ha	
	% TRR	mg/kg	% TRR	mg/kg
PCA (M-H)	1.14	0.001	--	--
Unknown 1	--	--	0.51	0.001
Unknown 2	1.27	0.001	0.76	0.001
Unknown 3	--	--	0.87	0.001
Unknown 4	2.51	0.001	0.60	0.001
Subtotal identified/characterized	24.6	0.013	10.2	0.015
Aqueous-2 – fraction	9.56	0.005	9.51	0.012
C3MSSA (M-A) & C2MSMaCy (M-D)	3.11	0.002	3.79	0.005
C3SA (M-E)	2.06	0.001	1.19	0.002
C4SA (M-F)	1.04	0.001	1.65	0.002
C5SA (M-G)	0.63	<0.001	--	--
Unknown 1	2.72	0.001	1.08	0.001
Unknown 2	--	--	0.77	0.001
Unknown 3	--	--	1.02	0.001
Subtotal identified/characterized	6.84	0.004	6.63	0.009
Hexane	4.63	0.002	3.50	0.005
PES (final)	9.18	0.004	8.98	0.013
Total identified	76.16	0.039	65.26	0.094
Total characterized	86.19	0.044	87.52	0.125

Table 49 Summary of identification/characterization of radioactivity in succeeding turnip roots at maturity (365-day PBI only)

Fraction/Compound	2.2 kg ai/ha		11.2 kg ai/ha	
	% TRR	mg/kg	% TRR	mg/kg
TRR	100	0.055	100	0.243
MeOH-3 – fraction	31.6	0.018	26.2	0.064
C3MSSA (M-A) & C2MSMaCy (M-D)	6.96	0.004	2.45	0.006
C2SA (M-B) & C3MSMaCy (M-C)	6.11	0.003	16.2	0.039
C3SA (M-E)	2.97	0.002	3.10	0.008
C4SA (M-F)	8.96	0.005	0.78	0.002
C5SA (M-G)	1.89	0.001	0.75	0.002
unknown 1	3.14	0.002	2.98	0.007
unknown 2	1.60	0.001	--	--
Subtotal identified/characterized	26.9	0.015	23.3	0.057
$CHCl_3$-2 – fraction	34.3	0.020	30.8	0.076
C3MSSA (M-A) & C2MSMaCy (M-D)	--	--	1.10	0.003
C3SA (M-E)	--	--	1.07	0.003
C3MS (M-I)	2.72	0.002	0.86	0.002
C3MS (M-J) & PCTASO (M-L)	3.09	0.002	1.79	0.004
C4MS (M-K)	--	--	1.34	0.003
PCP (M-M)	1.41	0.001	1.49	0.004
PCA (M-H)	26.2	0.015	22.0	0.054
PCTA (M-N)	0.85	<0.001	1.13	0.003
Subtotal identified/characterized	34.3	0.02	30.8	0.076
Hexane/ethyl acetate – fraction	20.0	0.010	25.4	0.060
PCA (M-H)	20.0	0.010	22.2	0.053
PCTA (M-N)	--	--	1.84	0.004
unknown 1	--	--	1.36	0.003
Subtotal identified/characterized	20.0	0.01	24.0	0.057
Aqueous-2	1.68	0.001	5.32	0.013
Aqueous-3	1.92	0.001	2.63	0.006
CH_2Cl_2	2.32	0.001	2.34	0.006
PES (final)	8.24	0.004	7.30	0.018

Fraction/Compound	2.2 kg ai/ha		11.2 kg ai/ha	
	% TRR	mg/kg	% TRR	mg/kg
Total identified	81.1	0.045	78.1	0.190
Total characterized	85.9	0.048	82.4	0.200

Wheat

Analyses of factions were performed using LC-MS (ESI) or GC-MS (NICI). Quintozene and metabolites were determined by the presence of chlorine isotope clusters, identified by comparison of spectra against authentic standards when available, and quantified by HPLC with fraction collection.

Twelve metabolites were isolated and identified.

- Quintozene at 0.023 mg/kg in straw (2.2 kg ai/ha, 120-day PBI);
- PCA at 0.013 mg/kg in straw (2.2 kg ai/ha, 120-day PBI);
- PCTA at 0.19 mg/kg in straw (2.2 kg ai/ha, 120-day PBI);
- C5SA at 0.14 mg/kg in straw (2.2 kg ai/ha, 120-day PBI);
- three isomers of C4SA at 0.08, 0.33 and 0.06 mg/kg in straw (2.2 kg ai/ha, 120-day PBI) and 0.22 mg/kg in forage (2.2 kg ai/ha, 120-day PBI);
- an isomer of C3SA at 0.17 mg/kg in straw ((2.2 kg ai/ha, 120-day PBI);
- C3MSSA at 0.11 mg/kg in straw (2.2 kg ai/ha, 120-day PBI);
- C3MSOH at 0.009 mg/kg in grain (2.2 kg ai/ha, 120-day PBI);
- C4SANH at 0.15 mg/kg in forage (11.2 kg ai/ha, 30-day PBI); and
- C3MS at 0.18 mg/kg in straw (33.6 kg ai/ha, 30-day PBI).

Identification of terminal metabolites indicated that reduction and conjugation as well as dichlorination are the main metabolic pathways of quintozene in wheat. The reduction product PCA was detected in forage and straw. Oxidation of PCTA, possibly derived from glutathione conjugates produced by plant metabolism or taken up by the roots from soil, yielded other major metabolites such as sulfonic acids and sulfones.

Table 50 Summary of identification/characterization of radioactivity in succeeding wheat forage (120-day PBI only)

Fraction/Compound	11.2 kg ai/ha	
	% TRR	mg eq/kg
TRR	100	3.3473
$CHCl_3$/MeOH - extract (non-polar)	5.50	0.1841
PCA	1.87	0.0625
C5MX		
PCTA	0.83	0.0278
C4MeAcCy	0.24	0.0080
Unknowns (minor)	2.56	0.0857
Subtotal identified/characterized	2.94	0.0983
MeOH/H_2O - extract (polar)	53.97	1.80661
tetrachlorobenzene sulfonic acid (F1a)	4.58	0.1532
tetrachlorosulfanilic acid (F1b)	2.61	0.0872
pentachlorobenzene sulfonic acid (F2)	5.36	0.1793
pentachlorophenyl glutathione conjugate (FC1)	0.52	0.0173

Fraction/Compound	11.2 kg ai/ha	
	% TRR	mg eq/kg
tetrachlorobenzene malonylcysteine-formylcysteine diconjugate (FC6)	0.88	0.0296
trichlorobenzene sulfonic acid (E1)	3.44	0.1152
tetrachlorobenzene sulfonic acid (E2)	2.17	0.0727
trichlorobenzene sulfonic acid (E3a)	1.77	0.0594
hydroxy trichlorobenzene sulfonic acid (E3b)	1.33	0.0444
hexose pentachlorophenyl sulfonate (EC1, EC3)	0.71	0.0238
	0.99	0.0332
trichlorosulfonic acid (EC4c; 1 of 3)	0.29	0.0096
tetrachlorosulfonic acid (EC4c; 2 of 3)	0.29	0.0096
trichlorosulfanilic acid (EC4c; 3 of 3)	0.29	0.0096
tetrachlorobenzene dichlorobenzene sulfonic acid (D3)	1.10	0.0369
dicysteine conjugate (C1b)	0.56	0.0189
trichlorobenzene sulfonic acid methyl sulfone (C2b)	0.82	0.0274
tetrachlorobenzene sulfonic acid methyl sulfoxide (C3a)	1.13	0.0377
Unknowns (minor)	25.13	0.8412
Subtotal identified/characterized	28.8	0.965
pH 5 enzymes	6.10	0.2042
pH 6 enzymes	4.06	0.1360
Protease enzymes	3.11	0.1041
Acid hydrolysis	9.89	0.3309
Alkaline hydrolysis	5.31	0.1778
PES (final)	1.66	0.0555
Total identified	31.78	1.0633
Total characterized	72.74	2.4345

For wheat straw, nineteen metabolites were identified from the extracts of the 2.2 and 11.2 kg ai/ha samples, eight of which were found in the non-polar fraction.

For the 120-day PBI wheat straw samples (2.2 and 11.2 kg ai/ha). The polar extracts were separated into eight fractions which were further separated by different HPLC systems and clean-up procedures to identify individual components. Eleven metabolites were found in the polar fraction. Moreover, in the polar fraction, a number of positional isomers exist for the trichlorobenzenesulfonic acid and tetrachlorobenzenesulfonic acid.

Field Rotational Crop Studies (Gaydosh, 1996, 900-RES-116; Gounaris, 1994, 900-RES-163; Gaydosh, 1996, 900-RES-146; Gounaris, 1994, 900-RES-162)

Two field trials were conducted in 1992–1993 in the typical peanut growing regions of the United States: one in Meigs, GA, and the other in Brookshire, TX. Unlabelled quintozene formulated as GR formulation was applied two times to bare soil as two split applications, 31 (Meigs) or 41 (Brookshire) days apart, each at a rate of 5.6 kg ai/ha (total 11 kg ai/ha) which simulated the then critical US GAP for peanut, which was not on the current label. Applications were made in 30 cm bands using common granular application equipment at a simulated pegging time for peanuts.

At plant-back intervals (PBI) of 30, 120, and 367 or 366 days after the second application, wheat and lettuce were planted. Turnips were planted at a PBI of approximately one year only, in accordance with the United States label restrictions on crop rotation of root vegetables. As a worst case, rotational crops were planted within the treated bands. At each location, from one control plot and two treated plots, duplicate crop samples were taken at each PBI with the exception of a 120-day PBI plot in Brookshire that was inadvertently treated at a higher rate and could not be used. Samples of wheat grain, wheat straw and lettuce were harvested at maturity while wheat forage was sampled immature (Table 51). Excessive soil

was typically shaken off the plant samples. All samples were frozen within 2 hours of harvest and kept frozen until analysis.

Table 51 Harvest timing of immature and mature rotational crops at each plant back interval

PBI	Crop	Immature	Mature
		Days after planting	Days after planting
Meigs, GA			
30-Day	Wheat	54	268
	Lettuce	--	90
120-Day	Wheat	70	178
	Lettuce	--	105
367-Day	Wheat	56	284
	Lettuce	--	106
	Turnip	--	56
Brookshire, TX			
30-Day	Wheat	54	231
	Lettuce	--	202
120-Day	Wheat	42	219
	Lettuce	--	104
366-Day	Wheat	78	255
	Lettuce	--	145
	Turnip	--	102

Samples were homogenized with dry ice. Samples after extraction and clean-up with Florisil column were analysed by GC-ECD for quintozene, PCA, PCTA, PB, HCB, TCA, TCTASOO, and PCTASO using the CAM-24-73 method with an LOQ of 0.005 mg/kg for each analyte. Samples were stored for up to 147 days from sampling until analysis.

The analytical results indicate that, after the application of quintozene two times at a rate of 5.6 kg ai/ha, in most samples of succeeding wheat forage, grain and straw and turnip tops, no residues were expected to occur above the LOQ of 0.005 mg/kg. In lettuce (30-day PBI and 120-day PBI) from the GA site showed quintozene above the LOQ but at the maximum within 3 times of the LOQ value while lettuce samples from the TX site did not contain quintozene above the LOQ. PCA was detected higher than the LOQ in 30-day PBI lettuce, 365-day PBI wheat forage and turnip roots (only planted at 365-day PBI) at the maximum of 0.016 mg/kg which was significantly lower than the residues in potato as a result of the approved use of quintozene to potato.

Table 52 Summary of identification/characterization of radioactivity in succeeding crops at various PBI

Location In United States	Crop	Part	TRR (mg eq/kg)							
			quintozene	PB	HCB	PCTA	PCA	TCA	TCTASOO	PCTASO
30-day Plant-Back Interval										
Meigs, GA	Wheat	Forage	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
		Grain	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
		Straw	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
	Lettuce		0.0096 0.0120 0.0133 0.0126	4× <0.005	4× <0.005	4× <0.005	<0.005 0.0060 0.0092 0.0092	4× <0.005	4× <0.005	4× <0.005
Brookshire, TX	Wheat	Forage	4× <0.005	4× <0.005	4× <0.005	4× <0.005	3x <0.005 0.0070	4× <0.005	4× <0.005	4× <0.005
		Grain	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
		Straw	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
	Lettuce		4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005

Location In United States	Crop	Part	TRR (mg eq/kg)							
			quintozene	PB	HCB	PCTA	PCA	TCA	TCTASOO	PCTASO
120-day Plant-Back Interval										
Meigs, GA	Wheat	Forage	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
		Grain	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
		Straw	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
	Lettuce		0.0080, 0.0053, 0.0075, <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
Brookshire, TX	Wheat	Forage	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005
		Grain	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005
		Straw	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005
	Lettuce		2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005	2x <0.005
366- or 367-day Plant-Back Interval										
Meigs, GA	Wheat	Forage	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
		Grain	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
		Straw	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
	Lettuce		4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
	Turnip	Tops	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
		Roots	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	<0.005 0.0104 0.0063 0.0071	4× <0.005	4× <0.005
Brookshire, TX	Wheat	Forage	4× <0.005	4× <0.005	4× <0.005	4× <0.005	2x <0.005 0.0155 0.0074	4× <0.005	4× <0.005	4× <0.005
		Grain	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
		Straw	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
	Lettuce		4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
	Turnip	Tops	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005	4× <0.005
		Roots	4× <0.005	4× <0.005	4× <0.005		0.0139 0.0136 0.0087 0.0120	4× <0.005	4× <0.005	4× <0.005

Summary of rotational crop studies

The uptake of quintozene by rotational crops was investigated through confined rotational crop studies and field rotational study using lettuce, turnip and wheat.

In the confined rotational crop studies using the high application rates (up to 34.6 kg ai/ha pre-plant), similar metabolites as plant metabolism studies were identified showing complex metabolite profile, except that parent quintozene was either not found or found at low levels in rotational crops.

In the field study with unlabelled quintozene (2 × 5.6 kg ai/ha), quintozene, PB, HCB, PCTA or PCTASO were not found above the LOQ of 0.005 mg/kg in lettuce, turnips or wheat planted 365 days PBI. PCA and TCA were found in the turnip root and top samples above the LOQ but at the maximum 0.014 mg/kg; and PCA in wheat forage at the maximum 0.016 mg/kg. At higher application rate to the soil (such as 25 kg ai/ha on the United States label for cabbage and broccoli), residues in the rotational crops may be higher. The metabolism of quintozene in rotational crops seems to follow similar pathway as in the plant metabolism.

Animal metabolism

The Meeting received information on animal metabolism in lactating goat and laying hens, in addition to metabolism in rats.

Rat

Metabolism studies on laboratory animals including rats were reviewed in the framework of toxicological evaluation by the current JMPR and the relevant information is summarized below.

In the rat metabolism studies, the main metabolite found was PCA. The studies also mention some additional metabolites, found in low levels. These included: PCTA, methyl pentachlorophenyl sulfone. An additional metabolism study identified PCA and pentachlorophenyl-N-acetylcysteine (PCC) as the major metabolites in rat. There was insufficient information on the quantity. The metabolism study reports do also mention other structures found in the analyses, but these were not further identified.

Lactating goats

Study 1 *(Cheng, 1989, 900-RES-008 and Cheng, 1991, 900-RES-009(supplement to 900-RES-008))*

In a goat metabolism study, two goats (varieties not described) were dosed orally with ^{14}C-quintozene in capsules for five consecutive days at levels 25 or 50 mg/kg bw, equivalent to 714 or 947 ppm in diet. The goats were milked by hand twice daily. Urinary and faecal samples were collected twice daily. The animals were sacrificed approximately 6 hours after the final dose. Liver, kidney, fats, muscle, blood, bile and gastrointestinal tract were collected, and they were analysed for radioactivity. All samples except blood were stored below 0 °C before and after analysis. Radioactivity in each sample was analysed by LSC or combustion/LSC.

The administered radioactivity was eliminated in feces (36 and 41 percent of total dose), followed by urine (20–30 percent of total dose). A small amount was excreted in milk (0.2 and 0.3 percent of total dose). Within 48 hours after the first dose, only 10 percent of the total administered radioactivity was eliminated in excreta. TRR in milk reached a plateau on Day 3 pm (a maximum of 5.98 mg eq/kg for the low dose goat, and 3.55 mg eq/kg for the high dose goat) and declined on Day 5 (2.37 mg eq/kg for the low dose goat and 1.13 mg eq/kg at the time of sacrifice).

TRR in tissues were: 10.3 and 11.2 mg eq/kg in kidney, 13.4 mg eq/kg in liver, 0.48 and 0.54 mg eq/kg in muscle, and 9.4 and 15.6 mg eq/kg in the fat. The TRR in bile was high (30.8 and 56.9 mg eq/kg), indicating importance of biliary excretion although 0.02 and 0.03 percent of total administered dose was recovered in bile.

The nature of radioactive residue was examined in milk and edible tissues of a lactating goat receiving daily oral dose of 25 mg/kg bw for 5 consecutive days, as the TRR values from this goat were higher than those from the goat receiving an oral daily dose of 50 mg/kg bw. The liver (hereafter, liver-1) and milk were homogenized separately using chloroform/methanol (1:1, v/v) in the presence of a small amount of zinc sulfate. The mixture was centrifuged and separated. The liver chloroform extract was cleaned up through a preparative gel permeation column. The liver (liver-2), kidney fat and muscle were homogenized with a mixture of chloroform, methanol and water. The mixture was centrifuged and separated. The chloroform fraction was evaporated to dryness and the residue was partitioned between acetonitrile and hexane. TRR was examined for each extract and PES. A PES from liver-1 was refluxed in 5 mol/L KOH/methanol overnight. The mixture was neutralized using concentrated HCl. The inorganic salt was removed by filtration, and the filtrate was concentrated and

reconstituted in methanol for chromatography. For each tissue, the chloroform extract was subjected to HPLC and TLC. Quantitation of radioactive metabolites in each tissue was accomplished by comparing the retention times of peaks with those of analytical standards. For milk sample, quantitation was done with TLC. The chloroform extracts of liver, kidney, muscle and fat were composited, concentrated and run through a preparative HPLC column. Fractions were collected and pooled and concentrated and analysed by TLC or GC. The radioactive components were collected from the preparative HPLC eluates. The isolated components were analysed by MS or GC.

From milk, kidney, muscle and fat, the majority of the radioactivity (70.8 and 84.0 percent TRR) was extracted by chloroform. A lessor amount of radioactivity (4.27 and 18.0 percent TRR) was found in the methanol/water fraction. The PES contained 0 (fat)–29.9 percent TRR. For liver, liver 2 showed higher extraction efficiency. The chloroform fractions of liver-1 and liver 2 contained 24.2 percent and 31.4 percent TRR respectively, methanol/water fractions 5.8 percent and 29.5 percent TRR, and PES 69.4 percent and 42.8 percent TRR. The accountability of radioactivity in these tissues and milk ranged from 99.7 to 109 percent.

PCA was identified as the main metabolite in milk (50.2 percent TRR, 3.00 mg eq/kg), kidney (31.2 percent TRR, 3.21 mg eq/kg), muscle (59.2 percent TRR, 0.32 mg eq/kg), and fat (49.4 percent TRR, 7.71 mg eq/kg). PCTA was also identified but at lessor amounts: in milk (4.68 percent TRR, 0.280 mg eq/kg), kidney (2.78 percent TRR, 0.286 mg eq/kg), muscle (6.43 percent TRR, 0.035 mg eq/kg) and fat (1.48 percent TRR, 0.231 mg eq/kg). Five unknown polar metabolites, each accounting for <5 percent TRR, were also isolated from kidney, fat and/or muscle.

As for liver, only liver-1 extracts were subjected to identification/characterization. PCA and PCTA accounted for 9.6 percent (1.34 mg eq/kg) and 1.3 percent TRR (0.180 mg eq/kg), respectively. However, the majority (40 percent TRR) of the radioactivity in liver was associated with an unknown polar metabolite released by base hydrolysis.

In these tissues and milk, parent quintozene was not detected. The radioactivity in the methanol/water fractions and PES was not identified.

Study 2*. (Daun, 1990, 900-RES-075; Daun, 1991, 900-RES-153)*

Two lactating goats (variety not described) were dosed with ^{14}C-quintozene in gelatin capsule once daily for five consecutive days, one at 20 and the other at 50 mg/kg bw/day. Urinary and fecal samples were collected twice daily. The goats were milked by hand twice daily. Animals were sacrificed approximately 6 hours after the final dose. Tissues (kidney, liver, muscle, fat, blood, bile and gastrointestinal tract and its contents) were obtained. All samples, except blood were stored below 0 °C before and after analysis. Radioactivity in each sample was analysed by LSC or combustion/LSC.

The administered radioactivity was eliminated in urine (33 and 38 percent of total dose), feces (19 and 25 percent of total dose) and in milk (0.35 and 0.41 percent of the total dose). TRR in milk reached a plateau on Day 3 pm (a maximum of 5.98 mg eq/kg for the low dose goat, and 3.55 mg eq/kg for the high dose goat) and declined on Day 5 (2.4 mg eq/kg for the low dose goat and 1.1 mg eq/kg at the time of sacrifice). TRR in milk reached the highest level of 3.9 mg eq/kg on Day 2 in the low dose goat, and of 8.4 mg eq/kg on Day 2 in the high dose goat. Toward the end of the study, the TRR in milk slightly decreased. Only about 1 percent of total dose remained in tissues, bile and urine in bladder.

Among the tissues, the highest TRR was found in kidneys (32 and 49 mg eq/kg for the low dose and high dose goat, respectively) followed by liver (26 and 46 mg eq/kg). Fat contained 17–18 and 27–

33 mg eq/kg with renal fat contained slightly higher radioactivity than omental fat. Muscle contained much lower radioactivity at 1.1 and 2.3 mg eq/kg.

Samples of fat, kidney, liver and milk were extracted, and the extracts were analysed by TLC and HPLC for identification/characterization. Fat sample was initially extracted with chloroform by homogenization. The chloroform extract was concentrated to dryness under a stream of nitrogen and dissolved in hexane saturated with acetonitrile. The acetonitrile fraction was partitioned with acetonitrile saturated with hexane. The acetonitrile layer obtained after repeated partition was concentrated for HPLC.

Kidney sample from the low-dose goat was immersed in water, methanol and chloroform (1:2:1, v/v) by homogenization. The chloroform phase was partitioned with water. After removing the chloroform layer, to the remaining aqueous layer chloroform was added. The chloroform layer thus obtained was combined with the previously mentioned chloroform phase. The aqueous layer was also combined with the aqueous layer from the first extraction. The combined aqueous fraction was partitioned with methanol and chloroform to obtain the chloroform fraction and water fraction. The PES was subjected to Type 1 protease by immersing the PES in the protease solution at 37 °C for 20 hours, and then filtrate and solid were separated by filtration. The chloroform and aqueous fractions were analysed by HPLC. These extracts and the protease filtrate were analysed by TLC. Liver sample from the low-dose goat was extracted in a similar way as the kidney sample.

Subsamples of milk from the low dose goat were each extracted with hexane. The combined hexane fractions were partitioned with methanol/water (7:3). The aqueous fractions were applied to Amberlite XAD-2 column for elution by gradient using water and methanol (from 100 percent water to 100 methanol). Fractions were collected by polarity. The fraction eluted by 100 percent water was taken up in sufficient methanol to make a solution of methanol/water (7:3). The solution was partitioned with hexane. Methanol/water was added to facilitate separation. The hexane and aqueous layers were separately concentrated. The hexane fraction was combined with the initial hexane extract. The pooled methanol/water fractions from XAD-2 column were partitioned with hexane and the hexane fraction was also combined with other hexane fractions and then concentrated *in vacuo*. The concentrated hexane fractions were subjected to alumina column clean-up with elution with hexane and then benzene. The hexane eluate was concentrated and re-applied to the same column. The benzene fraction was combined with the benzene fraction from the first attempt. The combined benzene fraction was subjected to TLC.

Majority of radioactivity in kidney and liver from the goat that received the lower dose was extracted in chloroform and water: totaling 68 percent TRR for kidney and 60 percent TRR for liver with 16 percent and 46 percent TRR remaining in the respective PES. Protease hydrolysis released about one half of radioactivity in the kidney PES but only about 5 percent in liver PES. Majority of radioactivity in milk was extracted in hexane and methanol/water (55 percent of TRR) with 31 percent TRR remaining in the PES.

Table 53 Extraction of radioactive residues in the kidney, liver, milk and fat of goat ora2lly administered ^{14}C-quintozene for 5 days at 20 mg/kg bw

Fraction	% TRR		Fraction	% TRR
	Kidney	Liver		Milk
Chloroform	28.8	34.1	Hexane (initial)	32.4
Aqueous	38.8	26.1	Methanol/water 1	3.3
PES	16.4	46.4	Aqueous (initial)	51.1
Filtrate after Protease hydrolysis	10.0	4.8	Methanol/water 2	9.9
Solid after protease hydrolysis	12.8	27.3	Methanol/water 3	8.2
Total [a]	90.3	92.3	Hexane (final)	34.0

Fraction	% TRR	
	Kidney	Liver
Fraction	% TRR	
	Renal fat	Omental fat
Hexane	24.0	22.7
Acetonitrile	68.9	48.2
PES	1.6	1.7
Total	94.5	72.6

Fraction	% TRR
	Milk
PES	30.7
Total	86.1

Notes:

[a] Sum of chloroform fraction, aqueous fraction and filtrate and solid after protease hydrolysis.

In the kidney, analysis of the chloroform extracts identified parent quintozene (9.1 percent TRR, 2.92 mg /kg) as a major component, followed by 2,3,4,5-TCNB (0.5 percent TRR, 0.161 mg eq/kg). There were three unknown peaks each at <3 percent TRR. In the aqueous extracts, a small amount of parent quintozene was identified (0.5 percent TRR, 0.16 mg/kg). One unknown peak accounted for 14 percent TRR and 4.63 mg eq/kg.

In the liver, the chloroform extracts contained parent quintozene and PCA (together 8.3 percent TRR, 2.15 mg eq/kg), PCTA (5.42 percent TRR, 1.40 mg eq/kg), and 2,3,4,5-TCNB (1.0 percent TRR, 0.26 mg eq/kg). In the aqueous extracts, a small amount of parent quintozene (1.4 percent TRR, 0.36 mg/kg) and 2,3,4,5-TCNB (0.7 percent TRR, 0.18 mg eq/kg) were identified. One unknown component was observed at the same retention time as the unknown in the aqueous extracts of kidney at 15.9 percent TRR (4.12 mg eq/kg).

The radioactive residues in milk were not characterized.

***Study 3**. (McManus, 1989, 900-RES-070; McManus, 1990, 900-RES-092)*

One lactating goat was orally dosed with ^{14}C-quintozene for five consecutive days at a level 50 mg/kg bw.

TRR in milk was 58.5 mg eq/kg. The TRR in tissues were 49.1 mg eq/kg in kidney, 45.5 mg eq/kg in liver, 21.8 mg eq/kg in fat, and 2.22 mg eq/kg in muscle.

The liver, kidneys, fat and muscle (investigated separately in 900-RES-092) showed radioactive levels above background and extracted for analysis of nature of residues. Six metabolites were identified in the kidneys, of which two together accounted for more than 80 percent TRR. They were identified as PCA and PCA glucuronide. The four minor metabolites were PCTP, tetrachloro(methylthio)benzenethiol, TCTA and tetrachloromethylsulfinylaniline (TCA sulfoxide). Six metabolites were detected in the liver, mainly PCA and a PCA glucuronide conjugate. Trace amounts of four other products were found: PCPT dimer, *N*-pentachlorophenylhydroxylamine, PCTP and tetrachloro(methylthio) benzenethiol.

Milk, omental fat and renal fat each contained only one metabolite which was identified as PCA. Quintozene was not detected in the tissues, milk or urine. Pentachloroaniline sulfamate (85 percent of the extracted radioactivity (ER)) and pentachloroaniline mercapturic acid (4 percent of ER) were found only in urine along with small amount of *N*-pentachlorophenylhydroxylamine (5 percent of ER) and Tetrachloro(methylthio)benzenethiol (6 percent of ER).

Table 54 Metabolites in the tissues and milk of goat orally administered quintozene at 50 mg/kg bw for 5 consecutive days

Compound	percent of the extracted radioactivity					
	Kidney	Liver	Fat (omental)	Fat (renal)	Milk	Muscle
PCA	26	17	100	100	96	46
PCA-Gluc	55	73				
PCTA						11
PCTP	3.3	2.9				
PCPT dimer		2.7				
TCTA	2.1					
TCTA / Tetrachlorophenyl methyl sulfoxide						42
N-Pentachlorophenylhydroxylamine		4.7				
Tetrachloro(methylthio)benzenethiol	4.5	1				
Pentachloroaniline mercapturic acid						
Tetrachloromethylsulfinylaniline	2.3					

Laying hen

Study 1. *(Cheng, 1989, 900-RES-006; Cheng, 1991, 900-RES-007)*

Two groups of laying hens were dosed orally with ^{14}C-quintozene in capsules for five consecutive days at 25 or 50 mg/kg bw equivalent to 309 or 554 ppm in the diet. Eggs were collected twice daily. Birds were sacrificed approximately 6 hours after the final dose and blood, liver, kidneys, thigh muscle, breast muscle, fat, skin with fat, and GI tract with contents were collected.

The majority of the total administered dose was recovered in excreta (65 and 71 percent for low and high dose hens), followed by in GI tract and contents (6.5 and 9.2 percent), liver (0.03 and 0.04 percent) and kidneys (0.02 percent). Only small amount of administered radioactivity was found in eggs, a total of 0.01–0.02 percent in all eggs, most of which was in the yolk. The elimination rate of radioactivity in excreta was relatively fast and constant, with approximately 70 percent of the dose eliminated within 24 hours after the first dose.

The highest levels of radioactivity in egg yolks were found at sacrifice (1.22 and 2.66 mg eq/kg). On the contrary, the corresponding egg white contained radioactivity at 0.038 and 0.071 mg eq/kg while before sacrifice radioactivity was not detected.

The highest TRR was found in kidneys at 4.52 mg eq/kg (low dose) and 5.45 mg eq/kg (high dose) followed by fat (2.11 and 4.25 mg eq/kg), liver (1.97 and 2.42 mg eq/kg) and skin with fat (1.12 and 2.18 mg eq/kg). In muscle, the TRR was low at 0.13–0.16 mg eq/kg and 0.15–0.31 mg eq/kg. A significant amount of radioactivity was found in GI tract and its contents.

Table 55 Radioactive residues in tissues and blood of hens orally administered ^{14}C-quintozene for 5 days

Tissue	Dose at 25 mg/kg bw		Dose at 50 mg/kg bw	
	mg eq/kg	percent TAR	mg eq/kg	percent TAR
Kidneys	4.52	0.02	5.45	0.02
Liver	1.97	0.04	2.42	0.03
Muscle (thigh)	0.127	<0.01	0.145	<0.01
Muscle (breast)	0.155	0.01	0.305	0.01
Fat (abdominal)	2.11	0.02	4.25	0.04

Tissue	Dose at 25 mg/kg bw		Dose at 50 mg/kg bw	
	mg eq/kg	percent TAR	mg eq/kg	percent TAR
Skin with fat	1.12	0.03	2.18	0.04
GI tract/contents	114.000	6.45	298.000	9.22
Blood	1.47	<0.01	1.85	>0.01

Egg yolk, liver, kidney, fat and muscle were extracted by homogenizing the sample with mixture of chloroform, methanol and water. The mixture was centrifuged and separated. The chloroform fraction was evaporated to dryness, and then partitioned between acetonitrile and hexane. The methanol/aqueous fraction (except for fat sample) was cleaned up using C-18 column with elution with water, methanol, acetonitrile and hexane. The distribution of radioactivity in various fractions was determined in each sample. Egg white was not extracted due to its low radioactivity.

Kidney homogenate (no indication about whether they were from the lower dose or higher dose) was refluxed in 5 mol/L KOH/methanol solution overnight. The mixture was neutralized using 6 mol/L HCl. The inorganic salts were removed by filtration and the filtrate was concentrated and reconstituted in methanol for chromatographic analysis. A liver sample was also hydrolysed using 6 mol/L HCl at 100 °C for 16 hours. The mixture was cooled, dried and reconstituted in acetonitrile for HPLC analysis.

The majority of radioactivity in liver, kidney, fat and muscle was extracted (72–103 percent TRR), with a minor proportion (2.7–37 percent TRR) remaining in the PES. Among the extracts, the highest level of radioactivity was recovered in either methanol layer of methanol/water fraction of liver and kidney (60–65 percent TRR, 1.16–2.92 mg eq/kg), or acetonitrile layer of the chloroform fraction of muscle and fat (70–71 percent TRR, 0.11–1.50 mg eq/kg) and hydrolysates of liver and kidney (48–59 percent TRR). Acid hydrolysis of liver and base hydrolysis of kidney releases soluble radioactivity quantitatively.

Table 56 Extraction of radioactive residues in tissues and egg yolk of hens orally administered ^{14}C-quintozene for 5 days at 25 mg/kg bw

Tissue	% TRR					
	Dose at 25 mg/kg bw			Dose at 50 mg/kg bw		
	Extracted	PES	Total	Extracted	PES	Total
Liver	86.3	25.2	111.5	84.1	16.1	100.2
Liver after acid hydrolysis	97.1	NA	97.1	--	--	--
Kidney	72.3	37.1	110.3	--	--	--
Kidney after 2nd base hydrolysis	91.4	NA	91.4	--	--	--
Fat	103.5	2.74	106.2	--	--	--
Muscle (breast)	97.0	22.4	119.4			
Egg yolk (at sacrifice)	33.5	79.3	112.7	25.9	89.3	115.0

Notes:

NB: there was no description about whether the acid hydrolysed sample was from the high dose or low dose.

Selected fractions were subjected to HPLC and TLC. In muscle and fat, parent quintozene was the major radioactive residue, accounting for 55.8 percent TRR (0.086 mg/kg) in muscle and 59.8 percent TRR (1.26 mg/kg) in fat. The metabolites PCA and PCTA were also detected in muscle at 4.7 percent (0.007 mg eq/kg) and 2.1 percent TRR (0.003 mg eq/kg), respectively, and in fat at 6.4 percent (0.14 mg eq/kg) and 10.3 percent TRR (0.22 mg eq/kg), respectively.

In liver fractions, quintozene, PCA and PCTA each accounted for 3.2 percent, 0.45 percent and 1.5 percent TRR, respectively, and after acid hydrolysis, only quintozene (3.8 percent TRR) and PCA (6.5 percent TRR) were detected. In kidney fractions, also quintozene, PCA and PCTA were detected, each accounting for 0.80 percent (0.063 mg/kg), 2.7 percent (0.12 mg eq/kg) and 0.23 percent (0.01 mg eq/kg) of TRR, respectively. After base hydrolysis, quintozene, PCA and PCTA were detected with slightly different ratio (quintozene at 2.4 percent TRR, PCA at 4.3 percent and PCTA at 1.9 percent TRR). The majority of the radioactivity in liver and kidney was not adequately identified/characterized.

Study 2. *(Parkins, 1990, 900-RES-090; Parkins, 1991, 900-RES-098)*

Three groups (five hens/group) of laying hens were dosed orally with ^{14}C-quintozene in capsule for six consecutive days at 15.8, 39.4 and 78.9 mg/hen/d, equivalent to 105 (low dose), 273 (medium dose) or 512 ppm (high dose) in the diet. Egg and excreta samples were collected daily and pooled. The birds were sacrificed approximately 6 hours after the final dose. Samples of blood, abdominal fat, skin with fat, muscle (thigh and breast), liver and kidney were taken and pooled by group.

The majority of the radioactivity was eliminated in the excreta (87–94 percent of the administered dose). The distribution of radioactivity was similar for all three treatment groups while as the administered dose was higher, TRRs were also higher. The highest TRR was found in fat in these three groups, followed by kidneys, skin with fat and egg yolk. The TRRs in muscles and egg white were much lower (Table 57)

Table 57 Radioactive residues in tissues and eggs of hens orally administered ^{14}C-quintozene for 5 days

Tissue	TRR (mg eq/kg)		
	Low dose	Medium dose	High dose
Liver	0.87	2.72	3.81
Kidneys	1.84	5.05	7.29
Muscle (Thigh)	0.13	0.36	0.71
Muscle (Breast)	0.07	0.17	0.30
Fat	2.64	6.17	10.1
Skin with fat	1.68	3.75	5.92
Egg yolk (Day 5)	1.74	3.52	5.75
Egg white (Day 5)	0.06	0.24	0.29

The tissues and egg yolks were extracted by blending with the proper amounts of chloroform, methanol and water. The chloroform layers were separated from the methanol/water layers. For cleaning up prior to HPLC, solid phase extractions were used. Fat extracts were cleaned up using SPE with Florisil and elution with hexane, and then 25, 50 and 75 percent dichloromethane in hexane, and finally with dichloromethane. Over 90 percent of the radioactivity was recovered in the first hexane eluate. C-18 SPE were used for methanol/water fractions of liver, and elution with water, and methanol. Approximately 75 percent of the radioactivity was in the methanol eluate.

Chromatographic separations were conducted using an HPLC system with reverse phase column and UV and radiological detectors. Mass spectra of isolated samples were obtained using mass spectrometer through the ionization (both positive and negative) source with a direct exposure probe, by thermospray, or by GC.

In fat, quintozene was identified as the major residue, accounting for 48 percent of the extracted radioactivity (ER). Other major metabolites identified in fat included PCA (16 percent ER) and tetrachloromethylsulfinylaniline (31 percent ER). In liver, PCTP (71 percent ER) and PCTASO (21 percent

ER) were identified as the major solvent-soluble residues. The major radioactive residues in base hydrolysates (37 percent TRR) of liver PES were identified as PCA and PCTA. In egg yolks, the majority of radioactivity (75 percent TRR) remained unextracted. Base hydrolysis of the egg yolk PES released 29 percent of the TRR, which was composed primarily of PCTA, along with trace amounts of PB, HCB, quintozene and PCA. In total, from the egg yolk, PCA (70 percent ER), PCTA (9 percent ER), and PCTP (18 percent ER) were identified. In muscle, the major radioactive residue was either PCP thioacetate or TCTA sulfone (88 percent ER), along with minor amount of PCTA (8 percent ER).

PCP-thiopyruvate and PCP-MalCys were only found in excreta at 26 and 19 percent ER respectively.

Table 58 Radioactive metabolites in tissues and eggs of hens orally administered ^{14}C-quintozene for 5 days

Compound	percent of extracted radioactivity					
	Fat	Liver	Kidney	Muscle	Egg yolk	Excreta [b]
Quintozene	48					
N-pentachlorophenylhydroxylamine			50			
PCA	16				70	
Tetrachloromethylsulfinylaniline	31					
PB					4	
PCTA				8	9	
PCTASO		21				
PCTP		71			18	30
PCP-Cys			8			
PCP thioacetate		7	35	88 [a]		17
TCTA	1					
TCTA sulfone			7	[a]		

Notes:

[a] Either PCP thioacetate or TCTA sulfone.

[b] Excluding those metabolites found only in the excreta.

Summary of animal metabolism

The metabolism of quintozene was investigated in lactating goats and laying hens. In general, the metabolic pathways in these species were similar to that in rats.

In goats, quintozene was metabolized mainly to PCA and its glucuronide conjugates (100 percent TRR in fat, 96 percent TRR in milk, 17 percent TRR in liver, and 26 percent TRR in kidney). Other metabolites were formed in much smaller amounts. They were TCTA, PCTA, C4MX. Parent quintozene was not detected in any of the tissues.

In hens, PCA, PCTA, PCTP, PCTP conjugated with cysteine, malonylcysteine, pyruvate and acetate were identified. Other metabolites identified included TCTA, TCTASOO, PCTASO and tetrachloromethylsulfinylaniline. Parent quintozene was detected only in fat at 48 percent ER.

The major metabolic pathway in animals involves (1) displacement of the nitro group by the sulfhydryl group of glutathione or SH-containing amino acids/peptides, followed by catabolic cleavage of the peptide, or by hydroxyl group; (2) reduction of the nitro group to produce N-hydroxypentachloroaniline and conjugated PCA; (3) dichlorination to yield tetrachloro- and trichloro-phenyl compounds. The pathway has some commonality with the metabolism in plants.

The proposed metabolic pathway for animals is shown in Figure 3, below.

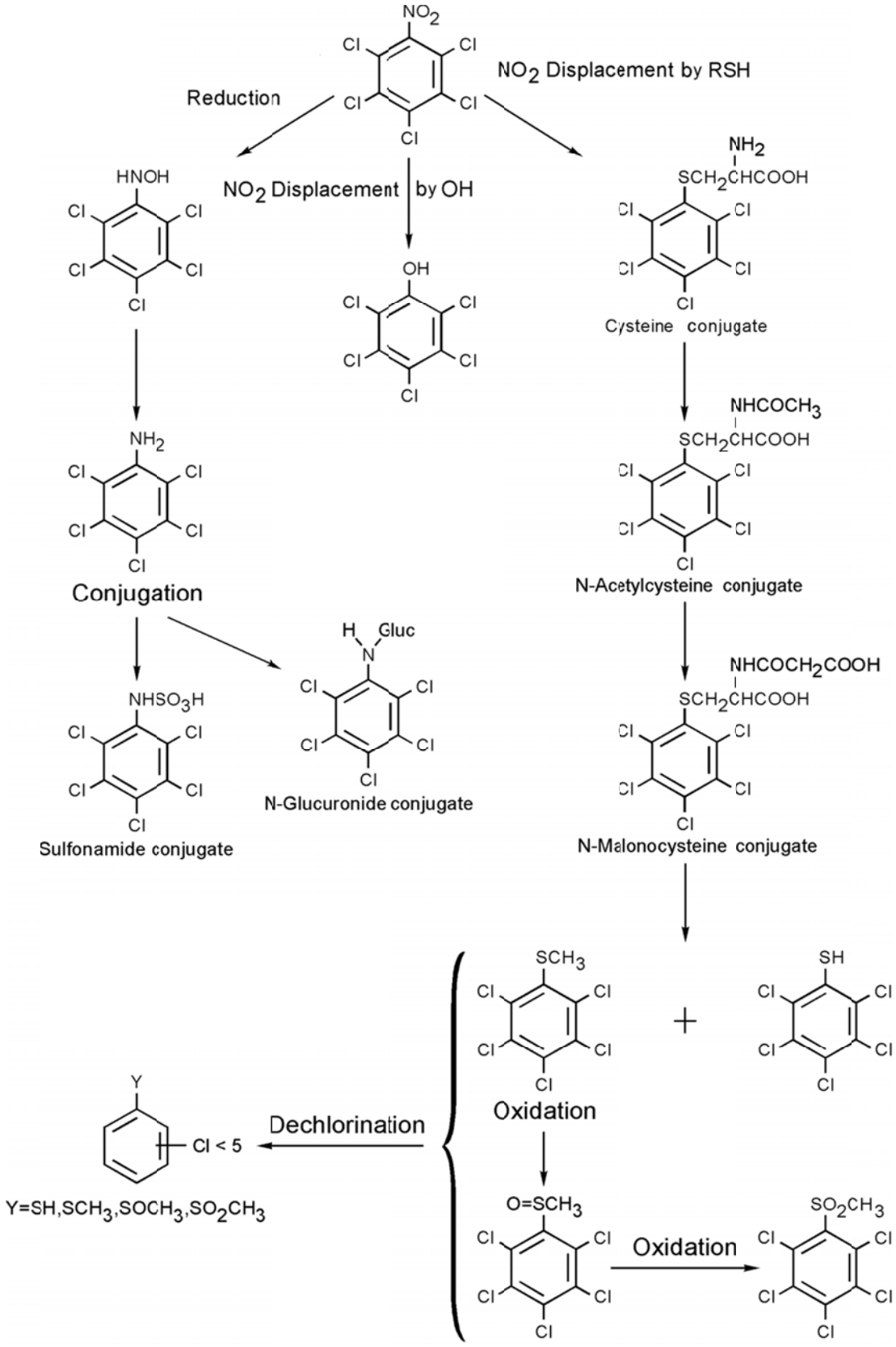


Figure 3 Proposed metabolic pathway of quintozene in lactating goat and laying hens after oral administration for 5 or 6 consecutive days

RESIDUE ANALYTICAL METHODS

The Meeting received information on: analytical methods developed and validated for the determination of quintozene and its metabolites in plant and animal matrices; and frozen storage stability of quintozene and its metabolites in high water content, high oil content, high protein content and high starch content category matrices.

Analytical Methods for Determination of Quintozene Residues–plant commodities

For data generation

CAM-24-73 Method (GC-ECD Method)(Griffith, 1973, 900-ANM-062, Vol.1)

Analyte: Quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB
Matrix: Cabbage, pepper, tomato, peanut (nutmeat and hulls)(applicable to various plant commodities)
LOQ: 0.01 mg/kg
Description: Cabbage, pepper, tomato
Ground samples (50 g) were extracted with 2-propanol/hexane (50:50; v/v) by homogenization. After filtration, the extract was washed 3-times with 5 percent sodium chloride solution. A 2.5-mL aliquot of the hexane phase was cleaned-up on a deactivated silica gel column. The hexane eluate was evaporated to 2.5 mL and residues of quintozene and its metabolites were determined by GC-ECD.
Peanut nutmeat and hulls
Ground samples (10 g) were extracted with hexane (3x 75 mL for nutmeat, 1x 100 mL for hulls) by homogenization. After centrifugation, the combined extracts were evaporated to approximately 5-mL. The extract of nutmeat was partitioned three times into acetonitrile and combined acetonitrile extracts were diluted with water and partitioned two times into petroleum ether. Extracts of nutmeat and also hulls were cleaned-up by gel permeation chromatography prior to determination of residues of quintozene and its metabolites by GC-ECD.

CAM-24-73 Method (modified 1) (GC-ECD Method)(no author name, 1988, 900-ANM-080; Yu, 1992, 900-RES-194)
Analyte: Quintozene, PCA, PCTA, PB, HCB
Matrix: Cabbage
LOQ: 0.001 mg/kg
Description: Ground samples (100 g) were extracted with 2-propanol/hexane (50:50; v/v; 200 mL) by blending for 4 minutes. After filtration, the extract was washed three times with 5 percent sodium chloride in water (1 L). The hexane layer was dried over sodium sulfate. An aliquot of the extract was cleaned-up on a Florisil column by elution with hexane. The cleaned-up extract was evaporated and adjusted to 5 mL. Residues of quintozene and its metabolites were determined by GC-ECD.

CAM-24-73 Method (modified 2) (GC-ECD Method)(Gaydosh, 1993, 900-RES-107; 1994, 900-RES-108; and 1992, 900-ANM-107)
Analyte: Quintozene, PCA, PCTA, PB, HCB
Matrix: Green bean, dry bean, cotton
LOQ: 0.0005 mg/kg

Description: Ground samples (e.g., 10 g) were extracted with acetone/hexane (50:50; v/v; 200 mL) by shaking for 20 minutes. After filtration, the extract was washed two times with water (adjusted to pH 12 with sodium hydroxide), which was extracted with hexane. The combined hexane layers were evaporated to 10 mL which was cleaned-up on a Florisil column by elution with ether/hexane (5:95; v/v). The cleaned-up extract was evaporated to 5 mL and the solvent was exchanged against toluene. Residues of quintozene and its metabolites were determined by GC-ECD.

CAM-24-73 Method (modified 3) (GC-ECD Method) (Gaydosh, 1994, 900-RES-149)

Analyte: Quintozene, PCA, PCTA, PB, HCB

Matrix: Lettuce, turnip roots and tops, wheat (whole plant, grain and straw)

LOQ: 0.005 mg/kg

Description: Ground samples (10 g; 5g for turnip tops) were extracted with acetone/hexane (50:50; v/v; 200 mL) by shaking for 20 minutes. After filtration, the extract was washed two times with water (adjusted to pH 12 with sodium hydroxide), which was extracted with hexane. The combined hexane layers were evaporated to 10 mL, which was cleaned-up on a Florisil column by elution with acetone/hexane (12:88; v/v). The cleaned-up extract was evaporated to 5 mL and the solvent was exchanged against toluene (10 mL). Residues of quintozene and its metabolites were determined by GC-ECD.

CAM-24-73 Method (modified 4) (GC-MS and GC-ECD Method)(Maselli, 1997, 900-RES-147)

Analyte: Quintozene, PCA, PCTA, PB, HCB

Matrix: Cotton (RAC and its processed commodities)

LOQ: 0.005 mg/kg

Description: Ground samples (10 g) were extracted three times by blending with hexane and anhydrous sodium sulfate. After filtration, the combined extract was reduced to the added keeper volume (iso-octane, 2 mL) and adjusted to volume with cyclohexane/dichloromethane (50:50; v/v). An aliquot of this solution is cleaned-up by gel-permeation chromatography. The solvent of the cleaned extract was exchanged against hexane, and the extract was cleaned-up on a Florisil column by elution with 3 percent ethyl ether in petroleum ether. The cleaned-up extract was evaporated to a volume of 2 mL for cotton seed or 4 mL for gin trash. Residues of quintozene and its metabolites were determined by GC-MS (cotton seed) or GC-ECD (gin trash).

MP-PCNC-MA1 method (GC-ECD Method)(LeRoy, 1989, 900-ANM-007)

Analyte: Quintozene, PCA, PCTA, PB, 2,3,4.5-TCNB, 2,3,5,6-TCNB, HCB

Matrix: Tomato, snap bean, potato, peanut (whole nut, shell, nutmeat)

LOQ: 0.005 mg/kg

Description: Samples were dried with anhydrous sodium sulfate and were extracted with ethyl acetate. The ethyl acetate extract was concentrated (not to dry up). To the concentrate, isooctane was added, and the mixture was concentrated. The concentrated extract was diluted with isooctane or petroleum ether and then cleaned up using Florisil column chromatography. Samples with high liquid content were cleaned up using gel permeation chromatography before Florisil column chromatography. The eluate (elution with 3 percent ethyl ether in petroleum ether solution) was concentrated, to which isooctane was added. The final solution was subjected to analysis for quintozene and its metabolites by GC-ECD.

MP-PCNC-MA2 Method (GC-ECD Method)(LeRoy, 1991, 900-RES-019, 900-RES-020, 900-RES-024, and 900-RES-026; and LeRoy and Cassidy, 1991, 9000-RES-032)

Analyte: Quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB
Matrix: Cabbage, broccoli, pepper, tomato, cotton processed fractions
LOQ: 0.01 mg/kg
Description: Pre-homogenized samples were extracted with acetonitrile/water (approximately 2:1, v/v), followed by a petroleum ether/water partitioning step. The combined petroleum ether layers were concentrated and cleaned-up on a Florisil column. The cleaned-up extract was concentrated. The solvent was exchanged with isooctane, and residues of quintozene and its metabolites were determined by GC-ECD.

Zweig Method (published)(GC-MS Method)(Thornton M., Feiler B.; 900-RES-030; "Analytical Methods for Pesticides and Plant Growth Regulators", ed by Sherma and Zweig, Volume VI, pp 578–580, 1972, Academic Press)

Analyte: Quintozene
Matrix: Potato tuber
LOQ: 0.01 mg/kg
Description: Samples were extracted with a mixture of isopropanol and hexane, the extracts were filtered and then washed with 5 percent NaCl, dried with Na_2SO_4 and passed through a silica gel column for clean-up. Eluate was collected and evaporated to approximately 1 ml and transferred for analysis. Residues of quintozene were determined by GC-MS by monitoring the ions *m/z* = 293, 295 and 249.

For monitoring

Battelle 100117568 Method (QuEChERS extraction + GC-MS method)(Thorn J., 2019, 900-RES-223)

Analyte: Quintozene, PCA, PCTA
Matrix: Broccoli, potato tuber
LOQ: 0.01 mg/kg
Description: A sample of 10 g is transferred into a 50 ml centrifuge tube to which acetonitrile (10 mL) was added for extraction by high-speed homogenization. After homogenization, the content of a QuEChERS Q-Sep Q110 package (4g $MgSO_4$, 1g NaCl, 1g TSCD, 0.5g DHS) was added. The sample was capped, shaken and high-speed homogenization was repeated. After centrifugation, 1 ml of the acetonitrile layer was transferred into the dispersive SPE tube and shaken for 2 minutes followed by centrifugation. An aliquot of the extract was transferred for analysis by GC-MS utilizing electronic ionization in Selected Ion Monitoring mode, monitoring the following three ions for detection of each analyte.

Quintozene: m/z = 295, 297, 293
PCA: m/z = 265, 267, 26
PCTA: m/z = 296, 294, 246

<u>*Analytical methods for animal commodities*</u>

For data generation

CAM-1-69 Method (GC-ECD Method)(Griffith, 1973, 900-ANM-062, Vol. 3)

Analyte: Quintozene, PCA, PB, HCB
Matrix: Milk
LOQ: 0.001 mg/kg for quintozene

Description: An aliquot of the milk sample was diluted with acetone and filtered into a separating funnel. Hexane, water and sulfuric acid were added and residues partitioned into the hexane phase. The hexane layer was dried over sodium sulfate, reduced to 25 mL using a stream of nitrogen and passed through a Florisil column for clean-up. An aliquot of the hexane extract was taken for determination of residues by GC-ECD.

Method for cow tissues (not numbered) (GC-ECD Method)(Griffith et al., 1969, 900-ANM-055)

Analyte: Quintozene, PCA, PB, PCTA, HCB

Matrix: Cow fat, kidney, liver, muscle(meat)

LOQ: 0.003–0.05 mg/kg

Description: Cow fat, kidney and liver

Samples were homogenized with acetonitrile containing sodium sulfate. An aliquot of the extract was diluted 1:1 with water and partitioned into hexane. After centrifugation, the hexane phase was dried over sodium sulfate in a separator funnel. The hexane phase was then used for determination of quintozene, its metabolites and HCB by GC-ECD.

Cow muscle

Samples were extracted by homogenization with hexane containing sodium sulfate. After separation of the solids, an aliquot of the hexane extract was taken for determination of quintozene, its metabolites and HCB by GC-ECD.

CAM-39-75 Method (GC-ECD Method)(Griffith, 1973, 900-ANM-062, Vol.2)

Analyte: Quintozene, PCA, PCTA, PB, HCB

Matrix: Poultry fat, liver, meat and egg

LOQ: 0.002–0.01 mg/kg

Description: Poultry fat, liver, egg white and egg yolk

Samples were extracted by homogenization with acetonitrile. After separation of the solids, an aliquot of the extract was transferred into a separating funnel. Egg white only was additionally diluted with water. Residues were partitioned into hexane. The hexane phase was dried over sodium sulfate. An aliquot of the hexane extract was taken for determination of residues by GC-ECD.

Poultry meat

Samples were extracted by homogenization with hexane containing sodium sulfate. After separation of the solids, an aliquot of the extract was taken for determination of residues by GC-ECD.

Method validation

Recovery data for the above methods are summarized in the following tables.

Table 59 Summary of recovery data of the above-mentioned methods for determination of quintozene and its metabolites in plant commodities

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
CAM-24-73 Method							
Cabbage (head)	Quintozene	0.05	2	81–90	86	--	900-ANM-106
		1.0	2	96–100	98	--	
		2.0	2	86–94	90	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCA	0.05	2	71–73	72	--	
		1.0	2	83–91	87	--	
		2.0	2	73–82	78	--	
	PCTA	0.001	2	105–115	110	--	
		0.005	2	112–114	113	--	
		0.05	2	89–98	94	--	
	PB	0.001	2	80–106	93	--	
		0.005	2	93–94	94	--	
		0.05	2	74–88	81	--	
	HCB	0.0005	2	85–118	102	--	
		0.005	2	113–114	114	--	
		0.05	2	81–90	86	--	
Pepper	Quintozene	0.05	2	87–93	90	--	900-ANM-106
		1.0	2	76–79	78	--	
		2.0	2	83–85	84	--	
	PCA	0.05	2	88–94	91	--	
		1.0	2	72–74	73	--	
		2.0	2	74–78	76	--	
	PCTA	0.0005	2	98–107	103	--	
		0.005	2	102–107	105	--	
		0.05	2	98–101	100	--	
	PB	0.0005	2	93–109	101	--	
		0.005	2	111–111	111	--	
		0.05	2	86–87	87	--	
	HCB	0.0005	2	98–109	104	--	
		0.005	2	89–94	92	--	
		0.05	2	87–90	89	--	
Tomato	Quintozene	0.05	2	95–114	105	--	900-ANM-106
		1.0	2	106–114	110	--	
		2.0	2	98–119	109	--	
	PCA	0.05	2	95–109	102	--	
		1.0	2	99–116	108	--	
		2.0	2	91–120	106	--	
	PCTA	0.0005	2	114–120	117	--	
		0.005	2	107–119	113	--	
		0.05	2	100–100	100	--	
	PB	0.0005	2	103–114	109	--	
		0.005	2	98–117	108	--	
		0.05	2	79–88	84	--	
	HCB	0.0005	2	82–120	101	--	
		0.005	2	92–101	97	--	
		0.05	2	91–93	92	--	
Peanut (nutmeat)	Quintozene	0.05	2	78–89	84	--	900-ANM-106
		1.0	2	72–74	73	--	
		2.0	2	70–71	71	--	
	PCA	0.05	2	84–120	102	--	
		1.0	2	80–83	82	--	
		2.0	2	77–77	77	--	
	PCTA	0.001	2	76–101	89	--	
		0.005	2	119–120	120	--	
		0.05	2	78–83	81	--	
	PB	0.001	2	76–81	79	--	
		0.005	2	106–114	110	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
		0.05	2	70–78	74	--	
	HCB	0.0005	2	102–104	103	--	
		0.005	2	112–118	115	--	
		0.05	2	73–81	77	--	
Peanut (hulls)	Quintozene	0.05	2	71–71	71	--	900-ANM-106
		1.0	2	78–86	82	--	
		2.0	2	76–86	81	--	
	PCA	0.05	2	76–77	77	--	
		1.0	2	80–91	86	--	
		2.0	2	79–92	86	--	
	PCTA	0.001	2	81–88	85	--	
		0.005	2	78–82	80	--	
		0.05	2	74–75	75	--	
	PB	0.001	2	104–109	107	--	
		0.005	2	79–89	84	--	
		0.05	2	70–75	73	--	
	HCB	0.0005	2	74–74	74	--	
		0.1	2	83–108	96	--	
		0.2	2	70–78	74	--	
Wheat	Quintozene	0.005	2	160, 140	150	--	900-RES-095
		0.025	2	88, 88	88	--	
		0.05	2	72, 76	74	--	
	PCA	0.005	2	72, 72	72	--	
		0.025	2	80, 80	80	--	
		0.05	2	72, 76	74	--	
	PCTA	0.005	2	88, 88	88	--	
		0.025	2	80, 72	76	--	
		0.05	2	74, 78	76	--	
	PB	0.005	2	72, 72	72	--	
		0.025	2	72, 72	72	--	
		0.05	2	76, 76	76	--	
	HCB	0.005	2	72, 72	72	--	
		0.025	2	72, 72	72	--	
		0.05	2	72, 76	74	--	
CAM-24-73 (modified 2) Method							
Snap Bean	Quintozene	0.0005	4	100	100	0.0	900-RES-107
		0.1	4	96–102	99	2.6	
		0.2	4	92–100	97	3.6	
	PCA	0.0005	4	100–120	115	8.7	
		0.1	4	98–106	103	3.3	
		0.2	4	94–102	98	3.7	
	PCTA	0.0005	4	80–120	90	22	
		0.1	4	84–94	89	4.7	
		0.2	4	86–96	89	5.3	
	PB	0.0005	4	100–120	105	9.5	
		0.1	4	80–98	83	2.3	
		0.2	4	80–84	82	2.8	
	HCB	0.0005	4	80–100	85	12	
		0.1	4	82–98	97	1.5	
		0.2	4	92–96	94	2.0	
Dry Bean	Quintozene	0.0005	3	80–120	93	25	900-RES-108
		0.1	4	90–96	93	2.8	
		0.2	4	86–98	91	5.8	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCA	0.0005	2	80, 100	90	--	
		0.1	4	92–96	94	2.0	
		0.2	4	84–98	90	6.5	
	PCTA	0.0005	4	100–100	100	0	
		0.1	4	90–104	98	7.7	
		0.2	4	86–98	91	5.5	
	PB	0.0005	4	80–120	105	18	
		0.1	4	90–92	91	1.1	
		0.2	4	84–90	87	3.0	
	HCB	0.0005	4	100–100	100	0	
		0.1	4	88–90	89	1.3	
		0.2	4	84–90	86	3.3	
Cotton	Quintozene	0.002	6	70–98	91	11	900-ANM-107
		0.01	6	71–88	80	8.3	
		0.1	6	77–84	81	4.5	
	PCA	0.002	6	73–99	85	10	
		0.01	6	69–90	78	11	
		0.1	6	71–88	77	8.5	
	PCTA	0.002	6	75–87	79	6.7	
		0.01	6	71–85	77	7.1	
		0.1	6	69–79	75	5.5	
	PB	0.002	6	93–108	102	5.0	
		0.01	6	82–89	84	3.3	
		0.1	6	79–82	81	1.6	
	HCB	0.002	6	61–90	78	13	
		0.01	6	71–81	77	5.6	
		0.1	6	70–82	77	6.5	
CAM-24-73 (modified 3) Method							
Wheat (forage)	Quintozene	0.005	4	84–114	103	13	900-RES-162
		0.05	4	86–91	88	2.3	
		1.0	4	92–96	94	1.8	
	PCA	0.005	4	116–126	120	4.1	
		0.05	4	84–89	87	2.6	
		1.0	4	86–94	91	3.9	
	PCTA	0.005	4	106–124	114	6.9	
		0.05	4	79–83	81	2.1	
		1.0	4	87–90	88	1.4	
	PB	0.005	4	102–120	110	7.1	
		0.05	4	85–93	87	4.4	
		1.0	4	89–92	90	1.7	
	TCA	0.005	4	92–120	106	11	
		0.05	4	84–109	98	12	
		1.0	4	90–94	92	2.1	
	TCTASO	0.005	4	96–108	102	6.3	
		0.05	4	76–89	82	8.0	
		1.0	4	86–101	92	7.0	
	TCTASOO	0.005	4	94–126	111	13	
		0.05	4	93–105	101	5.4	
		1.0	4	87–98	91	5.2	
	HCB	0.005	4	92–124	109	16	
		0.05	4	97–98	98	0.6	
		1.0	4	90–92	91	1.0	
Wheat	Quintozene	0.005	4	56–74	68	12	900-RES-162

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
(Grain)		0.05	4	72–79	76	4.1	
		1.0	4	77–83	81	3.3	
	PCA	0.005	4	70–76	74	3.8	
		0.05	4	92–98	95	2.6	
		1.0	4	85–89	88	2.2	
	PCTA	0.005	4	82–84	83	1.4	
		0.05	4	81–87	84	3.2	
		1.0	4	83–88	86	2.5	
	PB	0.005	4	88–98	94	4.7	
		0.05	4	83–85	84	1.0	
		1.0	4	81–85	83	2.2	
	TCA	0.005	4	72–102	87	16	
		0.05	4	84–90	88	3.0	
		1.0	4	83–87	86	2.2	
	TCTASO	0.005	4	82–88	87	3.5	
		0.05	4	73–80	77	4.6	
		1.0	4	75–81	79	3.3	
	TCTASOO	0.005	4	78–84	81	3.2	
		0.05	4	66–86	74	12	
		1.0	4	81–85	83	2.3	
	HCB	0.005	4	70–78	73	5.2	
		0.05	4	75–78	76	2.0	
		1.0	4	80–84	83	2.1	
Wheat (straw)	PB	0.005	4	76–88	84	6.3	900-RES-162
		0.05	4	78–89	83	5.5	
		1.0	4	79–85	83	3.3	
	TCA	0.005	4	68–90	82	12	
		0.05	4	80–89	85	4.4	
		1.0	4	82–87	86	2.8	
	HCB	0.005	4	58–86	77	17	
		0.05	4	73–80	76	4.3	
		1.0	4	77–87	82	5.0	
	Quintozene	0.005	4	58–74	67	13	
		0.05	4	65–75	70	5.9	
		1.0	4	74–79	77	3.2	
	PCA	0.005	4	76–106	97	14	
		0.05	4	76–105	89	14	
		1.0	4	82–88	86	3.3	
	PCTA	0.005	4	72–94	83	14	
		0.05	4	74–83	79	4.7	
		1.0	4	81–86	84	2.8	
	TCTASOO	0.005	4	64–84	79	12	
		0.05	4	68–80	76	7.0	
		1.0	4	77–86	82	4.9	
	TCTASO	0.005	4	56–80	65	17	
		0.05	4	63–73	68	6.2	
		1.0	4	66–80	74	7.9	
Lettuce	PB	0.005	4	78–102	92	11	900-RES-162
		0.05	4	85–94	89	4.6	
		1.0	4	81–88	86	3.9	
	TCA	0.005	4	90–98	94	3.7	
		0.05	4	87–98	91	5.7	
		1.0	4	83–90	88	3.6	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	HCB	0.005	4	80–86	83	3.1	
		0.05	4	85–91	88	3.0	
		1.0	4	85–89	87	2.0	
	Quintozene	0.005	4	74–96	82	12	
		0.05	4	86–92	88	3.1	
		1.0	4	89–92	91	1.7	
	PCA	0.005	4	76–92	84	8.4	
		0.05	4	83–89	85	3.2	
		1.0	4	88–92	90	2.3	
	PCTA	0.005	4	88–120	102	15	
		0.05	4	74–79	76	2.9	
		1.0	4	78–81	79	1.9	
	TCTASOO	0.005	4	84–96	89	5.9	
		0.05	4	86–102	94	7.8	
		1.0	4	87–90	89	1.7	
	TCTASO	0.005	4	98–120	110	8.3	
		0.05	4	94–104	100	4.3	
		1.0	4	89–100	93	5.6	
Turnip (tops)	PB	0.005	4	94–100	96	2.9	900-RES-162
		0.05	4	78–86	82	4.1	
		1.0	4	80–86	84	3.1	
	TCA	0.005	4	80–88	83	4.6	
		0.05	4	83–92	87	4.3	
		1.0	4	87–92	90	2.5	
	HCB	0.005	4	66–106	84	19.9	
		0.05	4	80–88	84	4.1	
		1.0	4	79–86	83	3.8	
	Quintozene	0.005	4	70–84	77	8.6	
		0.05	4	66–97	85	16	
		1.0	4	92–99	96	3.2	
	PCA	0.005	4	74–78	77	2.5	
		0.05	4	72–96	87	12	
		1.0	4	89–107	96	8.5	
	PCTA	0.005	4	82–88	85	3.0	
		0.05	4	85–93	90	4.0	
		1.0	4	92–96	95	1.8	
	TCTASOO	0.005	4	70–108	85	20	
		0.05	4	93–105	98	5.7	
		1.0	4	93–102	97	4.6	
	TCTASO	0.005	4	102–118	111	6.3	
		0.05	4	79–120	100	17	
		1.0	4	78–115	92	19	
Turnip (roots)	PB	0.005	4	74–104	87	15	900-RES-162
		0.05	4	81–90	86	4.9	
		1.0	4	82–83	83	0.6	
	TCA	0.005	4	100–118	111	7.3	
		0.05	4	91–94	92	1.4	
		1.0	4	88–90	89	1.1	
	HCB	0.005	4	102–108	106	2.4	
		0.05	4	84–89	87	2.8	
		1.0	4	84–111	94	13	
	Quintozene	0.005	4	70–108	82	22	
		0.05	4	75–78	77	1.7	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
		1.0	4	82–93	86	5.8	
	PCA	0.005	4	70–100	86	16	
		0.05	4	77–79	78	1.0	
		1.0	4	82–87	85	2.6	
	PCTA	0.005	4	82–92	85	5.6	
		0.05	4	80–83	82	1.5	
		1.0	4	85–90	88	2.7	
	TCTASOO	0.005	4	78–88	83	7.0	
		0.05	4	73–79	76	3.9	
		1.0	4	74–85	82	6.4	
	TCTASO	0.005	4	88–116	106	12	
		0.05	4	99–103	102	1.7	
		1.0	4	78–91	85	6.9	
CAM-24-73 (modified 4) Method							
Cotton (seeds)	Quintozene	0.0005	3	80–100	87	13	900-RES-114
		0.10	3	86–94	89	5.2	
		0.25	3	88–90	89	1.3	
	PCA	0.0005	3	100–120	113	10	
		0.10	3	88–100	93	6.5	
		0.25	3	88–94	91	3.4	
	PCTA	0.0005	3	80–100	87	13	
		0.10	3	78–88	83	6.1	
		0.25	3	84–96	90	6.7	
	PB	0.10	3	78–80	79	1.5	
		0.25	3	74–78	75	3.1	
	HCB	0.0005	3	80–100	93	12	
		0.10	3	80–84	83	2.8	
		0.25	3	82–88	85	3.6	
Cotton (meal)	Quintozene	0.0005	3	100–120	107	11	900-RES-114
		0.10	3	82–88	85	3.6	
		0.25	3	86–88	87	1.3	
	PCA	0.0005	3	40–100	73	42	
		0.10	3	76–86	82	6.5	
		0.25	3	86–86	86	0.0	
	PCTA	0.0005	3	80–120	93	25	
		0.10	3	76–84	80	5.0	
		0.25	3	80–82	81	1.4	
	PB	0.0005	3	100–120	107	11	
		0.10	3	80–86	83	3.7	
		0.25	3	80–82	81	1.4	
	HCB	0.0005	3	120–140	127	9.1	
		0.10	3	79–82	81	2.1	
		0.25	3	78–80	79	1.5	
Cotton (hulls)	Quintozene	0.0005	3	80–100	93	12	900-RES-114
		0.10	3	74–80	77	4.0	
		0.25	3	84–88	87	2.7	
	PCA	0.0005	3	80–120	93	25	
		0.10	3	68–76	73	5.7	
		0.25	3	84–90	87	3.5	
	PCTA	0.0005	3	80–100	87	13	
		0.10	3	72–76	73	3.1	
		0.25	3	84–86	85	1.4	
	PB	0.0005	3	80–100	93	12	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
		0.10	3	72–80	77	5.4	
		0.25	3	80–82	81	1.4	
	HCB	0.0005	3	80–80	80	0.0	
		0.10	3	72–76	74	2.7	
		0.25	3	80–82	81	1.4	
Cotton (soapstock)	Quintozene	0.0005	3	100–200	140	38	900-RES-114
		0.10	3	80–86	84	4.1	
		0.25	3	84–86	85	1.4	
	PCA	0.0005	3	80–80	80	0.0	
		0.10	3	82–88	85	3.6	
		0.25	3	88–88	88	0.0	
	PCTA	0.0005	3	60–80	73	16	
		0.10	3	76–82	79	3.9	
		0.25	3	78–82	79	2.9	
	PB	0.0005	2	20–40	30	47	
		0.10	3	80–88	83	5.0	
		0.25	3	78–80	79	1.5	
	HCB	0.0005	3	100–100	100	0.0	
		0.10	3	74–78	76	2.6	
		0.25	3	76–80	77	3.0	
Cotton (oil crude)	Quintozene	0.0005	3	40–80	67	35	900-RES-114
		0.10	3	72–84	76	9.1	
		0.25	3	58–86	77	21	
	PCA	0.0005	3	100–200	133	43	
		0.10	3	72–84	77	8.4	
		0.25	3	78–100	86	14	
	PCTA	0.0005	3	120–140	127	9.1	
		0.10	3	58–72	65	11	
		0.25	3	70–76	73	4.2	
	PB	0.0005	3	80–120	93	25	
		0.10	3	68–86	77	12	
		0.25	3	82–98	91	9.1	
	HCB	0.0005	3	80–80	80	0.0	
		0.10	3	66–70	67	3.4	
		0.25	3	70–88	77	12	
Cotton (oil refined)	Quintozene	0.0005	3	60–140	93	45	900-RES-114
		0.10	3	54–96	80	28	
		0.25	3	40–98	68	43	
	PCA	0.0005	3	120–300	233	42	
		0.10	3	68–86	79	12	
		0.25	3	72–108	91	20	
	PCTA	0.0005	2	120–120	120	0.0	
		0.10	3	84–116	101	16	
		0.25	3	84–108	98	13	
	PB	0.0005	3	100–100	100	0.0	
		0.10	3	82–106	94	13	
		0.25	3	74–94	87	13	
	HCB	0.0005	3	40–500	200	130	
		0.10	3	76–80	77	3.0	
		0.25	3	64–96	82	20	
Cotton (seed)	Quintozene	0.005	6	61–144	104	31	900-RES-147
		0.02	6	80–128	102	17	
		0.20	6	81–97	90	7.1	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCA	0.005	6	101–123	107	7.9	
		0.02	6	98–136	113	11	
		0.20	6	97–110	103	4.5	
	PCTA	0.005	6	118–134	124	4.5	
		0.02	6	104–138	118	10	
		0.20	6	95–111	102	5.8	
	PB	0.002	6	64–95	81	13	
		0.02	6	57–88	78	14	
		0.20	6	73–100	82	12	
	HCB	0.002	6	89–121	99	12	
		0.02	6	86–113	98	9.0	
		0.20	6	90–96	93	2.9	
Cotton (gin trash)	Quintozene	0.05	6	71–78	75	3.1	900-RES-147
		0.20	6	76–93	84	9.0	
		0.50	6	77–99	92	8.4	
	PCA	0.05	6	85–97	92	4.5	
		0.20	6	98–106	101	3.6	
		0.50	6	86–112	106	10	
	PCTA	0.05	6	75–87	81	5.2	
		0.20	6	86–97	90	5.6	
		0.50	6	81–107	100	9.3	
	PB	0.02	6	83–90	86	3.1	
		0.20	6	80–99	88	9.3	
		0.50	6	77–97	91	8.3	
	HCB	0.02	6	76–85	80	3.9	
		0.20	6	73–88	80	9.0	
		0.50	6	72–92	87	8.6	
MP-PCNC-MA1 Method							
Potato	Quintozene	0.01	3	86–88	87	1.3	900-RES-037, 900-RES-044 (same author)
		0.5	3	80–88	83	5.3	
	PCA	0.01	3	80–83	82	2.1	
		0.1	3	81–87	84	3.7	
	PCTA	0.01	3	82–84	83	1.2	
		0.1	3	79–89	83	6.2	
Potato	Quintozene (primary)	0.01	2	87, 87	87	--	900-RES-023
		0.05	2	85, 85	85	--	
		5.0	2	76, 79	77	--	
	Quintozene (confirmatory)	0.01	2	88, 89	89	--	
		0.025	2	83, 85	84	--	
		0.05	2	83, 84	83	--	
	PCA (primary)	0.01	2	96, 100	98	--	
		0.05	2	99, 99	99	--	
		5.0	2	89, 89	89	--	
	PCA (confirmatory)	0.01	2	98, 108	103	--	
		0.025	2	92, 93	92	--	
		0.05	2	93, 95	94	--	
	PCTA (primary)	0.01	2	99, 103	101	--	
		0.05	2	100, 100	100	--	
		5.0	2	85, 93	89	--	
	PCTA (confirmatory)	0.01	2	95, 100	97	--	
		0.025	2	93, 95	94	--	
		0.05	2	95, 95	95	--	
	PB	0.01	2	75, 84	80	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	(primary)	0.05	2	92, 93	93	--	
		5.0	2	75, 77	76	--	
	PB (confirmatory)	0.01	2	76, 79	78	--	
		0.025	2	70, 72	71	--	
		0.05	2	72, 75	74	--	
	2,3,4,5-TCNB (primary)	0.01	2	87, 87	87	--	
		0.05	2	90, 90	90	--	
		5.0	2	86, 88	87	--	
	2,3,4,5-TCNB (confirmatory)	0.01	2	87, 93	90	--	
		0.025	2	85, 88	87	--	
		0.05	2	87, 88	88	--	
	2,3,5,6-TCNB (primary)	0.01	2	89, 90	90	--	
		0.05	2	88, 89	89	--	
		5.0	2	82, 82	82	--	
	2,3,5,6-TCNB (confirmatory)	0.01	2	87, 94	91	--	
		0.025	2	86, 91	88	--	
		0.05	2	90, 91	90	--	
	HCB (primary)	0.01	2	88, 89	89	--	
		0.05	2	91, 93	92	--	
		5.0	2	85, 86	85	--	
	HCB (confirmatory)	0.01	2	90, 91	91	--	
		0.025	2	85, 88	86	--	
		0.05	2	87, 88	87	--	
Snap Bean	Quintozene (primary)	0.01	2	98, 104	101	--	900-RES-023
		0.05	2	87, 87	87	--	
		5.0	2	76, 76	76	--	
	Quintozene (confirmatory)	0.01	2	96, 96	96	--	
		0.025	2	87, 90	89	--	
		0.05	2	83, 86	84	--	
	PCA (primary)	0.01	2	98, 102	100	--	
		0.05	2	95, 95	95	--	
		5.0	2	86, 89	88	--	
	PCA (confirmatory)	0.01	2	91, 98	94	--	
		0.025	2	92, 94	93	--	
		0.05	2	91, 95	93	--	
	PCTA (primary)	0.01	2	105, 109	107	--	
		0.05	2	98, 100	99	--	
		5.0	2	89, 89	89	--	
	PCTA (confirmatory)	0.01	2	98, 100	99	--	
		0.025	2	98, 99	98	--	
		0.05	2	93, 97	95	--	
	PB (primary)	0.01	2	75, 80	78	--	
		0.05	2	97, 97	97	--	
		5.0	2	75, 77	76	--	
	PB (confirmatory)	0.01	2	79, 79	79	--	
		0.025	2	75, 77	76	--	
		0.05	2	78, 81	80	--	
	2,3,4,5-TCNB (primary)	0.01	2	86, 90	88	--	
		0.05	2	89, 89	89	--	
		5.0	2	85, 85	85	--	
	2,3,4,5-TCNB (confirmatory)	0.01	2	84, 84	84	--	
		0.025	2	88, 88	88	--	
		0.05	2	88, 92	90	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	2,3,5,6-TCNB (primary)	0.01	2	90, 93	92	--	
		0.05	2	89, 91	90	--	
		5.0	2	78, 80	79	--	
	2,3,5,6-TCNB (confirmatory)	0.01	2	85, 90	88	--	
		0.025	2	86, 88	87	--	
		0.05	2	90, 94	92	--	
	HCB (primary)	0.01	2	87, 93	90	--	
		0.05	2	88, 90	89	--	
		5.0	2	82, 84	83	--	
	HCB (confirmatory)	0.01	2	87, 87	87	--	
		0.025	2	88, 90	89	--	
		0.05	2	87, 90	88	--	
Tomato	Quintozene (primary)	0.01	2	82, 85	83	--	900-RES-023
		0.05	2	91, 91	91	--	
		5.0	2	76, 81	78	--	
	Quintozene (confirmatory)	0.01	2	86, 86	86	--	
		0.025	2	86, 90	88	--	
		0.05	2	89, 89	89	--	
	PCA (primary)	0.01	2	95, 99	97	--	
		0.05	2	102, 105	104	--	
		5.0	2	89, 96	93	--	
	PCA (confirmatory)	0.01	2	98, 103	101	--	
		0.025	2	96, 96	96	--	
		0.05	2	100, 101	101	--	
	PCTA (primary)	0.01	2	94, 99	96	--	
		0.05	2	101, 101	101	--	
		5.0	2	85, 96	91	--	
	PCTA (confirmatory)	0.01	2	100, 107	104	--	
		0.025	2	102, 102	102	--	
		0.05	2	101, 106	104	--	
	PB (primary)	0.01	2	76, 81	78	--	
		0.05	2	89, 89	89	--	
		5.0	2	74, 80	77	--	
	PB (confirmatory)	0.01	2	80, 80	80	--	
		0.025	2	72, 75	74	--	
		0.05	2	83, 85	84	--	
	2,3,4,5-TCNB (primary)	0.01	2	86, 88	87	--	
		0.05	2	95, 95	95	--	
		5.0	2	86, 90	88	--	
	2,3,4,5-TCNB (confirmatory)	0.01	2	92, 92	92	--	
		0.025	2	91, 91	91	--	
		0.05	2	95, 97	96	--	
	2,3,5,6-TCNB (primary)	0.01	2	88, 92	90	--	
		0.05	2	98, 98	98	--	
		5.0	2	87, 87	87	--	
	2,3,5,6-TCNB (confirmatory)	0.01	2	91, 91	91	--	
		0.025	2	91, 93	92	--	
		0.05	2	97, 101	99	--	
	HCB (primary)	0.01	2	84, 93	89	--	
		0.05	2	95, 96	96	--	
		5.0	2	82, 90	86	--	
	HCB (confirmatory)	0.01	2	92, 94	93	--	
		0.025	2	88, 92	90	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
		0.05	2	96, 96	96	--	
Peanut	Quintozene (primary)	0.01	2	83, 88	86	--	900-RES-023
		0.025	2	89, 91	90	--	
		0.05	2	92, 92	92	--	
		5.0	2	91, 93	92	--	
	PCA (primary)	0.01	2	87, 97	92	--	
		0.025	2	101, 101	101	--	
		0.05	2	104, 104	104	--	
		5.0	2	93, 101	97	--	
	PCTA (primary)	0.01	2	93, 95	94	--	
		0.025	2	101, 103	102	--	
		0.05	2	106, 106	106	--	
		5.0	2	102, 108	105	--	
	PB (primary)	0.01	2	78, 84	81	--	
		0.025	2	82, 88	85	--	
		0.05	2	84, 89	87	--	
		5.0	2	93, 96	94	--	
	2,3,4,5-TCNB (primary)	0.01	2	113, 117	115	--	
		0.025	2	100, 106	103	--	
		0.05	2	100, 102	101	--	
		5.0	2	100, 100	100	--	
	2,3,5,6-TCNB (primary)	0.01	2	84, 113	99	--	
		0.025	2	90, 93	91	--	
		0.05	2	95, 95	95	--	
		5.0	2	99, 102	100	--	
	HCB (primary)	0.01	2	89, 92	91	--	
		0.025	2	93, 95	94	--	
		0.05	2	96, 98	97	--	
		5.0	2	99, 101	100	--	
Peanut (whole nut)	Quintozene (primary)	0.005	2	85, 89	87	--	900-RES-023
		0.01	2	89, 103	96	--	
		5.0	2	96, 100	98	--	
	Quintozene (confirmatory)	0.005	2	82, 100	91	--	
		0.01	2	92, 102	97	--	
		5.0	2	97, 100	99	--	
	PCA (primary)	0.005	2	81, 87	84	--	
		0.01	2	107, 118	113	--	
		5.0	2	103, 106	105	--	
	PCTA (primary)	0.005	2	80, 85	83	--	
		0.01	2	88, 102	95	--	
		5.0	2	94, 98	96	--	
	PCTA (confirmatory)	0.005	2	109, 120	115	--	
		0.01	2	88, 113	100	--	
		5.0	2	96, 109	103	--	
	PB (primary)	0.005	2	83, 116	100	--	
		0.01	2	90, 102	96	--	
		5.0	2	93, 96	95	--	
	PB (confirmatory)	0.005	2	80, 118	99	--	
		0.01	2	91, 102	97	--	
		5.0	2	96, 98	97	--	
	2,3,4,5-TCNB (primary)	0.005	2	84, 103	93	--	
		0.01	2	86, 103	94	--	
		5.0	2	99, 101	100	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	2,3,5,6-TCNB (primary)	0.005	2	75, 85	80	--	
		0.01	2	88, 90	89	--	
		5.0	2	97, 97	97	--	
	HCB (primary)	0.005	2	85, 108	96	--	
		0.01	2	92, 105	99	--	
		5.0	2	97, 99	98	--	
Peanut (shells)	Quintozene (primary)	0.005	2	81, 82	82	--	900-RES-023
		0.01	2	88, 104	96	--	
		5.0	2	97, 99	98	--	
	Quintozene (confirmatory)	0.01	2	72, 75	73	--	
		5.0	2	100, 101	101	--	
	PCA (primary)	0.005	2	86, 92	89	--	
		0.01	2	92, 93	93	--	
		5.0	2	98, 99	99	--	
	PCTA (primary)	0.005	2	75, 84	79	--	
		0.01	2	96, 101	99	--	
		5.0	2	99, 101	100	--	
	PCTA (confirmatory)	0.005	2	86, 96	91	--	
		0.01	2	91, 106	98	--	
		5.0	2	97, 97	97	--	
	PB (primary)	0.005	2	104, 107	106	--	
		0.01	2	96, 110	103	--	
		5.0	2	97, 101	99	--	
	PB (confirmatory)	0.005	2	89, 93	91	--	
		0.01	2	91, 97	94	--	
		5.0	2	99, 100	100	--	
	2,3,4,5-TCNB (primary)	0.005	2	84, 88	86	--	
		0.01	2	91, 98	94	--	
		5.0	2	99, 103	101	--	
	2,3,5,6-TCNB (primary)	0.005	2	70, 77	73	--	
		0.01	2	82, 83	82	--	
		5.0	2	93, 96	95	--	
	HCB (primary)	0.005	2	84, 89	87	--	
		0.01	2	89, 95	92	--	
		5.0	2	100, 100	100	--	
Peanut (nutmeat)	Quintozene (primary)	0.005	2	92, 92	92	--	900-RES-023
		0.01	2	90, 93	92	--	
		5.0	2	100, 101	101	--	
	Quintozene (confirmatory)	0.005	2	93, 95	94	--	
		0.01	2	93, 93	93	--	
		5.0	2	100, 101	101	--	
	PCA (primary)	0.005	2	94, 95	94	--	
		0.01	2	98, 102	100	--	
		5.0	2	98, 103	101	--	
	PCTA (primary)	0.005	2	87, 89	88	--	
		0.01	2	90, 95	93	--	
		5.0	2	98, 100	99	--	
	PCTA (confirmatory)	0.005	2	106, 113	110	--	
		0.01	2	95, 95	95	--	
		5.0	2	98, 99	98	--	
	PB (primary)	0.005	2	90, 92	91	--	
		0.01	2	91, 93	92	--	
		5.0	2	95, 96	96	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PB (confirmatory)	0.005	2	92, 93	93	--	
		0.01	2	91, 93	92	--	
		5.0	2	100, 100	100	--	
	2,3,4,5-TCNB (primary)	0.005	2	85, 87	86	--	
		0.01	2	93, 97	95	--	
		5.0	2	103, 104	104	--	
	2,3,5,6-TCNB (primary)	0.005	2	76, 87	82	--	
		0.01	2	83, 84	83	--	
		5.0	2	101, 104	103	--	
	HCB (primary)	0.005	2	99, 100	100	--	
		0.01	2	95, 96	96	--	
		5.0	2	99, 100	100	--	
Battelle 100117568 Method (QuEChERS extraction)							
Broccoli	Quintozene (m/z = 295)	0.01	5	96–112	103	5.8	900-RES-223
		0.1	5	89–98	96	3.9	
	Quintozene (m/z = 297)	0.01	5	90–113	104	8.7	
		0.1	5	90–99	96	3.7	
	Quintozene (m/z = 293)	0.01	5	95–107	99	4.9	
		0.1	5	88–97	95	4.2	
	PCA (m/z = 265)	0.01	5	95–105	101	3.8	
		0.1	5	90–101	97	4.3	
	PCA (m/z = 267)	0.01	5	91–111	103	7.4	
		0.1	5	90–99	97	3.9	
	PCA (m/z = 263)	0.01	5	96–105	102	3.4	
		0.1	5	91–101	97	3.8	
	PCTA (m/z = 296)	0.01	5	90–94	92	1.8	
		0.1	5	83–93	90	4.4	
	PCTA (m/z = 294)	0.01	5	89–92	90	1.4	
		0.1	5	82–92	89	4.7	
	PCTA (m/z = 246)	0.01	5	89–94	91	2.6	
		0.1	5	84–92	90	3.9	
Potato (tubers)	Quintozene (m/z = 295)	0.01	5	85–100	95	6.5	900-RES-223
		0.1	5	72–84	80	6.3	
	Quintozene (m/z = 297)	0.01	5	85–108	98	9.0	
		0.1	5	72–83	79	6.1	
	Quintozene (m/z = 293)	0.01	5	85–97	93	5.8	
		0.1	5	73–84	80	5.8	
	PCA (m/z = 265)	0.01	5	92–96	94	1.9	
		0.1	5	77–89	84	5.9	
	PCA (m/z = 267)	0.01	5	92–102	96	4.1	
		0.1	5	77–87	84	5.5	
	PCA (m/z = 263)	0.01	5	90–96	93	2.5	
		0.1	5	78–90	86	5.8	
	PCTA (m/z = 296)	0.01	5	86–90	88	1.7	
		0.1	5	73–84	80	6.0	
	PCTA (m/z = 294)	0.01	5	87–92	90	2.4	
		0.1	5	74–85	81	5.9	
	PCTA (m/z = 246)	0.01	5	85–92	89	3.1	
		0.1	5	75–86	82	5.7	

Procedural recovery data obtained in relation to the supervised residue trials and processing studies were presented in Table 60. While no validation data were available for animal commodities, procedural recovery data were provided for animal commodities and are presented in Table 61.

Table 60 Summary of procedural recovery data for determination of quintozene and its metabolites in plant commodities in relation to the supervised residue residue trials and processing studies

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
CAM-24-73 Method							
Cabbage (head)	Quintozene	0.005	2	82, 88	85	--	900-RES-059
		0.01	10	76–107	91	11	
		0.02	7	82–104	90	9.7	
		0.05	1	85	--	--	
	PCA	0.005	2	72, 84	78	--	
		0.01	10	75–99	82	11	
		0.02	7	52–92	74	17	
		0.05	1	74	--	--	
	PCTA	0.005	2	76, 84	80	--	
		0.01	10	78–106	90	9.4	
		0.02	7	70–99	85	12	
		0.05	1	91	--	--	
MP-PCNC-MA2 Method							
Cabbage (head)	Quintozene	0.011-0.038	10	75–114	84	14	900-RES-020
	PCA	0.010-0.034	10	66–98	80	12	
	PCTA	0.010-0.033	10	83–109	96	8.1	
CAM-24-73 Method							
Cabbage (head)	Quintozene	0.001	6	82–120	98	15	900-RES-194
		0.5	6	87–120	101	12	
	PCA	0.001	6	78–117	103	13	
		0.5	6	81–111	97	12	
	PCTA	0.001	6	90–118	104	11	
		0.5	6	85–118	101	13	
Cabbage (head)	Quintozene	0.001	4	70–105	87	17	900-RES-201
		0.5	4	78–105	93	13	
	PCA	0.001	4	75–110	94	16	
		0.5	4	72–106	89	16	
	PCTA	0.001	4	76–106	90	17	
		0.5	4	87–105	96	9.9	
Cabbage (head)	Quintozene	0.001	3	85–118	100	17	900-RES-202
		0.5	3	101–116	111	7.6	
	PCA	0.001	3	75–103	89	16	
		0.5	3	93–120	110	14	
	PCTA	0.001	3	105–120	111	7.2	
		0.5	3	106–114	110	3.7	
Broccoli	Quintozene	0.004	2	102, 125	114	--	900-RES-059
		0.01	1	96	--	--	
		0.02	3	76–88	82	7.3	
		0.05	1	78	--	--	
	PCA	0.004	2	100, 110	105	--	
		0.01	1	85	--	--	
		0.02	3	64–77	70	9.6	
		0.05	1	61	--	--	
	PCTA	0.004	2	82, 112	97	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
		0.01	1	79	--	--	
		0.02	3	74–88	80	8.8	
		0.05	1	65	--	--	
MP-PCNC-MA2 Method							
Broccoli	Quintozene	0.011-0.038	4	75–100	85	14	900-RES-019
	PCA	0.010-0.034	4	77–104	87	14	
	PCTA	0.010-0.033	4	88–121	103	14	
Battelle 100117568 Method (QuEChERS extraction)							
Broccoli	Quintozene	0.01	1	99	--	--	900-RES-224
		0.1	1	90	--	--	
	PCA	0.01	1	96	--	--	
		0.1	1	91	--	--	
	PCTA	0.01	1	82	--	--	
		0.1	1	82	--	--	
MP-PCNC-MA2 Method							
Pepper	Quintozene	0.011-0.055	4	85–95	89	5.0	900-RES-024
	PCA	0.010-0.050	4	80–107	97	13	
	PCTA	0.010-0.048	4	99–109	103	4.3	
CAM-24-73 Method							
Pepper	Quintozene	0.05	3	98–109	103	5.5	900-RES-062
		1.0	3	101–109	104	4.6	
	PCA	0.05	3	93–111	101	8.9	
		1.0	3	99–106	101	3.5	
	PCTA	0.05	3	96–104	100	4.2	
		1.0	3	102–108	102	4.0	
Pepper	Quintozene	0.05	1	111	--	--	900-RES-081 (addendum)
		1.0	1	104	--	--	
	PCA	0.05	1	116	--	--	
		1.0	1	102	--	--	
	PCTA	0.05	1	114	--	--	
		1.0	1	103	--	--	
Pepper	Quintozene	0.002	2	90–100	95	--	900-RES-080
		0.02	3	85–91	89	3.7	
	PCA	0.002	3	74–99	85	15	
		0.02	3	86–90	88	2.1	
	PCTA	0.002	3	80–108	94	15	
		0.02	3	91–96	94	3.1	
MP-PCNC-MA2 Method							
Tomato	Quintozene	0.011-0.056	4	87–91	89	2.4	900-RES-027
	PCA	0.025-0.050	4	98–103	100	2.3	
	PCTA	0.024-0.048	4	103–112	107	3.9	
CAM-24-73 Method							
Tomato	Quintozene	0.05-0.06	7	89–116	103	7.7	900-RES-126
		1.0-1.3	8	99–106	102	2.4	
	PCA	0.05-0.07	7	90–112	102	7.7	
		1.0-1.5	8	100–123	107	8.6	
	PCTA	0.05-0.06	7	98–109	103	3.2	
		1.0-1.3	8	101–106	104	1.6	
Tomato (unwashed)	Quintozene	0.05	1	98	--	--	900-RES-126
		1.0	1	97	--	--	
	PCA	0.05	1	103	--	--	
		1.0	1	97	--	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCTA	0.05	1	100	--	--	
		1.0	1	98	--	--	
Tomato (washed)	Quintozene	0.05	1	103	--	--	900-RES-126
		1.0	1	100	--	--	
	PCA	0.05	1	101	--	--	
		1.0	1	100	--	--	
	PCTA	0.05	1	105	--	--	
		1.0	1	101	--	--	
Tomato (canned)	Quintozene	0.05	1	103	--	--	900-RES-126
		1.0	1	99	--	--	
	PCA	0.05	1	123	--	--	
		1.0	1	104	--	--	
	PCTA	0.05	1	114	--	--	
		1.0	1	101	--	--	
Tomato (puree)	Quintozene	0.05	1	108	--	--	900-RES-126
		1.0	2	102, 96	99	--	
	PCA	0.05	1	102	--	--	
		1.0	2	106, 95	100	--	
	PCTA	0.05	1	104	--	--	
		1.0	2	103, 97	100	--	
Tomato (paste)	Quintozene	0.05	1	108	--	--	900-RES-126
		1.0	1	102	--	--	
	PCA	0.05	1	106	--	--	
		1.0	1	105	--	--	
	PCTA	0.05	1	116	--	--	
		1.0	1	103	--	--	
Tomato (pomace wet)	Quintozene	0.05	1	156	--	--	900-RES-126
		1.0	2	103, 89	96	--	
	PCA	0.05	1	121	--	--	
		1.0	2	95, 88	91	--	
	PCTA	0.05	1	126	--	--	
		1.0	2	99, 94	97	--	
Tomato (pomace dry)	Quintozene	0.05	2	107, 109	108	--	900-RES-126
		1.0	1	98	--	--	
	PCA	0.05	2	82, 101	91	--	
		1.0	1	82	--	--	
	PCTA	0.05	2	97, 113	105	--	
		1.0	1	92	--	--	
Tomato (ketchup)	Quintozene	0.05	1	109	--	--	900-RES-126
		1.0	1	117	--	--	
	PCA	0.05	1	112	--	--	
		1.0	1	118	--	--	
	PCTA	0.05	1	104	--	--	
		1.0	1	119	--	--	
Tomato	Quintozene	0.002	2	101, 93	97	--	900-RES-160
		0.02	2	101, 99	100	--	
	PCA	0.002	3	78–102	88	14	
		0.02	3	103–108	105	2.6	
	PCTA	0.002	3	94–111	103	8.3	
		0.02	3	104–112	107	4.0	
Tomato (juice)	Quintozene	0.002	1	84	--	--	900-RES-160
		0.02	1	92	--	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCA	0.002	1	84	--	--	
		0.02	1	95	--	--	
	PCTA	0.002	1	94	--	--	
		0.02	1	96	--	--	
Tomato (canned)	Quintozene	0.002	1	92	--	--	900-RES-160
		0.02	1	97	--	--	
	PCA	0.002	1	88	--	--	
		0.02	1	103	--	--	
	PCTA	0.002	1	100	--	--	
		0.02	1	103	--	--	
MP-PCNC-MA1 Method							
Lima beans (whole pod)	Quintozene	0.006	2	71, 82	77	--	900-RES-017
		0.028	5	73–96	82	11	
	PCA	0.005	2	88, 88	88	--	
		0.025	5	79–106	89	11	
	PCTA	0.005	2	92, 108	100	--	
		0.024	5	83–112	95	11	
Snap beans (whole pod)	Quintozene	0.028	5	73–87	83	7.2	900-RES-018
		0.111	3	74–78	76	2.5	
	PCA	0.025	5	79–92	88	5.9	
		0.099	3	83–90	87	4.1	
	PCTA	0.024	5	91–100	95	4.1	
		0.097	3	87–93	90	3.6	
CAM-24-73 Method							
Bean (green seed: green and lima bean)	Quintozene	0.05	6	90–118	102	9.0	900-RES-061
		0.5	1	93	--	--	
		1.0	6	92–104	98	5.7	
	PCA	0.05	6	87–106	95	6.4	
		0.5	1	97	--	--	
		1.0	6	91–106	98	6.8	
	PCTA	0.05	6	77–110	96	12	
		0.5	1	93	--	--	
		1.0	6	88–105	98	7.7	
Bean (green pods: snap beans)	Quintozene	0.05	8	95–111	104	4.2	900-RES-061
		0.5	1	85	--	--	
		1.0	6	94–104	99	3.9	
	PCA	0.05	8	95–109	101	4.6	
		0.5	1	90	--	--	
		1.0	6	94–103	97	4.0	
	PCTA	0.05	8	90–106	100	5.0	
		0.5	1	87	--	--	
		1.0	6	94–102	99	3.3	
Bean (dry seed: dry bean, kidney, navy and pinto beans)	Quintozene	0.05	6	94–124	106	9.3	900-RES-061
		1.0	9	82–113	99	8.6	
	PCA	0.05	6	89–116	101	9.0	
		1.0	9	80–108	95	7.8	
	PCTA	0.05	6	98–119	17	7.9	
		1.0	9	63–113	98	15	
Snap Bean (green pods)	Quintozene	0.05	3	88–102	95	7.4	900-RES-107
		0.25	3	92–104	96	7.2	
	PCA	0.05	3	86–98	90	7.7	
		0.25	3	92–98	95	3.2	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCTA	0.05	3	78–96	86	11	
		0.25	3	82–106	92	14	
Bean (dry seed)	Quintozene	0.05	3	86–96	91	5.5	900-RES-108
		0.25	3	88–104	95	8.8	
	PCA	0.05	3	80–92	84	8.2	
		0.25	3	84–98	89	8.5	
	PCTA	0.05	3	80–94	88	8.2	
		0.25	3	86–104	92	11	
Potato (tubers)	Quintozene	0.004	3	78–152	112	34	900-RES-063
		0.01	2	93–111	102	--	
		0.02	1	77	--	--	
		0.05	1	72	--	--	
		0.1	7	89–110	99	7.4	
	PCA	0.004	3	68–155	111	39	
		0.01	2	86–95	91	--	
		0.02	1	72	--	--	
		0.05	1	82	--	--	
		0.1	7	81–102	91	8.2	
	PCTA	0.004	3	82–190	127	45	
		0.01	2	96–114	105	--	
		0.02	1	80	--	--	
		0.05	1	84	--	--	
		0.1	7	89–116	101	10	
MP-PCNC-MA1 Method							
Potato (tubers)	Quintozene	0.028	4	81–92	87	6.5	900-RES-025
		0.056	1	98	--	--	
		0.089	2	97, 101	99	--	
		0.111	10	68–105	86	11	
	PCA	0.025	4	96–105	101	4.6	
		0.050	1	109	--	--	
		0.079	2	119, 120	120	--	
		0.099	10	79–110	96	9.0	
	PCTA	0.024	4	93–104	99	4.5	
		0.048	1	111	--	--	
		0.078	2	109, 112	111	--	
		0.097	10	80–116	98	11	
Zweig Method							
Potato (tubers)	Quintozene	0.01	6	100–116	107	5.0	900-RES-030
		0.1	6	95–111	100	6.3	
MP-PCNC-MA1 Method							
Potato (tubers)	Quintozene	0.01	1	92	--	--	900-RES-037
		0.5	1	88	--	--	
	PCA	0.01	1	70	--	--	
		0.5	1	88	--	--	
	PCTA	0.01	1	77	--	--	
		0.5	1	87	--	--	
Potato (tubers)	Quintozene	0.01	1	91	--	--	900-RES-038
		0.5	1	77	--	--	
	PCA	0.01	1	77	--	--	
		0.5	1	82	--	--	
	PCTA	0.01	1	80	--	--	
		0.5	1	84	--	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
Potato (tubers)	Quintozene	0.01	2	77, 86	82	--	900-RES-044
		0.05	1	77	--	--	
		0.5	1	71	--	--	
	PCA	0.01	2	83, 94	89	--	
		0.05	1	82	--	--	
		0.5	1	76	--	--	
	PCTA	0.01	2	83, 87	85	--	
		0.05	1	82	--	--	
		0.5	1	76	--	--	
Battelle 100117568 Method (QuEChERS extraction)							
Potato (tubers)	Quintozene (m/z = 293)	0.01	7	72–96	83	12	900-RES-225
		0.10	2	94, 99	97	--	
		0.50	3	75–98	85	14	
		1.0	2	77, 83	80	--	
	PCA (m/z = 265)	0.01	7	71–98	82	11	
		0.10	2	85, 92	89	--	
		0.50	3	79–110	93	17	
		1.0	2	74, 81	78	--	
	PCTA (m/z = 296)	0.01	7	60–88	77	15	
		0.10	2	81, 83	82	--	
		0.50	3	75–101	86	16	
		1.0	2	70, 77	74	--	
CAM-24-73 Method							
Wheat	Quintozene	0.01	5	100–121	108	8.2	900-RES-095
		0.025	2	108, 100	104	--	
	PCA	0.01	5	88–101	93	6.3	
		0.025	2	96, 100	98	--	
	PCTA	0.01	5	97–105	100	3.6	
		0.025	2	100, 100	100	--	
MP-PCNC-MA1 Method							
Cotton (seeds)	Quintozene	0.011	1	87	--	--	900-RES-018
		0.028	3	89–125	103	18	
	PCA	0.010	1	97	--	--	
		0.025	3	100–116	107	7.8	
	PCTA	0.010	1	100	--	--	
		0.024	3	106–116	108	6.3	
CAM-24-73 Method							
Cotton (seeds)	Quintozene	0.01	5	82–107	93	9.8	900-RES-067
		0.02	5	94–105	101	4.2	
	PCA	0.01	5	84–107	94	9.4	
		0.02	5	94–113	105	7.2	
	PCTA	0.01	5	84–97	90	5.2	
		0.02	5	88–105	97	6.6	
Cotton (seeds)	Quintozene	0.005	1	116	--	--	900-RES-068
		0.01	1	107	--	--	
		0.02	1	105	--	--	
		0.05	3	84–92	89	4.7	
	PCA	0.005	1	98	--	--	
		0.01	1	90	--	--	
		0.02	1	110	--	--	
		0.05	3	90–98	95	4.4	
	PCTA	0.005	1	92	--	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
		0.01	1	89	--	--	
		0.02	1	110	--	--	
		0.05	3	92–100	96	4.2	
Cotton (soapstock)	Quintozene	0.05	1	98	--	--	900-RES-068
	PCA	0.05	1	86	--	--	
	PCTA	0.05	1	94	--	--	
Cotton (refined oil)	Quintozene	0.005	1	132	--	--	900-RES-068
	PCA	0.005	1	98	--	--	
	PCTA	0.005	1	90	--	--	
Cotton (crude oil)	Quintozene	0.02	1	95	--	--	900-RES-068
	PCA	0.02	1	95	--	--	
	PCTA	0.02	1	96	--	--	
Cotton (reclaimed solvent)	Quintozene	0.005	1	86	--	--	900-RES-068
	PCA	0.005	1	90	--	--	
	PCTA	0.005	1	94	--	--	
Cotton (hulls)	Quintozene	0.05	1	80	--	--	900-RES-068
	PCA	0.05	1	86	--	--	
	PCTA	0.05	1	88	--	--	
Cotton (meal)	Quintozene	0.05	1	84	--	--	900-RES-068
	PCA	0.05	1	80	--	--	
	PCTA	0.05	1	84	--	--	
Cotton (seeds)	Quintozene	0.01	2	96, 94	95	--	900-RES-110
		0.02	2	92, 89	91	--	
	PCA	0.01	2	81, 80	81	--	
		0.02	2	92, 80	86	--	
	PCTA	0.01	2	100, 91	96	--	
		0.02	2	91, 90	91	--	
Cotton (seeds)	Quintozene	0.01	3	100–123	112	10	900-RES-152
		0.05	1	102	--	--	
	PCA	0.01	3	108–129	118	9.0	
		0.05	1	89	--	--	
	PCTA	0.01	3	112–119	115	3.3	
		0.05	1	103	--	--	
Cotton (seeds)	Quintozene	0.01	1	102	--	--	900-RES-197
		0.2	1	90	--	--	
	PCA	0.01	1	97	--	--	
		0.2	1	101	--	--	
	PCTA	0.01	1	98	--	--	
		0.2	1	107	--	--	
MP-PCNC-MA1 Method							
Peanuts	Quintozene	0.0111	4	92–111	101	9.4	900-RES-022
		0.0555	6	74–100	88	9.7	
		0.111	1	95	--	--	
	PCA	0.0099	4	99–106	102	3.2	
		0.0495	6	83–103	95	7.6	
		0.099	1	104	--	--	
	PCTA	0.0097	4	99–115	106	6.8	
		0.0484	6	83–104	100	8.2	
		0.0969	1	104	--	--	
CAM-24-73 Method							
Peanuts (nutmeat)	Quintozene	0.01	1	82	--	--	900-RES-078
		0.02	3	80–85	83	3.2	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
		0.05	4	84–108	94	11	
		0.10	2	84, 106	95	--	
	PCA	0.01	1	81	--	--	
		0.02	3	80–89	85	5.4	
		0.05	4	86–106	93	9.5	
		0.10	2	84, 104	94	--	
	PCTA	0.01	1	82	--	--	
		0.02	3	80–88	85	5.1	
		0.05	4	88–92	90	2.6	
		0.10	2	85, 102	94	--	
Peanuts (hulls)	Quintozene	0.02	2	90, 90	90	--	900-RES-078
		0.05	4	88–100	94	5.5	
		0.10	4	66–88	78	14	
	PCA	0.02	2	90, 90	90	--	
		0.05	4	90–102	95	5.6	
		0.10	4	87–90	89	1.5	
	PCTA	0.02	2	85, 90	88	--	
		0.05	4	90–98	94	3.7	
		0.10	4	87–94	90	3.4	
Peanuts (Soap stock)	Quintozene	0.01	1	96	--	--	900-RES-078
	PCA	0.01	1	72	--	--	
	PCTA	0.01	1	102	--	--	
Peanuts (refined deodorized oil)	Quintozene	0.02	1	97	--	--	900-RES-078
	PCA	0.02	1	93	--	--	
	PCTA	0.02	1	90	--	--	
Peanuts (refined oil)	Quintozene	0.01	1	113	--	--	900-RES-078
	PCA	0.01	1	95	--	--	
	PCTA	0.01	1	102	--	--	
Peanuts (crude oil)	Quintozene	0.05	1	92	--	--	900-RES-078
	PCA	0.05	1	124	--	--	
	PCTA	0.05	1	108	--	--	
Peanuts (crude oil expeller)	Quintozene	0.005	1	106	--	--	900-RES-078
	PCA	0.005	1	88	--	--	
	PCTA	0.005	1	92	--	--	
Peanuts (reclaimed solvent)	Quintozene	0.005	1	94	--	--	900-RES-078
	PCA	0.005	1	94	--	--	
	PCTA	0.005	1	92	--	--	
Peanuts (press cake expeller)	Quintozene	0.005	1	42	--	--	900-RES-078
		0.05	1	88	--	--	
	PCA	0.005	1	78	--	--	
		0.05	1	88	--	--	
	PCTA	0.005	1	80	--	--	
		0.05	1	90	--	--	
Peanuts (press cake, solvent extracted)	Quintozene	0.005	1	88	--	--	900-RES-078
		0.02	1	95	--	--	
	PCA	0.005	1	104	--	--	
		0.02	1	100	--	--	
	PCTA	0.005	1	52	--	--	
		0.02	1	90	--	--	
Peanuts (nutmeat)	Quintozene	0.01	1	76	--	--	900-RES-111
		0.5	1	83	--	--	
	PCA	0.01	1	88	--	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
		0.5	1	81	--	--	
	PCTA	0.01	1	74	--	--	
		0.5	1	84	--	--	
Peanuts (hulls)	Quintozene	0.005	1	84	--	--	900-RES-111
		0.5	1	93	--	--	
	PCA	0.005	1	108	--	--	
		0.5	1	96	--	--	
	PCTA	0.005	1	91	--	--	
		0.5	1	96	--	--	
Peanuts (nutmeat)	Quintozene	0.01	2	102, 70	86	--	900-RES-109
		0.5	2	84, 75	80	--	
	PCA	0.01	2	109, 76	93	--	
		0.5	2	86, 73	80	--	
	PCTA	0.01	2	94, 72	83	--	
		0.5	2	90, 77	84	--	
Peanuts (hulls)	Quintozene	0.005	2	90, 96	93	--	900-RES-109
		0.5	2	75, 88	82	--	
	PCA	0.005	2	92, 94	93	--	
		0.5	2	81, 84	83	--	
	PCTA	0.005	2	91, 92	92	--	
		0.5	2	93, 101	97	--	
Peanuts (nutmeat)	Quintozene	0.01	3	80–98	91	10	900-RES-113
		0.5	3	78–96	86	11	
	PCA	0.01	3	77–102	93	15	
		0.5	3	71–99	84	17	
	PCTA	0.01	3	70–90	83	14	
		0.5	3	76–102	85	17	
Peanuts (hulls)	Quintozene	0.01	3	74–94	85	12	900-RES-113
		0.5	3	85–89	87	2.4	
	PCA	0.01	3	77–92	87	9.7	
		0.5	3	82–83	83	0.7	
	PCTA	0.01	3	70–90	82	13	
		0.5	3	86–88	87	1.3	
MP-PCNC-MA1 Method							
Peanuts (whole nuts)	Quintozene	0.01	1	94	--	--	900-RES-023
		2.0	1	104	--	--	
		5.0	5	93–99	97	2.6	
	PCA	0.01	1	99	--	--	
		2.0	1	104	--	--	
		5.0	5	91–102	98	4.6	
	PCTA	0.01	1	92	--	--	
		2.0	1	101	--	--	
		5.0	5	93–99	97	2.7	
Peanuts (hulls)	Quintozene	0.01	2	96, 102	99	--	900-RES-023
		4.0	1	95	--	--	
		5.0	3	97–102	99	2.6	
	PCA	0.01	2	89, 104	97	--	
		4.0	1	97	--	--	
		5.0	4	96–106	101	4.8	
	PCTA	0.01	2	85, 89	87	--	
		4.0	1	94	--	--	
		5.0	4	95–104	98	4.2	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
Peanuts (nutmeat)	Quintozene	0.01	3	91–109	102	9.9	900-RES-023
		0.1	2	95, 99	97	--	
		0.2	3	93–100	96	3.4	
	PCA	0.01	3	97–99	98	1.5	
		0.1	2	97, 101	99	--	
		0.2	3	97–104	100	3.8	
	PCTA	0.01	3	87–98	92	5.9	
		0.1	1	92	--	--	
		0.2	3	91–100	96	5.0	
CAM-24-73 (modified) Method							
Wheat (forage)	Quintozene	0.01	5	90–111	103	9.3	900-RES-116, 900-RES-162
		0.1	5	86–96	92	4.0	
	PCA	0.01	5	85–100	93	5.8	
		0.1	5	84–97	90	6.0	
	PCTA	0.01	5	77–101	89	9.9	
		0.1	5	80–92	87	6.2	
Wheat (grain)	Quintozene	0.01	5	77–84	81	3.4	900-RES-116, 900-RES-162
		0.1	5	74–78	76	2.2	
	PCA	0.01	5	81–118	98	13	
		0.1	5	68–94	83	13	
	PCTA	0.01	5	79–93	86	6.9	
		0.1	5	74–87	81	6.1	
Wheat (straw)	Quintozene	0.01	5	61–91	78	14	900-RES-116, 900-RES-162
		0.1	5	68–82	74	7.0	
	PCA	0.01	5	82–115	100	16	
		0.1	5	80–102	90	11	
	PCTA	0.01	5	83–91	87	3.9	
		0.1	5	72–90	81	9.1	
Lettuce	Quintozene	0.01	5	80–97	87	7.8	900-RES-116, 900-RES-162
		0.1	5	90–97	92	3.3	
	PCA	0.01	5	83–88	85	2.3	
		0.1	5	85–88	87	1.8	
	PCTA	0.01	5	72–80	76	4.4	
		0.1	5	76–82	80	3.4	
Turnip (tops)	Quintozene	0.01	2	100, 101	101	--	900-RES-162
		0.1	2	100, 102	101	--	
	PCA	0.01	2	75, 78	77	--	
		0.1	2	90, 93	92	--	
	PCTA	0.01	2	91, 94	93	--	
		0.1	2	93, 94	94	--	
Turnip (roots)	Quintozene	0.01	2	84, 114	99	--	900-RES-162
		0.1	2	83, 91	87	--	
	PCA	0.01	2	77, 81	79	--	
		0.1	2	80, 90	85	--	
	PCTA	0.01	2	73, 87	80	--	
		0.1	2	84, 88	86	--	
MP-PCNC-MA1 Method							
Cotton (seeds)	Quintozene	0.009–0.056	4	75–92	87	9.4	900-RES-032
	PCA	0.008–0.050	4	82–101	91	12	
	PCTA	0.008–0.048	4	75–112	100	17	
Cotton (hulls)	Quintozene	0.056–0.111	2	82–91	86	--	900-RES-032
	PCA	0.05–0.099	2	90–96	93	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCTA	0.048–0.097	2	87–94	91	--	
MP-PCNC-MA2 Method							
Cotton (lints)	Quintozene	0.007–0.028	2	72–106	89	--	900-RES-032
	PCA	0.006–0.025	2	85–110	98	--	
	PCTA	0.006–0.021	2	85–143	114	--	
MP-PCNC-MA1 Method							
Cotton (crude oil)	Quintozene	0.011–0.056	4	89–93	91	1.9	900-RES-032
	PCA	0.01–0.05	4	86–110	97	13	
	PCTA	0.01–0.049	4	87–105	94	8.3	
Cotton (refined oil)	Quintozene	0.017–0.028	3	100–122	112	10	900-RES-032
	PCA	0.015–0.025	3	113–122	118	3.8	
	PCTA	0.015–0.024	3	113–122	119	4.2	
Cotton (hydrogenated oil)	Quintozene	0.011–0.056	2	89–108	99	--	900-RES-032
	PCA	0.01–0.05	2	87–107	97	--	
	PCTA	0.01–0.049	2	80–104	92	--	
Cotton (bleached oil)	Quintozene	0.011–0.028	2	88–88	88	--	900-RES-032
	PCA	0.01–0.025	2	83–86	84	--	
	PCTA	0.01–0.024	2	89–90	89	--	
Cotton (deodorized oil)	Quintozene	0.028	1	86	--	--	900-RES-032
	PCA	0.025	1	83	--	--	
	PCTA	0.024	1	81	--	--	
Cotton (meal)	Quintozene	0.056–0.111	4	84–90	86	3.3	900-RES-032
	PCA	0.05–0.099	4	77–105	94	13	
	PCTA	0.048–0.097	4	98–103	101	2.5	
Cotton (delinted seed)	Quintozene	0.011–0.056	4	85–100	90	7.9	900-RES-032
	PCA	0.01–0.05	4	97–115	103	8.1	
	PCTA	0.01–0.048	4	99–115	104	7.2	
Cotton (kernels)	Quintozene	0.028–0.111	3	85–93	87	5.1	900-RES-032
	PCA	0.025–0.099	3	95–102	98	3.6	
	PCTA	0.024–0.097	3	93–103	98	5.3	
Cotton (soap stock)	Quintozene	0.011–0.044	6	76–86	81	5.1	900-RES-032
	PCA	0.01–0.04	6	74–96	88	9.2	
	PCTA	0.01–0.039	6	71–103	90	12	
MP-PCNC-MA2 Method							
Cotton (linter motes)	Quintozene	0.028–0.111	4	85–114	100	12	900-RES-032
	PCA	0.025–0.099	4	76–112	89	18	
	PCTA	0.024–0.097	4	96–121	108	12	
Cotton (gin trash)	Quintozene	0.056–0.111	2	71–83	77	--	900-RES-032
	PCA	0.05–0.099	2	72–81	77	--	
	PCTA	0.048–0.097	2	78–87	83	--	
MP-PCNC-MA1 Method							
Cotton (seeds)	Quintozene	0.028–0.056	4	78–111	92	16	900-RES-032
	PCA	0.025–0.05	4	88–121	101	15	
	PCTA	0.024–0.048	4	85–125	98	19	
MP-PCNC-MA2 Method							
Cotton (linters)	Quintozene	0.028–0.056	2	71–74	72	--	900-RES-032
	PCA	0.025–0.05	2	70–75	73	--	
	PCTA	0.024–0.048	2	79–80	79	--	
CAM-24-73 (modified) Method							
Cotton (seeds)	Quintozene	0.05–0.25	2	86, 94	90	--	900-RES-114
	PCA	0.05–0.25	2	78, 88	83	--	
	PCTA	0.05–0.25	2	80, 86	83	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
Cotton (meal)	Quintozene	0.05–0.25	2	90, 92	91	--	900-RES-114
	PCA	0.05–0.25	2	92, 96	94	--	
	PCTA	0.05–0.25	2	88, 92	90	--	
Cotton (hulls)	Quintozene	0.05–0.25	2	88, 90	89	--	900-RES-114
	PCA	0.05–0.25	2	84, 88	86	--	
	PCTA	0.05–0.25	2	82, 88	85	--	
Cotton (soap stock)	Quintozene	0.05–0.25	4	78–92	85	7.1	900-RES-114
	PCA	0.05–0.25	4	72–92	80	11	
	PCTA	0.05–0.25	4	76–96	85	9.9	
Cotton (crude oil)	Quintozene	0.25	1	84	--	--	900-RES-114
	PCA	0.25	1	110	--	--	
	PCTA	0.25	1	112	--	--	
Cotton (refined oil)	Quintozene	0.05–0.25	2	4, 88	46	--	900-RES-114
	PCA	0.05–0.25	2	30, 92	61	--	
	PCTA	0.05–0.25	2	30, 106	68	--	
Cotton (seed)	Quintozene	0.01	1	69	--	--	900-RES-147
		0.05	1	87	--	--	
Cotton (gin trash)	Quintozene	0.1	1	94	--	--	900-RES-147
		0.5	1	105	--	--	
MP-PCNC-MA1 Method							
Whole peanuts (seeds)	Quintozene	0.011–0.111	12	68–111	90	16	900-RES-033
	PCA	0.01–0.099	12	80–116	97	11	
	PCTA	0.01–0.097	12	86–121	102	10	
Peanut (hulls)	Quintozene	0.028–0.111	5	76–180	109	38	900-RES-033
	PCA	0.05–0.099	4	83–116	101	15	
	PCTA	0.024–0.097	5	86–157	119	24	
Peanut (kernels)	Quintozene	0.028–0.111	6	82–122	97	15	900-RES-033
	PCA	0.025–0.099	6	96–294	138	56	
	PCTA	0.024–0.097	6	97–240	133	41	
Peanut (presscake)	Quintozene	0.056–0.111	4	81–117	97	18	900-RES-033
	PCA	0.05–0.099	4	92–126	108	15	
	PCTA	0.048–0.097	4	95–101	99	2.9	
Peanuts (crude oil)	Quintozene	0.028–0.056	3	75–107	87	20	900-RES-033
	PCA	0.025–0.05	3	75–108	92	18	
	PCTA	0.024–0.048	3	79–111	95	17	
Peanut (soapstock)	Quintozene	0.017–0.056	3	80–98	90	11	900-RES-033
	PCA	0.015–0.05	3	94–115	105	9.9	
	PCTA	0.015–0.048	3	93–109	103	8.6	
Peanut (roasted)	Quintozene	0.056–0.111	4	81–94	87	6.5	900-RES-033
	PCA	0.05–0.099	4	92–107	99	6.3	
	PCTA	0.048–0.097	4	86–109	100	11	
Peanut (roasted oil)	Quintozene	0.056–0.111	3	84–92	89	5.2	900-RES-033
	PCA	0.05–0.099	3	101–107	104	2.9	
	PCTA	0.048–0.097	2	101–106	104	--	
Peanut (oil solvent extracted)	Quintozene	0.056–0.134	2	89–104	96	--	900-RES-033
	PCA	0.05–0.12	2	88–109	99	--	
	PCTA	0.048–0.117	2	96–116	106	--	
Peanut (refined oil)	Quintozene	0.056–0.139	2	84–102	93	--	900-RES-033
	PCA	0.05–0.124	2	88–104	96	--	
	PCTA	0.048–0.121	2	92–118	105	--	
Peanut (deodorized oil)	Quintozene	0.017–0.028	3	87–88	87	0.8	900-RES-033
	PCA	0.015–0.025	3	87–97	92	5.6	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCTA	0.015–0.024	3	75–85	82	6.8	
Peanut (hydrogenated oil)	Quintozene	0.011–0.028	3	87–92	89	2.5	900-RES-033
	PCA	0.01–0.025	3	83–109	99	14	
	PCTA	0.01–0.024	3	79–101	93	13	
Potato (RAC tubers)	Quintozene	0.011–0.222	4	73–123	93	23	900-RES-034
	PCA	0.01–0.198	4	80–119	103	16	
	PCTA	0.01–0.194	4	92–110	103	7.9	
Potato (flakes)	Quintozene	0.011–0.069	4	86–104	95	7.9	900-RES-034
	PCA	0.01–0.062	4	95–117	106	8.8	
	PCTA	0.01–0.061	4	97–120	107	9.0	
Potato (chips)	Quintozene	0.011–0.056	4	73–117	95	20	900-RES-034
	PCA	0.01–0.05	4	84–113	98	12	
	PCTA	0.01–0.048	4	86–111	100	11	
Potato (granules)	Quintozene	0.011–0.028	2	90–90	90	--	900-RES-034
	PCA	0.01–0.025	2	99–101	100	--	
	PCTA	0.01–0.024	2	97–101	99	--	
Potato (wet peel–flakes)	Quintozene	0.011–0.111	10	60–155	82	38	900-RES-034
	PCA	0.01–0.099	10	76–136	92	21	
	PCTA	0.01–0.097	10	75–128	95	17	
Potato (wet peel–chips)	Quintozene	0.011–6.66	5	53–95	77	22	900-RES-034
	PCA	0.01–5.94	5	86–97	92	5.5	
	PCTA	0.01–5.81	5	95–112	104	7.0	
Potato (dry peel–flakes)	Quintozene	0.011–6.66	4	74–93	85	10	900-RES-034
	PCA	0.01–5.94	4	88–108	101	9.2	
	PCTA	0.01–5.81	4	89–118	102	12	
Potato (dry peel–chips)	Quintozene	0.028	1	79	--	--	900-RES-034
	PCA	0.025	1	90	--	--	
	PCTA	0.024	1	95	--	--	
Tomato (RAC fruit)	Quintozene	0.01–0.111	4	84–96	90	6.4	900-RES-035
	PCA	0.011–0.099	4	68–108	94	20	
	PCTA	0.01–0.097	4	94–104	99	4.7	
Tomato (puree)	Quintozene	0.011–0.028	5	83–110	97	13	900-RES-035
	PCA	0.01–0.025	5	94–128	111	14	
	PCTA	0.01–0.024	5	94–111	104	7.9	
Tomato (ketchup)	Quintozene	0.011–0.028	2	91–105	98	--	900-RES-035
	PCA	0.01–0.025	2	93–101	97	--	
	PCTA	0.01–0.024	2	76–108	92	--	
Tomato (juice)	Quintozene	0.011–0.028	2	70–84	77	--	900-RES-035
	PCA	0.01–0.025	2	78–100	89	--	
	PCTA	0.01–0.024	2	95–106	100	--	
Tomato (wet pomace)	Quintozene	0.028–4.44	3	71–76	73	3.1	900-RES-035
	PCA	0.025–3.96	3	85–86	85	0.9	
	PCTA	0.024–3.88	3	86–87	86	0.6	
Tomato (dry pomace)	Quintozene	0.028–6.66	3	84–114	98	15	900-RES-035
	PCA	0.025–5.94	3	95–129	107	18	
	PCTA	0.024–5.81	3	95–112	106	9.4	
CAM-24-73 Method							
Tomato	Quintozene	0.01	1	97	--	--	900-RES-218
		0.5	1	98	--	--	
	PCA	0.01	1	109	--	--	
		0.5	1	108	--	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCTA	0.01	1	69	--	--	
		0.5	1	78	--	--	
Tomato (juice)	Quintozene	0.01	1	101	--	--	900-RES-218
		0.5	1	97	--	--	
	PCA	0.01	1	110	--	--	
		0.5	1	105	--	--	
	PCTA	0.01	1	70	--	--	
		0.5	1	72	--	--	
Tomato (puree)	Quintozene	0.01	1	97	--	--	900-RES-218
		0.5	1	110	--	--	
	PCA	0.01	1	106	--	--	
		0.5	1	122	--	--	
	PCTA	0.01	1	91	--	--	
		0.5	1	101	--	--	
Tomato (ketchup)	Quintozene	0.01	1	96	--	--	900-RES-218
		0.5	1	112	--	--	
	PCA	0.01	1	106	--	--	
		0.5	1	124	--	--	
	PCTA	0.01	1	86	--	--	
		0.5	1	102	--	--	
Tomato (dry pomace)	Quintozene	0.01	1	101	--	--	900-RES-218
		0.5	1	112	--	--	
	PCA	0.01	1	104	--	--	
		0.5	1	117	--	--	
	PCTA	0.01	1	99	--	--	
		0.5	1	110	--	--	
Tomato (wet pomace)	Quintozene	0.01	1	87	--	--	900-RES-218
		0.5	1	89	--	--	
	PCA	0.01	1	97	--	--	
		0.5	1	94	--	--	
	PCTA	0.01	1	69	--	--	
		0.5	1	69	--	--	
Potato (uncooked fries)	Quintozene	0.005	1	108	--	--	900-RES-198
		0.05	1	98	--	--	
	PCA	0.005	1	76	--	--	
		0.05	1	75	--	--	
	PCTA	0.005	1	100	--	--	
		0.05	1	102	--	--	
Potato (French fries)	Quintozene	0.02	1	68	--	--	900-RES-198
		0.05	1	61	--	--	
	PCA	0.02	1	80	--	--	
		0.05	1	69	--	--	
	PCTA	0.02	1	72	--	--	
		0.05	1	67	--	--	
Potato (uncooked chips)	Quintozene	0.005	1	96	--	--	900-RES-198
		0.05	1	90	--	--	
	PCA	0.005	1	94	--	--	
		0.05	1	79	--	--	
	PCTA	0.005	1	100	--	--	
		0.05	1	91	--	--	
Potato (chips)	Quintozene	0.02	1	94	--	--	900-RES-198
		0.05	1	91	--	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCA	0.02	1	98	--	--	
		0.05	1	80	--	--	
	PCTA	0.02	1	97	--	--	
		0.05	1	73	--	--	
Potato (uncooked cubes)	Quintozene	0.005	1	102	--	--	900-RES-198
		0.05	1	81	--	--	
	PCA	0.005	1	94	--	--	
		0.05	1	71	--	--	
	PCTA	0.005	1	106	--	--	
		0.05	1	86	--	--	
Potato (dried flakes)	Quintozene	0.005	1	116	--	--	900-RES-198
		0.05	1	101	--	--	
	PCA	0.005	1	132	--	--	
		0.05	1	97	--	--	
	PCTA	0.005	1	126	--	--	
		0.05	1	93	--	--	
CAM-24-73 (modified) Method							
Potato (tuber)	Quintozene	0.002	3	50–165	92	69	900-RES-056
		0.01	3	72–114	96	22	
		0.2	1	108	--	--	
	PCA	0.002	3	15–100	67	68	
		0.01	3	56–92	79	25	
		0.2	1	104	--	--	
	PCTA	0.002	3	65–120	92	30	
		0.01	3	78–102	93	14	
		0.2	1	109	--	--	
Potato (raw chips)	Quintozene	0.002	2	90, 115	113	--	900-RES-056
		0.01	2	106, 127	117	--	
		0.2	1	114	--	--	
	PCA	0.002	3	80–110	93	16	
		0.01	3	88–117	101	15	
		0.2	1	108	--	--	
	PCTA	0.002	3	90–110	103	11	
		0.01	3	106–127	115	9.4	
		0.2	1	117	--	--	
Potato (chips, cooked)	Quintozene	0.002	3	75–260	155	76	900-RES-056
		0.01	3	116–130	121	6.7	
		0.2	1	106	--	--	
	PCA	0.002	3	60–85	77	19	
		0.01	3	62–92	80	20	
		0.2	1	70	--	--	
	PCTA	0.002	3	80–140	100	35	
		0.01	3	80–86	83	3.7	
		0.2	1	90	--	--	
Potato (slurry)	Quintozene	0.002	3	70–120	92	28	900-RES-056
		0.01	3	85–108	98	12	
		0.2	1	105	--	--	
	PCA	0.002	3	85–105	93	11	
		0.01	3	81–96	89	8.5	
		0.2	1	102	--	--	
	PCTA	0.002	3	95–110	103	7.4	
		0.01	3	88–109	102	12	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
		0.2	1	108	--	--	
Potato (flakes)	Quintozene	0.002	3	70–145	103	37	900-RES-056
		0.01	3	78–96	87	10	
		0.2	1	81	--	--	
	PCA	0.002	3	30–130	83	60	
		0.01	3	37–109	70	52	
		0.2	1	76	--	--	
	PCTA	0.002	3	75–110	95	19	
		0.01	3	59–99	84	26	
		0.2	1	84	--	--	
Potato (granules)	Quintozene	0.002	3	110–125	115	7.5	900-RES-056
		0.01	3	91–128	112	17	
		0.2	1	107	--	--	
	PCA	0.002	3	75–165	122	37	
		0.01	2	106, 129	118	--	
		0.2	1	64	--	--	
	PCTA	0.002	2	75, 75	75	--	
		0.01	3	69–90	83	14	
		0.2	1	109	--	--	
Potato (tubers)	Quintozene	0.0005-0.005	2	137, 95		--	900-RES-144
	PCA	0.001	2	90, 94	92	--	
	PCTA	0.001	2	118, 124	121	--	
Potato (peeled tubers)	Quintozene	0.0005	2	116, 120	118	--	900-RES-144
	PCA	0.001	2	78, 97	88	--	
	PCTA	0.001	2	107, 122	116	--	
Potato (wet peels)	Quintozene	0.005	2	88, 90	89	--	900-RES-144
	PCA	0.001	2	94, 105	100	--	
	PCTA	0.001	2	99, 120	110	--	
Potato (dry peels)	Quintozene	0.05	2	108, 109	109	--	900-RES-144
	PCA	0.01	2	71, 80	76	--	
	PCTA	0.01	2	91, 92	92	--	
Potato (tubers)	Quintozene	0.01	1	89	--	--	900-RES-206
		0.5	1	99	--	--	
	PCA	0.01	1	88	--	--	
		0.5	1	99	--	--	
	PCTA	0.01	1	87	--	--	
		0.5	1	96	--	--	
Potato (peeled)	Quintozene	0.01	1	94	--	--	900-RES-206
		0.5	1	97	--	--	
	PCA	0.01	1	99	--	--	
		0.5	1	99	--	--	
	PCTA	0.01	1	93	--	--	
		0.5	1	99	--	--	
Potato (wet peel)	Quintozene	0.5	2	87, 78	83	--	900-RES-206
	PCA	0.5	2	94, 84	89	--	
	PCTA	0.5	2	91, 85	88	--	
Potato (dried peel)	Quintozene	0.5	2	95, 91	93	--	900-RES-206
	PCA	0.5	2	94, 94	94	--	
	PCTA	0.5	2	95, 96	96	--	
Peanut (nutmeat)	Quintozene	0.5	1	81	--	--	900-RES-115
	PCA	0.5	1	93	--	--	
	PCTA	0.5	1	92	--	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
Peanut (meal)	Quintozene	1.0	1	85	--	--	900-RES-115
	PCA	1.0	1	96	--	--	
	PCTA	1.0	1	79	--	--	
Peanut (shells)	Quintozene	0.5–1.0	2	99, 102	101	--	900-RES-115
	PCA	0.5–1.0	2	94, 96	95	--	
	PCTA	0.5–1.0	2	94, 101	97	--	
Peanut (soapstock)	Quintozene	0.5–1.0	2	105, 120	113	--	900-RES-115
	PCA	0.5–1.0	2	88, 98	93	--	
	PCTA	0.5–1.0	2	95, 104	100	--	
Peanut (crude oil)	Quintozene	0.5	1	84	--	--	900-RES-115
	PCA	0.5	1	89	--	--	
	PCTA	0.5	1	92	--	--	
Peanut (refined oil)	Quintozene	1.0	1	85	--	--	900-RES-115
	PCA	1.0	1	89	--	--	
	PCTA	1.0	1	91	--	--	

Table 61 Summary of procedural recovery data of CAM-1-69 method for determination of quintozene and its metabolites in animal commodities

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
Bovine milk	PB	0.001-0.002	8	67–108	88	18	900-ANM-055 (CAM 1-69 Method)
	HCB	0.001-0.10	8	74–92	82	8.8	
	Quintozene	0.007	1	118	--	--	
		0.009	2	68, 67	68	--	
		0.010	3	45, 48, 69	54	13	
		0.019	1	88	--	-	
	PCA	0.004	2	78, 88	83	--	
		0.005	1	100	--	--	
		0.006	2	83, 93	88	--	
		0.008	1	70	--	--	
		0.021	1	82	--	--	
	PCTA	0.005	2	67, 67	67	--	
		0.007	2	82, 100	91	--	
		0.008	2	50, 81	66	--	
		0.020	1	50	--	--	
Bovine kidney	PB	0.004	1	100	--	--	
	HCB	0.009	1	100	--	--	
	Quintozene	0.049	1	100	--	--	
	PCA	0.048	1	100	--	--	
	PCTA	0.079	1	100	--	--	
Bovine liver	PB	0.003	1	67	--	--	
	HCB	0.020	1	92	--	--	
	Quintozene	0.055	1	85	--	--	
	PCA	0.050	1	88	--	--	
	PCTA	0.047	1	85	--	--	
Bovine muscle	PB	0.003	1	58	--	--	
	HCB	0.020	1	103	--	--	
	Quintozene	0.095	1	79	--	--	
	PCA	0.076	1	74	--	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCTA	0.104	1	75			
Bovine fat	PB	0.008	1	78	--	--	
	HCB	0.019	1	63	--	--	
	Quintozene	0.051	1	71	--	--	
		0.101	1	76	--	--	
		0.133		91	--	--	
	PCA	0.047	1	64	--	--	
		0.088	1	90	--	--	
		0.094	1	68	--	--	
	PCTA	0.052	1	75	--	--	
		0.070	1	75	--	--	
		0.104	1	75	--	--	
Chicken egg yolk	PB	0.002-0.03	7	59–100	74	24	900-RES-55
	HCB	0.008-0.47	7	55–100	70	23	(CAM-39-75
	Quintozene	0.01	2	69, 108	88	--	Method)
		0.015	1	86	--		
		0.03	3	100, 83, 52	78	24	
		0.113	1	75	--	--	
	PCA	0.01	1	82	--	--	
		0.02	1	101	--	--	
		0.08	1	101	--	--	
		0.10	1	66	--	--	
		0.12	1	65	----	--	
		0.13	2	73, 82	78	--	
	PCTA	0.01	2	71, 76	74	--	
		0.015	1	53	--	--	
		0.02	2	61, 86	74	--	
		0.112		69	--	--	
Chicken egg white	PB	0.002	1	70	--	--	
	HCB	0.005	1	73	--	--	
	Quintozene	0.011	1	68	--	--	
	PCA	0.009	1	63	--	--	
	PCTA	0.008	1	76	--	--	
Chicken fat	PB	0.006, 0.061	2	71, 79	75	--	
	HCB	0.013, 0.251	2	117, 96	107	--	
	Quintozene	0.027	1	90	--	--	
		0.268	1	84	--	--	
	PCA	0.024	1	89	--	--	
		0.236	1	88	--	--	
	PCTA	0.021	1	109	--	--	
		0.212	1	72	--	--	
Chicken liver	PB	0.006, 0.055	2	91, 95	93	--	
	HCB	0.012, 0.234	2	100, 95	98	--	
	Quintozene	0.0032	1	75	--	--	
		0.272	1	88	--	--	
	PCA	0.016	1	89	--	--	
		0.206	1	85	--	--	
	PCTA	0.020	1	105	--	--	
		0.303	1	95	--	--	
Chicken muscle	PB	0.009, 0.067	2	100, 100	100	--	
	HCB	0.014, 0.139	2	100, 98	99	--	
	Quintozene	0.037	1	100	--	--	
		0.298	1	93	--	--	

Matrix	Analyte	Fortification Level (mg/kg)	N	Recoveries (percent)		RSD (percent)	Reference
				Range	Mean		
	PCA	0.036	1	108	--	--	
		0.262	1	87	--	--	
	PCTA	0.033	1	104	--	--	
		0.236	1	100	--	--	

Summary of analytical method validation

Information was available for analytical methods for quintozene and its metabolites including PCA, PCTA PB, 2,3,4,5-TCNB and 2,3,5,6-TCNB, and an impurity HCB in the crops for which supervised residue trials were provided to this Meeting. The methods employ extraction of samples with acetone, acetone/hexane, acetonitrile, ethyl acetate, hexane, or isopropanol/hexane, with or without partition, clean-up using Florisil, silica gel, gel permeation or SPE column, and separation and quantification with GC-ECD or GC-MS. The LOQ varied from 0.0005 to 0.01 mg/kg for each analyte.

The results of recovery tests for analytical methods for plant commodities mostly showed acceptable recoveries and RSD, except that for CAM-24-73 (modified 4) method for cotton and its processed commodities, the recoveries at 0.0005 mg/kg in various commodities did not result in acceptable recoveries.

An analytical method using the QuEChERS extraction, clean-up with SPE column, and separation/quantification by GC-MSD was tested for broccoli and potato resulting in recoveries and RSD in the acceptable range.

Procedural recovery result data were also provided for those crops as well as for animal commodities. For analytical methods for animal commodities, there have been no sufficient data to indicate validity of these methods for animal commodities. Also there were not sufficient number of recovery data for each concentration/commodity.

Storage Stability under Frozen Conditions

Plant commodities

The storage stability of quintozene and its metabolites under frozen conditions was investigated in high water content, high oil content, high protein content and high starch content plant commodities.

Study 1 (Ball J.O., 1990, 900-RES-050; and Ball J.O., 1990, 900-RES-112)

The stability of quintozene and its metabolites PCA, PCTA and PB, and the impurity HCB was investigated in fortified control samples of kidney bean, pepper, tomato, tomato ketchup, dried tomato pomace, wheat grain, corn and soya bean during freezer storage (-20 °C or below) for 0–14 months and wheat grain, corn and soya bean during freezer storage (-20 °C or below) for 0–8 months. Control samples of kidney bean, pepper, tomato and its processed commodities, wheat grain, corn and soya bean were individually fortified in duplicate with quintozene and its metabolites at levels of 0.2 mg/kg (kidney bean, pepper, tomato including the processed commodities) or 0.025 mg/kg (wheat grain, corn and soya bean) and stored frozen for 2–14 or 2–8 months, respectively. Day zero samples were extracted immediately after fortification. All samples were analysed by CAM-24-73 (modified) method with an LOQ of 0.005 mg/kg for each analyte. The results of the storage stability study on quintozene and its metabolites and impurities in these commodities are presented in the following table.

Table 62 Storage stability of quintozene, PCA, PCTA, PB and HCB in kidney beans, pepper, and tomato and its processed commodities at -20 °C or below

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
Kidney beans	Quintozene	0.2	0	102, 107	105	--
			2	102, 93	98	107
			4	80, 80	80	90
			6	85, 95	90	102
			9	66	66	104
			14	74, 78	76	100
	PCA	0.2	0	100, 103	102	--
			2	102, 93	98	105
			4	88, 88	88	90
			6	91, 97	94	100
			9	76	76	100
			14	91, 97	94	98
	PCTA	0.2	0	103, 106	104	--
			2	107, 97	102	108
			4	91, 91	91	93
			6	94, 102	98	102
			9	74	74	102
			14	84, 89	87	101
	PB	0.2	0	99, 104	102	--
			2	92, 87	90	106
			4	74, 69	72	92
			6	75, 88	81	99
			9	55	55	100
			14	52, 52	52	92
	HCB	0.2	0	102, 106	104	--
			2	100, 92	96	106
			4	84, 82	83	93
			6	86, 97	91	102
			9	67	67	101
			14	71, 76	73	95
Pepper	Quintozene	0.2	0	98, 96	97	--
			2	63, 68	65	98
			4	75, 69	72	94
			6	63, 56	59	105
			9	50	50	101
			14	43, 47	45	104
	PCA	0.2	0	95, 93	94	--
			2	68, 74	71	102
			4	77, 80	78	95
			6	64, 61	63	105
			9	54	54	101
			14	64, 50	57	102
	PCTA	0.2	0	99, 98	98	--
			2	64, 72	68	102
			4	82, 82	82	96
			6	64, 63	64	106
			9	53	53	101
			14	54, 49	51	105
	PB	0.2	0	95, 92	93	--
			2	66, 43	54	93

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			4	78, 34	56	95
			6	60, 39	50	103
			9	49	49	95
			14	14, 35	25	96
	HCB	0.2	0	97, 95	96	--
			2	66, 70	68	96
			4	84, 72	78	96
			6	66, 55	60	105
			9	55	55	98
			14	32, 47	39	99
Tomato	Quintozene	0.2	0	96, 81	88	--
			2	83, 86	84	105
			4	56, 72	64	93
			6	73, 69	71	99
			9	52	52	107
			14	52, 23	37	104
	PCA	0.2	0	93, 78	86	--
			2	85, 88	87	106
			4	64, 77	70	97
			6	81, 73	77	102
			9	60	60	107
			14	62, 28	45	102
	PCTA	0.2	0	96, 84	90	--
			2	87, 91	89	108
			4	64, 82	73	98
			6	85, 76	81	103
			9	61	61	108
			14	57, 26	42	99
	PB	0.2	0	95, 81	88	--
			2	81, 75	78	106
			4	53, 62	58	98
			6	57, 64	61	101
			9	28	28	106
			14	13, 3.7	8.1	94
	HCB	0.2	0	96, 83	90	--
			2	83, 83	83	105
			4	59, 75	67	97
			6	73, 74	73	103
			9	54	54	106
			14	41, 14	27	94
Tomato ketchup	Quintozene	0.2	0	101, 91	96	--
			2	68, 79	73	93
			4	69, 53	61	101
			6	58, 58	58	98
			14	10, 4.9	7.5	107
	PCA	0.2	0	98, 83	90	--
			2	67, 76	71	97
			4	77, 65	71	103
			6	66, 52	59	99
			14	4.7, 0.0	2.4	106
	PCTA	0.2	0	99, 89	94	--
			2	68, 81	75	94
			4	79, 63	71	102

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			6	63, 65	64	97
			14	9.4, 0.0	4.7	108
	PB	0.2	0	98, 87	92	--
			2	67, 67	67	78
			4	59, 45	52	101
			6	38, 32	35	95
			14	13, 0.0	6.4	99
	HCB	0.2	0	99, 89	94	--
			2	67, 75	71	89
			4	71, 56	63	102
			6	54, 51	53	97
			14	9.5, 0.0	4.8	101
Tomato, dry pomace	Quintozene	0.2	0	90, 91	90	--
			2	79, 77	78	107
			4	56, 56	56	97
			6	55, 44	50	84
	PCA	0.2	0	54, 62	58	--
			2	61, 59	60	93
			4	52, 45	48	87
			6	49, 37	43	76
	PCTA	0.2	0	86, 85	85	--
			2	75, 71	73	103
			4	58, 55	56	94
			6	53, 44	49	78
	PB	0.2	0	91, 89	90	--
			2	77, 75	76	107
			4	56, 58	57	99
			6	52, 42	47	82
	HCB	0.2	0	95, 93	94	--
			2	80, 77	78	106
			4	58, 62	60	100
			6	55, 45	50	83

Table 63 Storage stability of quintozene, PCA, PCTA, PB and HCB in wheat, corn and soya bean at -20 °C or below

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
Wheat grain	Quintozene	0.025	0	88, 92, 84, 74	85	96
			2	72, 44, 64, 68	62	76
			3	56, 64, 88, 96	76	96
			4	100, 88, 100, 96	96	96
			6	88, 92, 96, 96	93	112
			8	104, 108, 104, 104	105	120
	PCA	0.025	0	76, 84	80	84
			2	72, 48	60	80
			3	72, 72	72	80
			4	80, 76	78	80
			6	76, 76	76	88
			8	84, 88	86	88
	PCTA	0.025	0	80, 84	82	84

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			2	72, 48	60	84
			3	72, 76	74	88
			4	84, 76	80	80
			6	80, 80	80	88
			8	80, 88	84	92
	PB	0.025	0	76, 76	76	84
			2	76, 48	62	88
			3	72, 80	76	88
			4	84, 80	82	80
			6	80, 80	80	88
			8	84, 96	90	72
	HCB	0.025	0	76, 80	78	84
			2	72, 48	60	84
			3	68, 80	74	92
			4	84, 80	82	84
			6	80, 80	80	88
			8	84, 88	86	88
Corn	Quintozene	0.025	0	80, 84, 84, 88	84	88
			2	48, 56, 48, 64	54	80
			3	72, 60, 56, 48	59	72
			4	60, 60, 72, 72	66	84
			6	60, 56, 80, 76	68	88
			8	64, 56, 72, 68	65	112
	PCA	0.025	0	76, 84	80	84
			2	56, 60	58	84
			3	76, 64	70	88
			4	64, 64	64	80
			6	60, 64	62	80
			8	68, 72	70	112
	PCTA	0.025	0	80, 84	82	92
			2	52, 56	54	92
			3	72, 60	66	96
			4	64, 64	64	88
			6	56, 56	56	88
			8	64, 64	64	104
	PB	0.025	0	68, 72	70	84
			2	48, 48	48	72
			3	80, 68	74	96
			4	60, 60	60	80
			6	56, 48	52	80
			8	56, 56	56	104
	HCB	0.025	0	76, 84	80	88
			2	48, 56	52	84
			3	68, 56	62	96
			4	64, 64	64	80
			6	56, 52	54	85
			8	60, 56	58	104
Soya bean	Quintozene	0.025	0	88, 84, 80, 80	83	84
			2	52, 44, 68, 64	57	96
			3	52, 56, 52, 64	56	96
			4	64, 68, 64, 72	67	88
			6	88, 56, 72, 72	72	96
			8	64, 76, 64, 60	66	104

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
	PCA	0.025	0	84, 76	80	84
			2	60, 52	56	96
			3	72, 64	68	100
			4	72, 68	70	84
			6	72, 68	70	84
			8	64, 72	68	100
	PCTA	0.025	0	88, 84	86	88
			2	52, 44	48	92
			3	60, 56	58	96
			4	64, 68	66	88
			6	64, 52	58	84
			8	64, 72	68	104
	PB	0.025	0	84, 76	80	80
			2	52, 40	46	96
			3	56, 56	56	96
			4	68, 68	68	84
			6	64, 48	56	80
			8	64, 72	68	92
	HCB	0.025	0	92, 84	88	88
			2	56, 48	52	96
			3	60, 56	58	96
			4	68, 68	68	88
			6	64, 48	56	84
			8	64, 72	68	104

When frozen storage stability was tested at the fortification level of 0.2 mg/kg at -20 °C or below in kidney beans, quintozene, PCA, PCTA and HCB were stable for at least 14 months (longest period tested). PB declined to below 70 percent after 6 months of frozen storage.

In pepper, when stored frozen at -20 °C or below, quintozene, PCA, PCTA and HCB were stable for up to 4 months. PB was found to be stable for less than 2 months.

In tomato, when stored frozen at -20 °C or below, quintozene, PCA, PCTA and HCB were stable for up to 6 months. PB was stable for up to 2 months. In tomato ketchup, quintozene and HCB were stable for up to 2 months, and PCA and PCTA for up to 4 months. PB was stable for less than 2 months. In tomato dry pomace, quintozene, PCTA, PB and HCB were stable for up to 2 months. For PCA, percent remaining values were all below 70 percent at any time points, even at day zero, and therefore, the results cannot be used.

When frozen storage stability was tested at the fortification level of 0.025 mg/kg at -20 °C or below, in wheat grain, all analytes, quintozene, PCA, PCTA, PB and HCB were stable for at least 8 months (longest period tested).

In corn, quintozene, PCA, PCTA, PB and HCB were stable for less than 2 months, except that PCA remained at 70 percent after storage for 3 months and 8 months. In this study, at day zero, procedural recoveries were in a range of 80–84 percent and if this was taken into consideration, the lowest percent remaining were close to 70 percent.

In soya bean, all analytes, quintozene, PCA, PCTA, PB and HCB were stable for less than 2 months, except that quintozene remained at 72 percent after 6 months of storage and PCA remained at 70 percent after 4 and 6 months of storage.

Study 2. (LeRoy R.L., 1991, 900-RES-016)

The stability of quintozene PCA, PCTA, PB, 2,3,4,5-TCNB and 2,3,5,6-TCNB, and the impurity HCB was investigated in fortified control samples of potato, tomato, snap beans, peanut and broccoli during freezer storage (below 0 °C) for 0–14 months (up to 23 months for snap beans). Homogenized control samples (25 g) of potato, tomato, snap beans, peanut and broccoli were fortified in duplicate with quintozene and separately with a mixture of the metabolites PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and the impurity HCB at a level of 0.2 mg/kg and stored frozen (below 0 °C) over the given time frame of 2–14 months (2–23 months for snap beans). Day zero samples were extracted immediately after fortification. All samples were analysed by MP-PCNC-MA1 method with an LOQ of 0.005 mg/kg for each analyte. The result of the storage stability of quintozene and its metabolites and impurities in these commodities is resented in the table below.

Table 64 Frozen storage stability of quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,35,6-TCNB and HCB in potato, tomato, snap beans, peanut and broccoli.

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
Potato	Quintozene	0.2	0/0	84, 79	81	82
			30/1	72, 70	71	80
			60/2	61, 67	64	77
			92/3	82, 80	81	83
			179/6	80, 75	78	84
			439/14	72, 66	69	75
	PCA	0.2	0/0	82, 94	88	94
			30/1	78, 83	80	91
			60/2	75, 79	77	89
			92/3	89, 89	89	95
			179/6	83, 83	83	96
			439/14	77, 76	77	87
	PCTA	0.2	0/0	81, 91	86	95
			30/1	80, 85	82	92
			60/2	78, 82	80	91
			92/3	91, 89	90	99
			179/6	86, 83	84	94
			439/14	76, 75	76	87
	PB	0.2	0/0	72, 80	76	76
			30/1	76, 82	79	77
			60/2	72, 73	73	76
			92/3	85, 83	84	81
			179/6	82, 80	81	84
			439/14	77, 76	76	78
	2,3,4,5-TCNB	0.2	0/0	79, 88	83	89
			30/1	67, 73	70	86
			60/2	66, 67	66	83
			92/3	81, 77	79	89
			179/6	77, 76	76	92
			439/14	69, 70	70	83
	2,3,5,6-TCNB	0.2	0/0	78, 89	83	89
			30/1	39, 40	39	86
			60/2	45, 45	45	82
			92/3	58, 48	53	84
			179/6	53, 54	54	89
			439/14	48, 54	51	84

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
	HCB	0.2	0/0	77, 87	82	85
			30/1	77, 83	80	84
			60/2	75, 76	75	84
			92/3	90, 88	89	91
			179/6	83, 81	82	91
			439/14	75, 74	75	80
Tomato	Quintozene	0.2	0/0	91, 86	89	80
			30/1	82, 90	86	84
			60/2	74, 83	78	79
			92/3	87, 85	86	85
			179/6	85, 89	87	86
			439/14	84, 90	87	70
	PCA	0.2	0/0	97, 96	96	92
			30/1	92, 85	88	95
			60/2	88, 87	87	93
			92/3	91, 96	94	99
			179/6	83, 79	81	96
			439/14	78, 79	79	81
	PCTA	0.2	0/0	102, 93	97	95
			30/1	92, 83	87	97
			60/2	91, 87	89	94
			92/3	89, 96	92	101
			179/6	89, 83	86	97
			439/14	80, 80	80	82
	PB	0.2	0/0	76, 84	80	69
			30/1	87, 79	83	80
			60/2	84, 81	82	76
			92/3	87, 90	88	81
			179/6	84, 78	81	86
			439/14	82, 79	80	70
	2,3,4,5-TCNB	0.2	0/0	89, 90	90	84
			30/1	90, 81	85	89
			60/2	84, 82	83	86
			92/3	88, 90	89	91
			179/6	82, 77	79	93
			439/14	79, 77	78	76
	2,3,5,6-TCNB	0.2	0/0	94, 92	93	87
			30/1	90, 80	85	90
			60/2	84, 83	84	87
			92/3	84, 87	85	89
			179/6	79, 76	77	92
			439/14	76, 75	75	77
	HCB	0.2	0/0	89, 91	90	83
			30/1	88, 81	85	88
			60/2	87, 83	85	86
			92/3	89, 94	91	92
			179/6	84, 80	82	92
			439/14	81, 77	79	76
Snap beans	Quintozene	0.2	0/0	91, 95	93	81
			30/1	75, 95	85	85
			63/2	69, 71	70	76
			93/3	71, 74	72	76
			182/6	71, 61	66	79

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			439/14	60, 63	61	86
			689/23	62, 62	62	94
	PCA	0.2	0/0	97, 96	96	93
			30/1	94, 90	92	101
			63/2	76, 65	70	91
			93/3	81, 68	74	90
			182/6	64, 81	72	91
			439/14	95, 62	78	79
			689/23	72, 75	73	91
	PCTA	0.2	0/0	99, 96	98	96
			30/1	97, 97	97	104
			63/2	74, 61	67	93
			93/3	82, 66	74	96
			182/6	60, 77	69	94
			439/14	58, 58	58	111
			689/23	50, 50	50	95
	PB	0.2	0/0	90, 88	89	79
			30/1	87, 83	85	83
			63/2	75, 94	85	82
			93/3	81, 65	73	75
			182/6	58, 76	67	81
			439/14	58, 57	57	88
			689/23	53, 52	52	89
	2,3,4,5-TCNB	0.2	0/0	93, 90	91	87
			30/1	92, 85	88	93
			63/2	72, 58	65	86
			93/3	80, 63	71	83
			182/6	58, 78	68	87
			439/14	58, 58	58	91
			689/23	57, 56	57	92
	2,3,5,6-TCNB	0.2	0/0	93, 91	92	88
			30/1	91, 87	89	94
			63/2	66, 54	60	79
			93/3	76, 60	68	82
			182/6	54, 71	62	84
			439/14	47, 54	51	94
			689/23	45, 50	47	93
	HCB	0.2	0/0	93, 92	92	88
			30/1	91, 88	89	93
			63/2	73, 58	66	86
			93/3	78, 62	70	85
			182/6	58, 77	67	86
			439/14	55, 55	55	95
			689/23	51, 49	50	89
Peanut	Quintozene	0.2	0/0	96, 94	95	85
			30/1	98, 98	98	91
			63/2	89, 94	92	82
			97/3	93, 92	92	88
			182/6	93, 91	92	89
			439/14	99, 96	98	89
	PCA	0.2	0/0	97, 97	97	97
			30/1	94, 97	95	101
			63/2	98, 108	103	92

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			97/3	94, 93	94	100
			182/6	102, 95	99	101
			439/14	117, 120	118	98
	PCTA	0.2	0/0	98, 98	98	101
			30/1	93, 96	94	105
			63/2	99, 85	92	94
			97/3	96, 95	95	103
			182/6	99, 99	99	105
			439/14	80, 83	81	103
	PB	0.2	0/0	96, 97	96	89
			30/1	91, 96	93	90
			63/2	94, 86	90	80
			97/3	96, 87	91	90
			182/6	96, 95	95	91
			439/14	84, 88	86	86
	2,3,4,5-TCNB	0.2	0/0	97, 97	97	95
			30/1	92, 98	95	99
			63/2	96, 87	91	88
			97/3	94, 89	91	97
			182/6	89, 89	89	95
			439/14	91, 93	92	96
	2,3,5,6-TCNB	0.2	0/0	89, 89	89	88
			30/1	92, 96	94	99
			63/2	97, 87	92	88
			97/3	89, 86	87	93
			182/6	88, 88	88	94
			439/14	74, 84	79	94
	HCB	0.2	0/0	97, 98	97	94
			30/1	92, 97	94	97
			63/2	96, 88	92	88
			97/3	97, 92	94	97
			182/6	95, 94	94	96
			439/14	79, 83	81	91
Broccoli	Quintozene	0.2	0/0	82, 86	84	77
			30/1	86, 85	85	87
			68/2	75, 80	77	70
			93/3	99	99	97
			97/3	91, 114	103	99
			182/6	79, 87	83	78
			439/14	88, 87	87	84
	PCA	0.2	0/0	84, 65	75	85
			30/1	90, 89	89	95
			68/2	91, 86	88	76
			93/3	104, 86	95	90
			97/3	93, 108	100	96
			182/6	83, 76	79	86
			439/14	95, 91	93	92
	PCTA	0.2	0/0	90, 94	92	92
			30/1	88, 94	91	100
			68/2	90, 87	88	78
			93/3	121, 96	109	119
			97/3	101, 124	112	117
			182/6	90, 81	86	93

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			439/14	103, 96	99	94
	PB	0.2	0/0	86, 89	87	79
			30/1	92, 95	93	85
			68/2	95, 91	93	73
			93/3	115, 90	102	98
			97/3	103, 128	115	104
			182/6	89, 81	85	80
			439/14	99, 97	98	84
	2,3,4,5-TCNB	0.2	0/0	85, 77	81	84
			30/1	91, 93	92	93
			68/2	89, 86	87	75
			93/3	106, 82	94	94
			97/3	103, 122	112	105
			182/6	90, 75	82	84
			439/14	99, 95	97	92
	2,3,5,6-TCNB	0.2	0/0	78, 65	71	76
			30/1	85, 84	85	92
			68/2	85, 79	82	77
			93/3	93, 71	82	92
			97/3	74, 87	80	84
			182/6	68, 61	64	82
			439/14	92, 87	89	96
	HCB	0.2	0/0	85, 90	87	84
			30/1	90, 94	92	91
			68/2	91, 88	89	75
			93/3	115, 93	104	107
			97/3	103, 129	116	122
			182/6	89, 80	84	86
			439/14	98, 96	97	90

When frozen storage stability was tested at the fortification level of 0.2 mg/kg, quintozene, PCA, PCTA, 2,3,4,5-TCNB and HCB were stable for at least 14 months (longest period tested) in samples of potato, tomato, peanut and broccoli. 2,3,5,6-TCNB was stable for at least 14 months in tomato, peanut and broccoli samples but for less than 1 month in potato sample.

In snap beans, quintozene, PCTA, PB, 2,3,4,5-TCNB, HCB declined to below 70 percent of the fortified level when stored for longer than 3 months, and 2,3,5,6-TCNB for at least 1 month whereas PCA remained stable for at least 23 months (longest period tested for snap beans).

Study 3. (Gaydosh K.A., 1999, 900-RES-149)

The stability of quintozene and its metabolites PCA, PCTA and PB, and the impurity HCB was investigated in fortified control samples of lettuce, turnip roots and tops, and wheat whole plant, grain and straw during freezer storage at -20±5 °C for 0–24 months. Ground control samples (10 g) of lettuce, turnip roots and tops, and wheat whole plant, grain and straw were fortified in duplicate with quintozene and the metabolites PCA, PCTA and PB, and the impurity HCB at a level of 1.0 mg/kg and stored frozen over the given time frame of 1–24 months. Day zero samples were extracted immediately after fortification. All samples were analysed by CAM-24-73 (modified) method with an LOQ of 0.005 mg/kg for each analyte. The results of the storage stability test on quintozene and its metabolites and impurities in these commodities are presented in the table below.

Table 65 Storage stability of quintozene, PCA, PCTA, PB and HCB in lettuce, turnip roots and tops, and wheat whole plant, grain and straw at -20±5 °C

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
Lettuce	Quintozene	1.0	0	88, 85	87	86 / 84
			1	77, 73	75	84 / 89
			2	79, 77	78	88 / 93
			4	71, 7	71	77 / 78
			6	86, 81	84	77 / 87
			9	75, 63	69	82 / 81
			12	98, 93	95	87 / 96
			15	93, 101	97	108 / 85
			18	102, 94	98	99 / 96
			24	65, 67	66	79 / 88
	PCA	1.0	0	78, 80	79	89 / 98
			1	82, 91	87	95 / 102
			2	94, 81	87	93 / 96
			4	83, 89	86	93 / 93
			6	96, 91	94	109 / 108
			9	82, 80	81	88 / 90
			12	83, 83	83	85 / 93
			15	93, 74	83	106 / 81
			18	102, 104	103	107 / 96
			24	71, 73	72	90 / 83
	PCTA	1.0	0	91, 91	91	107 / 99
			1	96, 83	90	110 / 109
			2	94, 90	92	88 / 95
			4	90, 90	90	86 / 85
			6	89, 81	85	100 / 88
			9	82, 78	80	89 / 87
			12	107, 91	99	85 / 98
			15	78, 83	80	87 / 86
			18	89, 80	85	85 / 84
			24	56, 59, 79, 79	68	74 / 75
	PB	1.0	0	88, 86	87	83 / 85
			1	84, 82	83	90 / 88
			2	89, 87	88	89 / 89
			4	81, 79	80	77 / 81
			6	80, 80	80	85 / 85
			9	76, 83	80	82 / 85
			12	65, 70	68	72 / 80
			15	82, 75	79	87 / 78
			18	85, 76	81	73 / 80
			24	81, 76	79	85 / 98
	HCB	1.0	0	94, 90	92	90 / 89
			1	84, 81	82	94 / 96
			2	86, 78	82	91 / 94
			4	80, 82	81	78 / 85
			6	83, 89	86	89 / 90
			9	76, 74	75	74 / 76
			12	93, 93	93	80 / 92
			15	92, 76	84	92 / 79
			18	99, 81	90	85 / 91
			24	79, 74	77	87 / 92

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
Turnip tops	Quintozene	1.0	0	70, 75	72	83 / 81
			1	85, 78	81	91 / 81
			2	78, 73	75	82 / 81
			4	70, 64	67	81 / 71
			6	61, 67	64	79 / 78
			9	72, 84	78	80 / 79
			12	93, 84	89	95 / 85
			15	101, 100	100	103 / 104
			18	89, 88	89	83 / 89
			24	73, 80	76	97 / 89
	PCA	1.0	0	78, 85	82	85 / 87
			1	87, 99	93	92 / 96
			2	92, 80	86	93 / 89
			4	84, 87	86	81 / 84
			6	87, 83	85	101 / 98
			9	98, 87	93	82 / 81
			12	80, 84	82	90 / 83
			15	89, 86	87	90 / 91
			18	81, 87	84	90 / 88
			24	93	93	101 / 109
	PCTA	1.0	0	70, 74	72	79 / 79
			1	84, 84	84	89 / 97
			2	89, 87	88	88 / 91
			4	76, 88	82	86 / 84
			6	88, 64	76	91 / 92
			9	71, 75	73	75 / 80
			12	82, 83	82	88 / 83
			15	79, 90	85	87 / 95
			18	85, 76	81	84 / 81
			24	68, 64	66	68 / 74
	PB	1.0	0	71, 63	67	77 / 81
			1	76, 76	76	81 / 84
			2	75, 69	72	82 / 81
			4	69, 62	66	78 / 80
			6	58, 54	56	69 / 71
			9	64, 48	56	58 / 73
			12	72, 73	72	78 / 77
			15	61, 62	62	77 / 82
			18	74, 67	70	84 / 80
			24	73	73	93 / 96
	HCB	1.0	0	74, 80	77	83 / 84
			1	76, 71	74	83 / 91
			2	75, 79	77	84 / 87
			4	75, 70	72	83 / 83
			6	74, 74	74	74 / 77
			9	68, 55	62	68 / 74
			12	80, 93	86	76 / 75
			15	77, 81	79	83 / 89
			18	73, 77	75	82 / 83
			24	80, 93	87	96 / 101
Turnip roots	Quintozene	1.0	0	72, 93	82	83 / 83
			1	75, 69	72	87 / 79
			2	63, 65	64	73 / 79

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			4	64, 65	65	92 / 72
			6	95, 80	88	88 / 88
			9	82, 75	78	73 / 86
			12	93, 90	91	85 / 99
			15	90, 88	89	77 / 80
			18	61, 98	80	81 / 86
			24	91, 92	91	82 / 82
	PCA	1.0	0	96, 85	91	96 / 95
			1	85, 87	86	91 / 94
			2	88, 104	96	83 / 90
			4	85, 82	83	82 / 82
			6	114, 99	106	109 / 100
			9	82, 87	84	76 / 93
			12	95, 94	94	91 / 98
			15	102, 97	100	85 / 90
			18	87, 94	90	86 / 90
			24	102, 99	101	90 / 92
	PCTA	1.0	0	88, 101	95	92 / 97
			1	90, 85	88	91 / 93
			2	79, 92	86	87 / 89
			4	97, 89	93	79 / 83
			6	89, 75	82	91 / 92
			9	72, 69	70	79 / 91
			12	89, 88	88	96 / 99
			15	83, 65	74	76 / 77
			18	71, 93	82	83 / 89
			24	86, 90	88	85 / 87
	PB	1.0	0	79, 79	79	82 / 82
			1	81, 89	85	92 / 93
			2	82, 85	84	80 / 88
			4	82, 86	84	78 / 89
			6	89, 80	85	91 / 93
			9	72, 80	76	72 / 82
			12	77, 84	80	93 / 91
			15	82, 80	81	80 / 79
			18	67, 75	71	83 / 84
			24	91, 84	87	83 / 85
	HCB	1.0	0	63, 84	74	74 / 71
			1	79, 87	83	90 / 93
			2	85, 86	86	76 / 83
			4	84, 85	84	72 / 81
			6	91, 82	87	88 / 91
			9	67, 68	68	70 / 83
			12	92, 103	98	100 / 102
			15	88, 81	85	78 / 82
			18	69, 96	82	82 / 89
			24	88, 100	94	84 / 88
Wheat whole plant	Quintozene	1.0	0	82, 81	81	79 / 84
			1	78, 80	79	79 / 82
			2	85, 89	87	90 / 90
			4	92, 75	84	88 / 102
			6	77, 86	81	77 / 94
			9	80, 78	79	102 / 97

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			12	70, 77	73	74 / 72
			15	98, 96	97	98 / 91
			18	105, 74	89	78 / 94
			24	75, 72	73	73 / 80
	PCA	1.0	0	92, 93	93	89 / 93
			1	76, 81	79	89 / 91
			2	101, 97	99	101 / 66
			4	92, 88	90	108 / 109
			6	99, 76	88	94 / 100
			9	85, 101	93	114 / 105
			12	83, 87	85	91 / 83
			15	100, 94	97	105 / 95
			18	92, 85	89	92 / 95
			24	79, 80	80	85 / 93
	PCTA	1.0	0	76, 75	75	76 / 81
			1	76, 77	76	79 / 83
			2	80, 80	80	86 / 88
			4	75, 75	75	91 / 94
			6	93, 74	83	92 / 100
			9	74, 75	74	87 / 90
			12	64, 64	64	76 / 73
			15	83, 84	83	85 / 81
			18	79, 76	78	79 / 87
			24	70, 76	73	75 / 79
	PB	1.0	0	80, 85	83	77 / 82
			1	86, 87	87	81 / 82
			2	84, 87	86	86 / 89
			4	83, 81	82	92 / 98
			6	78, 75	77	80 / 86
			9	77, 72	75	92 / 92
			12	72, 75	73	81 / 81
			15	80, 71	76	74 / 71
			18	71, 71	71	74 / 81
			24	80, 74	77	80 / 79
	HCB	1.0	0	75, 76	75	72 / 80
			1	81, 79	80	80 / 84
			2	82, 79	80	81 / 90
			4	83, 85	84	91 / 103
			6	68, 71	70	75 / 92
			9	80, 67	74	83 / 88
			12	74, 72	73	74 / 78
			15	90, 89	89	90 / 94
			18	98, 83	91	81 / 97
			24	64, 74	69	70 / 77
Wheat grain	Quintozene	1.0	0	73, 81	77	81 / 88
			1	82, 74	78	72 / 80
			2	84, 82	83	84 / 92
			4	79, 73	76	84 / 85
			6	65, 67	66	78 / 82
			9	89, 79	84	101 / 101
			12	92, 76	84	77 / 83
			15	74, 73	74	89 / 91
			18	73, 65	69	69 / 74

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			24	74, 69	72	72 / 86
	PCA	1.0	0	76, 84	80	84 / 88
			1	71, 72	72	74 / 75
			2	97, 98	97	92 / 98
			4	76, 73	75	83 / 87
			6	82, 64	73	87 / 85
			9	98, 89	94	105 / 104
			12	77, 79	78	72 / 78
			15	82, 81	81	100 / 101
			18	77, 75	76	74 / 78
			24	84, 80	82	67 / 87
	PCTA	1.0	0	84, 92	88	88 / 92
			1	84, 71	78	78 / 86
			2	98, 101	99	91 / 100
			4	76, 71	74	80 / 87
			6	74, 64	69	77 / 85
			9	89, 78	84	95 / 96
			12	76, 80	78	78 / 82
			15	80, 74	77	95 / 96
			18	76, 69	73	77 / 78
			24	86, 72	79	73 / 90
	PB	1.0	0	80, 79	80	81 / 84
			1	71, 77	74	80 / 80
			2	77, 71	74	75 / 81
			4	80, 71	76	81 / 84
			6	66, 67	66	80 / 85
			9	67, 68	67	82 / 82
			12	75, 75	75	70 / 78
			15	69, 74	72	90 / 86
			18	71, 73	72	81 / 77
			24	78, 73	75	83 / 89
	HCB	1.0	0	81, 79	80	80 / 85
			1	72, 73	73	77 / 79
			2	82, 84	83	82 / 91
			4	69, 64	66	76 / 81
			6	70, 67	69	80 / 84
			9	89, 84	87	99 / 97
			12	73, 73	73	74 / 81
			15	79, 72	76	90 / 90
			18	71, 68	69	74 / 77
			24	73, 69	71	77 / 89
Wheat straw	Quintozene	1.0	0	83, 78	80	72 / 65
			1	81, 79	80	77 / 85
			2	92, 84	88	96 / 98
			4	72, 72	72	90 / 87
			6	64, 67	66	79 / 80
			9	87, 92	89	113 / 108
			12	108, 81	95	102 / 96
			15	66, 74	70	74 / 78
			18	75, 66	71	61 / 81
			24	81, 72	77	74 / 81
	PCA	1.0	0	96, 87	92	84 / 73
			1	75, 78	76	71 / 82

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (months)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			2	91, 85	88	93 / 99
			4	78, 81	80	78 / 93
			6	86, 87	87	89 / 95
			9	84, 90	87	101 / 103
			12	95, 67	81	84 / 83
			15	53, 74, 74	67	80 / 83
			18	76, 62	69	74 / 88
			24	82, 82	82	76 / 87
	PCTA	1.0	0	95, 92	94	83 / 74
			1	72, 69	71	66 / 73
			2	77, 72	74	77 / 80
			4	79, 72	76	80 / 82
			6	75, 72	73	81 / 84
			9	89, 91	90	98 / 99
			12	91, 81	86	81 / 81
			15	66, 72	69	77 / 80
			18	75, 75	75	69 / 80
			24	80, 80	80	74 / 73
	PB	1.0	0	84, 81	82	79 / 67
			1	75, 76	75	78 / 81
			2	76, 72	74	78 / 81
			4	74, 71	73	87 / 87
			6	72, 74	73	82 / 88
			9	88, 82	85	97 / 98
			12	77, 77	77	90 / 85
			15	62, 71	67	86 / 85
			18	72, 68	70	76 / 79
			24	74, 78	76	80 / 89
	HCB	1.0	0	86, 84	85	78 / 69
			1	81, 80	80	74 / 81
			2	92, 77	85	83 / 87
			4	75, 79	77	87 / 90
			6	77, 76	76	83 / 85
			9	107, 96	101	101 / 106
			12	98, 87	93	96 / 91
			15	66, 75	71	80 / 85
			18	75, 69	72	65 / 79
			24	75, 79	77	80 / 86

When frozen storage stability was tested at the fortification level of 1.0 mg/kg at -20±5 °C, quintozene, PCA, PCTA, PB and HCB were found to be stable in lettuce, turnip roots and tops and wheat whole plant, grain and straw for at least 24 months (longest period tested).

Study 4. (Gaydosh, 1991, 900-RES-097; Ruhland, 1991, 900-RES-167a; and Keller, 1991, 900-RES-167b)

The stability of quintozene and its metabolites PCA, PCTA and PB, and the impurity HCB was investigated in fortified control samples of cotton seed during freezer storage at below 0 °C for 0–18 months. Ground control samples of cotton seed (10 g) were fortified in duplicate with quintozene and the metabolites PCA, PCTA and PB, and the impurity HCB at a level of 0.2 mg/kg and stored frozen over the given time frame of 1–18 months. Day zero samples were extracted immediately after fortification. All samples were analysed

by MP-Quintozene-MA method with an LOQ of 0.005 mg/kg for each analyte. The results of the storage stability test on quintozene and its metabolites and impurities in cotton seed are presented in the table below.

Table 66 Storage stability of quintozene, PCA, PCTA, PB and HCB in cotton seed at below 0 °C

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
Cotton seed	Quintozene	0.2	0/0	94, 92	93	91
			30/1	90, 93	92	96
			60/2	91, 81	86	104
			90/3	90, 90	90	96
			180/6	89, 87	88	95
			360/12	95, 92	93	95
			540/18	82, 81	82	87
	PCA	0.2	0/0	84, 90	87	88
			30/1	88, 92	90	92
			60/2	100, 76	88	103
			90/3	90, 86	88	85
			180/6	88, 87	88	107
			360/12	99, 96	98	90
			540/18	76, 75	76	85
	PCTA	0.2	0/0	80, 92	86	92
			30/1	91, 94	92	95
			60/2	91, 83	87	110
			90/3	95, 95	95	93
			180/6	94, 93	93	92
			360/12	91, 98	94	78
			540/18	86, 82	84	89
	PB	0.2	0/0	96, 96	96	94
			30/1	91, 92	91	94
			60/2	91, 83	87	99
			90/3	97, 98	97	97
			180/6	92, 91	91	106
			360/12	99, 96	97	98
			540/18	88, 90	89	90
	HCB	0.2	0/0	87, 88	88	86
			30/1	85, 86	86	87
			60/2	84, 73	78	92
			90/3	85, 88	86	84
			180/6	83, 81	82	97
			360/12	88, 85	86	86
			540/18	85, 83	84	88

When frozen storage stability was tested at the fortification level of 0.2 mg/kg at below 0 °C, in cotton seeds, quintozene, PCA, PCTA, PB and HCB were found to be stable for at least 18 months (longest time tested).

Study 5. (Ball, 1990, 900-RES-144)

The stability of quintozene and its metabolites PCA, PCTA and PB, and the impurity HCB was investigated in fortified potato control samples during freezer storage at -20 °C±2 °C for 0–12 months. Ground control potato samples were fortified in duplicate with quintozene and the metabolites PCA, PCTA and PB, and the impurity HCB (fortification level not reported) and stored frozen over the given time frame of 1–12

months. Day zero samples were extracted immediately after fortification. All samples were analysed by CAM-24-73 (modified) method with an LOQ of 0.0005 mg/kg for each analyte. The results of the storage stability test on quintozene and its metabolites and impurities in potato tubers are presented in the table below.

Table 67 Storage stability of quintozene, PCA, PCTA, PB and HCB in potato at -20±2 °C

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
Potato tuber	Quintozene	Unknown	0/0	81, 86, 83, 85	84	--
			33/1	78, 89	84	96, 100
			70/2	90, 94	92	111, 112
			95/3	72, 80	76	87, 90
			186/6	83, 87	85	105, 104
			279/9	83, 84	84	115, 116
			368/12	103, 98	100	116, 116
	PCA	Unknown	0/0	78, 83, 89, 88	85	--
			33/1	100, 107	104	110, 109
			70/2	79, 85	83	86, 90
			95/3	103, 89	76	81, 94
			186/6	89, 91	90	88, 90
			279/9	88, 88	88	94, 87
			368/12	92, 94	93	83, 86
	PCTA	Unknown	0/0	101, 98, 90, 110	100	--
			33/1	104, 104	104	108, 107
			70/2	93, 96	94	94, 92
			95/3	85, 85	85	86, 88
			186/6	102, 101	102	101, 104
			279/9	91, 95	93	99, 101
			368/12	97, 92	94	83, 83
	PB	Unknown	0/0	110, 110, 105, 101	107	--
			33/1	84, 102	93	104, 106
			70/2	84, 88	86	83, 93
			95/3	93, 100	96	106, 115
			186/6	88, 91	90	101, 100
			279/9	93, 93	93	94, 85
			368/12	104, 100	102	101, 100
	HCB	Unknown	0/0	99, 106, 101, 100	102	--
			33/1	71, 73	72	86, 84
			70/2	77, 77	77	101, 104
			95/3	98, 102	100	114, 109
			186/6	77, 83	80	90, 101
			279/9	90, 88	89	98, 94
			368/12	75, 73	74	94, 91

In potato tubers, quintozene, PCA, PCTA, PB and HCB were found to be stable for at least 12 months when stored frozen at -20±2 °C. However, since the fortification level was unknown, whether the stability in this study applies to the residues arising in the supervised residue trials.

Study 6. (Gaydosh, 1991, 900-RES-096)

The stability of quintozene and its metabolites PCA, PCTA and PB, and the impurity HCB was investigated in fortified control samples of peanut seed during freezer storage at below 0 °C for 0–12 months. Ground control samples (10 g) of peanuts were fortified in duplicate with quintozene, the metabolites and the

impurity HCB at a level of 0.2 mg/kg and stored frozen over the given time frame of 1–12 months. Day zero samples were extracted immediately after fortification. All samples were analysed by a slightly modified MP-PCNC-MA1 method using hexane as the extraction solvent with an LOQ of 0.005 mg/kg for each analyte. The results of the storage stability test on quintozene and its metabolites and impurities in peanut seed are presented in the table below.

Table 68 Storage stability of quintozene, PCA, PCTA, PB and HCB in peanut at below 0 °C

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
Peanut, seed	Quintozene	0.2	0/0	86, 84	85	88
			30/1	81, 85	83	93
			60/2	74, 72	73	86
			90/3	85, 84	84	94
			120/4	81, 78	79	88
			180/6	81, 85	83	93
			360/12	96, 91	93	89
	PCA	0.2	0/0	84, 84	84	88
			30/1	82, 84	93	123
			60/2	72, 61	66	74
			90/3	78, 85	81	86
			120/4	83, 81	82	95
			180/6	78, 85	81	98
			360/12	100, 94	97	87
	PCTA	0.2	0/0	84, 84	84	88
			30/1	87, 87	87	92
			60/2	69, 82	75	71
			90/3	88, 83	85	87
			120/4	82, 84	83	95
			180/6	83, 89	86	96
			360/12	89, 92	91	85
	PB	0.2	0/0	86, 85	86	84
			30/1	80, 85	82	87
			60/2	63, 68	65	61
			90/3	83, 81	82	84
			120/4	75, 73	74	81
			180/6	81, 87	84	96
			360/12	98, 92	95	90
	HCB	0.2	0/0	81, 80	80	83
			30/1	77, 80	79	85
			60/2	66, 69	67	62
			90/3	82, 77	79	78
			120/4	77, 73	75	84
			180/6	78, 82	80	92
			360/12	99, 94	96	89

When frozen storage stability was tested at the fortification level of 0.2 mg/kg at below 0 °C, in peanut seed, quintozene, PCA, PCTA, PB and HCB were stable for at least 12 months (longest period tested).

Study 7. *(Stenner et al, 1992, 900-RES-023)*

The stability of quintozene and its metabolites PCA, PCTA, PB, 2,3,4,5-TCNB and 2,3,5,6-TCNB, and the impurity HCB was investigated in fortified control samples of peanut whole nut, shells and nutmeat during

freezer storage (temperature not reported) for 0–6 months. Homogenized control samples (25 g) of peanut whole nut, shells and nutmeat were fortified in duplicate with quintozene and separately with a mixture of PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and the impurity HCB at a level of 0.2 mg/kg and stored frozen for a maximum of 6 months (176 days). Day zero samples were extracted immediately after fortification. All samples were analysed by MP-PCNC-MA1 method with an LOQ of 0.005 mg/kg for each analyte. The results of the storage stability test on quintozene and its metabolites and impurity in peanut whole nut, shells and nutmeat are presented in the table below.

Table 69 Storage stability of quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB in peanut when frozen

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
Peanut, Whole nut	Quintozene	0.2	0/0	98, 95	97	94
			176/6	81, 81	81	84
	PCA	0.2	0/0	99, 107	103	99
			176/6	90, 86	88	89
	PCTA	0.2	0/0	94, 103	99	96
			176/6	87, 85	86	86
	PB	0.2	0/0	94, 101	98	95
			176/6	82, 82	82	82
	2,3,4,5-TCNB	0.2	0/0	90, 97	93	92
			176/6	85, 84	84	87
	2,3,5,6-TCNB	0.2	0/0	91, 101	96	94
			176/6	80, 77	78	81
	HCB	0.2	0/0	95, 102	98	96
			176/6	82, 82	82	83
Peanut, Shell	Quintozene	0.2	0/0	84, 81	82	93
			176/6	76, 67	71	93
	PCA	0.2	0/0	84, 87	85	92
			176/6	64, 75	69	97
	PCTA	0.2	0/0	84, 87	86	94
			176/6	69, 78	73	96
	PB	0.2	0/0	85, 88	87	93
			176/6	65, 72	69	94
	2,3,4,5-TCNB	0.2	0/0	86, 87	86	95
			176/6	57, 68	62	91
	2,3,5,6-TCNB	0.2	0/0	81, 83	82	90
			176/6	55, 66	60	93
	HCB	0.2	0/0	86, 90	88	93
			176/6	70, 78	74	91
Peanut, Nutmeat	Quintozene	0.2	0/0	103, 94	98	98
			176/6	90, 84	87	85
	PCA	0.2	0/0	98, 97	97	99
			176/6	90, 89	89	92
	PCTA	0.2	0/0	94, 97	95	98
			176/6	88, 88	88	89
	PB	0.2	0/0	94, 96	95	97
			176/6	83, 85	84	84
	2,3,4,5-TCNB	0.2	0/0	91, 107	99	109
			176/6	89, 89	89	88
	2,3,5,6-TCNB	0.2	0/0	92, 95	93	96
			176/6	85, 86	85	81
	HCB	0.2	0/0	92, 96	94	96

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (percent)
				Individual	Mean	
			176/6	84, 86	85	84

When frozen storage stability was tested at the fortification level of 0.2 mg/kg, quintozene, PCA, PCTA, PB and HCB were stable for at least 6 months (longest period tested) in frozen samples of peanut whole nut, shells and nutmeat. 2,3,4,5-TCNB and 2,3,5,6-TCNB were stable for at least 6 months in whole nut and nutmeat but in shells, degraded to less than 70 percent of the fortified levels after storage for 6 months. However, the residue levels at time zero were as low as 86 percent and 82 percent of fortified level, respectively.

Study 8. (Gaydosh, 1991, 900-RES-097; Ruhland , 1991, 900-RES-167a; Keller J.F., 1991, 900-RES-167b)

The stability of quintozene and its metabolites PCA and PCTA was investigated in fortified control samples of broccoli and potato tubers during freezer storage at below -20 °C for 0–12 months. Ground control samples (10 g) of broccoli or potato tubers were fortified in duplicate with quintozene and the metabolites PCA and PCTA at a level of 0.1 mg/kg and stored frozen over 1–12 months. Day zero samples were extracted immediately after fortification. All samples were analysed by the QuEChERS method as reported in Battelle Study Number: 100117568 with an LOQ of 0.01 mg/kg for each analyte. The results of the storage stability test on quintozene and its metabolites in broccoli and potato tubers are presented in table below.

Table 70 Storage stability of quintozene, PCA and PCTA in broccoli and potato at below -20 °C

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (mean)(percent)
				Individual	Mean	
Broccoli	Quintozene m/z = 295	0.1	0/0	95, 85	90	90, 91 (91)
			32/1	90, 91	91	94, 93 (93)
			186/6	80, 74	77	90, 86 (88)
			355/12	109, 89	99	107, 90 (98)
	Quintozene m/z = 297	0.1	0/0	89, 83	86	90, 91 (91)
			32/1	90, 91	90	95, 93 (94)
			186/6	70, 63	67	87, 77 (82)
			355/12	107, 91	99	101, 84 (93)
	Quintozene m/z = 293	0.1	0/0	95, 84	89	90, 91 (91)
			32/1	89, 91	90	94, 94 (94)
			186/6	73, 79	76	74, 71 (72)
			355/12	103, 85	94	102, 77 (90)
	PCA m/z = 265	0.1	0/0	89, 83	86	90, 88 (89)
			32/1	87, 88	88	94, 92 (93)
			186/6	72, 72	72	81, 72 (76)
			355/12	96, 92	94	89, 87 (88)
	PCA m/z = 267	0.1	0/0	87, 82	85	92, 87 (89)
			32/1	86, 86	86	92, 91 (91)
			186/6	74, 76	75	89, 74 (81)
			355/12	90, 90	90	93, 100 (96)
	PCA m/z = 263	0.1	0/0	90, 83	86	90, 89 (90)
			32/1	88, 88	88	94, 92 (93)
			186/6	78, 72	75	82, 70 (76)
			355/12	91, 96	94	82, 85 (83)
	PCTA	0.1	0/0	83, 73	78	78, 78 (78)

Matrix	Analyte	Fortification Level (mg/kg)	Storage Interval (d/month)	percent Remaining		Procedural Recovery (mean)(percent)
				Individual	Mean	
	m/z = 296		32/1	77, 78	78	83, 82 (82)
			186/6	70, 68	69	70, 69 (70)
			355/12	80, 77	78	76, 75 (76)
	PCTA m/z = 294	0.1	0/0	81, 74	77	78, 79 (78)
			32/1	79, 79	79	84, 83 (84)
			186/6	66, 63	65	70, 61 (65)
			355/12	80, 74	77	74, 75 (75)
	PCTA m/z = 246	0.1	0/0	79, 75	77	76, 78 (77)
			32/1	81, 80	80	86, 84 (85)
			186/6	121, 92	107	93, 109 (101)
			355/12	85, 84	85	87, 78 (83)
Potato tuber	Quintozene m/z = 295	0.1	0/0	90, 89	89	89, 80 (84)
			32/1	91, 99	95	97, 97 (97)
			186/6	79, 88	84	92, 91 (91)
			355/12	104, 74	89	72, 97 (84)
	Quintozene m/z = 297	0.1	0/0	90, 89	89	90, 78 (84)
			32/1	90, 98	93	96, 97 (96)
			186/6	88, 85	86	109, 99 (104)
			355/12	102, 77	89	75, 97 (86)
	Quintozene m/z = 293	0.1	0/0	87, 89	88	92, 80 (86)
			32/1	92, 102	96	97, 97 (97)
			186/6	76, 79	78	88, 84 (86)
			355/12	97, 74	85	73, 94 (84)
	PCA m/z = 265	0.1	0/0	88, 88	88	88, 82 (85)
			32/1	94, 104	99	92, 95 (93)
			186/6	87, 88	87	81, 87 (84)
			355/12	119, 87	103	77, 99 (88)
	PCA m/z = 267	0.1	0/0	88, 89	89	87, 82 (84)
			32/1	93, 104	98	90, 93 (92)
			186/6	71, 85	78	84, 81 (82)
			355/12	118, 80	99	77, 99 (88)
	PCA m/z = 263	0.1	0/0	89, 90	89	89, 84 (87)
			32/1	95, 105	100	92, 94 (93)
			186/6	79, 83	81	86, 80 (83)
			355/12	115, 82	98	74, 93 (83)
	PCTA m/z = 296	0.1	0/0	85, 86	85	85, 80 (82)
			32/1	88, 99	93	86, 89 (88)
			186/6	70, 69	70	75, 73 (74)
			355/12	111, 78	95	70, 85 (78)
	PCTA m/z = 294	0.1	0/0	87, 85	86	85, 80 (82)
			32/1	87, 98	92	87, 90 (88)
			186/6	65, 69	67	71, 71 (71)
			355/12	106, 79	93	70, 83 (77)
	PCTA m/z = 246	0.1	0/0	87, 87	87	85, 80 (83)
			32/1	85, 97	91	88, 90 (89)
			186/6	66, 65	66	74, 70 (72)
			355/12	119, 76	97	70, 83 (76)

When frozen storage stability was tested at the fortification level of 0.1 mg/kg at -20 °C or below, quintozene, PCA and PCTA were stable in broccoli and potato tubers for at least 12 months (longest period tested).

Summary of frozen storage stability of quintozene, PCA, PCTA, PB and HCB is shown below

Category	Commodity	Duration of study (month)	Stable period (months) [a] Quintozene	PCA	PCTA	PB	HCB
High water content	Broccoli	14	14	14	14	14	14
	Peppers	14	Up to 4	Up to 4	Up to 4	<2	Up to 4
	Tomato	14	14	14	14	14	14
	Lettuce	24	24	24	Up to 18	24	24
	Snap bean	23	Up to 3	23	Up to 3	Up to 3	Up 3
	Turnip tops	24	24	24	Up to 18	24	24
	Wheat immature whole plant	24	24	24	24	24	Up to 18
High protein content	Kidney bean	14	14	14	14	Up to 6	14
	Soya bean	8	<2	<2	<2	<2	<2
High starch content	Potato	14	14	14	14	14	14
	Turnip root	14	24	24	24	24	24
	Corn	8	<2	<2	<2	<2	<2
	Wheat grain	24	24	24	24	24	24
High oil content	Cotton seed	18	18	18	18	18	18
	Peanut	14	14	14	14	14	14
Dry sample	Wheat straw	24	24	24	24	24	24

Notes:

[a] Where the value of stable period is the same as the duration of study, this indicates that the fortified compound was stable at least for the months in this table. Where the term "up to" precedes the months of stable period, the fortified compound would not be stable after the months specified. The month value with the "<" symbol indicate that significant degradation occurred by the months described.

USE PATTERN

Quintozene is registered in many countries. For the purposes of estimating maximum residue levels, only the registered uses on those crops for which supervised trials were conducted and provided to the Meeting are recorded in the following table. Quintozene is allowed for pre-plant treatment as a broadcast application to bare soil followed by soil incorporation; treatment at planting as an in-furrow application directed to the seed; a post-emergence banded application directed to the soil; or as a transplant solution after transplanting of the crop.

Table 71 Registered uses of Quintozene for the crops for which supervised trials were provided

Crop	country	Form g ai/L or kg	Application Method	Rate kg ai/ha	Max rate/season kg ai/ha	Conc kg ai/ hL	No	PHI days	Note
Broccoli and Cabbage [b]	US	FL 480 [a]	Transplant solution.	1.68	25.2	0.18	-	--	Thoroughly incorporate solution into the soil. Up to 0.35 L of solution per plant.
			Banded soil application, pre-planting	25.2	25.2	10.8	1	--	Spray as a 12-inch band centred on row and incorporate to a depth of 4 to 6 inches immediately prior to planting. May be used on direct seeded cole crops. Thoroughly incorporate solution into the

Crop	country	Form g ai/L or kg	Application					PHI days	Note
			Method	Rate kg ai/ha	Max rate/season kg ai/ha	Conc kg ai/ hL	No		
									soil.
			Broadcast application, pre-plant	25.2	25.2	8.98	1	--	Thoroughly incorporate to a depth of 4 to 6 inches using a disc or other suitable equipment. Thoroughly incorporate solution into the soil.
			Broadcast drench application	12.5–16.8	25.2	2.69–3.60	-	--	Apply as a soil drench at the time of or immediately after seeding.
			Row drench treatments	8.53–12.5	25.2	2.61–3.84	-	--	Spray as an 8-inch band centered on the row at time of or immediately after seeding.
Broccoli, cabbage, Brussel sprout and Cauliflower	MX	FL 480 [c]	Total ground treatment, Band application at sowing, Transplant application	14.4–19.2			1	--	For total ground treatment, incorporate to a depth of 10–15 cm. Apply in band at sowing. At transplant, apply 0.17 kg ai per plant
Cabbage	MY	WP 750	Soil application after transplanting	12		0.20	1	--	
Tomato	MX	FL 480 [c]	Application in nursery	9.6–14.4		0.34	1	--	Apply 40 ml in 5L of water per m^2 of nursery.
Tomato (kidney tomato)	EC	WP 750	Greenhouse use	0.56		0.093		1	Use under greenhouse conditions.
Tomato	TH	EC 240	Soil application.	--		1.23–-1.50	≥2	--	Spray the soil all over: first time, immediately after planting young tomato plant; and repeat spraying every 14 days at least 2 times.
Chile pepper	MX	FL 480 [c]	Application in nursery	9.6–14.4		0.34	-	--	Apply 40 ml in 5L of water per m^2 of nursery.
Beans	Mx	FL 480 [c]	Band application at sowing	0.72–1.44			1	--	Application at sowing.
			Band application, at flowering	4.8–8.64			1	--	Apply to both sides of the row of plants covering the basal part of the stem and furrow, at the beginning of flowering.
Beans	MX	WP 750	Mix with seeds Hopper box	0.75–1.50			1	--	Indirect treatment to the soil using higher doses than those uses in a simple seed treatment. In hopper box application, the seed is also used as a vehicle.
			Band application	2.25–4.50			1	--	For established crops, application in bands of 20–30 cm on both sides of the row, perfectly covering the crowns of the plants after cultivation and immediately before raising the furrow to irrigate. The application can be made in powder form or by means of sprays in water. Not for

Crop	country	Form g ai/L or kg	Application					PHI days	Note
			Method	Rate kg ai/ha	Max rate/season kg ai/ha	Conc kg ai/ hL	No		
									treatment of seeds, but for soils.
Beans	BR	WP 750	Seed treatment	0.113–0.225 kg ai/100 kg seed			1	--	
Beans (Phaseolus vulgaris)	EC	WP 750	Mix with seeds Hopper box	1.5			-	1	Mix with the seed in doses higher than those used in a simple treatment of seed. seed. Hopper box: The seed is used as a vehicle.
Potato	US	FL 480 [a]	Band application at planting.	2.81–5.61	5.61	1.50–6.00	1	--	Spray an 8.5-inch band in seed furrow at time of planting. Direct spray into furrow over the seed and cover as a part of the hilling operation during planting.
			Chemigation	1.68–2.81	5.61		2	28	If disease persists, a second foliar application may be made 10 days or more after the first application. Do not make chemigation application, if quintozene products were applied as a band application in furrow at planting.
Potato	MX	FL 480 [c]	Total ground treatment	12–21.6			1	--	Incorporate to a depth of 10–15 cm. Apply in band at sowing.
Potato	MX	WP 750	Total application	18.8–30.0			1	--	Use sufficient water to allow a uniform distribution in the ground. Directly sprinkle or spray in water, and then incorporate to a depth of 10-12 cm.
Potato	MX	WP 750	Pre-emergence band application	11.25–22.5			1	--	Use sufficient water to allow a uniform distribution. Sprinkle directly or in water sprinklers, ensuring that it is distributed evenly on the ground, and later incorporate to a depth of 10-12 cm, which offers better protection to the crop.
Potato	EC	WP 750		1.88			-	1	
Potato	ZA	WP 750	Overall application at planting	30			1	--	Mix thoroughly to a depth of minimum 10 to 20 cm. In not less than 500 to 1000 L water/ha.
Potato	ZA	WP 750	Band application to soil	0.22 kg ai/row			1	--	In not less than 500 L water/ha. Band spray to ensure mixing with the soil in the zone where the potato crop will be formed, as well as with the soil which surround and cover the seed tuber.
Cotton	MX	FL 480 [c]	Band application	0.72–1.44			1	--	Apply in band spray at sowing.
Cotton	MX	WP 750	Mix with seeds Hopper box	0.75–1.50			1	--	Indirect treatment to the soil using higher doses than those uses in a simple seed treatment. In hopper box application, the seed is also used as a vehicle.
Cotton	BR	WP	Seed	0.225–0.45 kg ai/100 kg seed			1	--	For seeds with linter, use higher

Crop	country	Form g ai/L or kg	Application					PHI days	Note
			Method	Rate kg ai/ha	Max rate/season kg ai/ha	Conc kg ai/ hL	No		
		750	treatment						dose.
Cotton	PE	WP 750	Seed treatment	0.652 kg ai/100 kg seed			1	--	
Cotton	TH	EC 240	Seed treatment	Mix 5 ml (1.2 g ai) with 1 kg of seeds			1	--	
Cotton	ZA	WP 750	Soil treatment (into furrow)	3.75–5.25		3.75–5.25			All the soil surrounding and covering the seed must be treated.
Peanut	MX	FL 480 [c]	Total application	0.96–1.92			1	--	Use sufficient water for uniform distribution. Apply in band at sowing.
Peanut	MX	WP 750	Mix with seeds Hopper box	0.75–1.50			-	--	Indirect treatment to the soil using higher doses than those uses in a simple seed treatment. In hopper box application, the seed is also used as a vehicle.
Peanut	BR	WP 750	Seed treatment	0.225 kg ai/100 kg seed			1	--	

Notes:

[a] 480 kg ai/L, 40 percent (w/w). Do not graze or feed clippings to livestock. Do not plant root crops in treated fields within 12 months of the last application unless quintozene is registered for use on those crops. Apply by ground boom application only except for greenhouse use.

[b] Listed vegetables together with broccoli and cabbage: Brussels sprouts, Chinese broccoli, cauliflower, Chinese cabbage (tight-headed varieties only), collards, kale, mustard greens.

[c] 370 kg ai/kg (W/W) according to the label.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised trials from the United States using either banded application at planting, in-furrow application at planting, post-emergence banded application, banded application at pegging or chemigation of quintozene were conducted on the following crops and their results are summarized in the tables:

Codex Group/Sub-group	Crop tested	Table No.
Brassica vegetables (except Brassica leafy vegetables)		
Subgroup of Flowerhead Brassicas	Broccoli	Table 72
Subgroup of head Brassicas	Cabbages, head	Table 73
Fruiting vegetables, other than Cucurbits		
Subgroup of tomatoes	Tomato	Table 74
Subgroup of pepper and pepper-like commodities	Peppers	Table 75
Legume vegetables		
Subgroup of beans with pods	Beans with pods	Table 76, Table 77
Subgroup of succulent beans without pods	Beans without pods	Table 78
Pulses		
Subgroup of dry beans	Beans (dry)	Table 79, Table 80
Root and tuber vegetables		

Codex Group/Sub-group		Crop tested	Table No.
	Subgroup of tuberous and corm vegetables	Potato	Table 81
Oilseed			
	Subgroup of cotton seed	Cotton seed	Table 82
	Subgroup of other oilseeds	Peanut	Table 83, Table 84

In addition to the descriptions and details of the field trials, each study report includes a summary of the analytical methods, together with the corresponding procedural recoveries (see Table 60), LOQ, LOD, and information on storage of samples. Duration of freezer storage between sampling and analysis were reported for all trials. If the duration is longer than the duration of proven frozen storage stability, this fact is stated before the related table below.

All appropriate trials are summarized and used. In many United States trials, where duplicate (replicate) replicate samples were taken from replicate plots at each sampling interval and were analysed separately, the mean values are presented in the residue tables and used for estimation of maximum residue levels and STMR. Where results from the same location with similar application timing and variety are reported, only the higher/highest results were used.

When residues were not quantifiable, they were shown as below the LOQ of the relevant analytical method (e.g., < 0.01 mg/kg). Application rates were generally rounded to three significant figures. For calculation of the mean residue values, available analytical results without rounding were used. For rounding the mean values from two values, the rule in ISO 80000-1 (round to the nearest even number) was used.

Although control plots were included in the trials, control data are not reported in the following tables unless residues in control samples exceeded the respective LOQ. Results were not corrected for concurrent method recoveries.

Residue values from the trials conducted according to the critical GAP were used for the estimation of maximum residue levels and STMR values. Those results included in the tables are underlined.

For the calculation of the "total" residues as expressed as quintozene equivalents in the following tables, the ratios of the molecular weights are used as follows:

	Molecular weight (g/mol)	MW Ratio Compared to MW of quintozene
Quintozene	295.32	1.00
PCA	265.34	1.11
PCTA	296.41	1.00

Where residue concentrations are below the LOQ, there are regarded to be at the LOQ values for summing up.

Brassica Vegetables

Broccoli

Twelve supervised trials were conducted on broccoli in the United States during the growing seasons 1987, 1988/89 and 2018/19.

In the four trials performed in 1987, the broadcast and banded applications were conducted with ground equipment in two parallel plots, each with a WP formulation or a GR formulation. The broadcast applications were done at a rate of 33.6 kg ai/ha whereas the banded applications were done at a rate of 25.2 kg ai/ha for the WP formulation and at 22.4 kg ai/ha for the GR formulation. Each trial comprised one plot where the WP formulation was applied as a soil drench application at a rate of 67.3 kg ai/ha after transplanting. In the two trials performed in 1987 in Oregon, a directed seed broadcast application was also performed.

In the 1987 trials, mature broccoli samples were obtained at 64–83 DAT and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.002 mg/kg for each analyte. PB and HCB were below the LOQ in all the trials.

In the 1988/89 trials, three different application techniques were compared in side-by-side plots, where one application to the soil was performed either as a broadcast spray application with soil incorporation pre-planting of the crop, a banded application at the time of planting, or as a soil drench application after transplanting of the crop.

Mature broccoli was sampled at 58–122 DAT and analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB using the method MP-PCNC-MA2 with an LOQ of 0.01 mg/kg for each analyte. PB and HCB were below the LOQ in all the trials.

In the trials performed in 2018/19 three trial locations received either a soil drench application, a banded application, or a broadcast spray application, each with a 480 g ai/L FL-formulation at a nominal rate of 25.2 kg ai/ha. Two trials, LR18357-01 and LR18357-05 were performed at the same location with treatment only 10 days apart. LR18357-01 was performed as a soil dench application whereas LR18357-05 was performed as a broadcast spray application to soil. Since the application techniques are not significantly different, the trials were not considered independent.

Mature broccoli was sampled at 89–132 DAT and analysed for quintozene, PCA and PCTA using the Battelle 100117568 method with an LOQ of 0.01 mg/kg for each analyte.

All the samples were stored frozen for periods shorter than the proven storage stability duration.

Applications by direct seed treatment are presented below for completeness but the use is not included in the permitted uses on the label and therefore the results are not used for estimation of maximum residue level or STMR.

Table 72 Magnitude of residues of quintozene in/on broccoli after broadcast application or banded application to soil, soil application after transplanting, or direct seed treatment in the supervised trials conducted in the United States in 1987, 1988/89, and 2018/19 (The portions analysed were head and stalk in all trials.)

Broccoli Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study Trial Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in United States	FL 480 g/L	25.2	1	-	Banded soil application or broadcast application, pre-plant.				
		1.68	-	-	Transplant solution. Maximum seasonal rate, 25.2 kg ai/ha.				
Gales Creek, OR, United States, 1988 (Cruiser)	WP 750 g/kg	33.6 (direct seed)	1	78	0.006	0.003	<0.002	0.011	UR-1408, 45038/45113, 900-RES-060
	GR 100 g/kg	33.6 (direct seed)	1	78	0.004	0.002	<0.002	0.009	
	WP 750 g/kg	33.6 (broadcast)	1	73	0.002 (<0.002)	0.002 (<0.002)	<0.002 (--)	0.006 (<0.006)	

Broccoli Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study Trial Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in United States	FL 480 g/L	25.2	1	-	Banded soil application or broadcast application, pre-plant.				
		1.68	-	-	Transplant solution. Maximum seasonal rate, 25.2 kg ai/ha.				
	GR 100 g/kg	33.6 (broadcast)	1	73	0.007 (0.006)	0.006 (0.004)	0.002 (<0.002)	0.016 (0.012)	
	WP 750 g/kg	25.2 (banded)	1	72	0.008	0.007	<0.002	0.018	
	GR 100 g/kg	22.4 (banded)	1	72	0.007 (0.008)	0.006 (0.007)	0.002 (0.003)	0.017 (0.019)	
	WP 750 g/kg	5.0 (transplant solution)	1	72	0.026	0.022	0.003	0.053	
Scholls, OR United States, 1988 (Cruiser)	WP 750 g/kg	33.6 (direct seed)	1	73	0.005	0.003	<0.002	0.010	UR-1408, 45114/44777, 900-RES-060
	GR 100 g/kg	33.6 (direct seed)	1	73	0.007	0.006	0.002	0.016	
	WP 750 g/kg	33.6 (broadcast)	1	64	0.004 (0.003)	0.004 (0.003)	<0.002 (--)	0.010 (0.008)	
	GR 100 g/kg	33.6 (broadcast)	1	64	0.006 (0.005)	0.006 (0.004)	<0.002 (--)	0.014 (0.011)	
	WP 750 g/kg	25.2 (banded)	1	64	0.004	0.004	<0.002	0.011	
	GR 100 g/kg	22.4 (banded)	1	64	0.008 (0.009)	0.004 (0.004)	<0.002 (--)	0.014 (0.016)	
	WP 750 g/kg	5.0 (transplant solution)	1	64	0.014	0.010	0.002	0.028	
Hollister, CA, United States, 1988 (Cruiser)	WP 750 g/kg	33.6 (broadcast)	1	83	<0.002	<0.002	<0.002	<0.006	UR-1408, 46241, 900-RES-060
	GR 100 g/kg	33.6 (broadcast)	1	83	0.006 (0.005)	0.004 (0.003)	0.004 (0.003)	0.014 (0.011)	
	WP 750 g/kg	25.2 (banded)	1	83	0.005	0.004	0.003	0.012	
	GR 100 g/kg	22.4 (banded)	1	83	0.005 (0.006)	0.006 (0.007)	0.002 (0.003)	0.014 (0.016)	
	WP 750 g/kg	5.0 (transplant solution)	1	83	0.018	0.022	0.006	0.049	
Holtville, CA, United States, 1988 (Cruiser)	WP 750 g/kg	33.6 (broadcast)	1	77	0.020 (0.015)	0.013 (0.010)	0.002 (<0.002)	0.037 (0.028)	UR-1408, 46007, 900-RES-060
	GR 100 g/kg	33.6 (broadcast)	1	77	0.027 (0.020)	0.012 (0.009)	0.002 (<0.002)	0.043 (0.032)	
	WP 750 g/kg	25.2 (banded)	1	77	0.022	0.013	<0.002	0.039	
	GR 100 g/kg	22.4 (banded)	1	77	0.023 (0.026)	0.014 (0.016)	0.002 (0.002)	0.041 (0.046)	
	WP 750 g/kg	5.0 (transplant solution)	1	77	0.044	0.033	0.004	0.085	

Broccoli Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study Trial Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in United States	FL 480 g/L	25.2	1	-	Banded soil application or broadcast application, pre-plant.				
		1.68	-	-	Transplant solution. Maximum seasonal rate, 25.2 kg ai/ha.				
Watsonville, CA, United States, 1988 (Futura)	WP 750 g/kg	36.6 (broadcast)	1	72	<0.01	<0.01	<0.01	<0.03	PAL-PB-BR, PB-BR-2508, 900-RES-019
	GR 100 g/kg	36.6 (broadcast)	1	72	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	36.6 (banded)	1	72	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	36.6 (banded)	1	72	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	67.3 (transplant solution)	1	72	<0.01	<0.01	<0.01	<0.03	
Center Point, TX. United States, 1988/89 (Green Duke F1)	WP 750 g/kg	36.6 (broadcast)	1	96	<0.01	<0.01	<0.01	<0.03	PAL-PB-BR, PB-BR-2509, 900-RES-019 (Quintozene at 0.013 mg/kg in control)
				110	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	36.6 (broadcast)	1	110	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	36.6 (banded)	1	96	0.013 (<0.01)	<0.01 (--)	<0.01 (--)	0.034 (<0.01)	
				110	0.012 (<0.01)	<0.01 (--)	<0.01 (--)	0.033 <0.01	
	GR 100 g/kg	36.6 (banded)	1	96	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	67.3 (transplant solution)	1	122	<0.01	<0.01	<0.01	<0.03	
Hillsboro, OR, United States, 1988 (Gem)	WP 750 g/kg	36.6 (broadcast)	1	58	0.024 (0.018)	0.017 (0.013)	<0.01 (--)	0.054 (0.040)	PAL-PB-BR, PB-BR-2510, 900-RES-019 (Quintozene at 0.012 mg/kg in control)
	GR 100 g/kg	36.6 (broadcast)	1	58	0.020 (0.015)	0.015 (0.011)	<0.01 (--)	0.046 (0.035)	
	WP 750 g/kg	36.6 (banded)	1	58	0.018 (0.013)	0.012 (<0.01)	<0.01 (--)	0.041 (0.031)	
	GR 100 g/kg	36.6 (banded)	1	58	0.032 (0.024)	0.023 (0.017)	<0.01 (--)	0.067 (0.050)	
	WP 750 g/kg	67.3 (transplant solution)	1	58	0.043 (0.016)	0.027 (0.010)	<0.01	0.083 (0.031)	
Fresno, CA, United States, 2018 (Imperial)	FL 480 g/L	25.3 (soil drench)	1	92	<0.01	<0.01	<0.01	<0.03	LR18357 LR18357-01 900-RES-224
King City, CA, United States, 2018/19 (Heritage)	FL 480 g/L	25.3 (soil drench)	1	112	<0.01	<0.01	<0.01	<0.03	LR18357 LR18357-02 900-RES-224
Guadalupe, CA, United States, 2018/19 (Heritage)	FL 480 g/L	25.3 (soil drench)	1	89	<0.01	<0.01	<0.01	<0.03	LR18357 LR18357-03 900-RES-224

Broccoli Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study Trial Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in United States	FL 480 g/L	25.2	1	-	Banded soil application or broadcast application, pre-plant.				
		1.68	-	-	Transplant solution. Maximum seasonal rate, 25.2 kg ai/ha.				
Madera, CA, United States, 2018/19 (Tradition)	FL 480 g/L	25.5 (banded)	1	132	0.010	0.013	<0.01	0.035	LR18357 LR18357-04 900-RES-224
Fresno, CA, United States, 2018/19 (Tradition)	FL 480 g/L	25.3 (broadcast)	1	106	<0.01	<0.01	<0.01	<0.03	LR18357 LR18357-05 [b] 900-RES-224

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets.

[b] Same location as LR18357-01 with the application date 10 days earlier prior to planting (LR18357-01 after planting).

Cabbage

Sixteen supervised residue trials were carried out on cabbage in the United States during the growing seasons 1987/88, 1988/89 and 1990/91.

In all trials, three different application techniques were compared in side-by-side plots, where one application to the soil was performed either as a broadcast spray application with soil incorporation pre-planting of the crop, a banded application at the time of planting, or as a soil drench application after transplanting of the crop.

In the seven trials performed in 1987/88, the broadcast and banded applications were conducted with ground equipment in two parallel plots each with a 750 g ai/kg WP formulation and a 100 g ai/kg GR formulation. The broadcast applications were done at a rate of 33.6 kg ai/ha. The banded applications were done at a rate of 25.2 kg ai/ha for the WP formulation and at 22.4 kg ai/ha for the GR formulation. Each trial also comprised one plot where the WP formulation was applied as a soil drench application at a rate of 67.3 kg ai/ha after transplanting. In the 1987 growing season, two trials, 45370-54373 (field 2) and 45374-54377 (field 6), were performed in the same location with the treatments on the same dates. These trials are not considered independent.

Mature cabbage heads were sampled at 67–125 DAT and analysed for quintozene, PCA, PCTA, PB and HCB with or without wrapper leaves using the method CAM-24-73 (modified) with an LOQ of 0.002 mg/kg for each analyte. In all trials, analytical results of HCB were below the LOQ. Analytical results of PB were mostly below the LOQ with in some cases at the maximum 0.003 mg/kg shown in the footnotes of the table.

In the five trials performed in 1988/89, the broadcast and banded applications were conducted with ground equipment in two parallel plots each with a 750 g ai/kg WP formulation and a 100 g ai/kg GR formulation, all at a rate of 36.6 kg ai/ha. Each trial also comprised one plot where the WP formulation was applied as a soil drench application at a rate of 67.3 kg ai/ha after transplanting.

Mature cabbage heads were sampled at 70–134 DAT and analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB with or without wrapper leaves using the method MP-PCNC-MA2 with an LOQ of 0.01 mg/kg for each analyte. In all trials, analytical results of HCB and PB were below the LOQ.

In the trials performed in 1990/91, the broadcast applications were conducted either in two parallel plots each with a 750 g ai/kg WP formulation and a 100 g ai/kg GR formulation (2 trials) or in a single plot with a 480 g ai/L flowable formulation, all at a rate of 36.6 kg ai/ha. The same setup was used for the banded application with exception that a reduced application rate of 22.4–25.2 kg ai/ha was used. Two trials, CRA-90-081 (1990/1991) and CRA-90-084 (1990) were performed in the same location. The application dates and harvest dates were available for CRA-90-081 but no date information was available for CRA-90-084. Since the harvest dates for CRA-90-081 were March and April 1091, it is likely that these dates may be more than 60 days earlier than those for CRA-90-084, and since both trials were presenting high residues, both trials were considered for estimating maximum residue level.

In the 1990/91 trials, mature cabbage heads were sampled at maturity (the DAT was reported only for one trial with 153–161 days) and analysed for quintozene, PCA, PCTA, PB and HCB with or without wrapper leaves using the method CAM-24-73 with an LOQ of 0.001 mg/kg for each analyte except HCB for which an LOQ was 0.0005 mg/kg. PB and HCB were found above the respective LOQ in some trials and some others in one of two samples from some trials, which were shown in the footnotes of the table. In one sample from one trial, HCB was found at 0.059 mg/kg but the other sample was found to contain HCB below the LOQ.

All the samples were stored frozen for periods shorter than the proven storage stability duration, except for 2 trials (shown in the following table).

Table 73 Magnitude of residues of quintozene in/on cabbage after broadcast application or banded application to soil, or soil application after transplanting in the supervised trials conducted in the United States in 1987/88, 1988/89, and 1990/91

Cabbage Location, Year (Variety)	Application			DAT	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
GAP in United States	FL 480 g/L	25.2	1	-	Banded soil application or broadcast application, pre-plant.					
		1.68	-	-	Transplant solution. Maximum seasonal rate, 25.2 kg ai/ha.					
Fresno, CA, United States, 1987/88 (Head Start)	WP 750 g/kg	33.6 (broadcast)	1	90	head w. leaves	0.037 (0.028)	0.022	<0.002	0.063 (0.047)	UR-1413, 46338, 900-RES-059
					head w/o leaves	0.003 (0.003)	0.003	<0.002	0.009 (0.006)	
	GR 100 g/kg	33.6 (broadcast)	1	90	head w. leaves	0.013 (0.010)	0.008	<0.002	0.024 (0.018)	
					head w/o leaves	0.010 (0.007)	0.002	<0.002	0.014 (0.011)	
	WP 750 g/kg	25.2 (banded)	1	90	head w. leaves	0.005	0.005	<0.002	0.012	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	22.4 (banded)	1	90	head w. leaves	0.009 (0.010)	0.009	<0.002	0.020 (0.023)	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP 750 g/kg	5.0 (transplant solution)	1	90	head w. leaves	0.006	0.006	<0.002	0.014	
					head w/o leaves	0.005	0.003	<0.002	0.010	
Madera, CA. United States, 1987/88 (Head Start)	WP 750 g/kg	33.6 (broadcast)	1	125	head w. leaves	0.030 (0.022)	0.022	<0.002	0.057 (0.043)	UR-1413, 47308, 900-RES-059
					head w/o leaves	0.002 (0.002)	0.002	<0.002	0.007 (0.005)	
	GR 100 g/kg	33.6 (broadcast)	1	125	head w. leaves	0.030 (0.022)	0.020	<0.002	0.054 (0.040)	
					head w/o leaves	0.012 (0.009)	0.008	<0.002	0.023 (0.017)	
	WP	25.2	1	125	head w. leaves [d]	0.015	0.015	<0.002	0.033	

Cabbage Location, Year (Variety)	Application			DAT	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
GAP in United States	FL 480 g/L	25.2	1	-	Banded soil application or broadcast application, pre-plant.					
		1.68	-	-	Transplant solution. Maximum seasonal rate, 25.2 kg ai/ha.					
	750 g/kg	(banded)			head w/o leaves	0.004	0.003	<0.002	0.008	
	GR 100 g/kg	22.4 (banded)	1	125	head w. leaves [e]	0.038 (0.042)	0.036	<0.002	0.080 (0.090)	
					head w/o leaves	0.007 (0.008)	0.007	<0.002	0.017 (0.019)	
	WP 750 g/kg	5.0 (transplant solution)	1	125	head w. leaves	0.050	0.039	<0.002	0.095	
					head w/o leaves	0.005	0.003	<0.002	0.010	
Downars Grove, IL, United States, 1987 (Early Copenhagen)	WP 750 g/kg	33.6 (broadcast)	1	69	head w. leaves	0.009 (0.007)	0.006	<0.002	0.017 (0.013)	UR-1413, 44884, 900-RES-059
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	33.6 (broadcast)	1	69	head w. leaves	0.007 (0.005)	0.006	<0.002	0.016 (0.012)	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP 750 g/kg	25.2 (banded)	1	69	head w. leaves	0.003	0.002	<0.002	0.007	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	22.4 (banded)	1	69	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP 750 g/kg	5.0 (transplant solution)	1	69	head w. leaves [f]	0.004	0.004	0.004	0.012	
					head w/o leaves [g]	<0.002	<0.002	<0.002	<0.006	
Verona, WI, United States, 1987 (Green Acres)	WP 750 g/kg	33.6 (broadcast)	1	67	head w. leaves	<0.002	<0.002	<0.002	<0.006	UR-1413, 45146, 900-RES-059
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	33.6 (broadcast)	1	67	head w. leaves	0.003 (<0.002)	0.002	<0.002	0.007 (<0.006)	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP 750 g/kg	25.2 (banded)	1	67	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	22.4 (banded)	1	67	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP 750 g/kg	5.0 (transplant solution)	1	67	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
Lake Harbor, FL, United States, 1987 (Bravo)	WP 750 g/kg	33.6 (broadcast)	1	77	head w. leaves	0.003 (<0.002)	<0.002	<0.002	0.007 (<0.006)	UR-1413, 46806-,46807 900-RES-059
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	33.6 (broadcast)	1	77	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP 750 g/kg	25.2 (banded)	1	77	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	22.4 (banded)	1	77	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP 750 g/kg	5.0 (transplant solution)	1	77	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
Field 2, Phelps, NY, United States, 1987 [b] (King Cole Hybrid)	WP 750 g/kg	33.6 (broadcast)	1	78	head w. leaves [h]	0.005 (0.004)	0.004	<0.002	0.012 (0.009)	UR-1413, 45370-54373, 900-RES-059
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	33.6 (broadcast)	1	78	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP	25.2	1	78	head w. leaves	0.002	0.002	<0.002	0.007	

Cabbage Location, Year (Variety)	Application			DAT	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
GAP in United States	FL 480 g/L	25.2	1	-	Banded soil application or broadcast application, pre-plant.					
		1.68	-	-	Transplant solution. Maximum seasonal rate, 25.2 kg ai/ha.					
	750 g/kg	(banded)			head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	22.4 (banded)	1	78	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP 750 g/kg	5.0 (transplant solution)	1	78	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
Field 6, Phelps, NY, United States, 1987 [b] (King Cole Hybrid)	WP 750 g/kg	33.6 (broadcast)	1	78	head w. leaves	<0.002	<0.002	<0.002	<0.006	UR-1413, 45374-54377, 900-RES-059
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	33.6 (broadcast)	1	78	head w. leaves	<0.002	0.002	<0.002	0.007 (<0.006)	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP 750 g/kg	25.2 (banded)	1	78	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	22.4 (banded)	1	78	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
	WP 750 g/kg	5.0 (transplant solution)	1	78	head w. leaves	<0.002	<0.002	<0.002	<0.006	
					head w/o leaves	<0.002	<0.002	<0.002	<0.006	
Watsonville, CA, United States, 1988 (Green Acres)	WP 750 g/kg	33.6 (broadcast)	1	70	head w. leaves	<0.01	<0.01	<0.01	<0.03	PAL-PB-CB, PB-CB-2511, 900-RES-020
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	33.6 (broadcast)	1	70	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	33.6 (banded)	1	70	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	33.6 (banded)	1	70	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	67.3 (transplant solution)	1	70	head w. leaves	0.011	<0.01	<0.01	0.032	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
Center Point, TX, United States, 1988/89 (Golden Cross)	WP 750 g/kg	33.6 (broadcast)	1	134	head w. leaves	<0.01	<0.01	<0.01	<0.03	PAL-PB-CB, PB-CB-2512, 900-RES-020
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	33.6 (broadcast)	1	134	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	0.013 (<0.01)	<0.01 (--)	<0.01 (--)	0.034 (<0.03)	
	WP 750 g/kg	33.6 (banded)	1	134	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	33.6 (banded)	1	134	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	67.3 (transplant solution)	1	129	head w. leaves	0.037	0.011	<0.01	0.059	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
Union Grove, WI, United States, 1988 (Gourmet)	WP 750 g/kg	33.6 (broadcast)	1	93	head w. leaves	<0.01	<0.01	<0.01	<0.03	PAL-PB-CB, PB-CB-2513, 900-RES-020
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	33.6 (broadcast)	1	93	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	33.6 (banded)	1	93	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	33.6 (banded)	1	93	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	WP	67.3	1	93	head w. leaves	<0.01	<0.01	<0.01	<0.03	

Cabbage Location, Year (Variety)	Application			DAT	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
GAP in United States	FL 480 g/L	25.2	1	-	Banded soil application or broadcast application, pre-plant.					
		1.68	-	-	Transplant solution. Maximum seasonal rate, 25.2 kg ai/ha.					
	750 g/kg	(transplant solution)			head w/o leaves	<0.01	<0.01	<0.01	<0.03	
Phelps, NY, United States, 1988 (Market Prize)	WP 750 g/kg	33.6 (broadcast)	1	98	head w. leaves	<0.01	<0.01	<0.01	<0.03	PAL-PB-CB, PB-CB-2514, 900-RES-020
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	33.6 (broadcast)	1	98	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	33.6 (banded)	1	98	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	33.6 (banded)	1	98	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	67.3 (transplant solution)	1	97	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
Loxahatchee, FL, United States, 1988/89 (Bravo)	WP 750 g/kg	33.6 (broadcast)	1	81	head w. leaves	<0.01	<0.01	<0.01	<0.03	PAL-PB-CB, PB-CB-2515, 900-RES-020
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	33.6 (broadcast)	1	81	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	33.6 (banded)	1	81	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	33.6 (banded)	1	81	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
	WP 750 g/kg	67.3 (transplant solution)	1	81	head w. leaves	<0.01	<0.01	<0.01	<0.03	
					head w/o leaves	<0.01	<0.01	<0.01	<0.03	
Uvalde, TX, United States, 1990/91 (Variety: not reported)	FL 480 g/L	5.04 (transplant solution)	1	153	head w. leaves [i]	0.099	0.054	0.002	0.16	RP-90025 CRA-90-081 900-RES-194
					head w/o leaves	0.001	<0.001	<0.001	<0.003	
	FL 480 g/L	24.7 (banded)	1	161	head w. leaves [j]	0.009	1.25	<0.001	1.40	
					head w/o leaves	<0.001	<0.001	<0.001	<0.003	
	FL 480 g/L	33.6 (broadcast)	1	161	head w. leaves	0.004 (0.003)	0.009	<0.001	0.015	
					head w/o leaves [k]	0.001	<0.001	<0.001	0.004	
Phelps, NY, United States, 1990 [c] (Variety:not reported)	FL 480 g/L	5.04 (transplant solution)	1	n/r	head w. leaves [l]	0.006	0.002	0.002	0.010	RP-90025 PWB-90-001 900-RES-194
					head w/o leaves [m]	<0.001	<0.001	0.002	0.004	
	FL 480 g/L	24.7 (banded)	1	n/r	head w. leaves	0.003	0.002	0.001	0.006	
					head w/o leaves [n]	0.003	0.003	0.004	0.010	
	FL 480 g/L	33.6 (broadcast)	1	n/r	head w. leaves	0.002 (<0.002)	0.002	0.001	0.006	
					head w/o leaves [o]	0.001	0.001	0.001	0.003	
Uvalde, TX, United States, 1990 [c] (Variety:not reported) NB: Dates of planting, treatment or harvest were not	GR 100 g/kg	22.4 (banded)	1	n/r	head w. leaves	0.004	0.004	<0.001	0.010	RP-90031 CRA-90-084 900-RES-201
					head w/o leaves	<0.001	<0.001	<0.001	0.003	
	WP 750 g/kg	25.2 (banded)	1	n/r	head w. leaves [p]	0.007	0.006	<0.001	0.015	
					head w/o leaves	<0.001	0.001	<0.001	0.003	
	GR 100 g/kg	33.6 (broadcast)	1	n/r	head w. leaves [q]	0.007	0.006	<0.001	0.014	
					head w/o leaves	<0.001	<0.001	<0.001	<0.003	
	WP 750 g/kg	33.6 (broadcast)	1	n/r	head w. leaves [r]	0.011 (0.008)	0.011	<0.001	0.024	
					head w/o leaves [s]	<0.001	<0.001	<0.001	0.004	
	WP	5.04	1	n/r	head w. leaves	0.032	0.019	<0.001	0.054	

Cabbage Location, Year (Variety)	Application			DAT	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
GAP in United States reported.	FL 480 g/L 750 g/kg	25.2	1	-	Banded soil application or broadcast application, pre-plant.					
		1.68	-	-	Transplant solution. Maximum seasonal rate, 25.2 kg ai/ha.					
		(transplant solution)			head w/o leaves	0.002	0.002	<0.001	0.005	
Sanford, FL, United States, 1990 [c] (Variety:not reported) NB: Dates of planting, treatment or harvest were not reported.	FL 480 g/L	5.04 (transplant solution)	1	n/r	head w. leaves [t]	0.056	0.032	0.001	0.093	RP-90065 PAK 90071 900-RES-202
					head w/o leaves	0.001	0.002	0.001	0.004	
	FL 480 g/L	24.7 (banded)	1	n/r	head w. leaves [u]	0.013	0.007	0.001	0.022	
					head w/o leaves [v]	0.214	0.008	0.003	0.226	
	FL 480 g/L	33.6 (broadcast)	1	n/r	head w. leaves [w]	0.010	0.010	<0.001	0.022	
					head w/o leaves [x]	<0.001	<0.001	<0.001	<0.003	

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

[b] Treatment was conducted on the same day. And therefore they are not regarded as independent from each other.

[c] The storage duration was not reported.

	PB (mg/kg)	HCB (mg/kg)
[d]	0.002 & <0.002	
[e]	0.003 & <0.002	
[f]	0.003, 0.002 & <0.002	
[g]	0.002 & <0.002	
[h]	0.002 × 2	
[i]	0.002 & <0.001	
[j]		0.059 & <0.0005.
[k]	0.005 & <0.001	0.005 & <0.0005
[l]	0.001 & <0.001	0.001 & <0.0005
[m]	0.002 & 0.001	
[n]	0.003 & <0.001	0.002 &0.004
[o]	0.003 & <0.001	0.003 & <0.0005
[p]	0.001 & <0.001	0.001 & <0.0005
[q]	0.001 & <0.001	
[r]	0.001 &<0.001	0.001 & <0.0005
[s]		0.001 & <0.0005
[t]	0.001 & <0.001	0.013 & 0.010
[u]	0.010 & <0.001	0.021 & 0.002
[v]	Both PB and HCB showed values of 0.097 and0.095 mg/kg but the second analysis indicated below the respective LOQ.	
[w]		0.009 & 0.018
[x]		0.001 & <0.0005

Fruiting vegetables, other than Cucurbits

Tomato

Fourteen supervised residue trials were carried out on tomato in the United States during the growing seasons 1987 and 1988/89.

In all trials two plots were established, where in the first plot an in-furrow application of a 750 g/kg WP formulation at a rate of 8.41 kg ai/ha was performed at the time of planting. In the second plot, a soil dench application after transplanting was performed using a transplanting solution at a concentration of 0.45 kg ai/hL at rates of about 25–29 kg ai/ha. In two of the 1987 trials an additional plot each was set-up with in-furrow application at an exaggerated rate of 42 kg ai/ha to obtain samples for processing.

In the 1987 trials, tomato fruits were sampled at 69–119 DAT and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.05 mg/kg for each analyte. As a follow-up, selected samples were re-analysed with a modified version of method CAM-24-73 with an LOQ of 0.002 mg/kg for each analyte. However, since samples were re-analysed after a storage of up to 574 days for which stability cannot be confirmed, the data from re-analysis were not considered for estimation of maximum residue level.

In the 1988/89 trials, mature tomato fruits were sampled at 81–108 DAT (also after planning) and analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB using the method MP-PCNC-MA2 with an LOQ of 0.01 mg/kg for each analyte.

All the samples were stored frozen for periods shorter than the proven storage stability duration (except for re-analysis as explained above).

Table 74 Magnitude of residues of quintozene in/on tomato after in-furrow application or soil application after transplanting in the supervised trials conducted in the United States in 1987 and 1988/89 (The portions analysed were whole fruits in all trials.)

Tomato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	14.4	1	-	Application in nursery				
GAP in Thailand	EC 240	1.50 kg ai/hL	≥2	-	Spray the soil all over: first time, immediately after planning young tomato plant; and repeat spraying every 14 days at least 2 times.				
GAP in EC	WP 750 g/kg	0.56	-	1	Use under greenhouse conditions. For kidney tomato.				
Madera, CA, United States, 1988 (P-19)	WP 750 g/kg	8.41 (in-furrow)	1	108	<0.01	<0.01	<0.01	<0.03	PAL-PB-TO PB-PP-2532 900-RES-027
	WP 750 g/kg	28.6 (transplant solution)	1	108	0.016	<0.01	<0.01	0.037	
Fresno, CA, United States, 1988 (P-19)	WP 750 g/kg	8.41 (in-furrow)	1	106	<0.01	<0.01	<0.01	<0.03	PAL-PB-TO PB-PP-2533 900-RES-027
	WP 750 g/kg	28.6 (transplant solution)	1	106	<0.01	<0.01	<0.01	<0.03	
Loxahatchee, FL, United States, 1988/89 (Sunny)	WP 750 g/kg	8.41 (in-furrow)	1	91	<0.01	<0.01	<0.01	<0.03	PAL-PB-TO PB-PP-2534 900-RES-027
	WP 750 g/kg	28.6 (transplant solution)	1	91	<0.01	<0.01	<0.01	<0.03	

Tomato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	14.4	1	-	Application in nursery				
GAP in Thailand	EC 240	1.50 kg ai/hL	≥2	-	Spray the soil all over: first time, immediately after planning young tomato plant; and repeat spraying every 14 days at least 2 times.				
GAP in EC	WP 750 g/kg	0.56	-	1	Use under greenhouse conditions. For kidney tomato.				
Haslett, MI, United States, 1988 (Mountain Pride)	WP 750 g/kg	8.41 (in-furrow)	1	81	<0.01	<0.01	<0.01	<0.03	PAL-PB-TO PB-PP-2535 900-RES-027
	WP 750 g/kg	28.6 (transplant solution)	1	81	<0.01	<0.01	<0.01	<0.03	
Hope, NJ, United States, 1988 (Rutgers)	WP 750 g/kg	8.41 (in-furrow)	1	89	<0.01	<0.01	<0.01	<0.03	PAL-PB-TO PB-PP-2536 900-RES-027
	WP 750 g/kg	28.6 (transplant solution)	1	90	<0.01	<0.01	<0.01	<0.03	
Groveland, FL United States, 1987 (Sunny)	WP 750 g/kg	8.41 (in-furrow)	1	69	<0.05	<0.05	<0.05	<0.155	UR-1411 900-RES-126
	WP 750 g/kg	25.2 (transplant solution)	1	69	<0.05	<0.05	<0.05	<0.155	
Marcellus, MI United States, 1987 (Pik Red)	WP 750 g/kg	8.41 (in-furrow)	1	73	<0.05	<0.05	<0.05	<0.155	UR-1411 900-RES-126
	WP 750 g/kg	0.45 kg ai/hL (transplant solution)	1	73	<0.05	<0.05	<0.05	<0.155	
	WP 750 g/kg	8.41 (in-furrow)	1	73	<0.05	<0.05	<0.05	<0.155	
	WP 750 g/kg	0.45 kg ai/hL (transplant solution)	1	73	<0.05	<0.05	<0.05	<0.155	
East Lansing, MI United States, 1987 (Heinz 1810)	WP 750 g/kg	8.41 (in-furrow)	1	94	<0.05	<0.05	<0.05	<0.155	UR-1411 900-RES-126
	WP 750 g/kg	0.45 kg ai/hL (transplant solution)	1	94	<0.05	<0.05	<0.05	<0.155	
Crown PT, FL United States, 1987 (Super Fantastic)	WP 750 g/kg	8.41 (in-furrow)	1	69	<0.05	<0.05	<0.05	<0.155	UR-1411 900-RES-126
Hope, NJ United States, 1987 (Supersonic)	WP 750 g/kg	8.41 (in-furrow)	1	91	<0.05	<0.05	<0.05	<0.155	UR-1411 900-RES-126
	WP 750 g/kg	0.45 kg/hL (transplant solution)	1	91	<0.05	<0.05	<0.05	<0.155	
Newton, NJ United States, 1987 (Early Gilr)	WP 750 g/kg	8.41 (in-furrow)	1	77	<0.05	<0.05	<0.05	<0.155	UR-1411 900-RES-126
	WP 750 g/kg	25.2 (transplant solution)	1	77	<0.05	<0.05	<0.05	<0.155	
Madera, CA United States,	WP 750 g/kg	8.41 (in-furrow)	1	113	<0.05	<0.05	<0.05	<0.155	UR-1411 900-RES-126

Tomato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	14.4	1	-	Application in nursery				
GAP in Thailand	EC 240	1.50 kg ai/hL	≥2	-	Spray the soil all over: first time, immediately after planning young tomato plant; and repeat spraying every 14 days at least 2 times.				
GAP in EC	WP 750 g/kg	0.56	-	1	Use under greenhouse conditions. For kidney tomato.				
1987 (Murietta)	WP 750 g/kg	0.45 kg/hL (transplant solution)	1	113	<0.05	<0.05	<0.05	<0.155	
	WP 750 g/kg	42.0 (transplant solution)	1	113	<0.05	<0.05	<0.05	<0.155	
Fresno, CA United States, 1987 (Murietta)	WP 750 g/kg	8.41 (in-furrow)	1	111	<0.05	<0.05	<0.05	<0.155	UR-1411 900-RES-126
	WP 750 g/kg	4.20 (transplant solution)	1	111	<0.05	<0.05	<0.05	<0.155	
	WP 750 g/kg	42.0 (transplant solution)	1	111	<0.05	<0.05	<0.05	<0.155	
Clewiston, FL United States, 1987 (Sunny)	WP 750 g/kg	8.41 (in-furrow)	1	119	<0.05	<0.05	<0.05	<0.155	UR-1411 900-RES-126
	WP 750 g/kg	0.45 kg/hL (transplant solution)	1	119	<0.05	<0.05	<0.05	<0.155	

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets.

Peppers

In total nine supervised residue trials were carried out on pepper in the United States during the growing seasons 1987/88 and 1988/89. Two trials in the 1987/88 season were performed on chili peppers (non-bell shape), all other trials on sweet pepper varieties (bell shape).

In all trials, two plots were established, where in the first plot an in-furrow application of a 750 g/kg WP formulation at a rate of 8.41 kg ai/ha was performed at the time of planting. In the second plot, a soil dench application after transplanting was performed using the same WP formulation at rates of 4.20–38.1 kg ai/ha.

In the 1987/88 trials, pepper fruits were sampled at 71–104 DAT and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.05 mg/kg for each analyte.

In the 1988/89 trials, mature pepper fruits were sampled at 49–105 DAT and analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB using the method MP-PCNC-MA2 with an LOQ of 0.01 mg/kg for each analyte.

As a follow-up, selected samples were re-analysed with a modified version of method CAM-24-73 with an LOQ of 0.002 mg/kg for each analyte. However, since samples were re-analysed after a storage of up to 567 days for which stability cannot be confirmed, the data from re-analysis was not considered for estimation of maximum residue level.

All the samples, except in one trial (see in the Table), were stored frozen for periods shorter than the proven storage stability duration. Those trials with the sample storage period longer than 230 days are not included in the following Table.

Table 75 Magnitude of residues of quintozene in/on peppers after in-furrow application or soil application after transplanting in the supervised trials conducted in the United States in 1987/88 and 1988/89 (The portions analysed were whole fruits in all trials.)

Peppers Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	14.4	1	-	Application in nursery. For chili peppers				
Fresno, CA, United States, 1988 [b] (Waxed Banana)(non-bell)	WP 750 g/kg	8.41 (in-furrow)	1	49	<0.01	<0.01	<0.01	<0.03	PAL-PB-PP PB-PP-2528 900-RES-024
	WP 750 g/kg	38.1 (transplant solution)	1	49	<0.01	<0.01	<0.01	<0.03	
Uvalde, TX United States, 1988 (Rio 66)(bell)	WP 750 g/kg	8.41 (in-furrow)	1	105	<0.01	<0.01	<0.01	<0.03	PAL-PB-PP PB-PP-2529 900-RES-024
	WP 750 g/kg	38.1 (transplant solution)	1	105	<0.01	<0.01	<0.01	<0.03	
Hope, NJ, United States, 1988 (Yolo Wonder) (bell)	WP 750 g/kg	8.41 (in-furrow)	1	82	0.016	0.012	<0.01	0.039	PAL-PB-PP PB-PP-2530 900-RES-024
	WP 750 g/kg	38.1 (transplant solution)	1	82	<0.01	<0.01	<0.01	<0.03	
Loxahatchee, FL, United States, 1988/89 (Early CalWonder) (bell)	WP 750 g/kg	8.41 (in-furrow)	1	76	0.039	0.012	<0.01	0.062	PAL-PB-PP PB-PP-2531 900-RES-024
	WP 750 g/kg	38.1 (transplant solution)	1	76	<0.01	<0.01	<0.01	<0.03	
Groveland, FL, United States, 1987 (Lady Bell) (bell)	WP 750 g/kg	8.41 (in-furrow)	1	71	<0.05	<0.05	<0.05	<0.155	UR-1410 PEP 900-RES-062
	WP 750 g/kg	25.2 (transplant solution)	1	71	<0.05	<0.05	<0.05	<0.155	
Hope, NJ, United States, 1987 (Lady Bell) (bell))	WP 750 g/kg	8.41 (in-furrow)	1	91	<0.05	<0.05	<0.05	<0.155	UR-1410 B-87 900-RES-062
	WP 750 g/kg	0.45 kg/hL (transplant solution)	1	91	<0.05	<0.05	<0.05	<0.155	
Overton, TX, United States, 1987 (experimental breeding line)	WP 750 g/kg	8.41 (in-furrow)	1	73	<0.05	<0.05	<0.05	<0.155	UR-1410 K-87 900-RES-062
	WP 750 g/kg	4.20 (transplant solution)	1	73	<0.05	<0.05	<0.05	<0.155	
Fresno, CA, United States, 1987 (Emerald Giant) (bell)	WP 750 g/kg	8.41 (in-furrow)	1	76	<0.05	<0.05	<0.05	<0.155	UR-1410 PAL 900-RES-062
	WP 750 g/kg	0.45 kg/hL (transplant solution)	1	76	<0.05	<0.05	<0.05	<0.155	
	WP 750 g/kg	8.41 (in-furrow)	1	77	<0.05	<0.05	<0.05	<0.155	

Peppers Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	14.4	1	-	Application in nursery. For chili peppers				
	WP 750 g/kg	0.45 kg/hL (transplant solution)	1	77	<0.05	<0.05	<0.05	<0.155	
Clewiston, FL, United States, 1987/88 (California Wonder)(bell)	WP 750 g/kg	8.41 (in-furrow)	1	104	<0.05	<0.05	<0.05	<0.155	A026.001-addendum A-87 900-RES-166a
	WP 750 g/kg	4.20 (transplant solution)	1	104	<0.05	<0.05	<0.05	<0.155	

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

[b] Stored frozen for184 days.

Legume vegetables

Beans, except broad bean and soya bean (green pods and immature seeds)–Directed band applications to soil or ground applications to soil

Twenty supervised residue trials were carried out on beans (all belong to the genus *Phaseolus*) in the United States during the growing seasons 1987 and 1988. In seventeen trials, whole green pods were sampled and in three trials, succulent seeds were sampled.

In all trials, two plots were established, where in the first plot a 750 g/kg WP formulation and in the second plot a 240 g/L EC formulation was used. In all trials/plots, except two, quintozene was applied four times at a nominal rate of 2.24 kg ai/ha with a nominal interval of 14 days. In other two trials, only three applications at rates ranging 0.56–5.60 kg ai/ha were performed.

In the 1987 trials, green pods and succulent seeds were sampled at 7–63 DALA and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.05 mg/kg for each analyte.

As a follow-up, selected samples were re-analysed with a modified version of method CAM-24-73 with an LOQ of 0.002 mg/kg for each analyte. However, since samples were re-analysed after a storage of up to 557 days for which stability could not be confirmed, the data from re-analysis were not considered for estimation of maximum residue level.

In the 1988 trials, green pods were sampled at 7–57 DALA and analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB using the method MP-PCNC-MA1 with an LOQ of 0.005 mg/kg or 0.01 mg/kg for each analyte. In trial PB-BS-2520 in which samples were obtained at 7 DALA, the last application was performed when pods were already present, which is not according to the label instructions. This trial was therefore not considered for estimating maximum residue level.

Samples in five trials (marked in the following table) were stored shorter than 3 months while all other samples were stored frozen for longer than 100 days but shorter than 250 days.

Table 76 Magnitude of residues of quintozene in/on immature bean pod after post-emergence directed band applications to soil or ground applications to soil in the supervised trials conducted in the United States in 1987 and 198. (The portions analysed were whole pods in all trials.)

Bean (pod) Location, Year (Variety)	Application			DALA	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application at sowing				
GAP in Mexico	FL 480 g/L	8.64	1	-	Band application at the beginning of flowering				
GAP in Mexico	WP 750 g/kg	4.50	1	-	Band application covering the crowns of the plants after cultivation and immediately before raising the furrow to irrigate.				
Porterville, CA, United States, 1988 [c] (Bush; lima bean)	EC 240 g/L	1.68 2.24 × 3 (banded)	4	42	<0.005	<0.005	<0.005	<0.016	PAL-PB-LB PB-PP-2516 900-RES-017
	WP 750 g/kg	1.68 2.24 × 3 (banded)	4	42	<0.005	<0.005	<0.005	<0.016	
Delavan, WI, United States, 1988 (Flo; lima bean)	EC 240 g/L	1.68 2.24 × 3 (banded)	4	47	0.028	<0.005	<0.005	0.038	PAL-PB-LB PB-PP-2517 900-RES-017
	WP 750 g/kg	1.68 2.24 × 3 (banded)	4	47	0.050	<0.005	<0.005	0.061	
Delamar, DE, United States, 1988 (Fordbook 242; lima bean)	EC 240 g/L	1.68 2.24 × 3 (banded)	4	57	<0.005	<0.005	<0.005	<0.016	PAL-PB-LB PB-PP-2518 900-RES-017
	WP 750 g/kg	1.68 2.24 × 3 (banded)	4	57	0.050	0.007	<0.005	0.062	
Hillsboro, OR, United States, 1988 (OS0 91 G; snap bean)	EC 240 g/L	1.68 2.24 × 3 (banded)	4	14	0.156	0.100	0.026	0.293	PAL-PB-BS PB-PP-2519 900-RES-018
	WP 750 g/kg	1.68 2.24 × 3 (banded)	4	14	0.142	0.072	0.022	0.252	
Phelps, NY, United States, 1988 (Improved Tendergreen; snap bean)	EC 240 g/L	1.68 2.24 × 3 (banded)	4	7	0.603	0.231	0.102	0.961	PAL-PB-BS PB-PP-2520 900-RES-018
	WP 750 g/kg	1.68 2.24 × 3 (banded)	4	7	1.29	0.140	0.034	1.48	
Knightdale, NC, United States, 1988 (Blue Lakes; snap bean)	EC 240 g/L	1.68 2.24 × 3 (banded)	4	20	0.017	<0.01	<0.01	0.038	PAL-PB-BS PB-PP-2521 900-RES-018
	WP 750 g/kg	1.68 2.24 × 3 (banded)	4	20	0.028	0.032	0.010	0.073	

Bean (pod) Location, Year (Variety)	Application			DALA	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application at sowing				
GAP in Mexico	FL 480 g/L	8.64	1	-	Band application at the beginning of flowering				
GAP in Mexico	WP 750 g/kg	4.50	1	-	Band application covering the crowns of the plants after cultivation and immediately before raising the furrow to irrigate.				
Loxahatchee, FL, United States, 1988 [d] (Triumph; snap bean)	EC 240 g/L	1.68 2.24 × 3 (banded)	4	10	0.166	0.057	0.032	0.261	PAL-PB-BS PB-PP-2522 900-RES-018
	WP 750 g/kg	1.68 2.24 × 3 (banded)	4	10	0.167	0.023	0.012	0.203	
Union Grove, WI, United States, 1987 (Improved Kingston; lima bean)	WP 750 g/kg	2.31 × 4 (ground)	4	36	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (ground)	4	36	<0.05	<0.05	<0.05	<0.155	
Hope, NJ, United States, 1987 [b] (Henderson Bush; lima bean)	WP 750 g/kg	2.31 × 4 (banded)	4	63	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (banded)	4	63	<0.05	<0.05	<0.05	<0.155	
Hope, NJ, United States, 1987 [b] (Henderson Bush; lima bean)	WP 750 g/kg	2.31 × 4 (banded broadcast)	4	35	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (banded broadcast)	4	35	0.051	<0.05	<0.05	0.157	
Phelps, NY, United States, 1987 (Improved Tendergreen; snap bean)	WP 750 g/kg	2.24 × 4 (ground)	4	14	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (ground)	4	14	<0.05	<0.05	<0.05	<0.155	
	WP 750 g/kg	2.24 × 4 (ground)	4	14	0.052	0.051	<0.05	0.159	900-RES-061
	EC 240 g/L	2.24 x4 (ground)	4	14	<0.05	<0.05	<0.05	<0.155	
Groveland, FL, United States, 1987 (Blue Lake Bush, snap bean)	WP 750 g/kg	2.24 × 4 (ground)	4	14	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (ground)	4	14	<0.05	<0.05	<0.05	<0.155	
Cornelius, OR, United States, 1987 (OSU 91, snap bean)	WP 750 g/kg	2.24 × 4 (ground)	4	14	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (ground)	4	14	<0.05	<0.05	<0.05	<0.155	

Bean (pod) Location, Year (Variety)	Application			DALA	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application at sowing				
GAP in Mexico	FL 480 g/L	8.64	1	-	Band application at the beginning of flowering				
GAP in Mexico	WP 750 g/kg	4.50	1	-	Band application covering the crowns of the plants after cultivation and immediately before raising the furrow to irrigate.				
Skippers, VA, United States, 1987 (Contender; snap bean)	WP 750 g/kg	2.24 × 4 (ground)	4	14	0.080	0.067	<0.05	0.204	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (ground)	4	14	0.067	0.057	<0.05	0.181	
Lake Harbor,FL, United States, 1987 [e] (Gator Green No. 5; snap bean)	WP 750 g/kg	2.24 × 4 (ground)	4	14	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (ground)	4	14	<0.05	<0.05	<0.05	<0.155	
Decatur, MI, United States, 1987 (Gallatin Valley 50, snap bean)	WP 750 g/kg	0.56 2.24 5.60 (ground)	3	14	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
Marcellus, Mi, United States, 1987 (Tender crop; snap bean)	WP 750 g/kg	0.56 2.24 5.60 (ground)	3	19	0.140	0.051	<0.05	0.246	UR-1409 900-RES-061

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

[b] Applications on the same days with 28 days difference in the harvest dates.

[c] Storage period of samples: 76 days.

[d] Storage period of samples: 69 days

[e] Storage period of samples: 72 days

Beans, except broad bean and soya bean (green pods and immature seeds)–In-furrow application

Additional three supervised residue trials were carried out on beans (all belong to the genus *Phaseolus*) in the United States during the growing season 1993 utilizing one in-furrow application at planting.

In all trials, three treated plots were established, where in the first plot a 750 g/kg WP formulation, in the second plot a 240 g/L EC formulation and in the third plot a 480 g/L FL formulation was used. In all trials/plots quintozene was applied once at a rate of 1.68 kg ai/ha.

In all three trials, green pods were sampled at 50–62 DAT and analysed for quintozene, PCA, PCTA, PB and HCB using a modified version of the method CAM-24-73 with an LOQ of 0.0005 mg/kg for each analyte. The method was validated within this study.

Table 77 Magnitude of residues of quintozene in/on immature bean pod after in-furrow application in the supervised trials conducted in the United States in 1993 (The portions analysed were whole pods in all trials.)

Bean (pod) Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application at sowing				
GAP in Mexico	FL 480 g/L	8.64	1	-	Band application at the beginning of flowering				
GAP in Mexico	WP 750 g/kg	4.50	1	-	Band application covering the crowns of the plants after cultivation and immediately before raising the furrow to irrigate.				
Waterloo, NY, United States, 1993 [b] (Improved Tendergreen; snap bean)	WP 750 g/kg	1.68 (in-furrow)	1	58	0.048	<0.0005	<0.0005	0.049	RP-93009 RGC-93-010 900-RES-107
	EC 240 g/L	1.68 (in-furrow)	1	58	0.074	0.010	0.008	0.093	
	FL 480 g/L	1.68 (in-furrow)	1	58	0.066	0.003	0.007	0.076	
Knightdala, NC, United States, 1993 (Continue; snap bean)	WP 750 g/kg	1.68 (in-furrow)	1	50	<0.0005	<0.0005	<0.0005	<0.0016	RP-93009 ABR-93-002 900-RES-107
	EC 240 g/L	1.68 (in-furrow)	1	50	<0.0005	<0.0005	<0.0005	<0.0016	
	FL 480 g/L	1.68 (in-furrow)	1	50	<0.0005	<0.0005	<0.0005	<0.0016	
Hillsboro, OR, United States, 1993 [c] (Oregon 91G)	WP 750 g/kg	1.68 (in-furrow)	1	62	0.013	0.007	<0.0005	0.021	RP-93009 DNJ-93-102 900-RES-107
	EC 240 g/L	1.68 (in-furrow)	1	62	0.017	0.007	<0.0005	0.026	
	FL 480 g/L	1.68 (in-furrow)	1	62	0.013	0.006	<0.0005	0.020	

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

[b] Storage period of samples: 49 days

[c] Storage period of samples: 53 days

Beans, Shelled

For the explanation on the trials, refer to the section on beans, except broad bean and soya bean (green pods and immature seeds).

Table 78 Magnitude of residues of quintozene in/on succulent bean (bean, shelled; portions analysed were succulent seeds) after banded applications to soil or ground applications to soil in the supervised trials conducted in the United States in 1987

Bean (succulent seed) Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application at sowing				
GAP in Mexico	FL 480 g/L	8.64	1	-	Band application at the beginning of flowering				
GAP in Mexico	WP 750 g/kg	4.50	1	-	Band application covering the crowns of the plants after cultivation and immediately before raising the furrow to irrigate.				

Bean (succulent seed) Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
Kenly, NC, United States, 1987 (Tenderette; green bean)	WP 750 g/kg	2.31 × 4 (ground)	4	16	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (ground)	4	16	<0.05	<0.05	<0.05	<0.155	
Waterford, CA, United States, 1987 (Through grain; lima bean)	WP 750 g/kg	2.24 × 4 (banded)	4	35	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (banded)	4	35	<0.05	<0.05	<0.05	<0.155	
Madera, CA United States, 1987 (Fordhook 242; lima bean)	WP 750 g/kg	2.31 × 4 (banded broadcast)	4	35	<0.05	<0.05	<0.05	<0.155	UR-1409 900-RES-061
	EC 240 g/L	2.24 × 4 (banded broadcast)	4	35	<0.05	<0.05	<0.05	<0.155	

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

Pulses

Beans (dry)–Directed band applications to soil or ground applications to soil

Thirteen supervised residue trials were carried out on beans (*Phaseolus*) in the United States during the growing seasons 1987, 1988 and 1989.

In all trials, two plots were established where in the first plot a 750 g/kg WP formulation and in the second plot a 240 g/L EC formulation was used. In all trials/plots, except one, quintozene was applied four times post-emergence at a nominal rate of 2.24 kg ai/ha with a nominal interval of 14 days. In one other trial, only three applications at rates ranging 0.56–5.60 kg ai/ha were performed.

In the 1987 trials, dry beans were sampled at 45–78 DALA and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.05 mg/kg for each analyte. As a follow-up, selected samples were re-analysed with a modified version of the method CAM-24-73 with an LOQ of 0.002 mg/kg for each analyte. However, since samples were re-analysed after a storage of up to 535 days for which stability could not be confirmed, the data from re-analysis were not considered for estimation of maximum residue level.

In the 1988/89 trials, dry beans were sampled at 41–64 DALA and analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB using the method MP-PCNC-MA1 with an LOQ of 0.01 mg/kg for each analyte.

All the samples were stored frozen for periods shorter than the proven storage stability duration.

Table 79 Magnitude of residues of quintozene in/on beans (dry) after post-emergence directed band applications to soil or ground applications to soil in the supervised trials conducted in the United States in 1987, 1988 and 1989 (The portions analysed were dry seeds in all trials.)

Bean (dry)	Application	DALA	Residues (mg/kg) [a]	Study,

Location, Year (Variety)	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	Trial, Reference
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application at sowing				
GAP in Mexico	FL 480 g/L	8.64	1	-	Band application at the beginning of flowering				
GAP in Mexico	WP 750 g/kg	4.50	1	-	Band application covering the crowns of the plants after cultivation and immediately before raising the furrow to irrigate.				
Chowchilla, CA, United States, 1988 (Dark red kidney)	EC 240 g/L	1.68 2.24×3 (banded)	4	42	0.014	<0.01	<0.01	0.035	PAL-PB-DB PB-DB-2523 900-RES-016
	WP 750 g/kg	1.68 2.24 x3 (banded)	4	42	0.012	<0.01	<0.01	0.033	
Minidoka, ID, United States, 1988 (Pinto 114)	EC 240 g/L	1.68 2.24 x3 (banded)	4	64	0.089	0.016	<0.01	0.117	PAL-PB-DB PB-DB-2524 900-RES-016
	WP 750 g/kg	1.68 2.24 x3 (banded)	4	64	0.023	0.011	<0.01	0.045	
Monte Vista, CO, United States, 1988 (Olaethe; pinto)	EC 240 g/L	1.68 2.24 x3 (banded)	4	41	0.019	<0.01	<0.01	0.040	PAL-PB-DB PB-DB-2525 900-RES-016
	WP 750 g/kg	1.68 2.24 x3 (banded)	4	41	0.029	<0.01	<0.01	0.050	
Haslett, MI, United States, 1988 (Seafarer; common bean)	EC 240 g/L	1.68 2.24 x3 (banded)	4	53	0.012	0.026	<0.01	0.051	PAL-PB-DB PB-DB-2527 900-RES-016
	WP 750 g/kg	1.68 2.24 x3 (banded)	4	53	0.012	<0.01	<0.01	0.034	
Hastings, MN, United States, 1989 (Red Kidney)	EC 240 g/L	1.68 2.24 x3 (banded)	4	50	0.061	0.087	<0.01	0.167	PAL-PB-DB PB-DB-2564 900-RES-016
	WP 750 g/kg	1.68 2.24 x3 (banded)	4	50	0.077	0.079	<0.01	0.175	
Colorado State University, CO, United States, 1987 (UI 114; common bean)	WP 750 g/kg	2.35x4 (ground)	4	73	<0.05	<0.05	<0.05	<0.155	A026.001C 900-RES-165
	EC 240 g/L	2.26x4 (ground)	4	73	<0.05	<0.05	<0.05	<0.155	
Waterford, CA, United States, 1987 (Red Kidney)	WP 750 g/kg	2.24×4 (tractor mounted)	4	45	<0.05	<0.05	<0.05	<0.155	A026.001C 900-RES-165
	EC 240 g/L	2.24×4 (tractor mounted)	4	45	<0.05	<0.05	<0.05	<0.155	
Minidoka, ID, United States, 1987 (Kidney)	WP 750 g/kg	2.31x4 (directed band)	4	62	<0.05	<0.05	<0.05	<0.155	A026.001C 900-RES-165
	EC 240 g/L	2.24×4 (directed band)	4	62	<0.05	<0.05	<0.05	<0.155	
Madera, CA, United States,	WP 750 g/kg	2.31x4 (ground)	4	45	<0.05	<0.05	<0.05	<0.155	A026.001C 900-RES-165

Bean (dry) Location, Year (Variety)	Application			DALA	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application at sowing				
GAP in Mexico	FL 480 g/L	8.64	1	-	Band application at the beginning of flowering				
GAP in Mexico	WP 750 g/kg	4.50	1	-	Band application covering the crowns of the plants after cultivation and immediately before raising the furrow to irrigate.				
1987 (Red Kidney)	EC 240 g/L	2.24×4 (ground)	4	45	<0.05	<0.05	<0.05	<0.155	
Northwood, ND, United States, 1987 (Fleetwood; navy bean)	WP 750 g/kg	2.31x4 (ground)	4	67	<0.05	<0.05	<0.05	<0.155	A026.001C 900-RES-165
	EC 240 g/L	2.31x4 (ground)	4	67	<0.05	<0.05	<0.05	<0.155	
Saginaw, MN, United States, 1987 (C-20; navy bean)	WP 750 g/kg	0.56 2.24 5.60 (banded soil application)	3	78	<0.05	<0.05	<0.05	<0.155	A026.001C 900-RES-165
Minidoka, ID, United States, 1987 (Pinto)	WP 750 g/kg	2.24×4 (directed band)	4	62	<0.05	<0.05	<0.05	<0.155	A026.001C 900-RES-165
	EC 240 g/L	2.24×4 (directed band)	4	62	<0.05	<0.05	<0.05	<0.155	
	WP 750 g/kg	2.24×4 (directed band)	4	62	0.058	<0.05	<0.05	0.163	A026.001C 900-RES-165
	EC 240 g/L	2.24×4 (directed band)	4	62	0.080	<0.05	<0.05	0.185	
Saginaw, MN, United States, 1987 (Pindak; pinto bean)	WP 750 g/kg	2.24×4 (banded soil)	4	64	<0.05	<0.05	<0.05	<0.155	A026.001C 900-RES-165
	EC 240 g/L	2.24×4 (banded soil)	4	64	<0.05	<0.05	<0.05	<0.155	

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

Beans (dry)–In-furrow application

Additional three supervised residue trials were carried out on beans (*Phaseolus*) in the United States during the growing season 1993 utilizing one in-furrow application at planting.

In all trials, three treated plots were established where in the first plot a 750 g/kg WP formulation, in the second plot a 240 g/L EC formulation and in the third plot a 480 g/L FL formulation was used. In all trials/plots quintozene was applied once at a rate of 1.68 kg ai/ha.

In all three trials, dry bean seeds were sampled at 77-106 DAT and analysed for quintozene, PCA, PCTA, PB and HCB using a modified version of the method CAM-24-73 with an LOQ of 0.0005 mg/kg for each analyte. The method was validated within this study.

Table 80 Magnitude of residues of quintozene in/on beans (dry) after in-furrow application at planting in the supervised trials conducted in the United States in 1993 (The portions analysed were dry beans in all trials.)

Bean (dry)	Application	DAT	Residues (mg/kg) [a]	Study,

Location, Year (Variety)	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total[b]	Trial, Reference
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application at sowing				
GAP in Mexico	FL 480 g/L	8.64	1	-	Band application at the beginning of flowering				
GAP in Mexico	WP 750 g/kg	4.50	1	-	Band application covering the crowns of the plants after cultivation and immediately before raising the furrow to irrigate.				
Conklin, MI, United States, 1993 (Midland; common bean)	WP 750 g/kg	1.68 (in-furrow)	1	91	<0.0005	<0.0005	<0.0005	<0.0016	RP-93010 JGC-93-016 900-RES-108
	EC 240 g/L	1.68 (in-furrow)	1	91	<0.0005	<0.0005	<0.0005	<0.0016	
	FL 480 g/L	1.68 (in-furrow)	1	91	<0.0005	<0.0005	<0.0005	<0.0016	
Northwood, ND, United States, 1993 (Othello Pinto)	WP 750 g/kg	1.68 (in-furrow)	1	106	0.005	0.001	<0.0005	0.007	RP-93010 AWW-93-016 900-RES-108
	EC 240 g/L	1.68 (in-furrow)	1	106	0.017	0.005	0.001	0.023	
	FL 480 g/L	1.68 (in-furrow)	1	106	0.006	0.002	<0.0005	0.009	
Hughson, CA, United States, 1993 (Light Red Kidney)	WP 750 g/kg	1.68 (in-furrow)	1	77	<0.0005	<0.0005	<0.0005	<0.0016	RP-93010 RCP-93-016 900-RES-108
	EC 240 g/L	1.68 (in-furrow)	1	77	<0.0005	<0.0005	<0.0005	<0.0016	
	FL 480 g/L	1.68 (in-furrow)	1	77	<0.0005	<0.0005	<0.0005	<0.0016	

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

Root and Tuber Vegetables

Potato

In total, forty-six supervised residue trials were carried out on potato in the United States during the growing seasons 1987, 1988/89, 2002, 2006 and 2019/20. Thirty-seven trials/plots utilizing an in-furrow application either at an exaggerated rate of 11.2–13.1 kg ai/ha in side-by-side plots using a 240 g ai/L EC or a 100 g ai/L GR formulation (1988/89 trials) or a 480 g ai/L FL formulation at a rate in the range of 4.12–6.04 kg ai/ha. Eight trials/plots are available utilizing two applications by chemigation at a rate of 2.8 kg ai/ha and at intervals of either 5 or 10–11 days and DALA in the range of 20–41 days.

Additional trials/plots were conducted utilizing a combination of an initial in-furrow application followed by one or two applications by chemigation. Since the United States label says, “Do not make chemigation application, if quintozene products were applied as a band application in furrow at planting”, and this type of use is not permitted by the available labels from other countries, these trials are not presented here.

In the 1987 trials, mature potato tubers were sampled at 82–140 DAT and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.002 mg/kg for each analyte.

In the 1988/89 trials, mature potato tubers were sampled at 89–135 DAT and analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB using the method MP-PCNC-MA1 with an LOQ of 0.01 mg/kg for each analyte.

In the 2002 trials, mature potato tubers were sampled at 20–41 DALA (chemigation) and analysed for parent quintozene using the published method of Zweig 1972 (Analytical Methods for Pesticides and Plant Growth Regulators, Zweig, Volume VI, pages 578–580,1972) with an LOQ of 0.01 mg/kg.

In the 2006 trials of study 900-RES-044, potato tubers were sampled at 96 and 99 DAT (in-furrow application) and 28 DALA (chemigation) and analysed for quintozene, PCA and PCTA using the method MP-PCNC-MA1 with an LOQ of 0.01 mg/kg for each analyte.

In the 2019/20 trials, potato tubers were sampled at 60–125 DAT (in-furrow application) and at 26 and 28 DALA (chemigation) and analysed for quintozene, PCA and PCTA using the Battelle 100117568 method (QuEChERS) method with an LOQ of 0.01 mg/kg for each analyte.

All the samples were stored frozen for periods shorter than the proven storage stability duration.

Table 81 Magnitude of residues of quintozene in/on potato tuber after in-furrow application at planting, chemigation applications, or other application methods, in the supervised trials conducted in the United States in 1987, 1988/89, 2002, 2006 and 2019/20 (The portions analysed were tubers in all trials.)

In-furrow application

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in US	FL 480 g/L	5.6	1	-	Band application by spraying an 8.5-inch band in seed furrow at planting.				
Clewiston, FL. United States, 1987 (Red Irish)	EC 240 g/L	11.2 (in-furrow)	1	126	0.027 (0.013)	0.039	0.014	0.083 (0.042)	UR-1414, 47418 900-RES-063
Hillsboro, OR, United States, 1987 (Russet)	EC 240 g/L	13.1 (in-furrow)	1	105	0.337 (0.144)	0.049	0.052	0.443 (0.189)	UR-1414, 45123 900-RES-063
Marcellus, MI, United States, 1987 (Superior)	EC 240 g/L	11.2 (in-furrow)	1	102	0.328 (0.164)	0.058	0.103	0.495 (0.247)	UR-1414, 46033 900-RES-063
Presque Isle, ME, United States, 1987 (Katahdin)	EC 240 g/L	11.2 (in-furrow)	1	140	0.186 (0.093)	0.040	0.051	0.281 (0.140)	UR-1414, 45642 900-RES-063
Hollandale, WA, United States, 1987 (Norkota)	EC 240 g/L	11.2 (in-furrow)	1	131	0.098 (0.049)	0.032	0.027	0.161 (0.080)	UR-1414, 45366 900-RES-063
Madera, CA, United States, 1987 (Red Baking Variety)	EC 240 g/L	11.2 (in-furrow)	1	114	0.889 (0.444)	0.223	0.205	1.341 (0.670)	UR-1414, 45321 900-RES-063
Bath, MI, United States, 1987 (Onaway)	EC 240 g/L	11.2 (in-furrow)	1	110	0.030 (0.015)	0.010	0.014	0.055 (0.027)	UR-1414, 45138 900-RES-063
Minidoka, ID, United States, 1987 (Russet Burbank)	EC 240 g/L	14.0 (in-furrow)	1	135	0.182 (0.073)	0.041	0.022	0.249 (0.099)	UR-1414, 45359 900-RES-063
	EC 240 g/L	14.0 (in-furrow)	1	135	0.143 (0.057)	0.027	0.022	0.196 (0.078)	
Northwood, ND, United States, 1987 (Norchip)	EC 240 g/L	11.2 (in-furrow)	1	119	0.025 (0.013)	0.019	0.012	0.059 (0.029)	UR-1414, 45194 900-RES-063

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in US	FL 480 g/L	5.6	1	-	Band application by spraying an 8.5-inch band in seed furrow at planting.				
Verona, WI, United States, 1987 (Norland)	EC 240 g/L	11.2 (in-furrow)	1	82	0.180 (0.090)	0.125	0.180	0.499 (0.249)	UR-1414, 45147 900-RES-063
Sherwood, OR, United States, 1987 (Russet)	EC 240 g/L	12.8 (in-furrow)	1	103	0.324 (0.142)	0.012	0.053	0.390 (0.171)	UR-1414, 45123 900-RES-063
Madera, CA, United States, 1988 (Centennial)	EC 240 g/L	13.1 (in-furrow)	1	105	0.478 (0.204)	0.072	0.078	0.636 (0.271)	PAL-PB-PO, PB-DB-2501 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	105	0.121 (0.052)	0.028	0.037	0.190 (0.081)	
Minidoka, ID, United States, 1988 (Russet Burbank)	EC 240 g/L	13.0 (in-furrow)	1	129	0.148 (0.064)	0.044	0.022	0.218 (0.094)	PAL-PB-PO, PB-DB-2502 900-RES-025
	GR 100 g/kg	1.39 (in-furrow)	1	129	0.397 (1.60)	0.013	<0.01	0.421 (1.70)	
Ephrata, WA, United States, 1988 (Russet Burbank)	EC 240 g/L	13.1 (in-furrow)	1	120	0.160 (0.068)	0.106	0.082	0.359 (0.153)	PAL-PB-PO, PB-DB-2503 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	120	<0.01 (--)	<0.01	<0.01	<0.03 (--)	
Union Grove, WI, United States, 1988 (Russet Burbank)	EC 240 g/L	13.1 (in-furrow)	1	119	0.239 (0.102)	0.020	0.057	0.317 (0.135)	PAL-PB-PO, PB-DB-2504 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	119	0.131 (0.056)	<0.01	0.028	0.170 (0.073)	
Hollandale, MN, United States, 1988 (Pontiac)	EC 240 g/L	13.1 (in-furrow)	1	109	0.355 (0.152)	0.041	0.084	0.484 (0.207)	PAL-PB-PO, PB-DB-2505 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	109	0.076 (0.033)	0.010	0.020	0.108 (0.046)	
Houlton, ME, United States, 1988 (Superior)	EC 240 g/L	13.1 (in-furrow)	1	115	0.380 (0.162)	0.041	0.079	0.504 (0.215)	PAL-PB-PO, PB-DB-2506 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	115	0.093 (0.040)	0.016	0.024	0.136 (0.058)	
Loxahatchee, FL, United States, 1988 (Pontiac)	EC 240 g/L	13.1 (in-furrow)	1	89	0.081 (0.035)	<0.01	0.016	0.109 (0.046)	PAL-PB-PO, PB-DB-2507 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	89	0.088 (0.038)	<0.01	<0.01	0.109 (0.047)	
Minidoka, ID, United States, 1989 (Russet Burbank)	EC 240 g/L	13.1 (in-furrow)	1	135	0.844 (0.360)	0.069	0.033	0.953 (0.407)	PAL-PB-PO, PB-DB-2560 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	135	0.052 (0.022)	<0.01	<0.01	0.073 (0.031)	
Rupert, ID, United States, 2006 (Russet Norkotah)	FL (480 g/L)	4.12 (in-furrow)	1	99	0.126 (0.171)	0.022	0.012	0.163 (0.221)	TCI-06-146, TCI-06-146-01 900-RES-044
Ephrata, WA, United States, 2006 (Russet Norkotah)	FL (480 g/L)	4.24 (in-furrow)	1	96	0.072 (0.095)	<0.01	0.012	0.095 (0.125)	TCI-06-146, TCI-06-146-02 900-RES-044
Germansville, PA. United States, 2019 (Dark Red Norland)	FL (480 g/L)	6.04 (in-furrow)	1	82	0.107 (0.099)	0.111	0.071	0.301 (0.279)	AMVAC LR19391 01 900-RES-225

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in US	FL 480 g/L	5.6	1	-	Band application by spraying an 8.5-inch band in seed furrow at planting.				
Chula, GA, United States, 2019 (Red Pontiac)	FL (480 g/L)	5.72 (in-furrow)	1	77	0.159 (0.156)	0.034	0.053	0.249 (0.244)	AMVAC LR19391 02 900-RES-225
Moberly, MO, United States, 2019 (Yukon Gold)	FL (480 g/L)	5.59 (in-furrow)	1	86	0.347 (0.347)	0.474	0.138	1.01 (1.01)	AMVAC LR19391 03 900-RES-225
Richland, IA, United States, 2019 (Yukon Gold)	FL (480 g/L)	5.63 (in-furrow)	1	118	0.391 (0.389)	0.231	0.078	0.726 (0.722)	AMVAC LR19391 04 900-RES-225
Yuma, AZ, United States, 2019 (Norkotah)	FL (480 g/L)	5.72 (in-furrow)	1	120	0.020 (0.020)	<0.01	<0.01	0.041 (0.041)	AMVAC LR19391 05 900-RES-225
Payette, ID, United States, 2019 (Ranger Russet)	FL (480 g/L)	5.83 (in-furrow)	1	133	0.229 (0.220)	0.113	0.060	0.414 (0.398)	AMVAC LR19391 06 900-RES-225
Jerome, ID, United States, 2019 (Ranger)	FL (480 g/L)	5.59 (in-furrow)	1	148	0.037 (0.037)	0.011	0.01	0.060 (0.060)	AMVAC LR19391 07 900-RES-225
Ephrata, WA, United States, 2019 [b] (Norland Dark Red Gen 3)	FL (480 g/L)	5.58 (in-furrow)	1	64	<0.01	<0.01	<0.01	<0.03	AMVAC LR19391 08 900-RES-225
Ephrata, WA, United States, 2019 [b] (Norkotah)	FL (480 g/L)	5.69 (in-furrow)	1	118	<0.01	<0.01	<0.01	<0.03	AMVAC LR19391 09 900-RES-225
North Rose, NY, United States, 2019 (French Fingerling)	FL (480 g/L)	5.69 (in-furrow)	1	125	0.104 (0.102)	0.041	0.015	0.164 (0.162)	AMVAC LR19391 14 900-RES-225
Winter Garden, FL, United States, 2019 (Yukon Gold)	FL (480 g/L)	5.62 (in-furrow)	1	75	0.790 (0.787)	0.215	0.193	1.22 (1.22)	AMVAC LR19391 15 900-RES-225
Moberly, MO, United States, 2019 (French Fingerling)	FL (480 g/L)	5.62 (in-furrow)	1	86	0.179 (0.178)	0.242	0.063	0.510 (0.509)	AMVAC LR19391 16 900-RES-225
York, NE, United States, 2019 (Yukon Gold)	FL (480 g/L)	5.64 (in-furrow)	1	78	0.276 (0.274)	0.124	0.040	0.453 (0.450)	AMVAC LR19391 17 900-RES-225
Fresno, CA, United States, 2019 (Yukon Gold)	FL (480 g/L)	5.64 (in-furrow)	1	60	0.377 (0.374)	0.171	0.090	0.657 (0.652)	AMVAC LR19391 18 900-RES-225
Jerome, ID, United States, 2019 (French Fingerling)	FL (480 g/L)	5.71 (in-furrow)	1	106	0.057 (0.056)	0.015	<0.01	0.084 (0.083)	AMVAC LR19391 19 900-RES-225

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in US	FL 480 g/L	5.6	1	-	Band application by spraying an 8.5-inch band in seed furrow at planting.				
Ephrata, WA, United States, 2019 [b] (Cheshire)	FL (480 g/L)	5.58 (in-furrow)	1	98	0.081 (0.081)	0.083	0.073	0.247 (0.248)	AMVAC LR19391 20 900-RES-225

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

[b] The application dates were 30 days or more apart in these three trials: trial 08 to 09, 30 days; trial 09 to 20, 33 days; and trial 08 to 20, 63 days.

[c] The application rate was inadvertently low. The irrigation was about one half of the frequency of normal practice and as a result, the harvest was poor. Therefore, the result of this trial was not used.

(Residues of PB, HCB, 2,34,5-TCNB and 2,3,5,6-TCNB in potato from in-furrow application with soil at planting)

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		PB	HCB	2,3,4,5-TCNB	2,3,5,6-TCNB	
Clewiston, FL. United States, 1987 (Red Irish)	EC 240 g/L	11.2 (in-furrow)	1	126	0.029	0.003	--	--	UR-1414, 47418 900-RES-063
Hillsboro, OR, United States, 1987 (Russet)	EC 240 g/L	13.1 (in-furrow)	1	105	0.022	0.011	--	--	UR-1414, 45123 900-RES-063
Marcellus, MI, United States, 1987 (Superior)	EC 240 g/L	11.2 (in-furrow)	1	102	0.036	0.019	--	--	UR-1414, 46033 900-RES-063
Presque Isle, ME, United States, 1987 (Katahdin)	EC 240 g/L	11.2 (in-furrow)	1	140	0.020	0.013	--	--	UR-1414, 45642 900-RES-063
Hollandale, WA, United States, 1987 (Norkota)	EC 240 g/L	11.2 (in-furrow)	1	131	0.028	0.006	--	--	UR-1414, 45366 900-RES-063
Madera, CA, United States, 1987 (Red Baking Variety)	EC 240 g/L	11.2 (in-furrow)	1	114	0.052	0.027	--	--	UR-1414, 45321 900-RES-063
Bath, MI, United States, 1987 (Onaway)	EC 240 g/L	11.2 (in-furrow)	1	110	0.008	0.003	--	--	UR-1414, 45138 900-RES-063

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		PB	HCB	2,3,4,5-TCNB	2,3,5,6-TCNB	
Minidoka, ID, United States, 1987 (Russet Burbank)	EC 240 g/L	14.0 (in-furrow)	1	135	0.029	0.005	--	--	UR-1414, 45359 900-RES-063
	EC 240 g/L	14.0 (in-furrow)	1	135	0.017	0.004	--	--	
Northwood, ND, United States, 1987 (Norchip)	EC 240 g/L	11.2 (in-furrow)	1	119	0.014	0.003	--	--	UR-1414, 45194 900-RES-063
Verona, WI, United States, 1987 (Norland)	EC 240 g/L	11.2 (in-furrow)	1	82	0.049	0.014	--	--	UR-1414, 45147 900-RES-063
Sherwood, OR, United States, 1987 (Russet)	EC 240 g/L	12.8 (in-furrow)	1	103	0.009	0.007	--	--	UR-1414, 45123 900-RES-063
Madera, CA, United States, 1988 (Centennial)	EC 240 g/L	13.1 (in-furrow)	1	105	0.031	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2501 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	105	0.022	<0.01	<0.01	<0.01	
Minidoka, ID, United States, 1988 (Russet Burbank)	EC 240 g/L	13.0 (in-furrow)	1	129	0.033	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2502 900-RES-025
	GR 100 g/kg	1.39 (in-furrow)	1	129	0.010	<0.01	<0.01	<0.01	
Ephrata, WA, United States, 1988 (Russet Burbank)	EC 240 g/L	13.1 (in-furrow)	1	120	0.099	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2503 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	120	<0.01	<0.01	<0.01	<0.01	
Union Grove, WI, United States, 1988 (Russet Burbank)	EC 240 g/L	13.1 (in-furrow)	1	119	0.012	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2504 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	119	<0.01	<0.01	<0.01	<0.01	
Hollandale, MN, United States, 1988 (Pontiac)	EC 240 g/L	13.1 (in-furrow)	1	109	0.015	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2505 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	109	<0.01	<0.01	<0.01	<0.01	
Houlton, ME, United States, 1988 (Superior)	EC 240 g/L	13.1 (in-furrow)	1	115	0.025	<0.01	0.011	<0.01	PAL-PB-PO, PB-DB-2506 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	115	0.012	<0.01	<0.01	<0.01	
Loxahatchee, FL, United States, 1988 (Pontiac)	EC 240 g/L	13.1 (in-furrow)	1	89	0.018	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2507 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	89	<0.01	<0.01	<0.01	<0.01	
Minidoka, ID, United States, 1989 (Russet Burbank)	EC 240 g/L	13.1 (in-furrow)	1	135	0.041	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2560 900-RES-025
	GR 100 g/kg	13.1 (in-furrow)	1	135	<0.01	<0.01	<0.01	<0.01	

Chemigation

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg)				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in US	FL 480 g/L	2.8	2	28	Chemigation. Second foliar application may be made 10 days of more after the first application.				
Eltopia, WA, United States, 2002 (Russet Burbank)	FL (480 g/L)	2.8 (chemigation)	1	20 30 41	<0.01 0.015 <0.01	not analysed	not analysed	--	ML02-1043-AMV, WM-Pot.2002, 900-RES-030
		5.6 (chemigation)	1	20 30 41	0.013 <0.01 0.021	not analysed	not analysed	--	
		8.4 (chemigation)	1	20 30 41	0.028 <0.01 <0.01	not analysed	not analysed	--	
		2.8 (chemigation)	2	20 30 41	0.013 <0.01 0.011	not analysed	not analysed	--	
		5.6 (chemigation)	2	20 30 41	0.016 0.037 0.020	not analysed	not analysed	--	
		8.4 (chemigation)	2	20 30 41	0.023 0.025 0.034	not analysed	not analysed	--	
Rupert, ID, United States, 2006 (Russet Norkotah)	FL (480 g/L)	2.8 (chemigation)	2	28	<0.01	<0.01	<0.01	<0.03	TCI-06-146, TCI-06-146-01 900-RES-044
Ephrata, WA, United States, 2006 (Russet Norkotah)	FL (480 g/L)	2.8 (chemigation)	2	28	0.011	<0.01	<0.01	0.032	TCI-06-146, TCI-06-146-02 900-RES-044
Richland, IA, United States, 2019 (Yukon Gold)	FL (480 g/L)	2.8 (chemigation)	2	26	0.080	0.042	0.020	0.147	AMVAC LR19391 10 900-RES-225
Yuma, AZ, United States, 2019 (Masquerade)	FL (480 g/L)	2.8 (chemigation)	2	28	0.026	<0.01	<0.01	0.047	AMVAC LR19391 11 900-RES-225
Ephrata, WA, United States, 2019 [b] (Russet Burbank)	FL (480 g/L)	2.8 (chemigation)	2	28	<0.01	<0.01	<0.01	<0.03	AMVAC LR19391 12 900-RES-225
Ephrata, WA, United States, 2019 [b] (Cheshire)	FL (480 g/L)	2.8 (chemigation)	2	28	0.017	0.011	0.013	0.043	AMVAC LR19391 13 900-RES-225
Winter Garden, FL, United States, 2019 (Red Lasoda)	FL (480 g/L)	2.8 (chemigation)	2	28	0.037	0.011	0.010	0.059	AMVAC LR19391 21 900-RES-225

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

[b] The applications dates were 49 days apart between these trials.

Broadcast application with soil incorporation before planting

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	WP 750 g/kg	30.0	1	-	Total application.				
Clewiston, FL, United States, 1987 (Red Irish)	EC 240 g/L	28.0 (broadcast)	1	126	0.152	0.101	0.050	0.314	UR-1414, 47418 900-RES-063
Hillsboro, OR, United States, 1987 (Russet)	EC 240 g/L	28.0 (broadcast)	1	125	0.048	0.017	0.023	0.090	UR-1414, 45123 900-RES-063
Marcellus, MI, United States, 1987 (Superior)	EC 240 g/L	28.0 (broadcast)	1	102	0.098	0.027	0.058	0.185	UR-1414, 46033 900-RES-063
Presque Isle, ME, United States, 1987 (Katahdin)	EC 240 g/L	28.0 (broadcast)	1	140	0.067	0.035	0.044	0.150	UR-1414, 45642 900-RES-063
Hollandale, WA, United States, 1987 (Norkota)	EC 240 g/L	28.0 (broadcast)	1	132	0.007	0.002	<0.002	0.012	UR-1414, 45366 900-RES-063
Burlington, WA, United States, 1987 [b] (Russet Burbank)	EC 240 g/L	26.9 (broadcast)	1	121	0.052	0.023	0.044	0.122	UR-1414, 45352 900-RES-063
Burlington, WA, United States, 1987 [b] (Norgold)	EC 240 g/L	26.9 (broadcast)	1	121	0.070	0.029	0.033	0.135	UR-1414, 45353 900-RES-063
Burlington, WA, United States, 1987 [b] (Red LaSota)	EC 240 g/L	26.9 (broadcast)	1	121	0.008	0.010	0.010	0.029	UR-1414, 45354 900-RES-063
Madera, CA, United States, 1987 (Red Baking Variety)	EC 240 g/L	28.0 (broadcast)	1	114	0.139	0.182	0.144	0.484	UR-1414, 45321 900-RES-063
Bath, OR, United States, 1987 (Onaway)	EC 240 g/L	28.0 (broadcast)	1	110	<0.002	<0.002	<0.002	<0.006	UR-1414, 45138 900-RES-063
Minidoka, ID, United States, 1987 (Russet Burbank)	EC 240 g/L	28.0 (broadcast)	1	135	0.016	0.051	0.008	0.080	UR-1414, 45359 900-RES-063
	EC 240 g/L	28.0 (broadcast)	1	135	0.018	0.019	0.007	0.046	

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	WP 750 g/kg	30.0	1	-	Total application.				
Northwood, ND, United States, 1987 (Norchip)	EC 240 g/L	28.0 (broadcast)	1	119	0.014	0.020	0.011	0.047	UR-1414, 45194 900-RES-063
Verona, WI, United States, 1987 (Norland)	EC 240 g/L	28.0 (broadcast)	1	82	0.009	0.038	0.028	0.079	UR-1414, 45147 900-RES-063
Sherwood, OR, United States, 1987 (Russet)	EC 240 g/L	28.0 (broadcast)	1	127	0.007	0.006	0.007	0.021	UR-1414, 45123 900-RES-063
Madera, CA, United States, 1988 (Centennial)	EC 240 g/L	28.0 (broadcast)	1	105	0.117	0.055	0.072	0.250	PAL-PB-PO, PB-DB-2501 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	105	0.046	0.018	0.022	0.088	
Minidoka, ID, United States, 1988 (Russet Burbank)	EC 240 g/L	22.4 (broadcast)	1	131	0.016	0.053	0.010	0.085	PAL-PB-PO, PB-DB-2502 900-RES-025
	GR 100 g/kg	2.9 (broadcast)	1	131	0.192	0.020	<0.01	0.224	
Ephrata, WA, United States, 1988 (Russet Burbank)	EC 240 g/L	28.0 (broadcast)	1	120	0.030	0.064	0.025	0.126	PAL-PB-PO, PB-DB-2503 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	120	0.044	0.067	0.021	0.139	
Union Grove, WI, United States, 1988 (Russet Burbank)	EC 240 g/L	28.0 (broadcast)	1	119	0.064	0.011	0.027	0.103	PAL-PB-PO, PB-DB-2504 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	119	0.078	<0.01	0.019	0.108	
Hollandale, MN, United States, 1988 (Pontiac)	EC 240 g/L	28.0 (broadcast)	1	109	0.087	0.010	0.027	0.125	PAL-PB-PO, PB-DB-2505 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	109	0.040	<0.01	0.013	0.064	
Houlton, ME, United States, 1988 (Superior)	EC 240 g/L	28.0 (broadcast)	1	117	0.114	0.023	0.051	0.191	PAL-PB-PO, PB-DB-2506 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	117	0.060	0.011	0.026	0.098	
Loxahatchee, FL, United States, 1988 (Pontiac)	EC 240 g/L	28.0 (broadcast)	1	89	0.663	0.091	0.136	0.900	PAL-PB-PO, PB-DB-2507 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	89	0.014	<0.01	<0.01	0.035	
Minidoka, ID, United States, 1989 (Russet Burbank)	EC 240 g/L	28.0 (broadcast)	1	135	0.074	0.017	0.031	0.124	PAL-PB-PO, PB-DB-2560 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	135	0.061	0.017	<0.01	0.090	

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

[b] Application on the same day; and sowing and harvesting on the same days.

(Residues of PB, HCB, 2,34,5-TCNB and 2,3,5,6-TCNB in potato from broadcast application with soil incorporation before planting)

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		PB	HCB	2,3,4,5-TCNB	2,3-5,6-TCNB	
Clewiston, FL, United States, 1987 (Red Irish)	EC 240 g/L	28.0 (broadcast)	1	126	0.055	0.008	--	--	UR-1414, 47418 900-RES-063
Hillsboro, OR, United States, 1987 (Russet)	EC 240 g/L	28.0 (broadcast)	1	125	0.025	0.004	--	--	UR-1414, 45123 900-RES-063
Marcellus, MI, United States, 1987 (Superior)	EC 240 g/L	28.0 (broadcast)	1	102	0.034	0.011	--	--	UR-1414, 46033 900-RES-063
Presque Isle, ME, United States, 1987 (Katahdin)	EC 240 g/L	28.0 (broadcast)	1	140	0.025	0.009	--	--	UR-1414, 45642 900-RES-063
Hollandale, WA, United States, 1987 (Norkota)	EC 240 g/L	28.0 (broadcast)	1	132	0.003	<0.002	--	--	UR-1414, 45366 900-RES-063
Burlington, WA, United States, 1987 [b] (Russet Burbank)	EC 240 g/L	26.9 (broadcast)	1	121	0.020	0.005	--	--	UR-1414, 45352 900-RES-063
Burlington, WA, United States, 1987 [b] (Norgold)	EC 240 g/L	26.9 (broadcast)	1	121	0.006	0.005	--	--	UR-1414, 45353 900-RES-063
Burlington, WA, United States, 1987 [b] (Red LaSota)	EC 240 g/L	26.9 (broadcast)	1	121	0.014	0.003	--	--	UR-1414, 45354 900-RES-063
Madera, CA, United States, 1987 (Red Baking Variety)	EC 240 g/L	28.0 (broadcast)	1	114	0.100	0.021	--	--	UR-1414, 45321 900-RES-063
Bath, OR, United States, 1987 (Onaway)	EC 240 g/L	28.0 (broadcast)	1	110	<0.002	<0.002	--	--	UR-1414, 45138 900-RES-063
Minidoka, ID, United States, 1987 (Russet Burbank)	EC 240 g/L	28.0 (broadcast)	1	135	0.062	0.004	--	--	UR-1414, 45359 900-RES-063
	EC 240 g/L	28.0 (broadcast)	1	135	0.019	0.002	--	--	

Potato Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		PB	HCB	2,3,4,5-TCNB	2,3-5,6-TCNB	
Northwood, ND, United States, 1987 (Norchip)	EC 240 g/L	28.0 (broadcast)	1	119	0.023	0.003	--	--	UR-1414, 45194 900-RES-063
Verona, WI, United States, 1987 (Norland)	EC 240 g/L	28.0 (broadcast)	1	82	0.029	0.004	--	--	UR-1414, 45147 900-RES-063
Sherwood, OR, United States, 1987 (Russet)	EC 240 g/L	28.0 (broadcast)	1	127	0.008	<0.002	--	--	UR-1414, 45123 900-RES-063
Madera, CA, United States, 1988 (Centennial)	EC 240 g/L	28.0 (broadcast)	1	105	0.044	<0.01	0.010	<0.01	PAL-PB-PO, PB-DB-2501 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	105	0.017	<0.01	<0.01	<0.01	
Minidoka, ID, United States, 1988 (Russet Burbank)	EC 240 g/L	22.4 (broadcast)	1	131	0.075	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2502 900-RES-025
	GR 100 g/kg	2.9 (broadcast)	1	131	0.028	<0.01	<0.01	<0.01	
Ephrata, WA, United States, 1988 (Russet Burbank)	EC 240 g/L	28.0 (broadcast)	1	120	0.087	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2503 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	120	0.066	<0.01	<0.01	<0.01	
Union Grove, WI, United States, 1988 (Russet Burbank)	EC 240 g/L	28.0 (broadcast)	1	119	0.011	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2504 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	119	<0.01	<0.01	<0.01	<0.01	
Hollandale, MN, United States, 1988 (Pontiac)	EC 240 g/L	28.0 (broadcast)	1	109	<0.01	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2505 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	109	<0.01	<0.01	<0.01	<0.01	
Houlton, ME, United States, 1988 (Superior)	EC 240 g/L	28.0 (broadcast)	1	117	0.027	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2506 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	117	0.010	<0.01	<0.01	<0.01	
Loxahatchee, FL, United States, 1988 (Pontiac)	EC 240 g/L	28.0 (broadcast)	1	89	0.062	<0.01	0.014	<0.01	PAL-PB-PO, PB-DB-2507 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	89	0.017	<0.01	<0.01	<0.01	
Minidoka, ID, United States, 1989 (Russet Burbank)	EC 240 g/L	28.0 (broadcast)	1	135	0.137	<0.01	<0.01	<0.01	PAL-PB-PO, PB-DB-2560 900-RES-025
	GR 100 g/kg	28.0 (broadcast)	1	135	0.032	<0.01	<0.01	<0.01	

Oilseeds

Cotton seed

In total twenty-three supervised residue trials were carried out on cotton in the United States during the growing seasons 1987 to 1992.

In all trials, two or more plots were established where the granules, emulsifiable concentrates and flowable formulations were compared by use as an in-furrow application at a rate of 2.24 kg ai/ha at the time of planting. In some trials in 1991 and 1992, a reduced rate of 1.12 or 1.68 kg ai/ha was also used. In all the trials on cotton, the dates of application are the same as the dates of sowing/planting.

In the 1987 trials, cotton seeds were sampled at 137–149 DAT and analysed for quintozene, PCA, PCTA, PB, and HCB using the method CAM-24-73 with an LOQ of 0.005 mg/kg for each analyte. At two locations, Bossier City, Louisiana, and Somerton, Arizona, in two trials each, application dates were the same. In this study an additional trial was established utilizing an exaggerated application rate of 11.2 kg ai/ha in-furrow to produce samples for processing.

In the 1988 trials reported in 900-RES-021, cotton seeds were sampled at 135–173 DAT and analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB using the method MP-PCNC-MA1 with an LOQ of 0.01 mg/kg for each analyte. In the 1988 trials reported in 900-RES-152, cotton seeds were sampled at 141–168 DAT and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.005 mg/kg for each analyte.

In the 1990 trials, cotton seeds were sampled at 142–160 DAT and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.002 mg/kg for each analyte.

In the 1991 trials, cotton seeds were sampled at 129–181 DAT and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.002 mg/kg for each analyte.

In the 1992 trial, cotton seeds were sampled at 142 DAT and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.002 mg/kg for each analyte.

All the samples were stored frozen for periods shorter than the proven storage stability duration.

Table 82 Magnitude of residues of quintozene in/on cotton seeds after in-furrow application at planting in the supervised trials conducted in the United States in 1987, 1988, 1990, 1991 and 1992 (The portions analysed were seeds in all trials.)

Cotton Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application on sowing.				
Bossier City, LA, United States, 1987 [b] (Deltapine 41)	EC 240 g/L	2.24 (in-furrow)	1	145	<0.005	<0.005	<0.005	<0.016	UR1412 PRN-87-005 900-RES-068
	GR 100 g/kg	2.24 (in-furrow)	1	145	<0.005	<0.005	<0.005	<0.016	
Bossier City, LA, United States, 1987 [b] (Deltapine 41)	EC 240 g/L	2.24 (in-furrow)	1	148	<0.005	<0.005	<0.005	<0.016	UR1412 PRN-87-006 900-RES-068
	GR 100 g/kg	2.24 (in-furrow)	1	148	<0.005	<0.005	<0.005	<0.016	
Senatobia, MS, United States, 1987 (Stoneville 825)	EC 240 g/L	2.24 (in-furrow)	1	137	<0.005	<0.005	<0.005	<0.016	UR1412 WSM-87-010 900-RES-068
	GR 100 g/kg	2.24 (in-furrow)	1	137	0.005	<0.005	<0.005	0.016	

Cotton Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application on sowing.				
Stoneville, MS. United States, 1987 (DES 119)	EC 240 g/L	2.24 (in-furrow)	1	149	0.012	<0.005	<0.005	0.023	UR1412 WSM-87-011 900-RES-068
	GR 100 g/kg	2.24 (in-furrow)	1	149	0.010	<0.005	<0.005	0.021	
Somerton, AZ, United States, 1987 [c] (DPL 91)	EC 240 g/L	2.24 (in-furrow)	1	140	<0.005	<0.005	<0.005	<0.016	UR1412 AZ Site 900-RES-068
	GR 100 g/kg	2.24 (in-furrow)	1	140	0.006	<0.005	<0.005	0.016	
Somerton, AZ, United States, 1987 [c] (DPL 91)	EC 240 g/L	2.24 (in-furrow)	1	140	<0.005	0.007	<0.005	0.018	UR1412 AZ Site 900-RES-068
	GR 100 g/kg	2.24 (in-furrow)	1	140	<0.005	0.006	<0.005	0.016	
Litchfield Park, Az, United States, 1988 (DPL-120)	EC 240 g/L	2.24 (in-furrow)	1	173	<0.01	<0.01	<0.01	<0.03	PAL-PB-CS PB-CS-2537 900-RES-021
	GR 100 g/kg	2.24 (in-furrow)	1	173	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	2.24 (banded soil)	1	173	<0.01	<0.01	<0.01	<0.03	
Kerman, CA, United States, 1988 (SJ-1)	EC 240 g/L	2.24 (in-furrow)	1	153	<0.01	<0.01	<0.01	<0.03	PAL-PB-CS PB-CS-2538 900-RES-021
	GR 100 g/kg	2.24 (in-furrow)	1	153	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	2.24 (banded soil)	1	153	<0.01	<0.01	<0.01	<0.03	
Friars Point MS, United States, 1988 (Stoneville 506)	EC 240 g/L	2.24 (in-furrow)	1	135	<0.01	<0.01	<0.01	<0.03	PAL-PB-CS PB-CS-2539 900-RES-021
	GR 100 g/kg	2.24 (in-furrow)	1	135	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	2.24 (banded soil)	1	135	<0.01	<0.01	<0.01	<0.03	
Washington, LA, United States, 1988 (Stoneville 506)	EC 240 g/L	2.24 (in-furrow)	1	140	<0.01	<0.01	<0.01	<0.03	PAL-PB-CS PB-CS-2540 900-RES-021
	GR 100 g/kg	2.24 (in-furrow)	1	140	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	2.24 (banded soil)	1	140	<0.01	<0.01	<0.01	<0.03	
Brookshire, TX, United States, 1988 (DPL-50)	EC 240 g/L	2.24 (in-furrow)	1	138	<0.01	<0.01	<0.01	<0.03	PAL-PB-CS PB-CS-2541 900-RES-021
	GR 100 g/kg	2.24 (in-furrow)	1	138	<0.01	<0.01	<0.01	<0.03	
	GR 100 g/kg	2.24 (banded soil)	1	138	<0.01	<0.01	<0.01	<0.03	
Kerman, CA, United States, 1988 (not reported)	EC 240 g/L	2.24 (in-furrow)	1	168	<0.005	<0.005	<0.005	<0.016	RP-88001 CEJ-88-003 900-RES-152
	FL 480 g/L	2.24 (in-furrow)	1	168	<0.005	0.007	<0.005	0.017	
Madera, CA, United States, 1988 (not reported)	EC 240 g/L	2.24 (in-furrow)	1	161	<0.005	<0.005	<0.005	<0.016	RP-88001 CEJ-88-004 900-RES-152
	FL 480 g/L	2.24 (in-furrow)	1	161	<0.005	<0.005	<0.005	<0.016	
Senatobia, MS, United States,	EC 240 g/L	2.24 (in-furrow)	1	141	<0.005	<0.005	<0.005	<0.016	RP-88001 WSM-88-011

Cotton Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application on sowing.				
1988 (DPL50)	FL 480 g/L	2.24 (in-furrow)	1	141	<0.005	<0.005	<0.005	<0.016	900-RES-152
Bossier, LA, United States, 1988 (DPL20)	EC 240 g/L	2.24 (in-furrow)	1	146	0.007	0.011	<0.005	0.024	RP-88001 PRN-88-011 900-RES-152
	FL 480 g/L	2.24 (in-furrow)	1	146	<0.005	0.007	<0.005	0.018	
Opelousas, LA, United States, 1988 (DPL20)	EC 240 g/L	2.24 (in-furrow)	1	150	<0.005	<0.005	<0.005	<0.016	RP-88001 PRN-88-010 900-RES-152
	FL 480 g/L	2.24 (in-furrow)	1	150	<0.005	<0.005	<0.005	<0.016	
Bishop, GA, United States, 1988 (Stoneville 825)	EC 240 g/L	2.24 (in-furrow)	1	146	<0.005	<0.005	<0.005	<0.016	RP-88001 KHG-88-5 900-RES-152
	FL 480 g/L	2.24 (in-furrow)	1	146	<0.005	<0.005	<0.005	<0.016	
Avon, MS, United States, 1988 (Stoneville 112)	EC 240 g/L	2.24 (in-furrow)	1	147	<0.005	<0.005	<0.005	<0.016	RP-88001 WSM-88-013 900-RES-152
	FL 480 g/L	2.24 (in-furrow)	1	147	<0.005	<0.005	<0.005	<0.016	
Hernando, MS, United States, 1990 (DPL50)	GR 100 g/kg	2.24 (in-furrow)	1	142	<0.002	0.002	<0.002	0.006	RP-90034 WSM-90-007 900-RES-110
	EC 240 g/L	2.24 (in-furrow)	1	142	<0.002	0.002	<0.002	0.006	
Plainview, TX, United States, 1990 (Pay-master 145)	GR 100 g/kg	2.24 (in-furrow)	1	160	<0.002	0.002	<0.002	0.006	RP-90034 CRA-90-089 900-RES-110
	EC 240 g/L	2.24 (in-furrow)	1	160	<0.002	<0.002	<0.002	<0.006	
Fresno, CA, United States, 1991 (Acala GC-510)	GR 100 g/kg	1.68 (in-furrow)	1	181	<0.002	0.004	<0.002	0.008	RP-91020 CEJ-91-005 900-RES-067
	GR 100 g/kg	2.24 (in-furrow)	1	181	<0.002	<0.002	<0.002	<0.006	
	EC 240 g/L	1.68 (in-furrow)	1	181	<0.002	<0.002	<0.002	<0.006	
	EC 240 g/L	2.24 (in-furrow)	1	181	<0.002	<0.002	<0.002	<0.006	
Avon, MS, United States, 1991 (DES 119)	GR 100 g/kg	1.12 (in-furrow)	1	129	<0.002	<0.002	<0.002	<0.006	RP-91020 WSM-91-001 900-RES-067
	GR 100 g/kg	1.68 (in-furrow)	1	129	<0.002	<0.002	<0.002	<0.006	
	GR 100 g/kg	2.24 (in-furrow)	1	129	<0.002	<0.002	<0.002	<0.006	
	EC 240 g/L	1.12 (in-furrow)	1	129	<0.002	<0.002	<0.002	<0.006	
	EC 240 g/L	1.68 (in-furrow)	1	129	<0.002	<0.002	<0.002	<0.006	
	EC 240 g/L	2.24 (in-furrow)	1	129	<0.002	<0.002	<0.002	<0.006	
	FL 480 g/L	1.12 (in-furrow)	1	129	<0.002	<0.002	<0.002	<0.006	
	FL 480 g/L	1.68 (in-furrow)	1	129	<0.002	<0.002	<0.002	<0.006	
Senatobia, MN, United States,	FL 240 g/L	1.68 (in-furrow)	1	142	<0.002	0.004	<0.002	0.008	RP-90035 WSM-88-013

Cotton Location, Year (Variety)	Application			DAT	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.		Quintozene	PCA	PCTA	Total	
GAP in Mexico	FL 480 g/L	1.44	1	-	Band application on sowing.				
1992 (DPL50)	EC 240 g/L	1.68 (in-furrow)	1	142	<0.002	0.004	<0.002	0.008	900-RES-197

Notes:

[a] Mean of the analytical results of two samples. Residues scaled to the critical GAP-rate are presented in brackets

[b] The application dates are the same in these applications with three day difference in the harvest dates. Therefore, they are not regarded as independent.

[c] The application dates are the same and the harvest dates are the same. Therefore, they are not regarded as independent.

Peanut

In total, thirty-six supervised residue trials were carried out on peanuts in the United States during the growing seasons 1987 to 1992.

The trials were performed with one or more plots treated with quintozene as a 100 g ai/kg GR formulation, a 750 g ai/kg WP formulation, a 240 g ai/L EC formulation or a 480 g ai/L FL formulation at pegging stage until full seed beginning to mature. Applications were performed either as a banded granular application or directed spray (banded or broadcast), or by overhead sprinkler application or chemigation. Two trials are available where quintozene as a granular formulation was applied by aerial broadcast application. Applications were done at a nominal rate of 11.2 kg ai/ha either in one application or split equally to 2 × 5.6 kg ai/ha or in the range of 3.4–7.9 kg ai/ha.

In the 1987 trials, quintozene was applied as a split application in the range of 3.36–7.85 kg ai/ha totalling to nominally 11.2 kg ai/ha at intervals of 21–63 days. Eight trials were performed by banded application of GR and WP formulations, six trials by sprinkler application of WP and EC formulations and finally two trials were performed by aerial broadcast application of a GR formulation. Samples were stored for up to 528 days from sampling to analysis. An additional trial was established with two plots treated twice at exaggerated application rates of 28.0 kg ai/ha and 56.0 kg ai/ha to produce samples for processing.

Peanuts were sampled at 37–54 DALA, separated into nutmeat and hulls, and analysed for quintozene, PCA, PCTA, PB, and HCB using the method CAM-24-73 with an LOQ of 0.005 mg/kg for each analyte. Samples were stored for 443–535 days from sampling to analysis while the longest duration tested for storage stability for peanut was 14 months. For cotton seed, other type of high oil content matrix, however, the longest duration tested was 18 months during which quintozene, PCA, PCTA, PB and HCB were stable during this period. Perhaps the results with storage stability study with cotton seed may be extrapolated to peanut. In many of these trials, residues were detected above the LOQ in untreated samples (see in Tables 83 and 84).

In the 1988 trials, quintozene was applied as one single application at nominally 11.2 kg ai/ha. Five trials were performed by banded application of GR and WP formulations and four trials by sprinkler application of WP and EC formulations. Peanuts, whole nuts, were sampled at 45 days and analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB using the method MP-PCNC-MA1 with an LOQ of 0.01 mg/kg for each analyte. Samples were stored for 31–70 days from sampling to analysis, shorter than the proved duration of storage stability.

In the 1990 trials reported in 900-RES-111, quintozene was applied as a split overhead sprinkler application at 4.5 and 6.7 kg ai/ha totalling to nominally 11.3 kg ai/ha at intervals of 26–27 days. Peanuts were sampled at 45 DALA, separated into nutmeat and hulls, and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.001 mg/kg for each analyte. Samples were stored for 401 or 409 days from sampling to analysis.

In the 1990 trials reported in 900-RES-109, quintozene was applied by banded application split equally to 2× 5.6 kg ai/ha or in the range of 3.4–7.9 kg ai/ha at intervals of 28–37 days. Peanuts were sampled at 43–45 DALA, separated into nutmeat and hulls, and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.001 mg/kg for each analyte. Samples were stored for 387 or 388 days from sampling to analysis.

In the 1991 trials, quintozene was applied by chemigation split equally to 2x 5.6 kg ai/ha at intervals of 14–30 days. Peanuts were sampled at 45–47 DALA, separated into nutmeat and hulls, and analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.001 mg/kg for each analyte. Samples were stored for 64–91 days from sampling to analysis.

In the 1991/1992 trials of study 900-RES-023, quintozene was applied as one single application at nominally 11.2 kg ai/ha. Four trials were performed by banded application of GR and WP formulations. In one trial an additional plot was established utilizing a sprinkler application of an EC formulation at 11.2 kg ai/ha. Peanuts were sampled at 44–50 DAT, separated into nutmeat and hulls, and analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5,6-TCNB and HCB using the method MP-PCNC-MA1 with an LOQ of 0.005 mg/kg for each analyte. Samples were stored for 93–182 days from sampling to analysis.

Table 83 Magnitude of residues of quintozene in/on peanut after banded applications at pegging in the supervised trials conducted in the United States in 1987, 1988, 1990, 1991 and 1991/92

Peanut Location, Year (Variety)	Application			DALA	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
No GAP related to the trials.										
Troy, AL, United States, 1987 (Florunner)	GR 100 g/kg	5.60 (banded) Pegging	2	54	Nutmeat	0.152	0.102	0.042	0.307	UN1421 WSM-87-012 900-RES-078
					Hulls	0.382	0.177	0.082	0.661	
	WP 750 g/kg	3.36 / 7.85 (banded) Pegging	2	54	Nutmeat	0.025	0.032	0.008	0.069	
					Hulls	0.074	0.036	0.019	0.133	
	Untreated				Nutmeat	0.030	0.049	0.020	-	Only >LOQ
					Hulls	0.094	0.083	0.038	-	
Mrianna, FL, United States, 1987 (Florunner)	GR 100 g/kg	5.60 (banded) Early peg.	2	40	Nutmeat	0.085	0.076	0.023	0.192	UN1421 KHG-87-(145-156) 900-RES-078
					Hulls	0.401	0.211	0.090	0.725	
	WP 750 g/kg	5.60 (banded) Early peg.	2	40	Nutmeat	0.062	0.044	0.011	0.120	
					Hulls	0.232	0.105	0.046	0.393	
	Untreated				Nutmeat	0.006	-	-	-	Only >LOQ
					Hulls	0.035	-	-	-	
Brinson, GA, United States, 1987 (Florunner)	GR 100 g/kg	5.60 (banded) Early peg.	2	44	Nutmeat	0.066	0.047	0.010	0.127	UN1421 KHG-87 (133-144) 900-RES-078
					Hulls	0.466	0.239	0.092	0.824	
	WP 750 g/kg	5.60 (banded) Early peg.	2	44	Nutmeat	0.042	0.072	0.024	0.145	
					Hulls	0.067	0.121	0.040	0.241	
Hawkinsville, GA,	GR	5.60	2	45	Nutmeat	0.061	0.025	0.012	0.100	UN1421

Peanut Location, Year (Variety)	Application			DALA	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
No GAP related to the trials.										
United States, 1987 (GK 7)	100 g/kg	(banded) Pegging			Hulls	1.44	0.108	0.042	1.606	KHG-87 (101-112) 900-RES-078
	WP 750 g/kg	7.85 / 3.36 (banded) Pegging	2	45	Nutmeat	0.074	0.033	0.015	0.125	
					Hulls	0.489	0.073	0.030	0.600	
Washington, NC, United States, 1987 (Florigiant)	GR 100 g/kg	5.60 (banded) Pegging	2	37	Nutmeat	0.008	0.015	<0.005	0.029	UN1421 ABR-87 (6-17) 900-RES-078
					Hulls	0.062	0.023	0.010	0.098	
	WP 750 g/kg	7.85 / 3.36 (broadcast) Pegging	2	37	Nutmeat	0.005	0.008	<0.005	0.019	
					Hulls	0.012	<0.005	<0.005	0.022	
	Untreated				Hulls	0.022	-	-	-	Only >LOQ
Holland, VA, United States, 1987 (VA81B)	GR 100 g/kg	5.60 (banded) Peg. Pod develop.	2	45	Nutmeat	0.106	0.057	0.059	0.227	UN1421 PP-87 (18-29) 900-RES-078
					Hulls	0.189	0.106	0.093	0.399	
	WP 750 g/kg	7.85 / 3.36 (banded) Peg. Pod develop.	2	45	Nutmeat	0.016	0.016	0.005	0.038	
					Hulls	0.026	0.015	0.015	0.056	
	Untreated				Nutmeat	0.005	0.005	0.005	-	Only >LOQ
					Hulls	0.008	-	-	-	
Kingston, OK, United States, 1987 [c] (Florunner)	GR 100 g/kg	5.60 (banded) n/r	2	42	Nutmeat	0.213	0.133	0.048	0.408	UN1421 PRN-87-009 900-RES-078
					Hulls	0.540	0.271	0.160	1.000	
	WP 750 g/kg	7.85 / 3.36 (banded) n/r	2	42	Nutmeat	0.216	0.038	<0.005	0.263	
					Hulls	0.506	0.111	0.019	0.648	
	Untreated				Nutmeat	0.043	0.036	0.008	-	Only >LOQ
					Hulls	0.222	0.040	-	-	
Kingston, OK, United States, 1987 [c] (Florunner)	GR 100 g/kg	5.60 (banded) n/r	2	42	Nutmeat	0.198	0.126	0.048	0.385	UN1421 PRN-87-010 900-RES-078
					Hulls	0.509	0.193	0.085	0.807	
	WP 750 g/kg	7.85 / 3.36 (banded) n/r	2	42	Nutmeat	0.201	0.027	<0.005	0.235	
					Hulls	0.380	0.079	0.016	0.483	
	Untreated				Nutmeat	0.116	0.056	0.008	-	Only >LOQ
					Hulls	0.404	0.088	0.004	-	
Eastman, GA, United States, 1988 (Florunner)	WP 750 g/kg	11.2 (banded) Late peg.	1	45	Whole nuts	0.016	<0.01	<0.01	0.037	PAL-PB-PT PB-PT-2545 900-RES-022
					Nutmeat [b]	0.003	n/a	n/a	0.016	
	GR 100 g/kg	11.2 (banded) Late peg.	1	45	Whole nuts	0.243	0.038	0.015	0.300	
					Nutmeat [b]	0.049	n/a	n/a	0.132	
Biship, GA, United States, 1988 (Florunner)	WP 750 g/kg	11.2 (banded) Late peg.	1	45	Whole nuts	0.206	0.097	0.069	0.383	PAL-PB-PT PB-PT-2544 900-RES-022
					Nutmeat [b]	0.041	n/a	n/a	0.168	
	GR 100 g/kg	11.2 (banded) Late peg.	1	45	Whole nuts	0.330	0.095	0.059	0.494	
					Nutmeat [b]	0.066	n/a	n/a	0.217	
Battleboro, NC, United States,	WP 750 g/kg	8.41 (banded)	1	45	Whole nuts	0.133	0.014	0.011	0.160	PAL-PB-PT PB-PT-2543

Peanut Location, Year (Variety)	Application			DALA	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	Max kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
No GAP related to the trials.										
1988 (Fiorigiant)		End prod. pegging			Nutmeat [b]	0.027	n/a	n/a	0.070	900-RES-022
	GR 100 g/kg	11.4 (banded) End prod. pegging	1	45	Whole nuts	0.152	0.015	0.012	0.181	
					Nutmeat [b]	0.030	n/a	n/a	0.080	
Eakly, OK, United States, 1988 (Spanish)	WP 750 g/kg	11.2 (banded) Pegging	1	45	Whole nuts	0.062	0.036	<0.01	0.113	PAL-PB-PT PB-PT-2558 900-RES-022
					Nutmeat [b]	0.012	n/a	n/a	0.050	
	GR 100 g/kg	11.2 (banded) Pegging	1	45	Whole nuts	1.22	0.168	0.063	1.47	
					Nutmeat [b]	0.243	n/a	n/a	0.645	
Brookshire, TX, United States, 1988 (Spanish)	WP 750 g/kg	13.0 (banded) Pegging	1	45	Whole nuts	0.012	0.050	<0.01	0.078	PAL-PB-PT PB-PT-2542 900-RES-022
					Nutmeat [b]	0.002	n/a	n/a	0.034	
	GR 100 g/kg	13.1 (banded) Pegging	1	45	Whole nuts	0.377	0.052	0.047	0.482	
					Nutmeat [b]	0.075	n/a	n/a	0.212	
Brookshire, TX, United States, 1990 (Spanish)	GR 100 g/kg	5.60 (banded) Pegging, pod full	2	43	Nutmeat	0.050	0.023	0.017	0.092	RP-90033 CRA-90-087 900-RES-109
					Hulls	0.894	0.178	0.197	1.29	
	WP 750 g/kg	7.85 / 3.36 (banded) Pegging, Pod full	2	43	Nutmeat	0.005	0.011	0.004	0.022	
					Hulls	0.041	0.027	0.037	0.107	
Alfalfa, OK, United States, 1990 (Spanco)	GR 100 g/kg	5.60 (banded) Late peg. Pod full	2	45	Nutmeat	0.026	0.030	0.006	0.064	RP-90033 CRA-90-088 900-RES-109
					Hulls	0.074	0.028	0.007	0.111	
	WP 750 g/kg	7.85 / 3.36 (banded) Late peg. Begin to mature	2	45	Nutmeat	0.002	0.009	0.001	0.013	
					Hulls	0.006	0.017	<0.001	0.026	
Caddo County, OK, United States, 1991 (Spanco)	GR 100 g/kg	11.4 (banded) Pegging	1	46	Whole nut	1.16	0.096	0.049	1.31	HWI 6274-109 6012-360 900-RES-023
					Hulls	2.14	0.139	0.090	2.38	
					Nutmeat	0.143	0.024	0.007	0.177	
Pulaski County, GA, United States, 1991 (Florunner)	GR 100 g/kg	11.2 (banded) Pegging	1	45	Whole nut	0.824	0.131	0.046	1.01	HWI 6274-109 6012-361 900-RES-023
					Hulls	3.20	0.496	0.172	3.92	
					Nutmeat	0.123	0.049	0.021	0.198	
Waller County, TX, United States, 1991 (Spanish)	WP 750 g/kg	11.3 (banded) Pegging	1	44	Whole nut	0.023	0.014	<0.01	0.049	HWI 6274-109 6012-362 900-RES-023
					Hulls	0.037	0.012	0.006	0.056	
					Nutmeat	0.005	0.013	<0.01	0.030	
	GR 100 g/kg	11.3 (banded) Pegging	1	51	Whole nut	0.485	0.290	0.149	0.956	
					Hulls	0.950	0.311	0.170	1.47	
					Nutmeat	0.211	0.180	0.136	0.546	
Camilla, GA, United States, 1991 (Florunner)	WP 750 g/kg	11.2 (banded) Pegging	1	50	Whole nut	0.176	0.048	0.017	0.247	HWI 6274-109 6012-381 900-RES-023
					Hulls	0.555	0.083	0.041	0.688	
					Nutmeat	0.030	0.036	0.009	0.078	

Notes:

[a] Mean of the analytical results of two samples.

[b] Calculated from "whole nuts" using the respective processing factor for peeling.

[c] The application dates and harvest dates are the same between these two trials and therefore they are not regarded independent from each other.

Table 84 Magnitude of residues of quintozene in/on peanuts after sprinkler, aerial broadcast or chemigation applications in the supervised trials conducted in the United States in 1987, 1988, 1990 and 1991

Peanut Location, Year (Variety)	Application			DALA or DAT	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
No corresponding GAP										
Hawkinsville, GA, United States, 1987 (Florunner)	EC 240 g/L	6.73 / 4.48 (sprinkler) Pegging	2	45	Nutmeat	0.482	0.288	0.212	1.01	UN1421 KHG-87 (93-100) 900-RES-078
					Hulls	1.21	0.641	0.277	2.203	
Pulaski county, GA, United States, 1987 (Florunner)	EC 240 g/L	6.73 / 4.48 (sprinkler) Pegging	2	45	Nutmeat	0.100	0.086	0.075	0.269	UN1421 KHG-87 (113-120) 900-RES-078
					Hulls	0.258	0.144	0.090	0.507	
Prague, OK, United States, 1987 (Spanish)	WP 750 g/kg	5.60 / 4.20 (sprinkler) n/r	2	45	Nutmeat	0.244	0.085	0.019	0.357	UN1421 K-87 (172-178) 900-RES-078
					Hulls	0.662	0.079	0.014	0.763	
	Untreated				Nutmeat	0.019	0.016	-	-	Only >LOQ
					Hulls	0.102	0.016	-	-	
Kingston, OK, United States, 1987 (Florunner)	WP 750 g/kg	6.73 / 4.20 (sprinkler) n/r	2	44	Nutmeat	0.082	0.076	0.010	0.175	UN1421 K-87 (160-170) 900-RES-078
					Hulls	0.354	0.131	0.032	0.531	
	EC 240 g/L	6.73 / 4.48 (sprinkler) n/r	2	43	Nutmeat	0.234	0.124	0.027	0.399	
					Hulls	1.14	0.435	0.143	1.770	
	Untreated				Nutmeat	0.267	0.089	0.015	-	Only >LOQ
					Hulls	0.303	0.160	0.032	-	
Comanche county, TX, United States, 1987 [b] (Runner)	WP 750 g/kg	5.60 (sprinkler) n/r	2	45	Nutmeat	0.078	0.284	0.082	0.475	UN1421 PRN-87-013 900-RES-078
					Hulls	0.057	0.118	0.056	0.243	
	EC 240 g/L	6.73 / 4.48 (sprinkler) n/r	2	45	Nutmeat	0.104	0.101	0.095	0.310	
					Hulls	0.073	0.068	0.059	0.206	
	Untreated				Nutmeat	0.019	0.050	0.030	-	Only >LOQ
					Hulls	0.028	0.036	0.034	-	
Troy, AL, United States, 1987 (Florunner)	EC 240 g/L	6.73 / 4.48 (sprinkler) Pegging	2	47	Nutmeat	0.025	0.052	0.015	0.097	UN1421 WSM-87-012 900-RES-078
					Hulls	0.089	0.106	0.041	0.247	
	Untreated				Nutmeat	0.029	0,051	0.021	-	Only >LOQ
					Hulls	0.105	0.100	0.042	-	
Comanche county, TX, United States, 1987 [b] (Spanish)	GR 100 g/kg	5.60 (aerial broadcast) Pegging	2	45	Nutmeat	0.021	0.012	0.005	0.038	UN1421 PRN-87-014 900-RES-078
					Hulls	0.108	0.026	0.019	0.155	
	Untreated				Nutmeat	0.015	0.012	-	-	Only >LOQ
					Hulls	0.147	0.036	0.019	-	
Comanche county,	GR	5.60	2	45	Nutmeat	0.018	0.018	0.005	0.042	UN1421

Peanut Location, Year (Variety)	Application			DALA or DAT	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
No corresponding GAP										
TX, United States, 1987 [b] (Runner)	100 g/kg	(aerial broadcast) Pegging			Hulls	0.059	0.025	0.021	0.107	PRN-87-015 900-RES-078
	Untreated				Nutmeat	0.024	0.014	0.005	-	Only >LOQ
					Hulls	0.166	0.046	0.025	-	
Eastman, GA, United States, 1988 (Florunner)	EC 240 g/L	11.2 (sprinkler) Late pegging	1	45	Whole nuts	1.97	0.204	0.117	2.31	PAL-PB-PT PB-PT-2549 900-RES-022
					Nutmeat [b]	0.394	n/a	n/a	1.02	
Howkinsville, GA, United States, 1988 (Florunner)	EC 240 g/L	11.2 (sprinkler) Late pegging	1	45	Whole nuts	1.79	0.379	0.316	2.53	PAL-PB-PT PB-PT-2548 900-RES-022
					Nutmeat [b]	0.358	n/a	n/a	1.11	
Eakly, OK, United States, 1988 (Spanco)	WP 750 g/kg	11.2 (sprinkler) Pegging	1	45	Whole nuts	0.528	0.099	0.047	0.685	PAL-PB-PT PB-PT-2546 900-RES-022
					Nutmeat [b]	0.106	n/a	n/a	0.301	
Brookshire, TX, United States, 1988 (Spanish)	WP 750 g/kg	11.2 (sprinkler) Pegging	1	45	Whole nuts	0.094	0.056	0.029	0.184	PAL-PB-PT PB-PT-2547 900-RES-022
					Nutmeat [b]	0.019	n/a	n/a	0.081	
	EC 240 g/L	11.2 (sprinkler) Pegging	1	45	Whole nuts	0.610	0.272	0.153	1.06	
					Nutmeat [b]	0.122	n/a	n/a	0.468	
Eakley, OK, United States, 1990 (Spanco)	WP 750 g/kg	6.73 / 4.48 (sprinkler) Late peg. Full seed	2	45	Nutmeat	0.007	0.016	0.004	0.029	RP-90032 CRA-90-085 900-RES-111
					Hulls	0.024	0.030	0.009	0.065	
Alfalfa, OK, United States, 1990 (Spanco)	WP 750 g/kg	6.73 / 4.48 (sprinkler) Late peg. Full seed	2	45	Nutmeat	0.005	0.020	0.002	0.030	RP-90032 CRA-90-086 900-RES-111
					Hulls	0.018	0.030	0.004	0.056	
Hawkinsville, GA, United States, 1991 (Florunner)	FL 480 g/kg	5.60 (chemigation) Pegging	2	45	Nutmeat	0.043	0.040	0.011	0.098	RP-91022 KHG-91-065 900-RES-113
					Hulls	0.053	0.046	0.021	0.125	
	EC 240 g/kg	5.60 (chemigation) Pegging	2	45	Nutmeat	0.115	0.157	0.063	0.352	
					Hulls	0.116	0.117	0.066	0.312	
Eakley, OK, United States, 1991 (Spanco)	FL 480 g/kg	5.60 (chemigation) R5/R7	2	45	Nutmeat	0.011	0.013	0.001	0.026	RP-91022 CRA-91-079 900-RES-113
					Hulls	0.070	0.043	0.006	0.124	
	EC 240 g/kg	5.60 (chemigation) R5/R7	2	45	Nutmeat	0.041	0.051	0.006	0.104	
					Hulls	0.341	0.273	0.033	0.678	
Brookshire, TX, United States, 1991 (Spanco)	FL 480 g/kg	5.60 (chemigation) Pegging	2	47	Nutmeat	0.063	0.081	0.049	0.203	RP-91022 CRA-91-080 900-RES-113
					Hulls	0.096	0.126	0.082	0.318	
	EC 240 g/kg	5.60 (chemigation) Pegging	2	47	Nutmeat	0.149	0.162	0.116	0.445	
					Hulls	0.285	0.269	0.167	0.751	
Waller County, TX, United States,	EC 240 g/kg	11.2 (sprinkler)	1	44	Whole nut	0.236	0.154	0.120	0.526	HWI 6274-109 6012-362
					Hulls	0.360	0.179	0.147	0.706	

Peanut Location, Year (Variety)	Application			DALA or DAT	Portion analysed	Residues (mg/kg) [a]				Study, Trial, Reference
	Form. (g ai/L)	kg ai/ ha	No.			Quintozene	PCA	PCTA	Total	
No corresponding GAP										
1991 (Spanish)		Pegging			Nutmeat	0.115	0.121	0.112	0.361	900-RES-023

Notes:

[a] The application dates and harvest dates are the same in these trials. Trial 013 used sprinkler application while 014 and 015 used aerial broadcast application.

[b] Calculated from "whole nuts" using the respective processing factor for peeling.

n/a, Not analysed.

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

The Meeting received information on the ratio of residues in peel and pulp of potato, and hulls and nutmeat of peanut, as well as on processing of tomato, green beans, potato, cotton seed and peanut.

Potato–Residues in Peel and pulp

Study 1. *(Ball, 1900, 900-RES-144)*

The effect of peeling on residue concentrations of quintozene in potato was investigated using two trials in the United States in 1988: one in Hollandale, MN and the other in Northwood, ND. Quintozene in EC formulation was applied as either in-furrow or broadcast application at 28 kg ai/ha at planting. The potato samples to be processed were collected at maturity 122–123 DAT. Potato (RAC) and all processed commodities were stored frozen for up to 4.3 months (132 days). The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 (modified) with an LOQ of 0.0005 mg/kg for each analyte.

The potatoes were processed to wet peel by abrasion, yielding about 10 percent weight of peel. Samples of the peeled potatoes and unpeeled potatoes were frozen for analysis. The wet peel samples were divided, and half were dried to about 13 percent moisture content. The wet peel and dry peel were frozen for analysis. The analytical results and calculated processing factors are summarized in the following table.

Table 85 Effect of peeling of potato on quintozene, PCA and PCTA concentrations (broadcast application at planting

Location, Year (Variety)	Application Rate Method DAT	Commodity	Residues (mg/kg)				Processing factor	
			Quintozene	PCA	PCTA	Total [a]	Quintozene	Total [a]
Hollandale, MN, United States, 1988 (Red Pontiac)	28 kg ai/ha In-furrow 123 days	Potato (RAC)	0.133	0.020	0.031	0.186	--	--
		Wet peel	1.010	0.066	0.121	1.204	7.6	6.5
		Dried peel	2.120	0.273	0.202	2.625	16	14
		Peeled potato	0.014	0.008	0.009	0.031	0.10	0.17
Northwood, ND, United States, 1988 (Russet Burbank)	28 kg ai/ha Broadcast 122 days	Potato (RAC)	0.012	0.008	0.010	0.032	--	--
		Wet peel	0.067	0.021	0.023	0.114	5.6	3.6
		Dried peel	0.168	0.091	0.051	0.319	14	10
		Peeled potato	0.002	0.003	0.003	0.008	0.14	0.24

Notes:

RAC: Raw agricultural commodity

[a] Sum of quintozene, PCA and PCTA expressed as quintozene

***Study 2**. (Zheng, 1992, 900-RES-206)*

The effect of peeling on residue concentrations of quintozene in potato was investigated in two trials in Bethany, IL in the United States. Quintozene in the EC formulation was applied as broadcast at a rate 140 kg ai/ha at planting. The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 (modified) with an LOQ of 0.001 mg/kg for each analyte. Detailed field trial and processing data are not available.

Table 86.Effect of peeling of potato on quintozene, PCA and PCTA concentrations (broadcast application at planting).

Location, Year (Variety)	Application Rate Method	Matrix	Residues (mg/kg) [a]				Processing Factor	
			Quintozene	PCA	PCTA	Total [b]	Quintozene	Total [b]
Bethany, IL, United States, Year unknown (variety unknown) Trial 1	140 kg ai/ha Broadcast	Potato (RAC)	0.045	0.122	0.023	0.203	--	--
		Peeled potato	0.003	0.055	0.003	0.067	0.06	0.33
		Wet peels	0.257	0.259	0.093	0.638	5.8	3.1
		Dry peels	3.32	3.90	1.33	8.99	75	44
Bethany, IL, United States, Year unknown (variety unknown) Trial 2	140 kg ai/ha Broadcast	Potato (RAC)	0.039	0.160	0.023	0.240	--	--
		Peeled potato	0.005	0.045	0.005	0.060	0.10	0.29
		Wet peels	0.465	0.525	0.197	1.25	10	6.1
		Dry peels	2.87	3.52	1.31	8.10	65	40

Notes:

RAC: Raw agricultural commodity

[a] Mean of three replicates

[b] Sum of quintozene, PCA and PCTA expressed as quintozene

Peanut–Residues in hulls and nutmeat (Stenner et al., 1992, 900-RES-023)

Using the samples obtained in the trials reported in 900-RES-023 (also Table 83 and Table 84), analysis of peanuts as whole nuts and separately as hulls and nutmeat was conducted as part of the residue trials.

Table 87 Residues of quintozene, PCA and PCTA in whole nut, hulls and nutmeat of peanuts (banded or sprinkler application at pegging)

Location, Year (Variety)	Application Rate Method DAT	Commodity	Residues (mg/kg) [a]				Processing Factor	
			Quintozene	PCA	PCTA	Total [b]	Quintozene	Total [b)]
Caddo County, OK, United States, 1991 (Spanco)	11.4 kg ai/ha Banded 46 days	Whole nut	1.16	0.096	0.049	1.31	--	--
		Hulls	2.14	0.139	0.090	2.38	1.9	1.8
		Nutmeat	0.143	0.024	0.007	0.177	0.12	0.14
Pulaski County, GA, United States, 1991 (Florunner)	11.2 kg ai/ha Banded 45 days	Whole nut	0.824	0.131	0.046	1.01	--	--
		Hulls	3.20	0.496	0.172	3.92	3.9	3.9
		Nutmeat	0.123	0.049	0.021	0.198	0.15	0.20
Waller County,	11.3 kg ai/ha	Whole nut	0.023	0.014	<0.01	0.049	--	--

Location, Year (Variety)	Application Rate Method DAT	Commodity	Residues (mg/kg) [a]				Processing Factor	
			Quintozene	PCA	PCTA	Total [b]	Quintozene	Total [b)]
TX, United States, 1991 (Spanish)	Banded 44 days	Hulls	0.037	0.012	0.006	0.056	1.6	1.2
		Nutmeat	0.005	0.013	<0.01	0.030	0.23	0.62
	11.3 kg ai/ha Banded 51 days	Whole nut	0.485	0.290	0.149	0.956	--	--
		Hulls	0.950	0.311	0.170	1.47	2.0	1.5
		Nutmeat	0.211	0.180	0.136	0.546	0.43	0.57
	11.2 kg ai/ha Sprinkler 44 days	Whole nut	0.236	0.154	0.120	0.526	--	--
		Hulls	0.360	0.179	0.147	0.706	1.5	1.3
		Nutmeat	0.115	0.121	0.112	0.361	0.49	0.69
Camilla, GA, United States, 1991 (Florunner)	11.2 kg ai/ha Banded 50 days	Whole nut	0.176	0.048	0.017	0.247	--	--
		Hulls	0.555	0.083	0.041	0.688	3.2	2.8
		Nutmeat	0.030	0.036	0.009	0.078	0.17	0.32

Notes:

[a] Mean of the two samples.

[b] Sum of quintozene, PCA and PCTA expressed as quintozene.

Residues in processed commodities

Tomato

Study 1. *(Ball, 1990, 900-RES-126 and 900-RES 084 (addendum))*

Two trials were performed in CA, United States (Madera and Fresno) during the growing season 1987. One in-furrow application of a 750 g/kg WP formulation of quintozene at a rate of 42 kg ai/ha was performed at the time of planting. The tomato samples to be processed were sampled at maturity 113 and 111 DAT.

The tomato samples were processed to canned tomato, juice, puree, paste, ketchup, as well as to the by-products, wet and dry pomace, as follows. Initially tomatoes were washed with chlorinated water to remove dirt, debris, and rotten fruit. A second chlorinated water wash preceded the final rinse with potable water.

Tomato Juice (separation of pomace (skin and seeds)): The tomatoes were fed through a disintegrator equipped with a 4 mm screen and then pumped into a plate heat exchanger for heating. The temperature of the tomato juice was raised to 107 °C and held for approximately 40 seconds. The hot tomato juice was then fed into a finisher for removal of skins and seeds (pomace). The finisher is fitted with a 0.8 mm screen for this separation. The hot juice was then collected for use in the following products.

Canned Tomato Juice: An aliquot of the hot juice was filled into cans, sealed and heated in boiling water for 10 minutes to ensure sterility.

Canned Tomato: The best red tomatoes were selected from each lot for use as whole canned tomatoes. The tomatoes were blanched in boiling water prior to removal of the skins, then received a light rinse with potable water before being packed into cans. Juice from the corresponding lots were used as cover juice. The cans were then vacuum sealed and cooked for 30 minutes in a rotary cooker before cooling.

Tomato Concentrate: The hot tomato juice from each lot was concentrated in a single pass wiped surface evaporator to the requested brix degree (about 30°). The concentrate was then filled into cans, sealed, and heated in boiling water for 30 minutes before cooling.

Sauces: Sauces were formulated to a refractive index of not less than 1.3461 and a Bostwick of not more than 14 cm in 30 seconds of 20 °C.

Ketchup: Ketchup was made to 33 percent solids and a Bostwick of 9 cm in 30 seconds at 20 °C.

Pomace: The wet pomace from juicing was weighed and divided into two equal lots. Half of the pomace was immediately frozen (wet pomace); the remaining pomace was dried in a hot air dehydrator at a temperature of 66 °C. The dry pomace was packaged to prevent rehydration.

Tomato (RAC) and all processed commodities were stored frozen up to 4 months prior to analysis. The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.05 mg/kg for each analyte. As a follow-up, selected samples were re-analysed with a modified version of method CAM-24-73 with an LOQ of 0.002 mg/kg for each analyte. However, since samples were re-analysed after a storage of up to 456 days for which storage stability could not be confirmed, the data from reanalysis were not considered for calculation of the processing factors.

The analytical results (4 values each) of quintozene, PCA and PCTA in tomato (RAC), washed tomato, canned tomato, tomato juice, tomato puree, tomato paste, and ketchup were all below the LOQ of 0.05 mg/kg. Quintozene was found above LOQ only in wet pomace (0.061–0.079 mg/kg) and dry pomace (0.097–0.186 mg/kg); and PCA only in dry pomace (0.063–0.098 mg/kg).

Study 2. *(LeRoy & Cassidy, 1991; 900-RES-035)*

Two separate field trials in CA (Madera and Fresno), United States, were performed during the growing season 1988. Tomatoes (var. UC-82) were treated with one 750 g/kg WP in-furrow application at transplanting at 8.4 or 84.1 kg ai/ha. Tomatoes were sampled at normal commercial harvest 113–114 days DAT.

Samples from the treatment at 84.1 kg ai/ha were used for processing to tomato juice, puree, ketchup, and wet and dry pomace in similar methods as in Study 1.

The samples were analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5, 6-TCNB and HCB using the method MP-PCNC-MA1 with an LOQ of 0.005 mg/kg for each analyte.

Processing factors are calculated for quintozene only and for the sum of quintozene, PCA and PCTA, expressed as quintozene in the following table. Significant concentrations of quintozene, PCA and PCTA were found in untreated wet pomace and dry pomace samples.

Table 88 Residues of quintozene, PCA and PCTA in tomato and its processed commodities following one in-furrow application at transplanting

Location, Year (Variety) DAT	Application Rate Method	Commodity	Residues (mg/kg) [a]				Processing factor	
			Quintozene	PCA	PCTA	Total [b]	Quintozene	Total [b]
Madera, CA, United States, 1988 (UC-82) DAT = 114	84.1 kg ai/ha	Tomato (RAC)	0.029	0.005	<0.005	0.040	--	--
		Puree	0.008	0.005	<0.005	0.019	0.28	0.48
		Ketchup	<0.005	<0.005	<0.005	<0.016	<0.17	<0.40
		Juice	<0.005	<0.005	<0.005	<0.016	<0.17	<0.40
		Wet pomace [c]	0.411	0.172	0.133	0.733	14	18
		Dry pomace [d]	0.959	0.442	0.336	1.79	33	45

Location, Year (Variety) DAT	Application Rate Method	Commodity	Residues (mg/kg) [a]				Processing factor	
			Quintozene	PCA	PCTA	Total [b]	Quintozene	Total [b]
Fresno, CA, United States, 1988 (UC-82) DAT = 113	84.1 kg ai/ha	Tomato (RAC)	0.046	0.008	<0.005	0.060	--	--
		Puree	0.037	0.013	<0.005	0.056	0.80	0. 93
		Ketchup	0.016	0.008	<0.005	0.030	0.35	0.50
		Juice	0.020	<0.005	<0.005	0.030	0.43	0.50
		Wet pomace [e]	1.960	0.324	0.051	2.37	43	40
		Dry pomace [f]	3.300	0.626	0.115	4.11	72	69

Notes:

[a] Mean values of 2-3 determinations.

[b] Sum of quintozene, PCA and PCTA expressed as quintozene.

[c] Residues in the untreated sample: quintozene, 0.121; PCA, 0.174; and PCTA, 0.075 mg/kg).

[d] Residues in the untreated sample: quintozene, 0.008; PCA, 0.340; and PCTA, 0.208 mg/kg).

[e] Residues in the untreated sample: quintozene, 0.006 mg/kg).

[f] Residues in the untreated sample: quintozene, 0.022; PCA, 0.0068 mg/kg).

Study 3. *(Brunk, 1994; 900-RES-218)*

Effect of processing on residues of quintozene in tomato and processed fractions was investigated in one trial in the United States in 1991 (no information on in-life part of the trial in the study report). The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.0005 mg/kg for each analyte. Most of untreated samples were found to contain no PB, HCB, quintozene, PCA or PCTA above the LOQ. However, the untreated dry pomace showed trace levels of quintozene and PCA near the LOQ level and the untreated wet pomace contained quintozene at a trace level.

Table 89 Residues of quintozene, PCA and PCTA in tomato and its processed commodities following one in-furrow application at transplanting

Commodity	Residues (mg/kg) [a]				Processing factor	
	Quintozene	PCA	PCTA	Total [b]	Quintozene	Total [b]
Tomato (RAC)	0.007	0.002	0.001	0.011	--	--
Juice	<0.0005	0.002	<0.0005	0.003	<0.07	0.26
Puree	0.001	0.004	0.001	0.007	0.18	0.65
Ketchup	<0.0005	0.002	<0.0005	0.003	<0.07	0.30
Wet pomace	0.124	0.057	0.027	0.214	17	19
Dry pomace	0.079	0.022	0.014	0.116	11	10

Notes:

[a] Mean values of 2 samples analysed

[b] Sum of quintozene, PCA and PCTA, expressed as quintozene

Green beans *(LeRoy & Cassidy, 1991, 900-RES-031)*

Information on the processing of green beans to canned beans was provided utilizing different application rates in two locations. However, where RAC was analysed, processed commodities were not analysed, vice

versa. Therefore, it was not possible to use the results for calculating the processing factors, and therefore the data are not presented here.

Potato

Study 1. *(Ball, 1987, 900-RES-198)*

Potatoes were selected from a commercial lot of potatoes which had been treated with quintozene EC formulation (containing 24 percent quintozene and 0.1 percent HCB), applied broadcast at the rate of 26.9 kg ai/ha. Potatoes were grown, harvested and stored under commercial conditions. Approximately 9–11 kg of each of two varieties of treated potatoes, Russet Burbank and Norgold Russet, and an untreated sample of Russet Burbank were shipped to the processing facility for processing into French fried potatoes, potato chips (crisps) and dried flakes as follows.

French fries: Unpeeled potatoes were washed, cut into slices, and immersed in water. The slices were washed with water two times to remove excess starch and sugar, drained and divided into two lots. One batch was frozen, uncooked. The other batch was cooked in corn oil for 2.5 min. at 185 °C. After cooling, the cooked fries were packaged.

Potato chips (potato crisps): Unpeeled potatoes were washed, sliced 1.3 mm thick and immersed in water. The twice washed slices were divided into two lots. One batch was frozen, uncooked. The other batch was fried in corn oil for 2.0 minutes at 185 °C. After cooling, the chips were packaged.

Dried flakes: Potatoes were peeled and immersed in water. The washed tubers were drained and cut into slices. The twice washed potato slices were drained for a short period of time, and a portion of the slices were frozen, uncooked. The remainder of the slices were cooked in water at 78 °C to a slurry consistency. The slurry was dried at 38 °C to a final moisture content of 5–6 percent and packaged.

The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.002 mg/kg in raw potatoes and dried potato flakes and 0.01 mg/kg in fried potato products.

Processing factors were calculated for quintozene only and for the sum of quintozene, PCA and PCTA, expressed as quintozene in the following table.

Table 90 Residues of quintozene, PCA and PCTA in commercially available potato and its processed commodities following broadcast application.

Location, Year (Variety)	Application Rate Method	Commodity	Residues (mg/kg) [a]				Processing factor	
			Quintozene	PCA	PCTA	Total [b]	Quintozene	total [b]
WA, United States, 1986/87 (Kennebeck)	26.9 kg ai/ha Broadcast	Potato (RAC)	0.014	0.011	0.030	0.055	--	--
		French fries (uncooked)	0.025	0.013	0.028	0.067	1.8	1.2
		French fries	0.026	0.012	0.038	0.077	1.9	1.4
		Crisps (uncooked)	0.015	0.040	0.019	0.078	1.1	1.4
		Crisps	0.027	0.013	0.031	0.072	1.9	1.3
		Uncooked cubes	0.002	0.004	0.008	0.014	0.14	0.25
		Dried flakes	0.002	0.003	0.006	0.011	0.14	0.20
Grand Forks, ND, United States, 1987	26.9 kg ai/ha Broadcast	Potato (RAC)	0.011	0.010	0.028	0.050	--	--
		French fries (uncooked)	0.038	0.015	0.036	0.091	3.5	1.8
		French fries	0.031	0.018	0.040	0.091	2.8	1.8

Location, Year (Variety)	Application Rate Method	Commodity	Residues (mg/kg) [a]				Processing factor	
			Quintozene	PCA	PCTA	Total [b]	Quintozene	total [b]
(Norchip)		Crisps (uncooked)	0.014	0.015	0.023	0.054	1.3	1.1
		Crisps	0.020	0.010	0.021	0.052	1.8	1.0
		Uncooked cubes	0.010	0.004	0.007	0.021	0.91	0.42
		Dried flakes	0.002	0.004	0.006	0.012	0.18	0.24

Notes:

[a] Mean of three replicates

[b] Sum of quintozene, PCA and PCTA, expressed as quintozene

Study 2. *(Ball, 1987, 900-RES-056)*

Two field trials were performed in CA, United States (Madera and Fresno) during the growing season 1987. Two formulations, the EC and GR formulations were selected for this potato processing study: EC formulation was applied broadcast at 140 and 280 kg ai/ha and GR formulation was applied in-furrow at 56.0 and 112 kg ai/ha at planting. The potato samples to be processed were sampled at maturity 139 or 133 DAT. Potato (RAC) and all processed commodities were stored frozen approximately one month prior to analysis.

Potato samples were processed into chips (crisps), flakes and granules as follows.

Potato chips (potato crisps): A difference from the descriptions in Study 1 is that, a portion of washed slices were fried in corn oil for 2.5–2.75 minutes at 185 °C to a final moisture content of approximately 2 percent.

Flakes and granules: Potato samples were lightly peeled in a washer/peeler for 45 seconds. The potatoes came into contact with a moving, abrasive stone which is constantly flushed with cold, running water. The amount of peel removed is proportionate to the length of contact time with the abrasive surface. In this study, peeling time was relatively short, which removed surface soil and grit with a minimal amount of peel removal. The washed, peeled potatoes were sliced to a thickness of 1.3 mm. The slices were washed and steam cooked. The cooked potato slices were immediately pulped and screened over a 2.4 mm screen recovering a smooth, uniform slurry which was utilized for the product of both flakes and granules. A portion of the slurry was dried in a thin layer on stainless steel at a temperature of 49 °C to a final moisture content of 5-6 percent. The dried flakes were packaged. Another portion of the slurry was homogenized at room temperature to a uniform consistency by being passed twice through a homogenizer. The finely divided slurry was pumped into a mixed flow spray drier, with a pressurized air feed at an inlet temperature of 150°C. The product rose in the chamber and the particles remained suspended until dry enough to drop into the collector. The final moisture content was approximately 4-6 percent. The dried granules were packaged.

The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 (modified) with an LOQ of 0.002 mg/kg for each analyte.

Processing factors were calculated for quintozene only and for the sum of quintozene, PCA and PCTA, expressed as quintozene in the following table.

Table 91 Residues of quintozene, PCA and PCTA in potato and its processed commodities following broadcast or in-furrow application at planting

Location, Year	Application	Commodity	Residues (mg/kg) [a]	Processing factor

(Variety) DAT	Rate Form.-Method		Quintozene	PCA	PCTA	Total [b]	Quintozene	Total [b]
Bethany, IL, United States, 1987 (Kennebeck) DAT: 139	140 kg ai/ha EC-Broadcast	Potato (RAC)	0.113	0.133	0.059	0.319	--	--
		Crisps (uncooked)	0.106	0.107	0.050	0.275	0.94	0.86
		Crisps (cooked)	0.147	0.024	0.094	0.267	1.3	0.84
		Slurry	0.002	0.030	0.004	0.040	0.02	0.12
		Flakes	0.008	0.073	0.015	0.104	0.07	0.33
		Granules	<0.002	0.047	0.007	0.061	<0.02	0.19
	280 kg ai/ha EC-Broadcast	Potato (RAC)	0.252	0.163	0.084	0.518	--	--
		Crisps (uncooked)	0.463	0.224	0.157	0.868	1.8	1.7
		Crisps (cooked)	0.435	0.053	0.156	0.650	1.7	1.3
		Slurry	0.006	0.054	0.012	0.077	0.02	0.15
		Flakes	0.022	0.125	0.039	0.200	0.09	0.39
		Granules	0.006	0.090	0.018	0.124	0.02	0.24
	56.0 kg ai/ha G - In-furrow	Potato (RAC)	0.085	0.077	0.040	0.210	--	--
		Crisps (uncooked)	0.080	0.037	0.024	0.145	0.95	0.69
		Crisps (cooked)	0.068	0.012	0.030	0.111	0.80	0.53
		Slurry	0.003	0.017	0.003	0.025	0.04	0.12
		Flakes	0.009	0.042	0.010	0.065	0.10	0.31
		Granules	0.004	0.041	0.007	0.056	0.04	0.27
	112 kg ai/ha G -In-furrow	Potato (RAC)	0.117	0.061	0.033	0.217	--	--
		Crisps (uncooked)	0.061	0.030	0.015	0.110	0.52	0.50
		Crisps (cooked)	0.116	0.026	0.048	0.192	0.99	0.88
		Slurry	<0.002	0.125	0.020	0.161	<0.02	0.74
		Flakes	0.005	0.038	0.005	0.052	0.04	0.24
		Granules	<0.002	0.013	<0.002	0.018	<0.02	0.08
Grand Forks, ND, United States, 1987 (Norchip) DAT: 133	140 kg ai/ha EC-Broadcast)	Potato (RAC)	0.606	0.064	0.047	0.723	--	--
		Crisps (uncooked)	0.624	0.053	0.042	0.724	1.0	1.0
		Crisps (cooked)	0.141	0.009	0.021	0.172	0.23	0.24
		Slurry	0.019	0.015	0.007	0.042	0.03	0.06
		Flakes	0.025	0.012	0.009	0.047	0.04	0.06
		Granules	0.002	0.018	0.002	0.025	0.00	0.03
	280 kg ai/ha EC-Broadcast)	Potato (RAC)	0.990	0.070	0.054	1.12	--	--
		Crisps (uncooked)	0.722	0.031	0.025	0.782	0.73	0.70
		Crisps (cooked)	0.127	0.011	0.028	0.167	0.13	0.15
		Slurry	0.016	0.009	0.005	0.031	0.02	0.03
		Flakes	0.024	0.015	0.007	0.047	0.02	0.04
		Granules	0.003	0.013	<0.002	0.019	0.00	0.02
	56.0 kg ai/ha G-In-furrow	Potato (RAC)	0.086	0.002	0.014	0.102	--	--
		Crisps (uncooked)	0.123	0.017	0.012	0.153	1.4	1.5
		Crisps (cooked)	0.040	0.005	0.008	0.054	0.47	0.53
		Slurry	0.003	0.005	<0.002	0.010	0.03	0.10
		Flakes	0.005	0.005	0.003	0.013	0.06	0.13
		Granules	<0.002	0.013	<0.002	0.019	<0.02	0.18
	112 kg ai/ha G-In-furrow	Potato (RAC)	0.283	0.047	0.025	0.360	--	--
		Crisps (uncooked)	0.329	0.029	0.020	0.381	1.2	1.1

Location, Year (Variety) DAT	Application Rate Form.-Method	Commodity	Residues (mg/kg) [a]				Processing factor	
			Quintozene	PCA	PCTA	Total [b]	Quintozene	Total [b]
		Crisps (cooked)	0.046	0.002	0.009	0.058	0.16	0.16
		Slurry	0.009	0.011	0.003	0.024	0.03	0.07
		Flakes	0.016	0.009	0.006	0.032	0.06	0.09
		Granules	0.002	0.014	0.002	0.020	0.01	0.06

Notes:

[a] Mean of three replicates.

[b] Sum of quintozene, PCA and PCTA, expressed as quintozene.

Study 3. *(LeRoy & Cassidy, 1991, 900-RES-034)*

Two field trials, one in Minidoka, ID and the other in Ephrata, WA, United States, were performed during the growing season of 1988. Potatoes (var. Russet Burbank) were treated with quintozene EC formulation in-furrow at planting at 56.0 kg ai/ha in Minidoka, and at 13.5 kg ai/ha in Ephrata. Potatoes were sampled at normal commercial harvest for processing as follows. Processed commodities were frozen immediately after processing.

The tubers were stored refrigerated at approximately 7 °C until processing. Prior to processing, the tubers were washed, destoned, and inspected.

Potato chips (potato crisps), wet peel and dried peel: One batch of tubers was sent through an abrasive peeler, then sliced and fried to produce chips. This process generated both wet and dried peels. A second batch of tubers was sent through a steam peeler to produce wet peels. The resulting peels were then dried.

Potato flakes and granules: The peeled tubers were precooked for 20 minutes then divided into two batches. One batch was used to prepare flakes, and the remaining batch was used to prepare granules.

The samples were analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5, 6-TCNB and HCB using the method MP-PCNC-MA1 with an LOQ of 0.005 mg/kg for each analyte.

Processing factors were calculated for quintozene only and for the sum of quintozene, PCA and PCTA, expressed as quintozene in the following table.

Table 92 Residues of quintozene, PCA and PCTA in potato and its processed commodities following in-furrow application at planting

Location, Year (Variety)	Application Rate (kg ai/ha)	Commodity	Residues (mg/kg)				Processing factor	
			Quintozene	PCA	PCTA	Total [a]	Quintozene	Total [a]
Minidoka,ID, United States, 1988 (Russet Burbank)	13.5	Potatoes (RAC)	0.422	0.093	0.045	0.570	--	--
	56.0	Potatoes (RAC)	0.244	0.014	0.006	0.265	--	--
		Granules	<0.005	<0.005	<0.005	<0.016	<0.02	<0.06
		Flakes	<0.005	0.005	<0.005	0.016	<0.02	0.06
		Crisps	0.006	0.007	0.007	0.020	0.02	0.08
		Dried Peel (flakes)	6.01	0.967	0.678	7.76	25	29
		Dried Peel (chips)	3.28	0.604	0.386	4.34	13	16
		Wet Peel (flakes)	3.14	0.321	0.204	3.70	13	14

Location, Year (Variety)	Application Rate (kg ai/ha)	Commodity	Residues (mg/kg)				Processing factor	
			Quintozene	PCA	PCTA	Total [a]	Quintozene	Total [a]
		Wet Peel (chips)	2.68	0.261	0.175	3.14	11	12
Ephrata,WA, United States, 1988 (Russet Burbank)	56.0	Potatoes (RAC)	0.137	0.203	0.081	0.444	--	--
		Granules	<0.005	<0.005	<0.005	<0.016	<0.04	<0.04
		Flakes	<0.005	0.029	0.006	0.043	<0.04	0.10
		Crisps	0.007	0.022	0.005	0.037	0.05	0.08
		Dried Peel (flakes)	1.76	3.39	1.81	7.34	13	17
		Dried Peel (chips)	0.320	0.462	0.137	0.971	2.3	2.2
		Wet Peel (flakes)	0.272	0.444	0.201	0.966	2.0	2.2
		Wet Peel (chips)	0.455	0.572	0.238	1.33	3.3	3.0

Notes:
[a] Sum of quintozene, PCA and PCTA expressed as quintozene

Cotton seed

Study 1. *(Ball, 1989, 900-RES-068)*

In one field trial performed in Stoneville, MS, United States during the growing season 1988, one in-furrow application of a GR formulation at a rate of 11.2 kg ai/ha was performed at the time of planting. The cotton seed samples to be processed were collected at maturity 149 days after application.

The effect of processing on residues in cotton seed was investigated. The cotton seed samples were processed to solvent-extracted crude and refined oil and meal, as well as to the by-products hulls and linter motes.

Separation of seed, lint and gin trash: A gin was used to separate these fractions.

Delinted seed, linters and motes: A saw delinter was used to remove the majority of the lint from ginned cottonseed. The fractions are linters, motes and delinted seed. The seed has small amounts of lint adhering to each end of the seed.

Hull and kernel fractions of seed: A bar huller was used to decorticate cottonseed. Either high lint cottonseed (directly from the gin) or delinted cottonseed can be decorticated. The cracked seed was passed across a shaker screen, which separates hull and kernel fractions. An aspirator was utilized if more complete separation was required.

Cotton seed meal (hexane extraction): Kernels were preheated to 74 °C and flaked by flaking rolls to 0.2–0.3 mm thickness. Flaked kernels were then placed in a steam-jacketed, stainless steel, batch extractor. Hexane (at approximately 63 °C) was added until the flake bed was flooded. After 30 minutes, the solvent was drained, and fresh solvent added for 5 additional cycles (3 hours total extraction time). At the end of this period, warm air was forced through the flake bed for approximately 4 hours for removal of the solvent.

Crude Cotton Oil (hexane extraction): The miscella (crude oil and hexane mixture) was separated in an evaporator. During this procedure, the crude oil reaches a temperature of 85 °C.

Refined oil and soapstock (following AOCS Method Ca9a52): Prior to refining, the percent free fatty acid was determined in the crude oil. A weighed oil sample was placed in a laboratory oil refining machine. A weighed amount of sodium hydroxide (14° Baume) was added to the crude oil, as calculated on the basis of the amount of free fatty acids present. The solution was mixed at 250 rpm (20–24 °C) for

15 minutes, followed by 70 rpm for 12 minutes (63–67 °C). After a settling period (1 hour; 60–65 °C), the oil was refrigerated for at least 12 hours. At the end of this period, the refined oil was decanted and filtered. The soapstock fraction was settling to the bottom of the refrigerated container.

Cotton seed (RAC) and all processed commodities were stored frozen up to 7 months prior to analysis. The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.005 mg/kg for each analyte.

Processing factors were calculated for quintozene only and for the sum of quintozene, PCA and PCTA, expressed as quintozene in the following table.

Table 93 Residues of quintozene, PCA and PCTA in cotton seed and its processed commodities following in-furrow application at planting

Location, Year (Variety)	Application Rate (kg ai/ha)	Commodity	Residues (mg/kg)				Processing factor	
			Quintozene	PCA	PCTA	Total [a]	Quintozene	Total [a]
Stoneville, MS, United States, 1987 (DES 119)	11.2	Cotton seed (RAC)	0.005	<0.005	<0.005	0.016	--	--
		Crude oil	0.024	0.006	<0.005	0.036	4.8	2.3
		Refined oil	0.022	0.006	<0.005	0.034	4.4	2.2
		Soapstock	<0.005	0.006	<0.005	0.017	<1	1.1
		Linters	<0.005	<0.005	<0.005	<0.016	<1	<1
		Linter motes	0.047	<0.005	<0.005	0.058	9.4	3.7
		Meal (solvent extracted)	<0.005	<0.005	<0.005	<0.016	<1	<1
		Hulls	<0.005	<0.005	<0.005	<0.016	<1	<1
		Reclaimed solvent	<0.005	<0.005	<0.005	<0.016	<1	<1

Notes:

[a] Sum of quintozene, PCA and PCTA, expressed as quintozene

Study 2. *(LeRoy R.L., Cassidy J.E., 1991, 900-RES-032)*

Two separate field trials, one in Kerman, CA, and the other in Brookshire, TX, United States, were performed during the growing season 1988/89. Cotton (var. SJ-1 and DPL-50) were treated with quintozene EC formulation in-furrow at planting at different rates: 2.24, 5.60 and 11.2 kg ai/ha in the Brookshire trials and at 6.73 and 11.2 kg ai/ha in the Kerman trials.

Cotton balls were sampled at normal commercial harvest at 139–153 DAT. Samples from the highest treatment scheme (11.2 kg ai/ha) were used for processing as follows (see Study 1 above).

The seed cotton was first ginned which produced cottonseed, gin trash, and lint cotton. Gin trash was not collected in one trial. The cottonseed was delinted into three fractions: linters, delinted seed, and linter motes. The delinted seed was hulled and separated into kernels and hulls. The kernels were extracted with hexane into crude oil and meal. The crude oil was then refined and soapstock was generated.

The refined oil was bleached and the bleached oil was hydrogenated. The hydrogenated oil was deodorized for the final processing step. All samples generated were stored frozen until analysis.

The samples were analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5, 6-TCNB and HCB using the method MP-PCNC-MA1 and MP-PCNC-MA2 with an LOQ of 0.005 mg/kg for each analyte.

Quintozene, PCA, or PCTA was not detected above the LOQ in the RAC (Kerman trial only) or any processed commodities in these trials. These compounds were not analysed in the RAC sample from the Brookshire trial. It was not possible to calculate processing factors for these process commodities.

Study 3. *(Gaydosh & Smudin, 1996, 900-RES-114)*

In one field trial in Hernando, MS, United States, during the growing season 1993, one in-furrow application of a GR formulation at a rate of 6.7 kg ai/ha was performed at the time of planting. The cotton ball samples to be processed were collected at maturity 156 DAT.

Samples of cotton balls were processed into linters, linter motes, delinted seed, hulls, kernels, meal, crude oil, refined oil, and soapstock as follows.

Delinted cotton seed and lint: The cottonseed was delinted to remove a majority of the existing lint.

Hull and kernel: The delinted seed was mechanically cracked and screened to separate the majority of the hull material from the kernel material.

Crude oil (hexane extraction): The kernel material with some hull material was heated, flaked, expanded into collets and exposed to hexane for the purpose of removing crude oil from the collets. The solvent was removed from the spent collets with warm air.

Refined oil and soapstock: After the crude oil and hexane mixture was adjusted to the proper ratio, the crude oil was miscella refined. The free fatty acid is determined and a known amount of sodium hydroxide was added to the miscella and the mixture stirred by heating. After reaction, the soapstock was removed. The refined oil and hexane were separated with an evaporator under vacuum at elevated temperature.

Cotton seed (RAC) and all processed commodities were stored frozen up to 5.3 months (162 days) prior to analysis. The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.0005 mg/kg for each analyte.

Analysis resulted in quintozene, PCA and PCTA below the LOQ, except that in soapstock PCA was found at 0.0064 mg/kg. Therefore, no processing factors were calculated.

Study 4. *(Maselli, 1997, 900-RES-147)*

Two separate field trials, one in Tunika, MS, and the other in East Bernard, TX, United States, were conducted during the growing season 1995. Cotton (var. Delta Pine 50) were treated with quintozene EC formulation in-furrow at planting at three different rates. Cotton balls harvested at maturity from the treatment at 7.2 kg ai/ha were used for processing.

Samples of cotton balls were processed into linters, gin-trash, delinted seed, hulls, kernels, crude oil, refined oil, and soapstock, in a similar method as in previous studies.

Cotton seed (RAC) and all processed commodities were stored frozen up to 14 months (427 days) prior to analysis. The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 (modified) with an LOQ of 0.002–0.005 mg/kg for cotton seeds and 0.02–0.05 mg/kg for gin-trash.

Results were presented for quintozene in cotton seed and gin-trash only. They were below the LOQ, except in one sample of gin trash the residue was 0.062 mg/kg. Therefore, no processing factors could be calculated.

Peanuts

Study.1. *(Ball, 1990, 900-RES-078)*

One trial was conducted in Tifton, GA, United States, during the growing season 1989. Peanut plants (variety Florunner) were treated with split applications of quintozene WP formulation at either 2 × 28.0 kg ai/ha or 2 × 56.0 kg ai/ha. The first application was made at pegging and the second application about 30 days later. The whole peanuts were dug and inverted by hand 45 DALA. After drying, whole peanuts from each treatment were collected, and shipped to a processing facility, where the peanut samples were placed in frozen storage. Approximately, one week after receipt, the whole peanuts were hulled, and a week later processed. Small-scale peanut processing equipment was used to process the peanuts.

Hull and kernel (nutmeat): If the unshelled peanut (whole peanut) sample was high in moisture, it was placed in a forced air oven for 30 minutes or longer at 66 °C. The peanut sample was cleaned by aspiration and/or screening. A peanut sheller was used to dehull the peanuts. Hull and kernel fractions were separated by aspiration.

Pressed oil from expeller: After determining the percent moisture of peanut kernels, tap water was added to adjust the moisture to 10 percent. The peanuts were cooked for approximately 45 minutes or until the temperature of the peanuts reached approximately 99 °C. At the end of the cooking period, the oil was mechanically removed with an expeller.

Extracted oil with hexane and presscake (meal): The residual oil in the presscake was extracted with hexane in a steam-jacketed, stainless steel, hatch extractor. Hexane was added until the presscake was flooded. The temperature of the hexane was raised to approximately 63 °C. After 30 minutes, the solvent was drained and fresh solvent was added to repeat the cycle 6 times (3 hours). At the end of this period, the extracted sample was drained, and warm air was forced through the presscake for approximately four hours to remove residual solvent.

Crude Oil: The miscella (crude oil and hexane) was separated in an evaporator. During this procedure, the crude oil reached a temperature of 85 °C. The crude oil accounted for approximately 50 percent of the whole peanut by weight.

Refined oil and soapstock (following AOCS Method Ca9a52) and refined and deodorized oil: After the percent free fatty acid was determined in the crude oil from the expeller (using AOCS Method CaSa-40), a weighed sample was placed in a laboratory oil refining machine. A weighed amount of 16° Baume sodium hydroxide was added to the crude oil as calculated on the basis of free fatty acids present. The solution was mixed for 30 minutes at 250 rpm at 20–24 °C, and then for additional 12 minutes at 70 rpm at 63–67 °C. The neutralized oil was allowed to settle at 60–65 °C for 1 hour. The oil solution was then refrigerated overnight (minimum of 12 hours); The neutralized oil was decanted and filtered. The fraction settling to the bottom of the refrigerated container was the soapstock.

A weighed refined oil sample as obtained above was heated in a steam bath for 1 hour under an absolute vacuum of 533 Pa. The temperature of the oil was kept between 220–230 °C. At the end of the 1-hour period, the oil sample was allowed to cool to 150 °C. Citric acid solution (0.005 percent) was added to the oil (at a rate of 1 mL/100 g-oil). The sample was allowed to cool, under vacuum, to 110 °C. The cooled oil sample was immediately transferred to a shipping container.

The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.005 mg/kg for each analyte.

Processing factors were calculated from the residue levels in kernel for quintozene only and for the sum of quintozene, PCA and PCTA, expressed as quintozene in the following table.

Table 94 Residues of quintozene, PCA and PCTA in peanut nutmeat and its processed commodities following two applications at begging

Location, Year (Variety)	Application Rate (kg ai/ha)	Commodity	Residues (mg/kg)				Processing factor	
			Quintozene	PCA	PCTA	Total [a]	Quintozene	Total [a]
Tifton, GA, United States, 1987 (Florunner)	2 × 28	Nutmeat	0.523	0.190	0.143	0.877	--	--
		Hulls	1.48	0.271	0.223	2.008	--	--
		Crude oil	1.42	0.626	0.400	2.519	2.7	2.9
		Crude oil expeller	1.18	0.432	0.282	1.942	2.3	2.2
		Refined oil	1.19	0.449	0.295	1.985	2.3	2.3
		Deodorized oil	0.006	0.013	0.011	0.031	0.01	0.04
		Presscake expeller	0.104	0.051	0.030	0.191	0.20	0.22
		Presscake extracted (meal)	0.047	0.025	0.012	0.087	0.09	0.10
		Soapstock	0.398	0.187	0.111	0.717	0.76	0.82
		Reclaimed solvent	<0.005	<0.005	<0.005	<0.016	<0.01	<0.02
	2 × 56	Nutmeat	1.02	0.382	0.310	1.753	--	--
		Hulls	4.85	0.723	0.468	6.124	--	--
		Crude oil	2.86	1.18	0.796	4.968	2.8	2.8
		Crude oil expeller	2.38	0.880	0.664	4.020	2.3	2.3
		Refined oil	2.30	0.875	0.648	3.918	2.3	2.2
		Deodorized oil	<0.005	0.008	0.009	0.023	0.005	0.01
		Presscake expeller	0.271	0.112	0.084	0.479	0.27	0.27
		Presscake extracted (meal)	0.097	0.043	0.025	0.170	0.10	0.10
		Soapstock	0.142	0.299	0.114	0.588	0.14	0.34
		Reclaimed solvent	<0.005	<0.005	<0.005	<0.016	<0.005	<0.01

Notes:

[a] Sum of quintozene, PCA and PCTA expressed as quintozene.

Study 2.*(LeRoy & Cassidy, 1991, 900-RES-033)*

Two separate processing trials were conducted, one in Brookshire, TX, and the other in Battleboro, NC, United States, during the 1988 growing season. Peanut plants (var. Pronto Spanish and Florigiant) were treated with quintozene WP formulation as a banded application at pegging stage at 11.2, 22.4 and 56.0 kg ai/ha in the Battleboro trial and at 11.2, 22.4 and 40.4 kg ai/ha in the Brookshire trial. Whole peanuts were harvested at normal commercial harvest 45 days after application. Samples from the trials with 40.4 and 56.0 kg ai/ha were used for processing.

The whole peanut samples (nut in shell) were kept frozen until separated into hulls and kernels, then the kernels were expelled into press-cake, crude oil, hexane-extracted press-cake, hexane-extracted

crude oil, refined oil, soapstock, dry roasted peanuts, oil roasted peanuts, deodorized oil, and hydrogenated oil. A RAC (nut in shell) sample was subsampled for analysis.

All samples generated were stored frozen until analysis. The samples were analysed for quintozene, PCA, PCTA, PB, 2,3,4,5-TCNB, 2,3,5, 6-TCNB and HCB using the method MP-PCNC-MA1 with an LOQ of 0.005 mg/kg for each analyte.

Processing factors were calculated from the residue levels in kernel for quintozene only and for the sum of quintozene, PCA and PCTA, expressed as quintozene in the following table.

Table 95 Residues of quintozene, PCA and PCTA in peanut nutmeat and its processed commodities following banded application at begging

Location, Year (Variety)	Application Rate (kg ai/ha)	Commodity	Residues (mg/kg)				Processing factor	
			Quintozene	PCA	PCTA	Total [a]	Quintozene	Total [a]
Brookshire, TX, United States, 1988 (Pronto Spanish)	11.2	Whole nuts (RAC)	0.014	0.008	<0.005	0.027	--	--
	40.4	Whole nuts (RAC)	0.475	0.151	0.108	0.751	--	--
		Hulls	1.170	0.299	0.237	1.74	--	--
		Kernel	0.144	0.135	0.113	0.407	--	--
		Presscake	<0.005	<0.005	<0.005	<0.016	<0.03	<0.04
		Crude oil	<0.005	<0.005	<0.005	<0.016	<0.03	<0.04
		Hexane extracted cake (meal)	0.009	<0.005	<0.005	0.019	0.06	0.05
		Hexane extracted oil	<0.005	<0.005	<0.005	<0.016	<0.03	<0.04
		Refined oil	<0.005	<0.005	<0.005	<0.016	<0.03	<0.04
		Soapstock	<0.005	<0.005	<0.005	<0.016	<0.03	<0.04
		Peanuts roasted	0.100	0.155	0.124	0.396	0.69	0.97
		Roasted peanut oil	0.100	0.131	0.114	0.359	0.69	0.88
		Hydrogenated oil	<0.005	<0.005	<0.005	<0.016	<0.03	0.04
		Deodorized oil	<0.005	<0.005	<0.005	<0.016	<0.03	<0.04
Battleboro, NC, United States 1988 (Florigiant)	11.2	Whole nuts (RAC)	0.110	0.013	0.011	0.136	--	--
	56.0	Whole nuts (RAC)	0.381	0.020	0.006	0.409	--	--
		Hulls	0.402	0.021	0.007	0.433	--	--
		Kernel	0.062	0.008	<0.005	0.075	--	--
		Presscake	0.007	<0.005	<0.005	0.017	0.11	0.23
		Crude oil	0.034	0.012	<0.005	0.053	0.55	0.70
		Hexane extracted cake (meal)	<0.005	<0.005	<0.005	<0.016	<0.08	<0.21
		Solvent extracted oil	<0.005	<0.005	<0.005	<0.016	<0.08	<0.21
		Refined oil	<0.005	<0.005	<0.005	<0.016	<0.08	<0.21
		Soapstock	<0.005	<0.005	0.006	0.017	<0.08	0.22
		Peanuts roasted	0.070	0.019	<0.005	0.096	1.1	1.3
		Roasted peanut oil	0.029	0.010	<0.005	0.045	0.48	0.60
		Hydrogenated oil	<0.005	<0.005	<0.005	<0.016	<0.08	<0.21
		Deodorized oil	0.006	<0.005	<0.005	0.017	0.10	0.22

Notes:

[a] Sum of quintozene, PCA and PCTA expressed as quintozene.

Study 3. *(Gaydosh & Smudin, 1996, 900-RES-115)*

In one field trial in Hawkinsville, GA, United States, during the 1992 growing season, two banded applications of quintozene EC formulation at a rate of 112 kg ai/ha was performed at the time of pegging and with an interval of 30 day. Whole peanuts to be processed were sampled at maturity 45 DALA.

The whole peanut samples were dried and then cleaned by aspiration and screening. A sheller was used to mechanically crack the hull surrounding the kernel (nutmeat). Aspiration was used to separate the hull and kernel fractions. The raw peanut kernels were heat conditioned and pressed in an expeller for the purpose of liberating a majority of crude oil. After pressing, the presscake was flaked. The residual crude oil remaining in the solid material (presscake) exiting the flaker was extracted with hexane. The solvent extracted presscake (meal) was desolventized. The crude oil recovered from the expeller and solvent extraction was combined and refined. Soapstock was recovered as a by-product of the refining process.

Peanuts (RAC) and all processed commodities were stored frozen up to 3.5 months (107 days) prior to analysis. The samples were analysed for quintozene, PCA, PCTA, PB and HCB using the method CAM-24-73 with an LOQ of 0.01 mg/kg for each analyte.

Processing factors were calculated from the residue levels in kernel for quintozene only and for the sum of quintozene, PCA and PCTA, expressed as quintozene in the following table.

Table 96 Residues of quintozene, PCA and PCTA in peanut nutmeat and its processed commodities following banded applications at begging

Location, Year (Variety)	Application Rate (kg ai/ha)	Commodity	Residues (mg/kg) [a]				Processing factor	
			Quintozene	PCA	PCTA	Total [b]	Quintozene	Total [b]
Hawkinsville, GA, United States 1992 (Florunner)	2 × 112	Peanut (RAC)	4.23	1.63	0.480	6.52	--	--
		Shells	10.5	1.98	0.380	13.1	--	--
		Nutmeat	1.24	0.984	0.550	2.88	--	--
		Meal (solvent extracted)	0.032	0.010	<0.010	0.053	0.03	0.02
		Soapstock	0.650	1.01	0.332	2.11	0.52	0.73
		Crude oil	1.82	1.73	0.776	4.52	1.5	1.6
		Refined oil	2.59	1.96	1.02	5.79	2.1	2.0

Notes:

[a] Mean of two samples analysed.

[b] Sum of quintozene, PCA and PCTA expressed as quintozene.

The following table summarizes the processing factors calculated from the data provided to the current Meeting.

Commodity	n	Processing factor for quintozene		Processing factor for total residue	
		Individual	Best estimate	Individual	Best estimate
Tomato					
Puree	2	0.18, 0.28	0.23	0.48, 0.65	
Ketchup	2	<0.07, <0.17	0.07	0.30, <0.40	
Juice	2	<0.07, <0.17	0.07	0.26, <0.40	
Wet pomace	2	14, 17	15.5	18, 19	18.5

Commodity	n	Processing factor for quintozene		Processing factor for total residue	
		Individual	Best estimate	Individual	Best estimate
Dry pomace	2	11, 33	33	10, 45	45
Potato		-		-	
Wet peel	8	2.0, 3.3, 5.6, 5.8, 7.6, 10, 11, 13	6.7	2.2, 3.0, 3.1, 3.6, 6.1, 6.5, 12, 14	4.85
Dried peel	8	2.3, 13, 13, 14, 25, 16, 65, 75	19.5	2.2, 10, 14, 16, 17, 29, 40, 44,	16.5
Peeled potato	4	0.06, 0.10, 0.10, 0.14	0.10	0.17, 0.24, 0.29, 0.33	0.265
French fries	2	1.9, 2.8	2.35	1.4, 1.8	1.6
Crisps	10	0.13, 0.16, 0.23, 0.47, 0.80, 0.99, 1.3, 1.7, 1.8, 1.9	0.90	0.15, 0.16, 0.24, 0.53, 0.53, 0.84, 0.88, 1.0, 1.3, 1.3	0.685
Dried flakes	2	0.14, 0.18	0.16	0.20, 0.24	0.22
Flakes	8	0.02, 0.04, 0.04, 0.06, 0.06, 0.07, 0.09, 0.10,	0.06	0.04, 0.06, 0.09, 0.13, 0.24, 0.31, 0.33, 0.39	0.185
Granules	8	0.00, 0.00, 0.01, <0.02, <0.02, <0.02, 0.02, 0.04	0.02	0.02, 0.03,0.06, 0.08, 0.18, 0.19, 0.24, 0.27	0.013
Cotton seed		-		-	
Crude oil	1	4.8	4.8	2.3	2.3
Refined oil	1	4.4	4.4	2.2	2.2
Meal	1	<1	1	<1	1
Linter motes	1	9.4	9.4	3.7	3.7
Peanut (whole nut)					
Hulls	6	1.5, 1.6, 1.9, 2.0, 3.2, 3.9	1.95	1.2, 1.3, 1.5, 1.8, 2.8, 3.9	1.65
Nutmeat	6	0.12, 0.15, 0.17, 0.23, 0.43, 0.49	0.20	0.14, 0.20,0.32, 0.57, 0.62, 0.69	0.445
Crude oil	5	<0.03, <0.03, <0.08, 0.55, 1.5	0.08	<0.04, <0.04, <0.21, 0.70, 1.6	0.21
Refined oil	3	<0.03, <0.08, 2.1	0.08	<0.04, <0.21, 2.9	0.21
Deodorized oil	2	<0.03, 0.10	0.065	<0.04, 0.22	0.13
Meal (presscake)	3	0.03, 0.06, <0.08	0.06	0.02, 0.05, <0.21	0.05
Roasted peanut	2	0.69, 1.1	0.895	0.97, 1.3	1.14
From peanut nutmeat					
Crude oil	4	2.3,2.3, 2.7, 2.8	2.5	2.2, 2.3, 2.8, 2.9	2.55
Refined oil	2	2.3, 2.3	2.3	2.2, 2.3	2.25
Deodorized oil	2	0.005, 0.01	0.0075	0.01, 0.04,	0.025
Meal (presscake)	2	0.09, 0.10	0.95	0.10, 0.10	0.10

Notes:

Total residue: sum of quintozene, PCA and PCTA, expressed as quintozene.

RESIDUES ON ANIMAL PRODUCTS

Livestock Feeding Studies

Dairy Cattle *(Griffith et al., 1969, 900-ANM-055)*

Groups of three lactating cows were fed quintozene (98.2 percent quintozene containing 0.1 percent PB and 1.4 percent HCB as impurities) at nominal levels of 0.1, 1 and 10 ppm in the diet for 12–15 weeks. Samples of the milk were taken on days 0, 1, 7, and then at weekly intervals to day 56. In each group, one cow was sacrificed (time between the last dose and slaughter was not reported) after 12 weeks and two others after 16 weeks. Samples of kidneys, muscle and fat were analysed at slaughter, 16 weeks after the start of feeding. Residues in milk were analysed using the method CAM-1-69. The LOQs of PB, PCA, HCB

and quintozene in milk were 0.001, 0.001, 0.005 and 0.01 mg/kg. PCTA could not be quantified because of interference.

Milk

Throughout the study period, parent quintozene and PB were not found in milk above the LOQ in any of three feeding levels. PCA was quantified in milk only from the 10-ppm feeding level from day 14, but at most 0.008 mg/kg. The impurity HCB was quantified in the medium and high feeding levels and seems to have reached plateau at about 2 weeks with the highest concentration of 0.31 mg/kg at 5 weeks.

Table 97 Residues of quintozene, PCA, PB and HCB in cow milk after oral administration of quintozene (containing (98.2 percent quintozene with 0.1 percent PB and 1.4 percent HCB as impurities)

Day	Mean residues (mg/kg)								
	Feeding level: 0.1 ppm			Feeding level: 1 ppm			Feeding level: 10 ppm		
	Quintozene	PCA	PB	Quintozene	PCA	PB	Quintozene	PCA	PB
0	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001
1	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001
7	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001
14	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001	<0.01	0.006	<0.001
21	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001
28	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001	<0.01	0.005	<0.001
35	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001	<0.01	0.005	<0.001
42	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001	<0.01	0.008	<0.001
49	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001
56	<0.01	<0.005	<0.001	<0.01	<0.005	<0.001	<0.01	0.006	<0.001
	HCB		Total [a]	HCB		Total	HCB		Total
0	<0.001		<0.017	<0.001		<0.017	<0.001		<0.017
1	<0.001		<0.017	<0.001		<0.017	0.002		<0.017
7	<0.001		<0.017	<0.001		<0.017	0.003		<0.017
14	<0.001		<0.017	0.001		<0.017	0.010		0.018
21	<0.001		<0.017	0.001		<0.017	0.008		<0.017
28	<0.001		<0.017	0.002		<0.017	0.012		0.017
35	<0.001		<0.017	0.002		<0.017	0.031		0.017
42	<0.001		<0.017	0.003		<0.017	0.016		0.020
49	<0.001		<0.017	0.001		<0.017	0.012		<0.017
56	<0.001		<0.017	0.003		<0.017	0.015		0.018

Notes:

[a] The total concentration was calculated as the sum of quintozene, PCA and PB, not including HCB which is an impurity. Where the concentration was below the LOQ, the LOQ value was used for calculation. It should be noted that PCTA was not quantified due to interference.

Tissues

Residues in tissues were analysed using the method CAM-1-69. When the concentrations of quintozene, PCA, PB and HCB in tissues were below the respective LOQ, they are shown in the following table with the LOQ values to which "<" is attached. Like for milk, PCTA could not be quantified owing to interference.

Quintozene was not detected in any of tissues at any of three feeding levels, except that quintozene was detected at higher level than the LOD but lower than LOQ in the control fat sample. PCA and impurity HCB were found at much higher levels in fat samples than muscle, kidney or liver. Acid hydrolysis by refluxing for 2 hours, portions of kidney or liver from the 10-ppm feeding level in

concentrated H_2SO_4 increased PCA in these tissues (kidney from 0.043 to 0.120 mg/kg; and liver from ND to 0.023 mg/kg). This implies the presence of PCA conjugates before the acid hydrolysis.

Table 98 Residues of PB, HCB, quintozene and PCA in cow tissues obtained at slaughter after oral administration of quintozene (containing (98.2 percent quintozene with 0.1 percent PB and 1.4 percent HCB as impurity) at 0.1–10 ppm in the diet

Tissue	Highest residues, mg/kg (Mean residue, mg/kg)											
	Feeding level: 0.1 ppm [a]				Feeding level: 1 ppm				Feeding level: 10 ppm			
	PB	HCB	quintozene	PCA	PB	HCB	quintozene	PCA	PB	HCB	quintozene	PCA
Kidney	<0.004	<0.02	<0.05	<0.05	<0.004	0.003 (0.002)	<0.05	<0.05	0.001 (<0.001)	0.059 (0.028)	<0.05	0.11 (0.061)
Liver	<0.003	<0.02	<0.05	<0.05	<0.003	0.005 (0.003)	<0.05	<0.05	<0.001	0.11 (0.039)	<0.05	0.03 (0.01)
Muscle	<0.003	<0.02	<0.05	<0.05	<0.003	0.008 (0.007)	<0.05	<0.05	0.004 (0.002)	0.11 (0.052)	<0.05	0.041 (0.020)
Fat, abdominal	<0.008	0.01	<0.1	0.005	<0.008	0.11 (0.075)	<0.1	0.038 (0.024)	0.004 (0.003)	0.80 (0.76)	<0.1	0.50 (0.34)
Fat, [b] subcutaneous	<0.008	0.01	<0.1	<0.08	<0.008	0.083 (0.063)	<0.1	<0.08	0.004 (0.003)	0.72 (0.66)	<0.1	0.38 (0.28)

Notes:

[a] One cow (sacrificed 12 weeks after the initiation of administration).

[b] Quintozene was found at 0.018 mg/kg in the control cow.

In an additional experiment, a single cow was fed at 1000 mg/kg and slaughtered after one month (time between the last administration and slaughter was not described). PCTA was detected in muscle and fat and quantified.

Table 99 Residues of PB, HCB, quintozene, PCA and PCTA in cow tissues obtained at slaughter after oral administration of quintozene (containing (98.2 percent quintozene, 0.1 percent PB, 1.4 percent HCB) at 1000 ppm in the diet. (one cow)

Tissue	Residue, mg/kg				
	PB	HCB	Quintozene	PCA	PCTA
Kidney	0.005	0.036	<0.05	0.25	<0.08
Liver	0.001	0.093	<0.05	0.029	<0.05
Muscle	0.004	0.095	<0.05	0.089	0.014
Fat, abdominal	0.049	2.32	<0.1	1.24	0.14
Fat, subcutaneous	0.036	1.26	<0.1	1.07	0.075

Laying hens *(Kuchar & Griffith, 1975, 900-RES-055)*

Hens (variety, Comet Red Chicken) were fed with quintozene (containing 98.1 percent quintozene, 0.06 percent PB, 0.2 percent 1,3,4,5-TCNB and 1.5 percent HCB and dissolved in corn oil) at levels of 0.05, 1, 5, 15, 75 and 300 ppm in the diet (commercial chicken feed) for four months. Eggs, collected every day, and fat, meat and liver, collected at slaughter, were analysed for quintozene, HCB, PB, PCA and PCTA, using the CAM-39-75 Method upon receipt or maintained at either -2.8 °C (eggs) or -29 °C (tissues) until analysis.

Residues of PB and HCB plateaued in egg yolks at about three weeks and in fat at about seven weeks, whereas quintozene, PCA and PCTA reached a plateau level in less than a week in egg yolk. All of these compounds were detected at much lower levels in egg white as anticipated from their fat solubility.

Table 100 Residues of PB, HCB, quintozene, PCA and PCTA in eggs and hen tissues obtained at slaughter after oral administration of quintozene at 0.05–300 ppm in the diet

Analyte	Residues, mg/kg, at various feeding levels						
	Control	0.05 ppm	1 ppm	5 ppm	15 ppm	75 ppm	300 ppm
Egg yolk (at equilibrium)							
PB	Highest <0.005	Highest <0.005	Highest <0.005	Mean <0.005 Highest 0.008	Mean 0.011 Highest 0.019	Mean 0.072 Highest 0.174	Mean 0.224 Highest 0.350
HCB	Highest <0.008	Mean <0.008 Highest 0.014	Mean 0.012 Highest 0.018	Mean 0.078 Highest 0.130	Mean 0.359 Highest 0.491	Mean 2.05 Highest 2.99	Mean 8.14 Highest 11.9
quintozene	Highest <0.01	Highest <0.01	Mean <0.01 Highest 0.020	Mean <0.01 Highest 0.023	Mean <0.01 Highest 0.017	Mean 0.019 Highest 0.131	Mean 0.024 Highest 0.141
PCA	Highest <0.01	Highest <0.01	Highest <0.01	Mean <0.01 Highest 0.024	Mean 0.014 Highest 0.034	Mean 0.084 Highest 0.206	Mean 0.174 Highest 0.383
PCTA	Highest <0.01	Highest <0.01	Highest <0.01	Highest <0.01	Highest <0.01	Mean 0.012 Highest 0.025	Mean0.024 Highest 0.045
Egg white (at equilibrium)							
PB	Highest<0.002	<0.002x1	Highest <0.002	<0.002x2	Highest <0.002	Mean <0.002 Highest 0.007	Mean <0.002 Highest 0.007
HCB	Highest<0.005	<0.005x1	Highest <0.005	<0.005x2	Highest <0.005	0.006 0.007 Mean 0.006	Mean 0.013 Highest 0.032
quintozene	Highest<0.01	<0.01x1	Highest <0.01	<0.01x2	Highest <0.01	<0.01x2	Mean <0.01 Highest 0.011
PCA	Highest<0.009	<0.009x1	Highest <0.009	<0.009x2	Highest <0.009	<0.009x2	Mean <0.009 Highest 0.014
PCTA	Highest<0.008	<0.008x1	Highest <0.008	<0.008x2	Highest <0.008	<0.008x2	Highest <0.008
Fat							
PB	<0.006 0.009 Mean <0.006	<0.006x2	0.007 0.010 Mean 0.008	0.026 0.024 Mean 0.025	0.061, 0.061, 0.071, 0.064 Mean 0.064	0.363 0.469 Mean 0.416	1.50, 1.14 Mean 1.32
HCB	0.087 0.049 Mean 0.068	0.052 0.046 Mean 0.049	0.087 0.064 Mean 0.076	0.403 0.349 Mean 0.376	1.22, 1.16, 1.37, 2.24 Mean 1.50	7.62 8.59 Mean 8.11	16.2 23.4 Mean 19.8
quintozene	<0.03x2	<0.03x2	<0.03x3	<0.03x2	0.082, 0.054 0.054, 0.108 Mean 0.075	<0.03x2	1.1 0.624 Mean 0.862
PCA	0.062 0.035 Mean 0.048	0.073 0.040 Mean 0.056	0.059 0.031 Mean 0.045	0.056 0.053 Mean 0.055	0.068, 0.057 0.053, 0.076 Mean 0.064	0.049 0.076 Mean 0.062	0.312 0.274 Mean 0.294
PCTA	<0.02x2	<0.02x2	<0.02x2	<0.02x2	<0.02x3, 0.078 Mean 0.028	<0.02 0.031 Mean 0.022	0.076 0.119 Mean 0.098
Muscle (described as white meat)							
PB	<0.01x3	<0.01x3	<0.01x3	<0.01x3	<0.01x3	<0.01x3	0.027 0.018 0.008 Mean 0.018
HCB	<0.01x3	<0.01x3	<0.01x3	<0.01x3	<0.01x3	0.047 0.047 0.031 Mean 0.042	0.703 0.375 0.293 Mean 0.457
quintozene	<0.04×3	<0.04×3	<0.04×3	<0.04×3	<0.04×3	<0.04×2	<0.04×3
PCA	<0.04×3	<0.04×3	<0.04×3	<0.04×3	<0.04×3	<0.04×2	<0.04×3
PCTA	<0.03x3	<0.03x3	<0.03x3	<0.03x3	<0.03x3	<0.03x2	<0.03x3
Liver							

Analyte	Residues, mg/kg, at various feeding levels						
	Control	0.05 ppm	1 ppm	5 ppm	15 ppm	75 ppm	300 ppm
PB	<0.006x3	<0.006x3	<0.006x3	<0.006x2	0.006 0.006 Mean 0.006	0.039 0.032 0.024 Mean 0.032	0.074 0.237 Mean 0.156
HCB	<0.01x3	<0.01x2, 0.017 Mean <0.01	0.015 0.033 0.018 Mean 0.022	0.076 0.123 Mean 0.10	0.166 0.156 Mean 0.161	1.02 0.748 0.560 Mean 0.776	2.08 6.63 Mean 4.36
quintozene	<0.03x3	<0.03x3	<0.03x3	<0.03x2	<0.03x2	<0.03x3	<0.03x2
PCA	<0.02x3	<0.02x3	<0.02x3	<0.02x2	<0.02x2	<0.02x3	0.033 0.171 Mean 0.102
PCTA	<0.02x3	<0.02x3	<0.02x3	<0.02x2	<0.02x2	<0.02x3	0.024 0.038 Mean 0.031

Notes:

Mean residues were calculated using values between the LOD and LOQ.

APPRAISAL

Quintozene (pentachloronitrobenzene–IUPAC name), is an aromatic fungicide, used as soil fungicide or for seed treatment of various vegetables, cereal grains, and oil seeds.

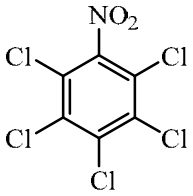
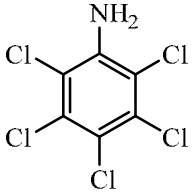
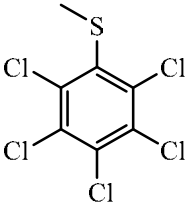
Quintozene was first evaluated by the JMPR in 1969 and reviewed under the CCPR periodic re-evaluation by the 1995 JMPR for toxicology and residues.

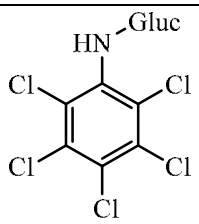
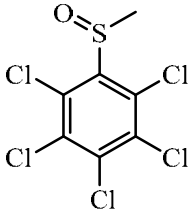
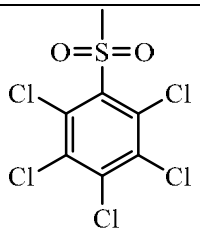
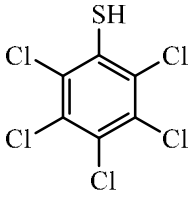
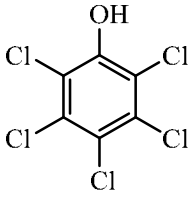
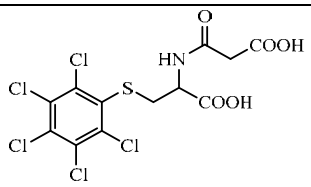
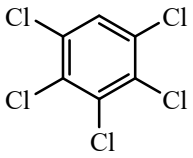
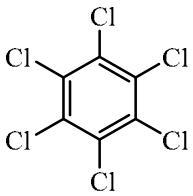
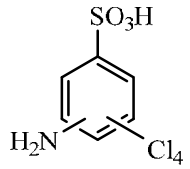
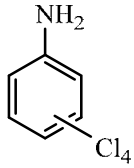
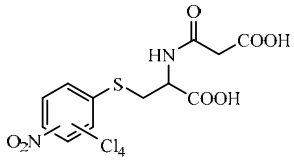
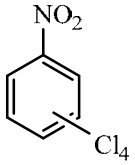
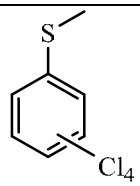
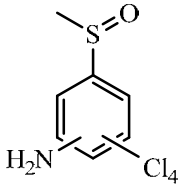
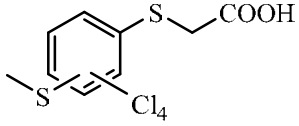
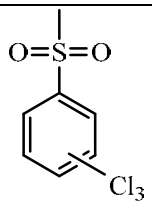
The Firty-third Session of the Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting, including periodic reevaluation of quintozene.

The current Meeting received information on identity; chemical and physical properties; plant, rotational crop, and animal metabolism; environmental fate; residue analytical methods and storage stability; use pattern; supervised trials; processing; and animal feeding.

The following abbreviated names were used for the metabolites commonly found in plants and animals in the appraisal of quintozene.

Table 101 List of some compounds that are the basic structures of metabolites and conjugated metabolites referred to in the appraisal

 Quintozene Pentachloronitrobenzene	 PCA Pentachloroaniline	 PCTA Methyl pentachlorophenyl sulfide

 PCA glucuronide N-(pentachloroaniline) glucuronide	 PCTASO=PCTA-sulfoxide	 PCTASOO=PCTA-sulfone
 PCTP Pentachlorothiophenol	 PCP Pentachlorophenol	 PCP-MayCys or PCPT-MayCys S-(Pentachlorophenyl) malonylcysteine
 PB Pentachlorobenzene	 HCB Hexachlorobenzene a	 AM TCB sulfonic acid Aminotetrachlorobenzene sulfonic acid
 TCA Tetrachloroaniline	 TCNP-MalCys S-(Tetrachloronitrophenyl) malonylcysteine	 TCNB isomers (2,3,4,5-, or 2,3,5,6-) tetrachloronitrobenzene
 TCTA Tetrachlorothioanisole	 TCA sulfoxide Tetrachloroaniline methy sulfoxide	 MTCP-TAA S-[(methylthio)tetrachlorophenyl]-2-thioacetic acid
 C3MS Trichlorophenyl methyl sulfone		

Notes:

[a] Known impurity of quintozene. According to available information, hexachlorobenzene as an impurity is allowed up to 0.03–0.05 percent in TGAI in the countries where quintozene is registered (the 1995 JMPR reported the maximum to be 0.1 percent.).

Based on the information on physical and chemical properties, quintozene is volatile and more soluble in organic solvents than in water with a Log Pow of 5–6 at pH 7 and 25 °C. Quintozene is hydrolytically stable. Aqueous photolysis is likely to be a major degradation pathway of quintozene in the environment. Photodegradation products after irradiation were isomeric mixture of chlorinated hydroxybenzenes and/or chloronitrophenols.

Plant metabolism

The Meeting received information on the fate of quintozene labelled uniformly with ^{14}C in the phenyl ring (hereafter described as ^{14}C-quintozene) in cabbage, potato and peanut after pre-plant soil application. Residues in harvested seeds of maize, peas, sugar beet, wheat and soya bean were investigated after seed application of ^{14}C-quintozene.

Cabbage

When cabbage was grown in soil treated with ^{14}C-quintozene, the highest radioactivity levels of mature cabbage were found in the wrapper (outer) leaves in both of two studies.

In one study, 28-day old cabbage plants were transplanted in soil treated at 53.8 kg ai/ha and grown in a greenhouse. Samples were taken 49, 70 and 154 days after the application. Total radioactive residue (TRR) from combustion were 7.80, 5.24, 1.14 and 13.9 mg eq/kg in immature whole plants (48 DAT and 70 DAT), mature head and mature wrapper leaves. Extraction of wrapper leaves with methanol/water (80:20) and acetone recovered 57.5 and 14.7 percent TRR, totaling 72.2 percent of TRR). Post-extraction solids (PES) were subjected to HCl hydrolysis.

Seven metabolites were identified in the wrapper leaf extracts, among which two major metabolites were tetrachlorophenyl methyl (TCPM) sulfide (36 percent TRR) and sulfone (42 percent TRR). In this study quintozene was not found. Other identified metabolites, ring hydroxylated C3MS, NOHPA, PCTASO and PCTA, each accounted for 0.5–8.9 percent TRR.

In a second study, cabbages was sown immediately after the soil was treated at 33.7 kg ai/ha and grown outdoors. Immature cabbage samples were taken at 120 DAT and mature cabbage samples at 209 DAT. The mature cabbage samples were separated into whole head with wrapper leaves, head without wrapper leaves and wrapper leaves. TRR from combustion were 3.37, 0.28, 0.11 and 2.03 mg eq/kg for immature cabbage, mature whole cabbage, mature head and mature wrapper leaves, respectively. Extraction with hexane followed by methanol recovered 40–70 percent TRR. Samples were extracted 4.7 months and 32 months after harvest, which might affect the metabolite profile. From the cabbage extracts of immature cabbage, mature wrapper leaves and mature whole cabbage, quintozene, 5 metabolites and HCB were identified but the total identified accounted for only 5–18 percent TRR. Quintozene was the most abundant residue accounting for 9.3 percent TRR (0.327 mg/kg) in the immature cabbage and was lower in the mature wrapper leaves. The only metabolites accounting for more than 10 percent TRR were the combined TCNP-MalCys and PCP-MalCys found mostly in methanol extract (12 percent TRR and 0.34 mg/kg in the wrapper leaves and lower in other portions). In this study, quintozene, PCTA, TCNP-MalCys and PCP-MalCys were found in all three matrices. PCA and PB were in the immature cabbage and mature wrapper leaves and 6-TCNB and HCB only in immature cabbage, which accounted for at most 1.1 percent TRR in mature cabbage.

Potato

Metabolism of quintozene in potato grown in soil treated with ^{14}C-quintozene pre-plant incorporation was investigated in three studies.

In the first study, potato plants were grown outdoor in soil treated with ^{14}C-quintozene at 21.1 kg ai/ha. Potatoes were harvested at early maturity, 11 weeks after planting. Subsamples were rinsed with water and separated into peel and flesh. TRR from combustion were 11.26, 0.76 and 2.39 mg eq/kg for peel, flesh and whole potato, respectively. Sum of TRR in the extracts and PES were 16.81, 0.41 and 0.63 mg eq/kg, differing significantly from the TRR values from combustion.

The samples were extracted with 80 percent methanol, and the extracts were partitioned with chloroform, ethyl ether and water, which recovered 50 percent, 78 percent and 95 percent TRR (based on the sum of the radioactivity in the extracts) from peel, flesh and whole potato, respectively.

In the peel, the most predominant residue was quintozene at 24 percent TRR (4.1 mg/kg) followed by PCA at 18 percent TRR (3.1 mg/kg), while in the whole potato tuber, quintozene was below the LOQ. In potato tuber, the most predominant residue was PCTP-MalCys at 32 percent TRR (0.20 mg/kg) followed by PCTP-Cys at 18 percent TRR (0.11 mg/kg) and PCTA at 9.5 percent TRR (0.06 mg/kg).

In the second study, potato plants were grown outdoor in soil treated with 30 kg ai/ha. Samples were collected at 92 DAT and 122 DAT for foliage, 122 DAT (early stage) and 154 DAT (mature stage) for potato tuber. TRR from combustion were 1.13–7.07 mg eq/kg in foliage, 11.25 mg eq/kg in immature tuber, and 1.37 mg eq/kg in mature tuber.

Samples were extracted with hexane and methanol at various timings up to 26 months after harvest, which might affect the metabolite profile. Extraction rates were 53–77 percent for all matrices. PES and extracts were subject to harsh HCl hydrolysis.

In potato foliage, level of identification was low, a total of 11.7 percent of TRR. PCA, PCTA and TCNP-MalCys and PCP-MalCys were found in minor amounts up to 7.5 percent TRR (0.37 mg eq/kg). In potato tubers, quintozene, 7 metabolites and HCB were identified. Parent quintozene was only detected in 122 DAT immature tuber at low levels (0.3–0.5 percent TRR, 0.02–0.06 mg/kg). In the mature tubers, the most abundant residue was PCA at 9.5 percent TRR (0.09 mg/kg) followed by TCNP-MalCys and PCP-MalCys together at 5.2 percent TRR (0.05 mg/kg) and PB at 4.7 percent TRR (0.05 mg/kg). The total identified was 23 percent TRR. In the immature tuber, PCA, PB and the sum of TCNP-MalCys and PCP-MalCys accounted for 6.0–15 percent TRR (0.68–1.08 mg/kg), 2.7–13 percent TRR (0.48–0.60 mg/kg) and 7.0–10 percent TRR (0.32–1.8 mg/kg), respectively with the total identified was 33–38 percent TRR.

The third study was conducted outdoor using the application at 22.4 kg ai/ha or 67.3 kg ai/ha. Samples of tuber, stem and foliage were collected at 95 DAT. However, only tuber samples were subjected to analysis. TRR in the whole tubers were 1.12 mg eq/ha and 3.54 mg eq/kg for the low and high rate, respectively.

A series of extraction (methanol, aqueous methanol and water) and partitioning (hexane and ethyl ether) extracted at least 90 percent TRR. The PES were treated with a variety of enzymes. Protease released about 40 percent of the TRR in PES and much of the radioactivity was associated with substances with molecular weight > 3000. The enzymes that hydrolyze carbohydrate released minimal amount of radioactivity.

Numerous metabolites at low levels were identified in the extracts/fractions. The total of identified residues accounted for 60–64 percent TRR. No single component accounted for higher than 10 percent TRR, except for PCP-MalCys from the high-rate application (10 percent TRR, 0.36 mg/kg). Although numerous metabolites (about 60) were identified at low levels, approximately one half of the identified radioactivity attributed to five components. The most abundant residues were: parent (7.5–8.0 percent TRR) and PCP-MalCys (9.8–10 percent TRR). Three other components were PCA (2.4–4.3 percent

TRR), PCTA (2.7–3.5 percent TRR) and AM TCB sulfonic acid (3.9–4.8 percent TRR). The hexane fractions contained quintozene as the major component and 11 metabolites.

Peanut

In three studies on peanut metabolism, ^{14}C-quintozene was applied as pre-plant soil incorporation treatment. In the third study, ^{14}C-quintozene was also applied twice as band application at around pegging time.

In the first study ^{14}C-quintozene was applied at 420 kg ai/ha (incorporation into top 15 cm of the soil), higher than the rates used in all other metabolism studies. Peanut plants were harvested 21 weeks after the application and separated into roots, vines, shells and nutmeat. TRR were 1520, 42.3, 128 and 5.16 mg eq/kg in roots, vines, shells and nutmeat, respectively.

Aqueous methanol extracted most of radioactive residues from vines, shells and nutmeat (94–100 percent TRR) while from roots, only 67 percent TRR was extracted. In the extracts, a total of three metabolites were identified. In nutmeat, only one metabolite was identified, which was PCP-MalCys at 6.7 percent TRR (0.26 mg/kg). PCP-MalCys was also a predominant metabolite in roots (33 percent TRR, 320 mg/kg), vines (20 percent TRR, 6.9 mg/kg) and shells (42 percent TRR, 47 mg/kg). TCA accounted for 25 percent (240 mg/kg), 14 percent (4.9 mg/kg), and 22 percent (26 mg/kg) of TRR, respectively in roots, vines and shells. In the same matrices, MTCP-TAA accounted for 8.7 percent (85 mg/kg), 14 percent (4.7 mg/kg) and 7.4 percent (3.4 mg/kg) respectively. Acid hydrolysis in methanolic HCl liberated more than 90 percent of radioactivity in the PES of vines, shells and nutmeat but the liberated radioactivity was not subject to identification.

In the second study, ^{14}C-quintozene was applied at 37.9 kg ai/ha (incorporation into the top 7.6 cm of the soil). Immediately after the application, peanut was sown. Foliage samples were collected at 92 and 154 DAT while hulls and nutmeat samples were collected at 185 DAT. TRR were 3.50–3.97 mg eq/kg, 26.3 mg eq/kg and 2.00 mg eq/kg in foliage, hulls and nutmeat, respectively.

Extraction with hexane followed by methanol recovered 87 percent from nutmeat but 50–55 percent TRR from vines and shells.

From nutmeat extracts, a total of 66 percent TRR was identified (quintozene and 7 metabolites in hexane fraction; and quintozene and 3 metabolites in methanol fraction). PCA and PB each accounted for 17 percent TRR (0.36 mg/kg). TCNP-MalCys and PCP-MalCys together accounted for 13 percent TRR (0.27 mg/kg). Parent quintozene accounted for 5.7 percent TRR (0.12 mg/kg) and PCTA 9.5 percent TRR (0.20 mg/kg).

In hulls, 41 percent TRR was identified (quintozene and 7 metabolites, same as in nutmeat). The predominant residues were TCNP-MalCys and PCP-MalCys together accounting for 34 percent TRR (8.3 mg/kg). Other identified metabolites and quintozene each accounted for <4.0 percent TRR.

In vines, 32 percent TRR was identified (quintozene and 6 metabolites). The predominant residues were TCNP-MalCys and PCP-MalCys, together accounting for 27 percent TRR (1.09 mg/kg). Other identified metabolites and quintozene each accounted for <2.7 percent TRR.

HCl hydrolysis of the PES from vines and nutmeat did not release the significant amounts of radioactivity from the PES. HCl hydrolysis of the iso-octane or methanol extract of vines released 60 percent or 42 percent of the radioactivity respectively but released radioactivity was not identified.

In the third study, ^{14}C-quintozene was applied in two treatment regimes: preplant soil incorporation treatment at 16.8 kg ai/ha after which peanut was sown; or two banded applications at 5.6 kg ai/ha, both at pegging time (68 days and 117 days after planting). Peanut was grown in greenhouse.

Mature plants were collected either 193 DAT (preplant application) or 76 DALA (banded applications) and separated into vines (hay), shells, nutmeat and roots.

TRR from the preplant application and banded application were within 3-fold difference without clear indication about which of these two regimes gave rise to higher residues: 13.0–16.3, 1.72–2.14, 166–211, and 49–122 mg eq/kg respectively in hay, nutmeat, shells and roots.

A total of 94–100 percent of TRR was extracted from nutmeat and hay by a series of extraction with methanol/water (20:80) followed by acetone.

After two banded applications at pegging, quintozene in the extracts and after hydrolysis with methanolic HCl accounted for 97 percent TRR with trace amount of PCA in nutmeat.

In hay from the two banded applications, 92 percent TRR was identified. PCTP-MalCys was the predominant residue at 53 percent TRR followed by N-malonyl-S-(tetrachloroaminophenyl)-cysteine at 20 percent TRR and parent quintozene at 19 percent TRR.

Treated seeds

Seeds of maize, peas, sugar beet, wheat and soya bean were treated with ^{14}C-quintozene and sown and grown in an open-sided greenhouse. After harvesting at respective commercial timing, radioactivity was measured in the harvested crops. There was uptake of radioactivity by all the crops with the highest radioactivity found in dry pea vines and soya bean stems at 1.8 and 1.5 mg eq/kg, respectively. While radioactivity above the LOQ was detected in vines, roots, hay, forage, and straw, none of the harvested seeds/grains from these crops contained residues above the minimum quantifiable limits. None of the samples were subjected to identification/ characterization.

Summary of plant metabolism

Metabolism of quintozene in plants after pre-plant soil incorporation application was studied on cabbage, potato and peanut. In the case of peanut, metabolism after two banded applications at pegging was also studied. In old studies on cabbage, potato and peanut, identified compounds were not consistent with newer studies and none of the compounds in the polar fraction was identified; or extraction took place 2 years or later after the harvest.

In the newer studies on potato, numerous compounds (about 60) were identified including those in the polar fractions. About 60–64 percent TRR were identified and approximately about one half of the identified radioactivity (27–30 percent TRR) was comprised of five compounds. PCP-MalCys and its esters together accounted for 10–13 percent TRR. This compound was also found in the old studies on cabbage, potato and peanut. Parent quintozene accounted for 7.5–8.0 percent TRR. PCA and PCTA accounted for 2.4–4.3 percent and 2.7–3.5 percent TRR respectively. AM TCB sulfonic acid accounted for 3.9–4.8 percent TRR. No other metabolites accounted for more than 2.8 percent TRR while the concentrations of many metabolites were higher than 0.01 mg/kg. PB was detected up to 1.9 percent TRR (up to 0.014 mg/kg).

Based on new data, the Meeting confirmed that the metabolism of quintozene in plants occurs through three routes: (1) dechlorination, replacing chlorine by hydrogen or hydroxyl to yield metabolites with less chlorine on the phenyl ring; (2) reduction of the nitro group to NHOH or elimination; or (3) displacement of the nitro group with sulfhydryl group of glutathione to give glutathione adducts or SH-containing amino acids, which are metabolized or oxidized to become sulfoxide, sulfone and sulfonic acid. They may be further metabolized to produce conjugates with glutathione, cysteine, malonylcysteine, glucose and others or incorporated into biomolecule.

Environmental fate

Aerobic degradation in soils

Quintozene in sandy loam soil degraded showing non-linear trend. A half-life of 189 days was calculated approximating first-order kinetics. The concentration of quintozene reached less than half of the initially applied in 60 days possibly due to its volatility. The major degradation products were PCA and PCTA. Other identified degradation products were PCTA sulfone and PCTA sulfoxide which occurred from oxidization of PCTA, and PB.

Quintozene was found to leach in small amounts (2–17 percent in four soil types) and only into the adjacent untreated soil zone.

Photodegradation in soil

There was gradual decline observed through one year in soil surface under photo-irradiation. A half-life of quintozene exposed to artificial sunlight was 28.5 days in sandy loam soil.

PCA and PCTA seemed major photodegradation products but they were found not only under irradiation but also in dark control and therefore in their formation photo irradiation does not play significant role.

Field dissipation

Field dissipation studies conducted in six locations in the United States with single application at rates ranging from 2.5 to 33.6 kg ai/ha or two applications each at 36.6 kg ai/ha indicated that quintozene dissipated with a geometric mean DT_{50} of 88 days and the total of quintozene, PCA, PCTA, PB and impurity HCB dissipated with a geometric mean DT_{50} of 360 days. These compounds were found in 0–15 cm layer soil at almost all the sampling intervals (up to 546 days). They were not found, or, if found. at very low levels in depths below 15 cm. The accumulation rates of the degradates were faster than their dissipation rates. These results indicate that quintozene itself is moderately persistent while the degradates are persistent.

Rotational crop metabolism

The Meeting received information on confined rotational crop studies and a field rotational crop study.

Confined rotational crop studies

Bare soil was treated with ^{14}C-quintozene with soil incorporation, and allowed to age for 30, 120 or 365 days as the plant-back intervals (PBI) before planting lettuce, turnips and wheat.

In the first study, after the 34 kg ai/ha application, succeeding crops were grown indoor and harvested at immature and mature growth stages. The TRR in mature plants after 31-day, 121-day or 365-day PBI were: lettuce, 1.6, 0.15 or 0.73 mg eq/kg; in turnip tops and roots, 3.6 and 20.3, 1.7 and 4.8, or 0.73 and 1.5 mg eq/kg respectively; and in wheat straw, hulls and grain, 22.9, 11.1 and 0.33; 22.2, 6.1 and 0.71; or 25.9, 8.0 and 0.38 mg eq/kg respectively. The TRR in 0–15 cm soil 2 hours after the application were in a range of 6.56–14.3 mg eq/kg and showed some tendency to gradually decrease with aging.

Samples of lettuce and turnip tops and roots were centrifuged to remove water (treated as “aqueous extract”) and the remaining moist tissues were extracted with methanol/water (40:60) and the extracts were partitioned with chloroform, and the methanol fraction was combined with the “aqueous extract” for analysis. Samples of wheat materials were extracted with methanol and then water, and after adjusting to methanol/water ratio to 40/60, the extract was partitioned with chloroform. PES fractions of

wheat straw were hydrolyzed with HCl, NaOH and cellulase. The extraction with methanol and acid hydrolysis of PES together recovered a total of 36.0–53.8 percent TRR from lettuce from all the PBIs; and 76.0 percent and 62.1 percent TRR from turnip roots and tops respectively from 365-day PBI; and 49.4–53.9 percent TRR in wheat hulls. The extraction with methanol and aci[d]base hydrolysis of PES of wheat straw recovered 61.6–67.5 percent TRR. While methanol extracted 36–67 percent TRR, acid hydrolysis released additional 9.4–19 percent TRR.

Residues of quintozene, PCA or PCTA were not found in any of lettuce, turnip tops and roots, or wheat straw and hulls. Those compounds found at higher than 0.01 mg/kg were: in all crops from all PBIs, N,N'-diacetyl-S,S'-(tetrachloro-p-phenylene)-dicysteine /C4CyCy; and in wheat straw and hulls from all PBIs, PCP-GSH; PCP-MalCys; TCP-diGSH; and N-acetyl-S-(pentachlorophenyl)-cysteine. The percentage of identified compounds in the TRR was low, 5.1–6.3 percent in lettuce; 4.3 percent in turnip roots, 12.3 percent in turnip tops, 12.1–16.9 percent in wheat straw, and 33.6–34.9 percent in wheat hulls.

In the second study, after 35 kg ai/ha application, succeeding crops were grown indoor and harvested at immature and mature growth stages. The TRR in mature plants after 31-day, 121-day or 365-day PBI were: mature lettuce, 3.0, 0.13, 0.45 mg eq/kg respectively, mature turnip tops and roots, 11.9 and 11.4, 1.3 and 5.8, or 0.91 and 1.9 mg eq/kg respectively; and mature wheat straw, hulls and grain, 27.7, 21.5 and 0.63, 13.4, 2.0 and 0.07 and 6.28, 19.3 and 1.2 mg/kg respectively. The TRR in soil immediately after treatment was in a range of 2.6–12.7 mg eq/kg.

Samples were extracted with hexane followed by methanol. These solvents extracted 77–80 percent TRR in lettuce from 121-day and 365-day PBI while only 10.8 percent TRR in lettuce from 30-day PBI; 47.1–67.0 percent and 50.1–71.8 percent TRR respectively from turnip tops and roots from all the PBIs; and 35.2–62.5 percent, 41.5–49.7 percent, and 29.6–34.2 percent TRR in wheat grain, hulls and straw respectively from all the PBIs. PES of turnip roots and tops from all PBIs and wheat matrices 365-day PBI were acid hydrolyzed. Acid hydrolysis released 5.6–7.9 percent TRR and <5 percent TRR from the PES of turnip tops and turnip roots, respectively; and <1 percent to 30 percent TRR from the PES of wheat matrices.

Detailed identification was performed only on turnip roots from 31-day PBI. Quintozene, PCA, PCTA and PB were identified in the hexane extract at the maximum of 4.1 percent TRR and 0.69 mg/kg. In the methanol extract, S-(tetrachlorothiophenol)-N-malonylcycteine and PCP-MalCys together accounted for 16 percent TRR and 2.7 mg/kg. The identified residue accounted for 11.8 percent TRR in hexane fraction and 15.6 percent TRR in methanol fraction, totalling 27.9 percent TRR.

In the third study, after application at rates of 2.2, 11 and 34 kg ai/ha, succeeding crops were grown outdoor and harvested at immature and mature growth stages. The application rate of 11 kg ai/ha was used for all crops. After the 11 kg ai/ha application, TRR in mature plants after 30-, 120- or 365-day PBI (only 365-day PBI for turnip) were: lettuce, 0.15, 0.10 or 0.43 mg eq/kg; turnip tops and roots, 1.1 and 0.77 mg eq/kg (1st retrial results); and wheat forage, grain, and straw, 3.0, 0.79 and 10.7; 3.4, 0.94 and 16.8; or 0.64, 0.14 and 4.6 mg eq/kg. The TRR in soil immediately after application of quintozene at 33.6 kg ai/ha were within a range of 2.6–12.7 mg eq/kg.

Samples were extracted using methanol followed by methanol/water (1:1), or methanol/water (1:1) alone, and the extracts were partitioned with chloroform, hexane followed by chloroform, or dichloromethane. Methanol and methanol/water, or methanol/water extracted a total of 56–77 percent TRR (27–44 percent TRR in chloroform fraction and 22–38 percent TRR in aqueous fraction) in lettuce; and 52 -76 percent and 59–70 percent TRR respectively from wheat forage and wheat straw. However, from wheat grain, only 9.8–33 percent TRR were released.

The PES remaining after extraction were treated with sequential digestions with three groups of enzymes (pH 5 compatible enzymes, pH 7 compatible or pH 6 compatible enzymes and proteases) followed by base hydrolysis. The hydrolysis with enzymes and strong acid and or base released <10 percent TRR in each fraction, except protease and base treatment released up to 10.1 percent TRR and 16.8 percent TRR respectively from lettuce PES; pH 5 compatible enzyme, pH 6 compatible enzyme and base treatment released up to 42.0 percent TRR, 13.7 percent TRR and 23.6 percent TRR from the wheat grain PES. After these treatments, < 10 percent TRR remained in the PES of these commodities.

Residues of quintozene were found in lettuce at 21 percent TRR (max conc. 0.032 mg/kg) from 30-day PBI and decreased in terms of percent TRR and concentration as the aging of soil got longer (after 365-day PBI, around 1 percent TRR and < 0.01 mg/kg). It was also found in wheat straw (2.2 kg ai/ha, 120-day PBI) at 0.023 mg/kg but not in turnip. PCA was found in all three cops. There were numerous compounds identified in lettuce and wheat matrices but most of them at very low levels.

Field rotational crop study

Unlabelled quintozene was applied to bare soil simulating the maximum US GAP for peanut (2 × 5.6 kg ai/ha; not valid at the time of this evaluation). Lettuce and wheat were planted 30, 120 and 365 days after the second application, and turnip 365 days after the second application. Samples were obtained at mature growth stage, except that wheat forage was obtained at immature stage. Samples were extracted with acetone/hexane (50:50) and partitioned with water to obtain the hexane layer which was cleaned up using Florisil column for analysis of quintozene, PCA, PCTA, PB, HCB, TCA, PCTASO and TCTASOO.

Quintozene was detected above the LOQ of 0.005 mg/kg in the 30-day PBI and 120-day PBI lettuce at a maximum 0.013 mg/kg. Quintozene was not found in any of wheat and related samples from any PBI or in turnip roots or tops from 365-day PBI (only PBI tested for turnip).

PCA was mostly below the LOQ but found above the LOQ in 30-day PBI lettuce (highest, 0.0092 mg/kg), 30-day PBI and 365-day PBI wheat forage (highest, 0.016 mg/kg) and turnip roots from 365-day PBI (highest, 0.014 mg/kg). PCTA was not found above the LOQ in any of the samples. PB, HCB, TCA, PCTASO, or TCTASOO were not found above the LOQ of 0.005 mg/kg in lettuce, turnip or wheat from all PBIs, except in 365-day PBI turnip roots TCA was found above the LOQ (highest, 0.010 mg/kg).

From 366- or 367-day PBI, no residues above the LOQ of 0.005 mg/kg of quintozene, PCTA, PCTASO, TCTASOO, PB or HCB were found in any portions of lettuce, turnip or wheat tested. PCA and TCA were found in the turnip roots at levels up to 0.014 mg/kg, PCA in wheat forage at the maximum 0.016 mg/kg.

Summary of rotational crop studies

In the confined rotational crop studies using the high application rates (up to 34.6 kg ai/ha pre-plant), similar metabolites as plant metabolism studies were identified showing complex metabolite profile, except that parent quintozene was either not found or found at low levels.

In the field study with unlabelled quintozene (2 × 5.6 kg ai/ha), quintozene was detected above the LOQ of 0.005 mg/kg in the 30-day PBI and 120-day PBI lettuce at a maximum 0.013 mg/kg. Quintozene was not found in any of wheat and related samples from any PBI or in turnip roots or tops from 365-day PBI (only PBI tested for turnip). PCA was mostly below the LOQ with sporadic detection up to 0.0092 mg/kg in 30-day PBI in lettuce and up to 0.016 mg/kg in wheat forage. PCTA was not found above the LOQ in any of the samples. PB, HCB, TCA, PCTASO, or TCTASOO were not found above the LOQ of 0.005 mg/kg in lettuce, turnips or wheat from all PBIs, except that up to 0.01 mg/kg TCA was found in

365-day PBI turnip roots. At higher single application rate to the soil (such as 25 kg ai/ha on the provided label for cabbage and broccoli), residues of quintozene were expected above 0.01 mg/kg in lettuce and other leafy vegetable.

The metabolism of quintozene in rotational crops seems to follow similar pathway as in the plant metabolism.

Animal metabolism

The Meeting received information on metabolism in lactating goats and laying hens, in addition to metabolism in rats.

Rat

Metabolism studies on laboratory animals including rats were reviewed in the framework of toxicological evaluation by the current JMPR.

Lactating goats

In three studies with goats, ^{14}C-quintozene was orally administered to lactating goats once daily for five consecutive days. The goats were sacrificed approximately 6 hours after the final dose.

In the first study, two goats were dosed orally with ^{14}C-quintozene in capsules at levels 25 or 50 mg/kg bw (equivalent to 714 and 947 ppm in the diet). The administered dose was eliminated in feces (36/41 percent of the total dose for 25 mg/kg bw goat/50 mg/kg bw goat) and urine (30/20 percent of total dose) within 48 h. A small amount was excreted in milk (0.34/0.21 percent of total dose). TRR in milk reached the highest level of 6.0/3.6 mg eq/kg on day 2 pm, after which TRR decreased. Liver, kidney and fat contained 13/13, 10/11, and 15–16/9.4–12.8 mg eq/kg respectively. Muscle contained much lower radioactivity of 0.54/0.48 mg eq/kg.

The samples of tissues were extracted with a mixture of chloroform, methanol and water, and the chloroform fraction was partitioned between acetonitrile and hexane. The milk samples and a portion of liver sample were extracted with chloroform/methanol (1:1) and the chloroform extracts were cleaned up with gel permeation column. From milk, kidney, muscle and fat following the administration of 25 mg/kg bw., 70.8–84.0 percent TRR were extracted in chloroform fraction while 4.3–18.0 percent TRR were found in the methanol/water fraction and PES contained 0–29.9 percent TRR. From liver using two slightly different extraction and partition systems, 24.2/31.4 percent TRR were extracted in chloroform fraction with 5.8/29.5 percent TRR in methanol/water fraction and PES contained 69.4/42.4 percent TRR, showing that the extraction system with chloroform/methanol was more efficient.

Chloroform fractions from the 25 mg/kg bw dose goat were subject to identification. Quintozene was extensively metabolized and it was not found in milk or tissues. PCA was identified as the main metabolite in milk (50 percent TRR, 3.0 mg/kg), kidney (31 percent TRR, 3.2 mg/kg), muscle (59 percent TRR, 0.32 mg/kg) and fat (49 percent TRR, 7.7 mg/kg). PCTA was identified at lessor amounts in milk (4.7 percent TRR, 0.28 mg/kg), kidney (2.8 percent TRR, 0.29 mg/kg), muscle (6.4 percent TRR, 0.04 mg/kg) and fat (1.5 percent TRR, 0.23 mg/kg). In liver, the majority of the radioactivity (40 percent TRR) was associated with an unknown polar metabolite released by base hydrolysis. As a result, PCA and PCTA accounted for 9.6 percent (1.3 mg/kg) and 1.3 percent (0.18 mg/kg) of TRR, respectively.

In the second study, two goats were dosed orally with ^{14}C-quintozene in capsule at 20 or 50 mg/kg bw. The administered dose was eliminated in urine (33/38 percent of total dose for 20 mg/kg bw goat/50 mg/kg bw goat) and feces (25/19 percent of the total dose). A small amount is excreted in milk (0.4 percent of total dose). TRR in milk reached a plateau on Day 2. Only 1.3/1.1 percent of total dose

remained in tissues, bile and urine in bladder. Kidney, liver, renal fat contained 32/49, 26/46 and 18–33 mg eq/kg. Muscle contained much lower levels of 1.1/2.3 mg eq/kg.

In milk from lower dose, hexane extracted 55 percent TRR with 31 percent TRR remaining in PES. In kidney and liver from lower dose, extraction with water/methanol/chloroform (1:2:1) recovered a total of 68 percent and 60 percent TRR leaving 16 percent and 46 percent TRR in the respective PES. Base hydrolysis of the PES released 10.0 percent and 4.8 percent TRR, respectively. In renal fat and omental fat from the lower dose, chloroform extracted 72.9 percent and 70.9 percent TRR respectively with 1.6 percent and 1.7 percent TRR in the PES. No extraction was conducted on muscle samples.

The extracts of kidney and liver of lower dose goat were subject to identification and characterization. The chloroform fraction of kidney contained quintozene (9.1 percent TRR, 2.9 mg/kg), followed 2,3,4,5-TCNB (0.5 percent TRR, 0.16 mg/kg). In the aqueous fraction, a smaller amount of quintozene was identified (0.5 percent TRR). The chloroform fraction of liver contained parent quintozene and PCA (together 8.3 percent TRR, 2.2 mg/kg), PCTA (5.4 percent TRR, 1.4 mg/kg) and 2,3,4,5-TCNB (1.0 percent TRR, 0.26 mg/kg). In the aqueous fraction, a smaller amount of quintozene and 2,3,4,5-TCNB were identified. Radioactivity in milk was not characterized.

In the third study, one goat was orally dosed with ^{14}C-quintozene at 50 mg/kg bw. The TRR in milk, kidney, liver, fat and muscle were 59, 49, 46, 22 and 2.2 mg eq/kg respectively.

Extraction of the 2nd day milk sample with ethyl acetate recovered 87 percent TRR. The kidney, liver and muscle samples were extracted with a mixture of methanol, chloroform and water and the PES was treated with protease. For kidney, chloroform fraction accounted for 46 percent TRR, aqueous fraction 28 percent TRR and PES 26 percent TRR of which 19 percent TRR was solubilized with protease (total of 93 percent TRR extracted). For liver, chloroform fraction accounted for 24 percent TRR, aqueous fraction 20 percent TRR and PES 56 percent TRR which was solubilized with protease. For muscle, 98 percent TRR was extracted. The renal and omental fat samples were extracted with chloroform and, after evaporation of chloroform, partitioned with acetonitrile and hexane. The acetonitrile fraction contained 84 percent of TRR. For milk, kidney, liver, muscle and fat, extractability was high from 84 to 100 percent TRR.

In kidneys, six metabolites were identified, among which PCA and PCA glucuronide accounted for 26 and 55 percent of the extracted residue. The four other metabolites were PCTP, tetrachloro(methylthio)benzenethiol, TCTA and TCA sulfoxide, each < 4.5 percent. In liver, also six metabolites were identified, mainly PCA (17 percent) and PCA-glucuronide (73 percent). Other four identified metabolites were PCPT dimer, N-pentachlorophenylhydroxyamine, PCPT and teterchloro(methylthio) benzenethiol, each < 4.7 percent. In muscle, four metabolites were identified: PCA (46 percent), PCTA (11 percent) and TCTA/tetrachlorophenyl methyl sulfoxide (together, 42 percent). In fat and milk, only one metabolite was identified as PCA, 96–100 percent of the extracted. In this study, quintozene was not detected in milk or tissues.

Laying hens

Laying hens were orally dosed with ^{14}C-quintozene for five (first study) or six (second study) consecutive days. Birds were sacrificed approximately 6 hours after the final dose.

In the first study, two groups of hens were dosed orally with ^{14}C-quintozene in capsule at 25 or 50 mg/kg bw (equivalent to 309 or 554 ppm in the diet). The majority of the total dose was recovered in excreta (65/71 percent TAR for 25 mg/kg bw group/50 mg/kg bw group) in four days, followed by GI tract and its contents (6.5/9.2 percent). Liver, kidneys, all eggs contained 0.03/0.04 percent, 0.02 percent and 0.01–0.02 percent of the total dose. In eggs, most of the radioactivity was found in egg yolk (1.2/2.7 mg eq/kg at sacrifice), much higher than that in corresponding egg white (0.038/0.071 mg eq/kg).

Kidneys, liver, abdominal fat and skin with fat contained 4.5/5.5, 2.0/2.4, 2.1/4.2 and 1.1–2.2 mg eq/kg respectively. Breast muscle contained 0.16/0.31 mg eq/kg, significantly lower than fat, as in the case of lactating goats.

Egg yolk, liver, kidney, fat and muscle were extracted with a mixture of chloroform, methanol and water. After evaporation, chloroform fraction was partitioned between acetonitrile and hexane. From liver of lower dose hens, 86.3 percent TRR were extracted and additional 10.8 percent TRR were released from PES by acid hydrolysis (total, 97.1 percent TRR). From kidney of the lower dose hens, 72 percent TRR was extracted and additional 19.1 percent TRR was released by base hydrolysis (total, 91.4 percent TRR). From fat, breast muscle and egg yolk at sacrifice, 103.5 percent, 97.0 percent and 33.5 percent TRR were extracted.

In liver extracts, quintozene, PCA and PCTA, each accounted for 3.2 percent, 0.45 percent and 1.5 percent TRR respectively. In kidney extracts, also quintozene, PCA and PCTA were detected, each accounting for 0.80 percent, 2.7 percent and 0.23 percent TRR respectively. The concentrations of these compounds were >0.01 mg/kg. The majority of the radioactivity in liver and kidney was not adequately identified or characterized.

In the second study, three groups of hens were dosed orally with ^{14}C-quintozene in capsule at rates equivalent to 105, 273 and 512 ppm in the diet. The majority of the total dose was eliminated in the excreta (87–94 percent of administered dose) for three dose groups. The TRR in liver, kidneys, thigh muscle and fat in three dose groups were 0.87/2.72/3.81, 1.84/5.05/7.29, 0.13/0.36/0.71, 2.64/6.17/10.1 mg eq/kg respectively. TRR in day-5 egg yolk and white were 1.74/3.52/5.75, and 0.06/0.24/0.29 mg eq/kg, respectively.

The tissue and egg yolk samples were extracted with a mixture of chloroform, methanol and water. Except for fat sample, significant radioactivity remained in PES: 32.8 percent TRR for kidney, 35.2 percent TRR for liver, 25.9 percent TRR for thigh muscle, and 75.3 percent TRR for egg yolk obtained from the highest dose. In order to solubilize the radioactivity in the PES, acid and base hydrolysis and proteolytic enzyme treatment were attempted. Base hydrolysis was the most efficient in solubilizing radioactivity in PES, releasing: 79.9 percent in kidney, 104 percent in liver, 75.9 percent in thigh muscle but only 38.2 percent in egg yolk. Among proteolytic enzymes with different optimal pH, 116 percent, 55 percent and 81 percent of radioactivity in kidney PES were released by pepsin, trypsin and protease treatment respectively. These enzymes released only up to 17.1 percent of radioactivity in liver PES and up to 7.4 percent in egg yolk PES.

In fat, quintozene was the predominant residue accounting for 48 percent of extracted radioactivity. Other major metabolites included PCA (16 percent) and teterchloromethylsulfinylaniline (31 percent). In liver, PCTP (71 percent) and PCTASO (21 percent) were identified. Base hydrolysis of the liver PES released PCA and PCTA. In egg yolks, PCA (70 percent), PCTA (9 percent) and PCTP (18 percent) were identified. In muscle, the major radioactive residues were PCP thioacetate or TCTA sulfone (88 percent) with minor amounts of PCTA (8 percent). Quintozene was found only in fat.

Summary of animal metabolism

The metabolism of quintozene was investigated in lactating goats and laying hens. In general, the metabolic pathways in these species were similar to that in rats.

In goats, quintozene was metabolized mainly to PCA and its glucuronide conjugates. Other metabolites were formed in much smaller amounts. They were TCTA, PCTA, C4MX. Parent quintozene was not detected in any of the tissues.

In hens, PCA, PCTA, PCTP, PCTP conjugated with cysteine, malonylcysteine, pyruvate and acetate were identified. Other metabolites identified included TCTA, TCTASOO, PCTASO and tetrachloromethylsulfinylaniline. Parent quintozene was detected in muscle (55.8 percent TRR) and fat (59.8 percent TRR) as predominant residue and in kidney and liver at low levels in one study, but detected only in fat at 48 percent of the extracted residue in another study.

The major metabolic pathway in animals involves (1) displacement of the nitro group by the sulfhydryl group of glutathione or SH-containing amino acids/peptides, followed by catabolic cleavage of the peptide, or by hydroxyl group; (2) reduction of the nitro group to produce N-hydroxypentachloroaniline and conjugated PCA; (3) dechlorination to yield tetrachloro- trichloro- phenyl compounds. The pathway has some commonality with the metabolism in plants.

Methods of analysis

The Meeting received information on the analytical methods using GC-ECD or GC-MS for the determination of residues of quintozene for data development and enforcement for broccoli, cabbage, peppers, tomato, green bean, dry bean, lettuce, potato, turnip roots and tops, wheat (forage, grain and straw), cotton seed and its processed products, peanut (whole, shell and nutmeat), cattle milk and tissues, and poultry eggs and tissues. The analytes include quintozene, PCA, PCTA, PB and impurity HCB. Some methods are capable of determining 2,3,4,5-TCNB and 2,3,5,6-TCNB.

In general, the methods for plant commodities employed extraction by homogenization with solvents containing hexane, such as 2-propanol/hexane, acetone/hexane or hexane alone, or acetonitrile/water, ethyl acetate and then partitioned into hexane fraction. After clean-up using either of Florisil, gel permeation, silica gel or SPE column, the hexane phase or reconstituted phase in other organic solvent was separated and quantified by GC-ECD or GC-MS. These methods were validated through recovery tests with the acceptable range of mean recoveries and RSD.. For the analytes mentioned above, the validated LOQ were in a range of 0.0005–0.01 mg/kg for each analyte.

One method for monitoring using the QuEChERS extraction, clean-up with SPE column, and GC-MS analysis was also validated for quintozene, PCA and PCTA with the LOQ of 0.01 mg/kg for broccoli and potato.

For quintozene, PCA, PCTA PB and HCB in animal commodities, the methods employed extraction by either acetone and then partitioned into hexane, or hexane alone, and without clean-up, analysis was conducted using GC-ECD with the LOQ in a range of 0.001–0.01 mg/kg. There was no full validation data provided to the Meeting. For analysis of these compounds in bovine and poultry commodities, procedural recovery data of the methods used in cattle and poultry feeding studies were available. In most of animal matrices, except bovine milk and egg yolk, recovery was examined by only single test on one or two fortification levels. For milk and egg yolk, four or five fortification levels were tested mostly in single or duplicate. In milk, quintozene showed recovery lower than 70 percent in 2 of 4 fortification levels. Therefore, the Meeting considered that the methods were not sufficiently validated and not fit-for-purpose.

Stability of pesticide residues in stored analytical samples

The Meeting received information on storage stability of quintozene, PCA, PCTA and PB and HCB in various commodities of high water content (broccoli, peppers, tomato, lettuce, snap bean, turnip tops and wheat immature whole plant), high protein content (kidney bean and soya bean), high starch content (potato, turnip root, corn and wheat grain), high oil content (cotton seed and peanut) and dry sample (wheat straw).

Table 102 Summary of frozen storage stability of quintozene, PCA, PCTA, PB and HCB is shown below

Category	Commodity	Duration of study (month)	Stable period (months) [a]				
			Quintozene	PCA	PCTA	PB	HCB
High water content	Broccoli	14	14	14	14	14	14
	Peppers	14	Up to 4	Up to 4	Up to 4	<2	Up to 4
	Tomato	14	14	14	14	14	14
	Lettuce	24	24	24	Up to 18	24	24
	Snap bean	23	Up to 3	23	Up to 3	Up to 3	Up 3
	Turnip tops	24	24	24	Up to 18	24	24
	Wheat immature whole plant	24	24	24	24	24	Up to 18
High protein content	Kidney bean	14	14	14	14	Up to 6	14
	Soya bean	8	<2	<2	<2	<2	<2
High starch content	Potato	14	14	14	14	14	14
	Turnip root	14	24	24	24	24	24
	Corn	8	<2	<2	<2	<2	<2
	Wheat grain	24	24	24	24	24	24
High oil content	Cotton seed	18	18	18	18	18	18
	Peanut	14	14	14	14	14	14
Dry sample	Wheat straw	24	24	24	24	24	24

Notes:

[a] Where the value of stable period is the same as the duration of study, this indicates that the fortified compound was stable at least for the months in this table. Where the term "up to" precedes the value of stable period, the fortified compound would not be stable after the months specified. The month value with the "<" symbol indicate that significant degradation occurred by the months described.

The storage periods in the storage stability studies on plant commodities cover the sample storage intervals in the residue trials.

Storage stability data were not available on animal commodities.

Definition of residue

Plant commodities

The plant metabolism of quintozene was studied in cabbage, potato and peanut. As for cabbage, there were inconsistent results in two studies. In the first study, quintozene or PCA were not detected but two metabolites were identified which were not detected in the other study. In the other study, quintozene, PCA and PCTA were detected in immature cabbage and mature wrapper leaves at the maximum 0.05 mg/kg and 1.6 percent TRR. The total identified radioactivity was only up to 18 percent TRR. The highest reported component was the sum of TCNP-MalCys and PCP-MalCys at the maximum 12 percent TRR and 0.34 mg/kg but no separate values were available for each of them.

As for potato, a new metabolism study identified numerous compounds, including conjugates with GSH, cysteine, malonylcysteine, sulfoxide, sulfone, and others, at low levels. A total of 60–64 percent TRR was identified. Quintozene, PCA and PCTA were identified and quantified but PCP-MalCys was the most abundant residue (10–12 percent TRR).

A new metabolism study on peanuts identified a total of 62 percent of TRR. Quintozene was the most abundant residue in nutmeat at 62 percent TRR. In the older studies, PCP-MalCys was identified as the predominant residue (20–42 percent TRR) with smaller amount of quintozene, PCA and PCTA.

In the supervised residue trials, quintozene was detected generally at higher concentrations than PCA and PCTA, or at similar or slightly lower concentrations than PCA. PCTA was detected generally at lower levels than quintozene or PCA. In the trials on potato, in addition to quintozene, PCA and PCTA, PB, HCB, 2,3,4,5-TCNB and 2,3,5,6-TCNB were analysed. 2,3,4,5-TCNB and 2,3,5,6-TCNB were, when analysed, below the LOQ. Quintozene was also detected at slightly higher than the LOQ of 0.005 mg/kg in succeeding lettuce in the field rotational crop study with the treatment of bare soil at a rate which is about one third of the maximum rate in available GAP.

Analytical methods were available to determine quintozene in plant commodities.

The Meeting therefore concluded that quintozene was a suitable marker for MRL-compliance.

For the residue definition for dietary risk assessment, the Meeting considered likely occurrence and toxicological relevance. While more than 80 compounds including many conjugates were identified, most of which were at low concentrations and contributions. The Meeting noted that while parent quintozene is not regarded as genotoxic, possibility of genotoxicity could not be excluded for all the metabolites, due to the lack of toxicological information on these metabolites and quantification of metabolites in rat metabolism. The Meeting considered that these metabolites could be assessed using the TTC approach for genotoxic compounds (0.0025 µg/kg bw/day).

The Meeting was unable to reach conclusion on residue definition for dietary risk assessment for plant commodities. due to concern of potential genotoxicity of all the metabolites.

Animal commodities

In the animal metabolism studies, parent quintozene was not detected in milk or any tissue of lactating goats in two of three studies. In laying hens, quintozene was detected at significant levels in tissues in one study but not in tissues other than fat in the other study. Therefore, quintozene alone is not a suitable marker.

PCA was the most predominant residue in tissues and milk, and egg yolk and fat. In chicken muscle, quintozene or PCA was not detected and the most predominant residue was PCP thioacetate or TCTA sulfone (88 percent of the extracted residue) followed by PCTA (8 percent of the extracted residue).

GC-ECD methods provided were not supported by full validation data and the Meeting considered it was not certain that these methods were fit-for-purpose to determine quintozene, PCA and PCTA in animal commodities. Therefore, it was not possible for the Meeting to establish residue definition for compliance with MRLs for animal commodities.

For the residue definition for dietary risk assessment, the Meeting considered likely occurrence and toxicological relevance. In the animal commodities, except egg yolk, the total of metabolites identified was relatively high in percentage of TRR, and comparing with plant commodities, the number of identified metabolites was relatively small. The Meeting noted that possibility of genotoxicity could not be excluded for all the metabolites, due to the lack of toxicological information on these metabolites and quantification of metabolites in rat metabolism. The Meeting considered that these metabolites could be assessed using the TTC approach for genotoxic compounds (0.0025 µg/kg bw/day).

The Meeting was unable to reach a conclusion on residue definition for dietary risk assessment for animal commodities. due to concern that exposure would exceed the TTC for genotoxicity of all the metabolites.

Conclusion

Based on the above, the Meeting recommended the following residue definition.

Definition of the residue for compliance with the MRL for plant commodities: *Quintozene*.

Results of supervised residue trials on crops

The Meeting received supervised trial data for quintozene residues on broccoli; cabbage, head; tomato; peppers; beans with and without pods; beans (dry); potato; cotton seed; and peanut conducted in the United States.

At the time of this evaluation, there were Codex MRLs for the following commodities but the current Meeting withdrew the previous recommendations: for barley; barley straw and fodder, dry; maize; maize fodder (dry); pea hay or pea fodder (dry); peas (dry); soya bean (dry); soya bean fodder; sugar beet; wheat; and wheat straw and fodder, dry since no GAP information or residue data were provided for these commodities.

The Meeting decided to withdraw the previous recommendations for quintozene on spices, fruits and berries; spices, roots and rhizomes, based on monitoring data .

Due to genotoxic concerns of all the metabolites, the Meeting estimated maximum residue levels based on provided supervised residue trials but they were not recommended for use as MRLs.

Broccoli

The critical GAP in the United States for broccoli (among the list of various Cole crops, but not for a group) allows one application of pre-plant banded soil application or broadcast application at 25 kg ai/ha.

The Meeting received 12 supervised trials conducted on broccoli in the United States during the growing season of 1988/89 and 2018/19.

In the trials, broadcast application and banded application of WP and GR formulations were used at rates (shown below for each residue value) that differ from the GAP rate. There were also direct seed treatment and transplant solution applications.

Since a number of the application rates used in the trials differed by more than 25 percent of the GAP rate, the Meeting decided to use the proportionality approach to estimate a maximum residue level and an STMR. Scaled residues were calculated using the formula below.

Scaled residue (mg/kg) = (residue in the trial, mg/kg) × 2.025 (kg ai/ha) / (rate used in the trial, kg ai/ha).

After each scaled residue value, the residue found in each trial and respective application rate are indicated in a pair of parentheses, e.g., (residue value in mg/kg, application rate in kg ai/ha).

Residues of quintozene from trials with banded application approximating the GAP in the United States were in rank order (n=7 scaled residues): 0.006 (0.005, 22.4), 0.008 (0.007, 22.4), 0.009 (0.008, 22.4), < 0.01 (< 0.01, 36.6), < 0.01 (0.13, 36.6), 0.024 (0.032, 36.6), and 0.026 (0.023, 22.4) mg/kg.

Residues of quintozene from trials with broadcast application approximating the GAP in the United States were in rank order (n=7 scaled residue): 0.005 (0.006. 33.6), 0.005 (0.006, 33.6), 0.006 (0.007, 33.6), < 0.01(< 0.01, 36.6), < 0.01(< 0.01, 36.6), 0.018 (0.024, 36.6), and 0.020 (0.027, 33.6) mg/kg.

Mann-Whitney U-test indicates that residues of quintozene from banded application and broadcast application were not significantly different. Therefore, the Meeting used the highest residue levels of quintozene from either banded or broadcast application in independent trials. Residues from independent trials were in rank order (n=7): 0.006, 0.008, 0.009, < 0.01, < 0.01, 0.024, and 0.026 mg/kg.

In a field rotational crop study with two applications each at 5.6 kg ai/ha, quintozene was detected above the LOQ of 0.005 mg/kg in lettuce from 30-day and 120-day PBI. The mean value was calculated using the residue data from all PBIs to be 0.0065 mg/kg (assuming that values < 0.005 mg/kg were at 0.005 mg/kg). After applying quintozene at 30 kg ai/ha, the highest maximum rate among available GAP, the mean residue in succeeding lettuce would be 0.017 mg/kg.

Adding 0.017 mg/kg, residue of quintozene from banded and broadcast application were: 0.023, 0.025, 0.026, < 0.027, < 0.027, 0.041 and 0.043 mg/kg.

The Meeting estimated a maximum residue level of 0.09 mg/kg for broccoli. The Meeting withdrew its previous recommendation of 0.05 mg/kg.

Cabbage, head

The critical GAP in the United States for cabbage (among the list of various Cole crops, but not for a group) is for one pre-plant banded soil application or broadcast application at 25 kg ai/ha.

The Meeting received 16 supervised trials conducted on cabbage in the United States during the growing season of 1987/88, 1988/89 and 1990/91.

In the trials, broadcast application and banded application of WP and GR formulations were used. There were also transplant solution applications.

Residues of quintozene from trials with banded application approximating the GAP in the United States were in rank order (n=15 including scaled residues): < 0.002, < 0.002, 0.002, 0.003, 0.003 (0.003,24.7), 0.007, 0.009 (0.009, /24.7), < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.010 (0.009, 22.4), 0.013 (0.013, 24.7) and 0.042 (0.038, 22.4) mg/kg.

Residues of quintozene from trials with broadcast application approximating the GAP in the United States were in rank order (n=15 scaled residue): < 0.002 (0.002, 33.6), < 0.002 (0.003, 33.6), < 0.002 (0.003, 33.6), 0.003 (0.004, 33.6), 0.004 (0.005, 33.6), 0.007 (0.009, 33.6), 0.008 (0.010, /33.6), 0.008 (0.011, 33.6), < 0.01, < 0.01, < 0.01, < 0.01, < 0.01 (0.013, 33.6), 0.022 (0.030, 33.6), and 0.028 (0.037, 33.6) mg/kg.

Mann-Whitney U-test indicates that residues of quintozene from banded application and broadcast application were not significantly different. Therefore, the Meeting used the highest residue levels of quintozene from either banded or broadcast application in independent trials. Residues from independent trials were in rank order (n=15): < 0.002, < 0.002, 0.003, 0.004, 0.007, 0.008 0.009, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.013, 0.028 and 0.042 mg/kg.

In a field rotational crop study with two applications each at 5.6 kg ai/ha, quintozene was detected above the LOQ of 0.005 mg/kg in lettuce from 30-day and 120-day PBI, with the mean value of 0.0065 mg/kg calculated assuming that values < 0.005 mg/kg were at 0.005 mg/kg. After applying quintozene at 30 kg ai/ha, the highest maximum rate among available GAP, the mean residue in succeeding lettuce would be 0.017 mg/kg.

Adding 0.017 mg/kg, residues from independent trials according to GAP in the United States were: 0.019, 0.019, 0.020, 0.021, 0.024, 0.025, 0.026, 0.027, 0.027, 0.027, 0.027, 0.027, 0.030, 0.045 and 0.059 mg/kg.

The Meeting estimated a maximum residue level of 0.08 mg/kg for cabbages, head. The Meeting withdrew its previous recommendation of 0.1 mg/kg for cabbages, head.

Tomato

Critical GAP for tomato was from Mexico, for a single application of quintozene at a maximum rate of 14.4 kg ai/ha in nursery. No trials matching this GAP were available.

GAP for tomato in Thailand allows spraying to soil, immediately after planting young tomato plant and repeating spraying every 14 days at least two times at a maximum spray concentration of 1.50 kg ai/hL. No trials matching this GAP were available.

GAP in Ecuador for kidney tomato allows the use of quintozene in greenhouse at 0.56 kg ai/ha with a PHI of 1 day (number of applications not specified). No trials matching this GAP were available.

The Meeting therefore decided to withdraw the previous recommendation of 0.02 mg/kg for tomato.

Peppers

The critical GAP for chili peppers is in Mexico which allows a single application in nursery situations.

The Meeting received nine supervised trials conducted in the United States during the growing seasons of 1987/1988 and 1988/89. In the trials, quintozene was applied to soil at the time of planting and only in two trials chili peppers were grown and harvested.

The Meeting considered that the trials did not match the GAP in Mexico. The Meeting withdrew the previous recommendation of 0.05 (*)mg/kg for peppers, sweet. Consequently, the Meeting also withdrew the previous recommendations on dried chili peppers (0.1 mg/kg).

Beans with pods, Beans, shelled and Beans (dry)

Critical GAP for beans in Mexico allows a single application at 8.64 kg ai/ha as band application at the beginning of flowering.

The Meeting received 20 supervised trials on beans with pods, three trials on shelled beans, 13 trials on beans (dry) conducted in the United States. Except for three trials on beans with pods using in-furrow application, other trials used 4 or 3 post-emergence applications. Finite residues were found in four trials on beans with pods and five trials on beans (dry). The Meeting considered that these trials did not match the critical GAP and could not be used for estimating a maximum residue level.

For in-furrow application at planting, critical GAP was from Mexico that allows a single banded application of 1.44 kg ai/ha at sowing.

In three in-furrow trials, the application was made once at 1.68 kg ai/ha and beans with pod were sampled. Residues of quintozene from trials approximating this GAP were: < 0.0005, 0.017 and 0.074 mg/kg. Since there were only three trials, the Meeting considered the information was not sufficient for estimating a maximum residue level.

The Meeting withdrew its previous recommendations for common bean (pods and/or immature seeds) at 0.1 mg/kg and for common beans (dry) at 0.02 mg/kg.

Potato

For in-furrow applications, the critical GAP is from the United States, which allows one application of quintozene at the maximum rate of 5.6 kg ai/kg, sprayed as a 22-cm band in the seed furrow at planting.

The Meeting received 37 supervised trials conducted on potato in the United States using in-furrow application at rates 11.2–13.1 kg ai/ha.

Residues of quintozene from the in-furrow application approximating US GAP with scaling (scaling factors from 5.6/14.0–5.6/4.12) were (n=37): < 0.01, < 0.01, 0.013, 0.013, 0.015, 0.020, 0.037, 0.038, 0.049, 0.056, 0.064, 0.068, 0.073, 0.081, 0.090, 0.093, 0.095, 0.099, 0.10, 0.10, 0.14, 0.14, 0.15, 0.16, 0.16, 0.16, 0.17, 0.18, 0.20, 0.22, 0.27, 0.35, 0.36, 0.38, 0.39, 0.44 and 0.79 mg/kg.

For chemigation, the critical GAP of United States allows two applications at the maximum rate of 2.8 kg ai/ha with a PHI of 28 days.

The Meeting received eight supervised trials conducted on potato in the United States using two chemigation applications at a rate of 2.8 kg ai/ha each.

Residues of quintozene from the chemigation application matching the US GAP were (n=8): < 0.01, < 0.01, 0.011, 0.017, 0.019 (0.037, 5.6), 0.026, 0.039 and 0.080 mg/kg.

For broadcast application, cGAP in Mexico allows one application at the maximum rate of 30.0 kg ai/ha. PHI was not specified. Twenty independent trials were conducted in the United States. Residues of quintozene from independent trials using the broadcast application and matching the GAP in Mexico were (n=20): < 0.002, 0.007, 0.007, 0.009, 0.014, 0.018, 0.044, 0.048, 0.067, 0.070, 0.074, 0.078, 0.087, 0.098, 0.11, 0.12, 0.14, 0.15, 0.19 and 0.66 mg/kg.

Among these three data populations, residues from chemigation were significantly lower than the other two populations. Residues from in-furrow application and those from broadcast applications were different according to Mann-Whitney U-test. As the residues from in-furrow application would lead to a higher maximum residue level, using this data population, the Meeting estimated a maximum residue level of 0.8 mg/kg for potato.

Cotton seed

Critical GAP in South Africa for cotton allowed one application of quintozene into furrow at the maximum rate of 5.25 kg ai/kg.

The Meeting received 23 supervised trials conducted on cotton in the United States during the growing seasons of 1987 to 1992. The trials used in-furrow application at planting or in five trials banded soil application at planting at a rate of 2.24 kg ai/ha. Residues of quintozene from the trials with banded soil application at 2.24 kg ai/ha were all below the LOQ of 0.01 mg/kg. The maximum residue level of quintozene from in-furrow application at 2.24 kg ai/ha was 0.012 mg/kg. Since residue levels were below the LOQ in most of the trials, there were only 3 finite values for scaling up to the GAP rate in South Africa. The Meeting concluded that information was insufficient to estimate maximum residue level for cotton seed.

Therefore, the Meeting decided to withdraw the previously recommended maximum residue level of 0.01 (*) mg/kg for cotton seed.

Peanut

Critical GAP for peanuts was from Mexico, which allows a single banded application at a rate of 1.92 kg ai/ha at sowing.

The Meeting received 36 supervised trials conducted on peanut in the United States conducted during the growing seasons of 1987 to 1992. In all the trials in the United States, two applications with quintozene were made during pegging time. The Meeting concluded that the trials did not match this GAP and decided to withdraw the previous recommendation of 0.5 mg/kg for peanut.

Residues arising from crop rotation

In a field rotational crop study using two applications of 5.6 kg ai/ha, quintozene was detected above the LOQ of 0.005 mg/kg in lettuce. Residues of quintozene were expected to occur in leafy vegetables and Brassica vegetables from crop rotation. Residues found from 30, 120 and 365-day PBI were: < 0.005 × 15, 0.053, 0.075, 0.0080, 0.0096, 0.0120, 0.0126 and 0.0133 mg/kg.

The critical maximum seasonal rate according to the available GAP is 30 kg ai/ha (Mexico). Finite residues were scaled to the rate of 30 kg ai/kg using the scaling factor of 30/11.2: 0.014, 0.020, 0.021, 0.026, 0.032, 0.034 and 0.037 mg/kg. Assuming that residues < 0.005 mg/kg were at 0.005 mg/kg, scaled residue of < 0.005 mg/kg would be 0.013 mg/kg

The Meeting estimated a maximum residue level of 0.08 mg/kg for a group of leafy vegetables and a group of Brassica vegetables (except Brassica leafy vegetables) (except broccoli and cabbage).

Fate of residues during processing

No information was available on high temperature hydrolysis.

Processing

The Meeting received information on residues in edible portions of potato and peanut, and processing of tomato, green beans, potato, cotton seed and peanut to various processed commodities.

Processing factors of quintozene for potato, for which maximum residue level was estimated, to its processed commodities potato are shown below.

Table 103 Potato processing factors

Commodity	n	Processing factor for quintozene	
		Individual	Best estimate
Potato		-	
Wet peel	8	2.0, 3.3, 5.6, 5.8, 7.6, 10, 11, 13	6.7
Dried peel	8	2.3, 13, 13, 14, 25, 16, 65, 75	19.5
Peeled potato	4	0.06, 0.10, 0.10, 0.14	0.10
French fries	2	1.9, 2.8	2.35
Crisps	10	0.13, 0.16, 0.23, 0.47, 0.80, 0.99, 1.3, 1.7, 1.8, 1.9	0.90
Dried flakes	2	0.14, 0.18	0.16
Flakes	8	0.02, 0.04, 0.04, 0.06, 0.06, 0.07, 0.09, 0.10,	0.06
Granules	8	0.00, 0.00, 0.01, < 0.02, < 0.02, < 0.02, 0.02, 0.04	0.02

Residues in animal commodities

Livestock feeding studies on lactating cows and laying hens were provided. However, due to the lack of full validation data on the analytical methods used, it was not possible to use the data for evaluation. Therefore, it was not possible to estimate maximum residue levels for animal commodities.

The Meeting withdrew its previous recommendations on chicken commodities: 0.1(*) mg/kg (fat) for chicken meat, 0.01(*) mg/kg for chicken, edible offal of, and 0.03 (*) mg/kg in eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting agreed to the following residue definition.

Definition of the residue for compliance with the MRL for plant commodities: *Quintozene*.

The Meeting was unable to establish residue definition for compliance with MRLs for animal commodities.

The Meeting was also unable to reach a conclusion on residue definition for dietary risk assessment for plant and animal commodities. due to concern that exposure would exceed the TTC for genotoxicity of all the metabolites.

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The Meeting confirmed an ADI of 0–0.01 mg/kg for quintozene and ARfD was unnecessary.

As the Meeting was unable to conclude on the toxicological relevance of all the metabolites, the Meeting could not reach a conclusion on a residue definition for dietary risk assessment. As a result, long-term dietary exposure assessments could not be conducted.

Threshold of toxicological concern (TTC) consideration for metabolites

The Meeting noted that possibilities of genotoxicity could not be excluded from all the metabolites. They could be assessed using the TTC approach for genotoxicity (threshold of 0.0025 µg/kg bw per day for genotoxic compounds).

In one potato metabolism study and one confined rotational crop study, numerous non-polar and polar metabolites were identified. However, the percentage of identified radioactivity in confined rotational crop studies was relatively low mostly below 50 percent of TRR, except that in turnip roots about 80 percent TRR was identified. In a field rotational crop study, quintozene, PCA, PCTA, TCA TCTASOO and PCTASO, PB and HCB were analysed, among which quintozene, PCA and TCA were detected in food commodities.

PCA and PCTA were also analysed in supervised residue trials provided to the Meeting.

Dietary exposure was calculated for a number of the metabolites using the rotational crop studies and the IEDI spreadsheet and compared with the threshold of 0.0025 µg/kg bw per day. The calculated exposure may be underestimated due to the low percentage of identification. For the metabolites calculated, TTC for genotoxic compounds was exceeded. Two examples are shown below.

1. PCA

PCA was detected in many of metabolism studies and confined rotational crop studies. PCA concentrations in the extracts in the confined rotational crop studies were scaled from the application rates used in the studies to the highest possible application rate of 30 kg ai/ha.

PCA was detected in turnip roots and lettuce at 0.023–0.155 mg/kg and 0.018–0.12 mg/kg, respectively, after scaling.

Using the lowest quantified concentrations, the calculated chronic exposure from root and tuber vegetables, leafy vegetables and Brassica vegetables were in a range of 0.063–0.24 ug/kg bw, higher than the TTC for genotoxic compounds (0.0025 ug/kg bw).

2. TCA

TCA was detected in the field rotational crop study in turnip root at < 0.005–0.010 mg/kg. Since in the study the application rate was 2 × 5.6 kg ai/ha, while the highest possible application rate was 30 kg ai/ha, the Meeting applied the proportionality principle to scale up the residues. The scaled finite residues were: 0.0134, 0.0169, 0.190 and 0.0279 mg/kg. TCA was < 0.005 mg/kg in other rotated crops and not detected in confined rotational crop studies.

Using the lowest quantified residue after scaling, the calculated chronic exposure from root and tuber vegetables were in a range of 0.021–0.15 µg/kg bw, higher than the TTC for genotoxic compounds (0.0025 ug/kg bw).

The Meeting concluded that the chronic dietary exposure of many of metabolites arising from uses of quintozene considered by the Meeting exceeded the TTC for genotoxic compounds of 0.0025 µg/kg bw/day and they may present a public health concern.

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900-RES-016	LeRoy, R.L.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues on Dry Beans; Pan-Agricultural Labs Report No. PAL-PB-DB AMVAC GLP, unpublished 21.03.1991
900-RES-017	LeRoy, R.L.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues on Lima Beans; Pan-Agricultural Labs Report No. PAL-PB-LB AMVAC GLP, unpublished 28.03.1991

Report Code	Author(s)	Year	Study Title Report Number, GLP, etc.
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900-RES-019	LeRoy, R.L.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues on Broccoli; Pan-Agricultural Labs Report No. PAL-PB-BR AMVAC GLP, unpublished 21.03.1991
900-RES-020	LeRoy, R.L.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues on Cabbage; Pan-Agricultural Labs Report No. PAL-PB-CB AMVAC GLP, unpublished 26.03.1991
900-RES-021	LeRoy, R.L.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues on Cottonseed; Pan-Agricultural Labs Report No. PAL-PB-CS AMVAC GLP, unpublished 21.03.1991
900-RES-022	LeRoy R.L.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues on Peanuts; Pan-Agricultural Labs Report No. PAL-PB-PT AMVAC GLP, unpublished 26.03.1991
900-RES-023	Stenner S.S., Stanek M., Grice M.	1992	Field Residue Study with PCNB on Peanuts; Hazelton Labs Report No. HWI 6274-109 AMVAC GLP, unpublished 03.04.1992
900-RES-024	LeRoy, R.L.	1991	Magnitude of the Residue: Pentachloronitrobenzene (PCNB) Residues on Pepper; Pan-Agricultural Labs Report No. PAL-PB-PP AMVAC GLP, unpublished 26.03.1991
900-RES-025	LeRoy, R.L.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues on Potato; Pan-Agricultural Labs Report No. PAL-PB-PO AMVAC GLP, unpublished 26.03.1991
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900-RES-030	Thornton M., Feiler W.A.	2003	Magnitude of Residue on Potatoes from Foliar Applications; Morse Laboratories, Inc. Report No. ML02-1043-AMV AMVAC GLP, unpublished 10.01.2003

Report Code	Author(s)	Year	Study Title Report Number, GLP, etc.
900-RES-031	LeRoy R.L., Cassidy J.E.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues in Processed Snap Beans; Pan-Agricultural Labs Report No. PAL-PB-SB-P AMVAC GLP, unpublished 10.07.1991
900-RES-032	LeRoy R.L., Cassidy J.E.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues in Processed Cottonseed; Pan-Agricultural Labs Report No. PAL-PB-CS-P AMVAC GLP, unpublished 10.07.1991
900-RES-033	LeRoy R.L., Cassidy J.E.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues in Processed Peanuts; Pan-Agricultural Labs Report No. PAL-PB-PT-P AMVAC GLP, unpublished 11.07.1991
900-RES-034	LeRoy R.L., Cassidy J.E.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues in Processed Potatoes; Pan-Agricultural Labs Report No. PAL-PB-PO-P AMVAC GLP, unpublished 23.07.1991
900-RES-035	LeRoy R.L., Cassidy J.E.	1991	The Magnitude of Pentachloronitrobenzene (PCNB) Residues in Processed Tomatoes; Pan-Agricultural Labs Report No. PAL-PB-TO-P AMVAC GLP, unpublished 10.07.1991
900-RES-037	Carringer S. J.	2005	Magnitude of the Residue of PCNB and its Metabolites in Potato Raw Agricultural Commodities; Morse Laboratories, Inc. Report No. TCI-04-085 AMVAC GLP, unpublished 17.01.2005
900-RES-038	Carringer S. J.	2006	Magnitude of the Residue of PCNB and its Metabolites in Potato Raw Agricultural Commodities - 2005; Morse Laboratories, Inc. Report No. TCI-05-121 AMVAC GLP, unpublished 22.02.2006
900-RES-042	McManus J.P., DeMatteo V.	1999	Distribution and Metabolism of ^{14}C(UL)-PCNB in Peanuts; Uniroyal Chemical Company, Inc. Project No. 97033 AMVAC GLP, unpublished 12.03.1999
900-RES-043	Fang N., Mertz J.	1999	Metabolism of ^{14}C-[UL]-PCNB in Potatoes as a Soil Treatment; Uniroyal Chemical Company, Inc. Report No. 97013 AMVAC GLP, unpublished 03.05.1999

Report Code	Author(s)	Year	Study Title Report Number, GLP, etc.
900-RES-044	Carringer, S. J.	2006	Magnitude of the Residue of PCNB and its Metabolites in Potato Raw Agricultural Commodities – 2006. Morse Laboratories, Inc. Report No. TCI-06-146 AMVAC GLP, unpublished 19.12.2006
900-RES-050	Ball, J.O.	1990	Stability of Terraclor and Allied Metabolites in Frozen Beans, Peppers, Tomatoes and Tomato Products; Huntingdon Analytical Services, Inc., Uniroyal Chemical Co. Report No. A026.001E, A026.005 AMVAC GLP, unpublished 22.05.1990
900-RES-055	Kuchar E.J., Griffith W.P.	1975	Analytical Investigations Concerned with Feeding Terraclor® to Chickens; Olin Mathieson Corp. Report No. CASR-8-75 AMVAC non-GLP 14.11.1975
900-RES-056	Ball J.O.	1987	Magnitude of the Residue Terraclor® Residues in Processed Potatoes; Uniroyal Study No. UR-1407 AMVAC GLP, unpublished 23.12.1987
900-RES-058	Selman F., Feutz E., Leak T.	1988	Determining the Uptake and Translocation of [^{14}C] PCNB Used in the Seed Treatment of Corn (Zea Mays), Wheat (Triticum Aestivum), Soybean (Glycine Max), Peas (Pisum Sativum), and Sugarbeets (Beta Vulgaris); ABC Laboratory Inc. Report No. 35972 AMVAC GLP, unpublished 28.03.1988
900-RES-059	Ball J.O.	1988	Magnitude of the Residue Terraclor and its Metabolites in Cabbage; Uniroyal Study No. UR-1413 AMVAC GLP, unpublished 10.08.1988
900-RES-060	Ball J.O.	1988	Magnitude of the Residue Terraclor and its Metabolites in Broccoli; Uniroyal Study No. UR-1408 AMVAC GLP, unpublished 19.08.1988
900-RES-061	Ball J.O.	1988	Magnitude of the Residue Terraclor and its Metabolites in Beans; Uniroyal Study No. UR-1409 AMVAC GLP, unpublished 31.08.1988
900-RES-062	Ball J.O.	1988	Magnitude of the Residue Terraclor and its Metabolites in Peppers; Uniroyal Study No. UR-1410 AMVAC GLP, unpublished 31.08.1988
900-RES-063	Ball J.O.	1988	Magnitude of the Residue Terraclor and its Metabolites in Potatoes; Uniroyal Study No. UR-1414 AMVAC GLP, unpublished 31.08.1988

Report Code	Author(s)	Year	Study Title Report Number, GLP, etc.
900-RES-067	Gaydosh K.A.	1991	Magnitude of the Residue: PCNB and Related Metabolites and Impurities in Cotton Treated with Terraclor 10G, Terraclor 2E and Terraclor Flowable; Uniroyal Study No. RP-91020 Centre Analytical Laboratories, Project No., 004-13, AMVAC GLP, unpublished 06.10.1992
900-RES-068	Ball J.O.	1989	Magnitude of the Residue in Terraclor Treated Cottonseed and Cottonseed Fractions; Uniroyal Study No. UR1412 Craven Laboratories Report No. UNRLCRV-2 AMVAC GLP, unpublished 15.11.1989
900-RES-070	McManus J.P.	1989	Metabolism of Pentachloronitrobenzene In the Goat: Identification of Metabolites; Uniroyal Project No. 8761 AMVAC GLP, unpublished 12.12.1989
900-RES-075	Daun R.J.	1990	Pentachloronitrobenzene: Nature of the Residue in Livestock - Lactating Goats; Hazelton Labs Report No. HLA 6111-118 Uniroyal Project no. 8761 AMVAC GLP, unpublished 19.01.1990
900-RES-078	Ball J.O.	1990	Magnitude of the Residue in Terraclor Treated Peanuts and Peanut Fractions; Uniroyal Study No. UR1421 Craven Laboratories Report No. UNRLCRV-1 AMVAC GLP, unpublished 07.03.1990
900-RES-080	Ball J.O.	1990	Determination of Terraclor and its Metabolites in Peppers at Two PPB Quantitation Limit; Huntingdon Analytical Services Report No. A026.005 AMVAC GLP, unpublished 05.05.1990
900-RES-081	Ball J.O.	1990	The Magnitude of Terraclor and its Metabolites in Peppers - Clewiston, Florida; Huntingdon Analytical Services Report No. A026.001B AMVAC GLP, unpublished 22.05.1990
900-RES-082	McManus J.P., Maisonet F.A.	1990	Metabolism of Pentachloronitrobenzene in Cabbage - Metabolite Identification; Uniroyal Chemical Company, Inc. Project No. 8756 AMVAC GLP, unpublished 24.04.1990

Report Code	Author(s)	Year	Study Title Report Number, GLP, etc.
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900-RES-085	Ball J.O.	1990	Determination of Terraclor and its Metabolites and Impurities in Beans and Bean Cannery Waste. at a Two PPM Quantitation Limit; Huntingdon Analytical Services Report No. A026.005.03 AMVAC GLP, unpublished 22.05.1990
900-RES-087	Halls T.D.J.	1990	Confined Accumulation Study of ^{14}C-PCNB on Rotational Crops Treatment, Sampling and Combustion Analysis; Analytical BioChemistry Laboratories Report No. 35971 AMVAC GLP, unpublished 25.06.1990
900-RES-090	Parkins M.D.	1990	Pentachloronitrobenzene: Nature of the Residue in Poultry-Laying Hens; Uniroyal Project No. 8762 AMVAC GLP, unpublished 12.11.1990
900-RES-092	McManus J.P.	1990	Metabolism of Pentachloronitrobenzene In the Goat: Identification of Metabolites in Muscle; Uniroyal Project No. 8761 AMVAC GLP, unpublished 13.12.1990
900-RES-095	Gaydosh K.A.	1991	Magnitude of the Residue: PCNB and Related Metabolites and Impurities in RTU-PCNB Treated Wheat; Uniroyal Study No. UR-1419 AMVAC GLP, unpublished 19.02.1991
900-RES-096	Gaydosh K.A.	1991	Storage Stability of PCNB and Its Allied Metabolites and Impurities in Peanuts; Uniroyal Study No. RP-89065 AMVAC GLP, unpublished 28.03.1991
900-RES-097	Gaydosh K.A.	1991	Storage Stability of PCNB and Its Allied Metabolites and Impurities in Cotton; Uniroyal Study No. RP-89066 AMVAC GLP, unpublished 28.03.1991
900-RES-098	Parkins M.D.	1991	Pentachloronitrobenzene: Nature of the Residue in Poultry-Laying Hens - Amendment 1: Identification of Bound Residues; Uniroyal Project No. 8762 AMVAC GLP, unpublished 02.05.1991

Report Code	Author(s)	Year	Study Title Report Number, GLP, etc.
900-RES-101	Ford C.M., Murty V.S.	1993	Volume IV: Characterization of Pentachloronitrobenzene Metabolites in Rotational Lettuce; Uniroyal Chemical Company, Inc. Project No. 9039 AMVAC GLP, unpublished 04.05.1993
900-RES-102	Ford C.M., Murty V.S.	1993	Volume V: Characterization of Pentachloronitrobenzene Metabolites in Rotational Turnip; Uniroyal Chemical Company, Inc. Project No. 9055 AMVAC GLP, unpublished 07.05.1993
900-RES-103	anonymous	1993	Volume II: In-Life Portion; AMVAC Non-GLP, unpublished 09.07.1993
900-RES-104	Putterman G.J.	1993	Volume I: Study Overview; AMVAC Non-GLP, unpublished 09.07.1993
900-RES-105	Ford C.M., Murty V.S.	1993	Volume III: Characterization of Pentachloronitrobenzene Metabolites in Rotational Wheat; Uniroyal Chemical Company, Inc. Project No. 9058 AMVAC GLP, unpublished 16.06.1993
900-RES-107	Gaydosh K.A.	1993	Magnitude of the Residue: Terraclor 75W, Terraclor 2EC and Terraclor Flowable on Snap Beans; Uniroyal Study No. RP-93009 AMVAC GLP, unpublished 22.12.1993
900-RES-108	Gaydosh K.A.	1993	Magnitude of the Residue: Terraclor 75W, Terraclor 2EC and Terraclor Flowable on Dry Beans; Uniroyal Study No. RP-93010 AMVAC GLP, unpublished 13.01.1994
900-RES-109	Gaydosh K.A.	1994	Terraclor 10G and Terraclor 75W on Peanuts - Magnitude of the Residue Study; Uniroyal Study No. RP-90033 AMVAC GLP, unpublished 10.08.1994
900-RES-110	Gaydosh K.A.	1994	Terraclor 10G and Terraclor 2E on Cotton - Magnitude of the Residue Study; Uniroyal Study No. RP-90034 Centre Analytical Laboratories, Project No., 004-07, AMVAC GLP, unpublished 20.09.1994
900-RES-111	Gaydosh K.A.	1994	Terraclor 75W on Peanuts - Magnitude of the Residue Study; Uniroyal Study No. RP-90032 AMVAC GLP, unpublished 31.10.1994

Report Code	Author(s)	Year	Study Title Report Number, GLP, etc.
900-RES-112	Ball, J.O.	1988	Stability of Terraclor and Allied Metabolites in Frozen Wheat, Corn, Soybeans, Kidney Beans, Peppers, Tomatoes, Catsup, and Dry Tomato Pomace; Uniroyal Study No. UR-1405 AMVAC GLP, unpublished 28.11.1988
900-RES-113	Gaydosh K.A.	1994	Terraclor Flowable and Terraclor 2E on Peanuts - Magnitude of the Residue Study; Uniroyal Study No. RP-91022 AMVAC GLP, unpublished 31.10.1994
900-RES-114	Gaydosh K.A., Smudin D.J.	1996	Terraclor 10G on Cotton: Processing Study; Uniroyal Chemical Company, Inc. Project No. RP-93008 AMVAC GLP, unpublished 22.03.1996
900-RES-115	Gaydosh K.A., Smudin D.J.	1996	Terraclor 2EC on Peanuts: Processing Study; Uniroyal Chemical Company, Inc. Project No. RP-92018 AMVAC GLP, unpublished 01.04.1996
900-RES-116	Gaydosh K.A.	1996	Terraclor 10G Rotational Crop Study. Residue Levels of PCNB and Related Metabolites and Impurities in Wheat, Turnip, and Lettuce Planted 30 and 120 Days After Terraclor 10G Applied to Bare Soil at Peanut Application Rates; Uniroyal Chemical Company, Inc. Report No. RP-92009 AMVAC GLP, unpublished 02.08.1996
900-RES-121	Harned W.H., Carter D.S., Yu W.C., Backer R.W., Regis R.R., Gu Z., Obrist J.J., Parkins M.D.	1998	Confined Rotational Crop Analytical and Field Study on PCNB: Amended Final Report; ABC Laboratory Inc., XenoBiotic Laboratories, Inc., Uniroyal Chemical Co. Report No. 9270; 92094; RPT00266 AMVAC GLP, unpublished 05.06.1998
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900-RES-126	Ball J.O.	1990	Magnitude of the Residue - Terraclor and its Metabolites and Impurities in Tomatoes and Processed Fractions; Uniroyal Report No. UR-1411 Huntingdon Analytical Services Report No. A026.001A AMVAC GLP, unpublished 22.05.1990
900-RES-144	Ball J.O.	1990	Residues of PCNB and its Metabolites in Processed Potatoes; Uniroyal Report No.RP-88003 AMVAC GLP, unpublished 28.03.1990

Report Code	Author(s)	Year	Study Title Report Number, GLP, etc.
900-RES-146	Gaydosh K.A.	1996	Terraclor 10G Rotational Crop Study. Residue Levels of PCNB and Related Metabolites and Impurities in Wheat, Turnip, and Lettuce Planted 365 Days After Terraclor 10G Applied to Bare Soil at Peanut Application Rates; Uniroyal Chemical Company, Inc. Report No. RP-91029 AMVAC GLP, unpublished 20.03.1996
900-RES-147	Maselli C.	1997	Terraclor Super × on Raw and Processed Cotton: Processing Study; ABC Laboratory Inc., Coastal Ag Research, S-L Agri-Development Company, Texas A&M University Report No. RP-95025, SL-95025, RP-95025, 42875 AMVAC GLP, unpublished 29.05.1997
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900-RES-152	Gaydosh K.A.	1992	Magnitude of the Residue: PCNB and Related Metabolites and Impurities in Cotton Treated with Terraclor 2E and Terraclor Flowable; Uniroyal Study No. RP-88001 AMVAC GLP, unpublished 06.10.1992
900-RES-153	Daun R.J.	1991	Pentachloronitrobenzene: Nature of the Residue in Livestock - Lactating Goats (Supplement No. 1 to Final Report); Hazelton Labs Report No. HLA 6111-118 Uniroyal Project no. 8761 AMVAC GLP, unpublished 22.11.1991
900-RES-156	Puglis J.M.	1990	Gas Chromatographic Determination of Terraclor and Its Metabolites in 1987 Pepper Study at a Two PPB Quantitation Limit; Huntingdon Analytical Services, Inc. Report No. A026.005.02 AMVAC GLP, unpublished 09.04.1990
900-RES-158	Puglis J.M.	1990	Gas Chromatographic Determination of 1987 Crop Residues of Terraclor and Metabolites at 5 PPB Quantitation Limit Using Uniroyal Chemical Company Method CAM-24-73: Peppers; Huntingdon Analytical Services, Inc. Report No. A026.001E.02 AMVAC GLP, unpublished 09.04.1990
900-RES-160	Ball J.O.	1990	Determination of Terraclor and its Metabolites and Impurities in Tomatoes and Processed Tomatoes at a Two PPM Quantitation Limit; Huntingdon Analytical Services Report No. A026.005.01 AMVAC GLP, unpublished 22.05.1990
900-RES-162	Gounaris E.K.	1994	Terraclor 10G in Soil, Turnip, Wheat, and Lettuce Rotated 365 Days after Terraclor Peanut Application; Centre Analytical Laboratories, Inc. Report No. RP-91029 AMVAC GLP, unpublished 30.08.1994

Report Code	Author(s)	Year	Study Title Report Number, GLP, etc.
900-RES-163	Gounaris E.K.	1994	Terraclor 10G Rotational Crop Study Peanuts Rotated with Wheat and Lettuce 30 and 120 Days After Terraclor 10G Application. Centre Analytical Laboratories, Inc. Report No. RP-92009 AMVAC GLP, unpublished 17.08.1994
900-RES-166a	Puglis J.M.	1988	Determination of Terraclor and Metabolites in Peppers – Addendum; Huntingdon Analytical Services, Inc. Report No. A026.001B Addendum AMVAC GLP, unpublished 14.10.1988
900-RES-167a	Ruhland J.H.	1991	Determination of the Stability of Terraclor (PCNB) and Allied Metabolites in Cottonseed - Amendment No. 1 to Final Report. Hazelton Labs Report No. 6012-309 AMVAC GLP, unpublished 27.03.1991
900-RES-167b	Keller J.F.	1991	Determination of the Stability of Terraclor (PCNB) and Allied Metabolites in Cottonseed - Amendment No. 2 to Final Report. Hazelton Labs Report No. 6012-309 AMVAC GLP, unpublished 06.09.1991
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900-RES-197	Gaydosh K.A.	1993	Magnitude of the Residue: Terraclor Super × Emulsifiable and Terraclor Super × Flowable on Cotton; Uniroyal Study No. RP-90035 AMVAC GLP, unpublished 09.08.1993
900-RES-198	Ball J.O.	1987	Magnitude of the Residue in Fried Treated Processed Potatoes PCNB and Its Metabolites and Impurities; Uniroyal Study No. UR-1404 Morse Laboratories, Inc. Report No. 42370, 42639 AMVAC GLP, unpublished 15.02.1987
900-RES-201	Yu W.C.	1992	Terraclor 10G and Terraclor 75W on Cabbage: Magnitude of the Residue Study; NET Atlantic Inc. Report No. RP-90031; 30700 AMVAC GLP, unpublished 10.02.1992
900-RES-202	Yu W.C.	1992	Terraclor Flowable on Cabbage: Magnitude of the Residue Study; NET Atlantic Inc. Report No. RP-90065; 32300 AMVAC GLP, unpublished 10.02.1992

Report Code	Author(s)	Year	Study Title Report Number, GLP, etc.
900-RES-205	Kebede E.	1993	Terraclor 75W and Terraclor Flowable on Sweet Peppers: Magnitude of the Residue; Spectralytix, Inc. Report No. 92006, Uniroyal Study No. RP-91023 AMVAC GLP, unpublished 02.09.1993
900-RES-206	Zheng S.	1992	Terraclor 2EC on Potatoes - Processing Study - Magnitude of the Residue Study; Uniroyal Project No. RP-91025 Centre Analytical Laboratories, Inc. Report No. 004-17 AMVAC GLP, unpublished 26.05.1992
900-RES-218	Brunk J.	1994	Terraclor 75W on Tomatoes (Processing Study): Magnitude of the Residue. Spectralytix, Inc. Report No. 92008 Uniroyal Study No. RP-91027 AMVAC GLP, unpublished 23.02.1994
900-RES-223	Thorn, J.	2019	Method Validation - Analytical Method for the Determination for Pentachloronitrobenzene and Two Metabolites in Crop Matrices; Battelle Report No. 100117568 AMVAC GLP, unpublished 25.03.2019
900-RES-224	Bennett R., Gibbs A.	2019	Blocker 4F Fungicide: Magnitude of the Residues on Broccoli Raw Agricultural Commodities After Application of Pentachloronitrobenzene Fungicide – California, 2018; Lange Research and Consulting, Inc. Report No. LR18357 AMVAC GLP, unpublished 26.10.2019
900-RES-225	Bennett, R., Rice, F.	2020	Blocker 4F Fungicide Magnitude of the Residues in or on Potato Raw Agricultural Commodities After Application of Pentachloronitrobenzene Fungicide – United States, 2019- Final Report; Lange Research and Consulting, Inc. Report No. LR19391 AMVAC GLP, unpublished 08.07.2020
900-RES-226	Lizotte R.	2020	Storage Stability Study for Pentachloronitrobenzene and Two Metabolites in Broccoli and Potato; Battelle Report No. 100131724 AMVAC GLP, unpublished 23.07.2020

SPIROMESIFEN (294)

The first draft was prepared by Dr Chris Anagnostopoulos, Benaki Phytopathological Institute, Greece

EXPLANATION

Spiromesifen is a contact insecticide-acaricide belonging to the titronic acid class of compounds. The mode of action is inhibition of lipid biosynthesis, especially triglycerides and free fatty acids.

Spiromesifen was first evaluated by the 2016 JMPR where an ADI of 0–0.03 mg/kg bw was established and an ARfD was determined to be unnecessary. The residue definition for compliance with MRLs for plant and animal commodities and for dietary risk assessment for animal commodities is sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen. For dietary risk assessment for plant commodities, the residue definition is sum of spiromesifen, spiromesifen-enol and 4-hydroxymethyl-spiromesifen-enol (free and conjugated), expressed as spiromesifen. The residue is fat-soluble.

Spiromesifen was scheduled at the Fifty-second Session of the CCPR for evaluation of additional uses by the 2022 JMPR. The Meeting received information on GAP, analytical methods, storage stability data, processing studies and residue trials on oranges, mango, papaya, legume vegetables and pulses.

RESIDUE ANALYSIS

Analytical methods

Method 00631 was evaluated by the 2016 JMPR and successfully validated in high water content (broccoli, cucumber, pepper, melon, beans and tomato), high starch content (corn/maize, sugar beet), high oil content (cotton), high acid content (strawberry) commodities and tea. Additional method validation data are available as part of the supervised residue trials relied upon in this submission, these data are summarised in Table 1. The initial method did not use a clean-up step if analysis was occurring using HPLC -MS/MS (due to high sensitivity of the instrument used). Modifications to the Method 00631, resulting in Method 00631/M001, were the inclusion of the clean-up step regardless of the instrumentation being used, with the addition of the deuterated internal standard occurring.

Method BS001-P09-01 is a modification of analytical method 00631. The method was evaluated by the 2016 JMPR and successfully validated in high starch content commodities (wheat: grain, aspirated grain fractions, bran, flour, germ, middling's, shorts; and sorghum: grain and aspirated grain fractions). Additional method validation data for the modified method BS001-P09-02 are available as part of the citrus fruit and soybean supervised residue trials relied upon in this submission and are summarised in Table 1.

Method BS001-P09-02

An analytical method was developed to determine the residues of BSN2060 (spiromesifen) and BSN2060-enol (spiromesifen-enol) in crop matrices. This analytical method encompasses minor modifications and best practices adopted since the original analytical method (Method 00631) was developed.

Spiromesifen and spiromesifen-enol residues were extracted from all orange matrices except oil by blending with an acetonitrile/water mixture (4/1, v/v) as the extraction solvent. The extract was filtered into a centrifuge tube. The filtered solids were washed with an additional aliquot of acetonitrile/water (4/1, v/v) and the solvent was added to the extract. The extract was amended with

stable isotopically labelled internal standards. A 0.5 mL aliquot of the extract was transferred to a HPLC vial and diluted with 0.75 mL of water. The samples were analysed by high performance liquid chromatography/tandem mass spectrometry (LC/MS/MS).

For the orange oil, a 0.5 g aliquot of sample was diluted with acetone and vortexed to mix. The extract was amended with stable isotopically labelled internal standards and further diluted with acetonitrile and mixed. An aliquot of the extract was purified by passing through tandem C18 cartridges, evaporated to dryness, and reconstituted in ACN/water (1:1) and analysed by LC/MS/MS.

The method was successfully validated by analysis of blank untreated control (UTC) orange whole fruit samples and blank untreated control (UTC) orange processed samples fortified with 0.010 ppm of BSN2060 and BSN2060-enol. Additionally, blank untreated control (UTC) orange oil samples fortified with 0.10 and 0.50 ppm of BSN2060 and BSN2060-enol were validated. These data support a method limit of quantitation (LOQ) of 0.010 ppm for BSN2060 and BSN2060-enol in orange fruit and all orange-processed commodities except oil, and an LOQ for both analytes of 0.1 ppm in orange oil. The LOQ is the lowest fortification level at which acceptable recovery was achieved.

The calibration was validated for spiromesifen (BSN2060) and BSN2060-enol with linear response in solvent over the range of 0.0050 ppm to 1.0 ppm, in parent equivalents (solution concentrations equivalent to 0.00020 to 0.040 µg/mL) ($R^2 \geq 0.99$). This validation was carried out alongside the analysis of residues in orange processed commodities. As part of this study procedural recoveries were carried out for whole fruit and processed commodities at the LOQ (n = 3-7) and a higher level (n = 4-5). These results are summarised in Table 1.

Table 1 Summary of validation data for methods BS001-P09-01, BS001-P09-02 and 00631

Commodity	Matrix	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Spiromesifen (BSN2060)								
Orange	Fruit	0.01	7	86-96	92	3.7	LC-MS/MS (BS001-P09-01)	M-627001-01-1
		0.25	5	92-99	96	2.7		
Orange	Dried pomace	0.01	5	84-107	90	11	LC-MS/MS (BS001-P09-02)	M-762109-01-1
		0.75	5	76-97	88	11		
	Dry pulp	0.01	5	79-83	80	2.3		
		0.75	5	86-96	92	4.7		
	Juice	0.01	5	99-100	100	0.9		
		0.10	5	83-101	95	7.8		
	Marmalade	0.01	7	87-105	99	5.8		
		0.10	5	97-103	100	2.2		
	Oil	0.50	7	100-108	103	2.6		
		72.5	5	88-97	93	4.7		
	Peel	0.01	5	89-92	91	1.7		
		1.00	5	84-94	90	4.3		
	Peel (washed)	0.01	5	93-99	97	2.2		
	Pulp	0.01	5	98-102	100	2.2		
		0.10	5	95-100	97	1.8		
	Wet pomace	0.01	5	91-95	93	1.6		
		0.20	5	92-94	93	1.3		
	Whole fruit (unwashed)	0.01	4	83-89	87	3.2		
	Whole fruit (washed)	0.01	3	87-91	90	2.8		
		0.40	5	94-97	96	1.9		

Commodity	Matrix	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Orange	Dried pulp	0.01	7	79-90	85	6	LC-MS/MS (BS001-P09-02)	M-635467-01-1
		0.80	5	93-98	95	2		
	Raw juice	0.01	7	90-102	97	4		
		0.10	5	99-103	101	2		
	Marmalade	0.01	7	88-97	94	4		
		0.10	5	92-99	96	3		
	Oil	0.1	7	96-105	99	3		
		150	5	93-95	94	1		
	Whole fruit	0.01	7	88-102	94	5		
		0.80	5	96-98	97	1		
Mango	Whole fruit	0.01	5	84-91	87	3	LC-MS/MS (00631)	M-675094-01-1
		0.1	5	83-91	86	4		
		2.0	5	79-85	83	3		
	Peel	0.01	3	91-100	94	5		
		0.1	3	88-95	91	4		
		2.0	3	96-90	88	2		
	Pulp	0.01	3	89-91	91	1		
		0.1	3	89-93	91	3		
Dry beans	Shelled beans	0.01	2	71-76	74	n/a	LC-MS/MS (00631)	M-282963-01-1
		0.10	3	70-92	81	14		
		5.0	3	77-82	79	3.3		
Succulent shelled beans	Beans	0.05	3	70-91	80	13		
		0.1	3	69-75	71	4.5		
Edible podded beans	Beans with pods	0.05	3	64-67	66	2.6		
		0.1	3	63-69	66	4.5		
Bean foliage	Vines	0.05	3	57-66	62	7.6		
		0.1	3	64-71	67	5.2		
		5.0	3	80-90	85	5.9		
Soybean	Dry seed	0.01	10	88-113	105	6.9	LC-MS/MS (BS001-P09-01)	M-600100-01-1
		1.0	8	103-132	113	10		
Spiromesifen-enol (BSN2060-enol), expressed as spiromesifen[a]								
Orange	Fruit	0.01	7	92-96	94	1.5	LC-MS/MS (BS001-P09-01)	M-627001-01-1
Orange	Dried pomace	0.014	5	81-86	83	2.4	LC-MS/MS (BS001-P09-02)	M-762109-01-1
		1.05	5	72-96	87	12		
	Dry pulp	0.014	5	77-82	80	2.4		
		1.05	5	89-96	92	3.4		
	Juice	0.014	5	94-99	97	1.9		
		0.14	5	81-100	94	7.9		
	Marmalade	0.014	7	83-101	97	6.5		
		0.154	5	97-101	99	1.4		
	Oil	0.68	7	94-103	100	3.0		
		98.6	5	89-98	94	4.4		
	Peel	0.014	5	91-97	93	2.3		
		1.4	5	85-94	91	3.6		
	Peel washed	0.014	5	89-99	95	4.5		
	Pulp	0.014	5	97-100	99	1.3		
		0.14	5	93-95	94	1.1		

Commodity	Matrix	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
	Wet pomace	0.014	5	94-101	97	2.9		
		0.28	5	90-92	91	1.0		
	Whole fruit (unwashed)	0.014	4	79-93	85	7.9		
	Whole fruit (washed)	0.014	3	81-89	85	4.7		
		0.54	5	92-95	93	1.2		
Orange	Dried pulp	0.014	7	82-96	90	5	LC-MS/MS (BS001-P09-02)	M-635467-01-1
		1.10	5	93-97	95	2		
	Raw juice	0.014	7	94-104	99	4		
		0.14	5	99-101	100	1		
	Marmalade	0.014	7	93-102	98	3		
		0.14	5	89-96	94	3		
	Oil	0.14	7	102-110	106	3		
		200	5	93-95	94	1		
	Whole fruit	0.014	7	93-114	102	7		
		1.10	5	98-101	99	1		
Mango	Whole fruit	0.014	5	83-90	86	4	LC-MS/MS (00631)	M-675094-01-1
		0.14	5	82-85	83	1		
		2.8	5	80-84	81	2		
	Peel	0.014	3	85-95	90	6		
		0.14	3	90-95	93	3		
		2.8	3	94-99	96	3		
	Pulp	0.014	3	92-95	93	2		
		0.14	3	90-96	92	3		
Dry beans	Shelled beans	0.014	2	91-95	93	n/a	LC-MS/MS (00631)	M-282963-01-1
		0.14	3	74-89	81	9.3		
		7.0	3	91-102	98	6.2		
Succulent shelled beans	Beans	0.07	3	91-136	114	20		
		0.14	3	92-102	98	7.4		
Edible podded beans	Beans with pods	0.07	3	97-99	98	1.2		
		0.14	3	88-101	96	7.1		
Bean foliage	Vines	0.07	3	100-120	108	10		
		0.14	3	93-95	94	1.2		
		7.0	3	96-102	100	3.2		
Soya bean	Dry, seed	0.010	7	84-103	95	6.4	LC-MS/MS (BS001-P09-01)	M-600100-01-1
		0.014	10	77-93	87	6.2		
		1.36	8	87-105	98	6.1		

Notes:

[a] Fortification values were often given in the reports expressed as analyte – here the fortification levels are expressed as parent spiromesifen – were appropriate, values have been calculated using a molecular weight correction factor of 1.36 (derived from the respective molecular weights of spiromesifen (370.5 g/mol) and spiromesifen-enol (272.3 g/mol))

STABILITY OF RESIDUES IN STORED ANALYTICAL SAMPLES

The Meeting received additional data on the storage stability of spiromesifen and spiromesifen-enol under frozen conditions (Sarti, A., 2016, M-553911-01-2).

The stability of total residues (sum of spiromesifen and spiromesifen-enol) on dry bean, coffee beans and citrus fruit is submitted for evaluation and summarised below. In this study, samples of dry

bean, coffee and citrus fruit were homogenised and fortified at 0.1 mg/kg with spiromesifen and Spiromesifen-enol, before being stored at <-18 °C for approximately 24 months. Samples were analysed at appropriate intervals using the validated analytical method 00631. Analysis of the samples showed that there also appeared to be a conversion of spiromesifen to spiromesifen- enol; up to 80 percent until 741 days of storage for dry bean samples and up to 27 percent until 755 days of storage for coffee samples. This conversion was not observed for citrus samples up to 728 days. Nevertheless, after a storage period of 24 months under deep-freezer conditions, the total residues of spiromesifen and spiromesifen-enol were well recovered from all matrices tested. Storage stability data on dry bean, coffee and citrus area summarised in Tables 2 to 10 below.

Table 2 Storage Stability Data for Spiromesifen in Dry bean (seed)

Sample Material	Storage Period [days]	Residue Level in Stored Samples				Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		mg/kg (ppm)	% of nominal spiking level	Average recovery [%]	RSD [%]			
Dry bean (seed)	**Spiromesifen**							
	0	0.0739 0.0839 0.0754 0.0764 0.0858	74 84 75 76 86	79	7.0	100	-	-
	30	0.0517 0.0509 0.0602	52 51 60	54	9.1	68	88	61
	58	0.0478 0.0320 0.0426	48 32 43	41	20.0	52	87	47
	90	0.0412 0.0414 0.0489	41 41 49	44	10.6	56	84	52
	275	0.0398 0.0413 0.0210	40 41 21	34	33.1	43	88	39
	377	0.0204 0.0191 0.0178	20 19 18	19	5.3	24	96	20
	544	0.0170 0.0152 0.0192	17 15 19	17	11.8	22	85	20
	741	0.0200 <0.010 0.0144	20 <10 14	<15	34.3	<19	99	<15

Notes:

Mean values were calculated with unrounded values. Therefore minor deviations may occur when the values given in the table are used.

[a] Day-0 Normalized Recovery [%] = (Average Recovery [%] / Average recovery at Day-0 [%])× 100

[b] Average Corrected Recovery [%] = (Average Recovery [%] / Average of Concurrent Recoveries [%])× 100

Table 3 Storage Stability Data for Spiromesifen-enol in Dry bean (seed)

Sample Material	Storage Period [days]	Residue Level in Stored Samples				Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		mg/kg (ppm)	% of nominal spiking level	Average recovery [%]	RSD [%]			
Dry bean (seed)	**Spiromesifen-enol**							
	0	0.0802 0.0810 0.0823 0.0804 0.0821	80 81 82 80 82	81	1.2	100	88	92
	30	0.0884 0.0929 0.0981	88 93 98	93	5.4	115	97	96
	58	0.0968 0.0684 0.0626	97 68 63	76	24.2	94	95	80
	90	0.0728 0.0623 0.0669	73 62 67	67	8.2	83	87	77
	275	0.0694 0.0590 0.0892	69 59 89	72	21.1	89	93	77
	377	0.0553 0.0595 0.0529	55 60 53	56	6.4	69	101	55
	544	0.0823 0.0781 0.0802	82 78 80	80	2.5	99	89	90
	741	0.0807 0.0910 0.0819	81 91 82	85	6.5	105	90	94

Notes:

Mean values were calculated with unrounded values. Therefore minor deviations may occur when the values given in the table are used.

[a] Day-0 Normalized Recovery [%] = (Average Recovery [%] / Average recovery at Day-0 [%]) × 100.

[b] Average Corrected Recovery [%] = (Average Recovery [%] / Average of Concurrent Recoveries [%])× 100.

Table 4 Storage Stability Data for Spiromesifen (Total Residue) in Dry bean (seed)

Sample Material	Storage Period [days]	Residue Level in Stored Samples					Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		Spirome-sifen-enol mg/kg (ppm)	Total Residue mg/kg (ppm)[c]	% of nominal spiking level	Average recovery [%]	RSD [%]			
Dry bean (seed)	**Spiromesifen (Total Residue)**								
	0	<0.010 <0.010 <0.010 <0.010 <0.010	0.0739 0.0839 0.0754 0.0764 0.0858	74 84 75 76 86	79	7.0	100	84	94
	30	0.0115 0.0111 0.0138	0.0672 0.0659 0.0789	67 66 79	71	10.2	90	88	81
		0.0234	0.0796	80					

Sample Material	Storage Period [days]	Residue Level in Stored Samples					Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		Spiromesifen-enol mg/kg (ppm)	Total Residue mg/kg (ppm)[c]	% of nominal spiking level	Average recovery [%]	RSD [%]			
	58	0.0149 0.0171	0.0523 0.0659	52 66	66	21.2	84	87	76
	90	0.0292 0.0244 0.0271	0.0809 0.0746 0.0858	81 75 86	81	6.8	103	84	96
	275	0.0307 0.0313 0.0438	0.0816 0.0839 0.0806	82 84 81	82	1.9	104	88	93
	377	0.0527 0.0501 0.0459	0.0921 0.0872 0.0802	92 87 80	86	7.0	109	96	90
	544	0.0468 0.0450 0.0366	0.0806 0.0764 0.0690	81 76 69	75	8.0	95	85	88
	741	0.0351 0.0587 0.0472	0.0677 0.0798 0.0786	68 80 79	76	8.8	96	99	77

Notes:

Mean values were calculated with unrounded values. Therefore minor deviations may occur when the values given in the table are used.

[a] Day-0 Normalized Recovery [%] = (Average Recovery [%] / Average recovery at Day-0 [%]) X 100

[b] Average Corrected Recovery [%] = (Average Recovery [%] / Average of Concurrent Recoveries [%]) X 100

[c] This value consider the sum of Spiromesifen and Spiromesifen-enol (expressed as Spiromesifen equivalents) to each sample when the Spiromesifen-enol measured is at least 95% of the LOQ. The conversion factor of Spiromesifen-enol to Spiromesifen is 1.36

Table 5 Storage Stability Data for Spiromesifen in Coffee (grain)

Sample Material	Storage Period [days]	Residue Level in Stored Samples				Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		mg/kg (ppm)	% of nominal spiking level	Average recovery [%]	RSD [%]			
Coffee beans	**Spiromesifen**							
	0	0.0738 0.0824 0.0821 0.0808 0.0790	74 82 82 81 79	80	4.2	100	87	92
	29	0.0899 0.0954 0.0881	90 95 88	91	4.0	114	98	93
	92	0.0822 0.0833 0.0772	82 83 77	81	4.0	101	96	84
	294	0.0788 0.0749 0.0826	79 75 83	79	5.1	99	93	85
	361	0.0799 0.0749 0.0726	80 75 73	76	4.7	95	92	83

Sample Material	Storage Period [days]	Residue Level in Stored Samples				Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		mg/kg (ppm)	% of nominal spiking level	Average recovery [%]	RSD [%]			
	559	0.0744 0.0680 0.0646	74 68 65	69	6.6	86	86	80
	755	0.0724 0.0854 0.0766	72 85 77	78	8.4	98	104	75

Notes:

Mean values were calculated with unrounded values. Therefore minor deviations may occur when the values given in the table are used.

[a] Day-0 Normalized Recovery [%] = (Average Recovery [%] / Average recovery at Day-0 [%]) X 100

[b] Average Corrected Recovery [%] = (Average Recovery [%] / Average of Concurrent Recoveries [%]) X 100

Table 6 Storage Stability Data for Spiromesifen-enol in coffee bean

Sample Material	Storage Period [days]	Residue Level in Stored Samples				Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		mg/kg (ppm)	% of nominal spiking level	Average recovery [%]	RSD [%]			
Coffee (grain)	**Spiromesifen-enol**							
	0	0.0760 0.0789 0.0771 0.0791 0.0816	76 79 77 79 82	79	2.9	100	93	85
	29	0.0834 0.0825 0.0881	83 83 88	85	3.4	108	106	80
	92	0.0777 0.0763 0.0806	78 76 81	78	3.2	99	98	80
	294	0.0619 0.0749 0.0696	62 75 70	69	9.5	87	87	79
	361	0.0779 0.0725 0.0790	78 73 79	77	4.2	97	91	85
	559	0.0733 0.0724 0.0723	73 72 72	72	0.8	91	86	84
	755	0.0842 0.0810 0.0885	84 81 89	85	4.8	108	99	86

Notes:

Mean values were calculated with unrounded values. Therefore minor deviations may occur when the values given in the table are used.

[a] Day-0 Normalized Recovery [%] = (Average Recovery [%] / Average recovery at Day-0 [%]) X 100.

[b] Average Corrected Recovery [%] = (Average Recovery [%] / Average of Concurrent Recoveries [%]) X 100.

Table 7 Storage Stability Data for Spiromesifen (Total Residue) in coffee bean

Sample Material	Storage Period [days]	Residue Level in Stored Samples					Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		Spiromesifen-enol mg/kg (ppm)	Total Residue mg/kg (ppm)[c]	% of nominal spiking level	Average recovery [%]	RSD [%]			
Coffee (grain)	**Spiromesifen (Total Residue)**								
0		<0.010	0.0738	74	80	4.2	100	87	92
		<0.010	0.0824	82					
		<0.010	0.0821	82					
		<0.010	0.0808	81					
		<0.010	0.0790	79					
29		<0.010	0.0899	90	91	4.0	114	98	93
		<0.010	0.0954	95					
		<0.010	0.0881	88					
92		<0.010	0.0822	82	81	4.0	101	96	84
		<0.010	0.0833	83					
		<0.010	0.0772	77					
294		<0.010	0.0788	79	79	5.1	99	93	85
		<0.010	0.0749	75					
		<0.010	0.0826	83					
361		0.0108	0.0946	95	90	4.6	113	92	98
		0.0101	0.0886	89					
		0.0105	0.0869	87					
559		<0.010	0.0744	74	81	7.9	101	86	94
		0.0135	0.0864	86					
		0.0139	0.0835	84					
755		0.0196	0.0991	99	99	0.6	124	104	95
		0.0109	0.1002	100					
		0.0161	0.0985	99					

Notes:

Mean values were calculated with unrounded values. Therefore minor deviations may occur when the values given in the table are used.

[a] Day-0 Normalized Recovery [%] = (Average Recovery [%] / Average recovery at Day-0 [%]) X 100

[b] Average Corrected Recovery [%] = (Average Recovery [%] / Average of Concurrent Recoveries [%]) X 100

[c] This value considers the sum of Spiromesifen and Spiromesifen-enol (expressed as Spiromesifen equivalents) to each sample when the Spiromesifen-enol measured is at least 95% of the LOQ. The conversion factor of Spiromesifen-enol to Spiromesifen is 1.36

Table 8 Storage Stability Data for Spiromesifen in Citrus (fruit)

Sample Material	Storage Period [days]	Residue Level in Stored Samples				Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		mg/kg (ppm)	% of nominal spiking level	Average recovery [%]	RSD [%]			
Citrus (fruit)	**Spiromesifen**							
0		0.0953	95	96	0.6	100	89	108
		0.0958	96					
		0.0960	96					
		0.0959	96					
		0.0946	95					
29		0.0878	88	88	0.7	92	91	97
		0.0877	88					

Sample Material	Storage Period [days]	Residue Level in Stored Samples				Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		mg/kg (ppm)	% of nominal spiking level	Average recovery [%]	RSD [%]			
		0.0872	87					
	91	0.0912 0.0908 0.0912	91 91 91	91	0.0	95	93	98
	181	0.0982 0.100 0.0918	98 100 92	97	4.3	101	101	96
	359	0.102 0.101 0.108	102 101 108	104	3.7	108	102	102
	531	0.0880 0.0889 0.0884	88 89 88	88	0.7	92	91	97
	728	0.0989 0.101 0.100	99 101 100	100	1.0	104	104	96

Notes:

Mean values were calculated with unrounded values. Therefore minor deviations may occur when the values given in the table are used.

[a] Day-0 Normalized Recovery [%] = (Average Recovery [%] / Average recovery at Day-0 [%]) X 100

[b] Average Corrected Recovery [%] = (Average Recovery [%] / Average of Concurrent Recoveries [%]) X 100

Table 9 Storage Stability Data for Spiromesifen-enol in Citrus (fruit)

Sample Material	Storage Period [days]	Residue Level in Stored Samples				Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		mg/kg (ppm)	% of nominal spiking level	Average recovery [%]	RSD [%]			
Citrus (fruit)	**Spiromesifen-enol**							
	0	0.107 0.108 0.104 0.107 0.104	107 108 104 107 104	106	1.8	100	105	101
	29	0.0951 0.0920 0.0917	95 92 92	93	1.9	88	105	89
	91	0.0968 0.0983 0.0947	97 98 95	97	1.6	92	100	97
	181	0.100 0.0988 0.102	100 99 102	100	1.5	94	100	100
	359	0.107 0.103 0.104	107 103 104	105	2.0	99	104	101
	531	0.0823 0.0877 0.0850	82 88 85	85	3.5	80	88	97
	728	0.0876 0.0888 0.0882	88 89 88	88	0.7	83	90	98

Notes:

Mean values were calculated with unrounded values. Therefore minor deviations may occur when the values given in the table are used.

[a] Day-0 Normalized Recovery [%] = (Average Recovery [%] / Average recovery at Day-0 [%]) × 100

[b] Average Corrected Recovery [%] = (Average Recovery [%] / Average of Concurrent Recoveries [%]) × 100

Table 10 Storage Stability Data for Spiromesifen (Total Residue) in Citrus (fruit)

Sample Material	Storage Period [days]	Residue Level in Stored Samples					Day-0 Normalized Recovery [%][a]	Average of Fresh Concurrent Recoveries [%]	Average Corrected Recovery [%][b]
		Spiromesifen-enol mg/kg (ppm)	Total Residue mg/kg (ppm)[c]	% of nominal spiking level	Average recovery [%]	RSD [%]			
Citrus (fruit)	**Spiromesifen (Total Residue)**								
	0	<0.010 <0.010 <0.010 <0.010 <0.010	0.0953 0.0958 0.0960 0.0959 0.0946	95 96 96 96 95	96	0.6	100	89	108
	29	<0.010 <0.010 <0.010	0.0878 0.0877 0.0872	88 88 87	88	0.7	92	91	97
	91	<0.010 <0.010 <0.010	0.0912 0.0908 0.0912	91 91 91	91	0.0	95	93	98
	181	<0.010 <0.010 <0.010	0.0982 0.100 0.0918	98 100 92	97	4.3	101	101	96
	359	<0.010 <0.010 <0.010	0.102 0.101 0.108	102 101 108	104	3.7	108	102	102
	531	<0.010 <0.010 <0.010	0.0880 0.0889 0.0884	88 89 88	88	0.7	92	91	97
	728	<0.010 <0.010 <0.010	0.0989 0.101 0.100	99 101 100	100	1.0	104	104	96

Notes:

Mean values were calculated with unrounded values. Therefore minor deviations may occur when the values given in the table are used.

[a] Day-0 Normalized Recovery [%] = (Average Recovery [%] / Average recovery at Day-0 [%]) X 100

[b] Average Corrected Recovery [%] = (Average Recovery [%] / Average of Concurrent Recoveries [%]) X 100

[c] This value considers the sum of Spiromesifen and Spiromesifen-enol (expressed as Spiromesifen equivalents) to each sample when the Spiromesifen-enol measured is at least 95% of the LOQ. The conversion factor of Spiromesifen-enol to Spiromesifen is 1.36

USE PATTERN

The Meeting received the GAP information on oranges, mango, papaya, legume vegetables and pulses. The information is summarised in Table 11.

Table 11 Registered uses of spiromesifen

Crop	Country	Formulation	Application					Max/season			PHI days	Remarks
			Method	Rate kg ai/ha (max)	Spray conc. kg as/hL (max)	Water L/ha (min)	timing	No.	Interval (days)	kg ai/ha		
	Citrus fruit											
Citrus	Brazil	Oberon® (Spiromesifen SC (240 g/L))	Broadcast spray	0.096-0.144	0.005-0.007	2000	at infestation BBCH not stated in label[1]	1	-	0.144	21	
	Tropical and sub-tropical fruits											
Mango	Brazil	Oberon® (Spiromesifen SC (240 g/L))	Broadcast spray	0.120-0.144	0.012-0.029	500-1000	at infestation BBCH not stated in label[2]	3	7	0.144	5	
Papaya	Brazil	Oberon® (Spiromesifen SC (240 g/L))	Broadcast spray	0.120-0.144	0.012-0.029	500-1000	at infestation BBCH not stated in label[2]	3	7	0.144	5	
	Legumes											
Succulent shelled and edible podded beans[3]	Canada	Oberon™ Flowable Insecticide-Miticide (Spiromesifen SC (240 g/L))	Broadcast spray	0.120-0.144	-	Min. 100 (ground) Min. 50 (aerial)	at infestation BBCH not stated in label	3	7	0.432	1	Forage from treated crops may be used for animal consumption
	Pulses											
Dry shelled beans[3]	Canada	Oberon™ Flowable Insecticide-Miticide (Spiromesifen SC (240 g/L))	Broadcast spray	0.120-0.144	-	Min. 100 (ground) Min. 50 (aerial)	at infestation BBCH not stated in label	3	7	0.432	10	Forage from treated crops may be used for animal consumption
Dry Beans	Brazil	Oberon® (Spiromesifen SC (240 g/L))	Broadcast spray	0.120-0.144	-	300 (ground) 50 (aerial)	at infestation BBCH not stated in label[4]	3	5	0.432	21	
Soybean (dry)	Brazil	Oberon® (Spiromesifen SC (240 g/L))	Broadcast spray	0.096-0.144	-	300 (ground) 50 (aerial)	at infestation BBCH not stated in label[5]	2	5	0.288	21	

Notes:

[1]For false spider mite, sample on a periodic basis and apply when the presence of mites is found in 3% of fruits or branches examined. For citrus rust mite, sample on a periodic basis and apply when the presence of mites is found in 20 to 30 mites/cm^2 in 5% of fruits examined.

[2]Applications must be started soon after crop emergence or transplantation, according to monitoring of leaves and inflorescences, early in the infestation.

[3]Lupinus spp., includes grain lupin, sweet lupin, white lupin, and white sweet lupin; Phaseolus spp., includes: field bean, kidney bean, lima bean, navy bean, pinto bean, runner bean, snap bean, tepary bean, wax bean; Vigna spp., includes: adzuki bean, asparagus bean, blackeyed pea, catjang, Chinese longbean, cowpea, Crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean, yardlong bean; broad bean (fava), chickpea (garbanzo bean), guar, jackbean, lablab bean (hyacinth bean), lentil, soybean (immature seed), sword bean.

[4]For broad mite, the application must be made early in the infestation, when the first forms of pest development are seen in monitoring. For white fly, monitor and start applications early in the infestation, when the presence of eggs, first "nymphs" or young forms is found, or 7-10 days after crop emergence with the presence of the pest. The lowest dose must be used in preventive applications, i.e., when the occurrence of the pest is anticipated in the crop, however, it is not present yet in the crop. The highest dose must be used under higher pressure conditions or when there is a history of pest occurrence. In case of reinfestation, re-apply within a 5-7-day interval.

[5]For two-spotted spider mite, the application must be made early in the infestation, when the first forms of pest development are seen in monitoring. For white fly, monitor and start applications early in the infestation, when the presence of eggs, first "nymphs" or young forms is found. The lowest dose must be used in preventive applications, i.e., when the occurrence of the pest is anticipated in the crop, however, it is not present yet in the crop. The highest dose must be used under higher pressure conditions or when there is a history of pest occurrence. In case of reinfestation, re-apply within a 5-7-day interval.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received residue trials on oranges, mango, papaya, legume vegetables and pulses. The detailed information is summarised below.

Citrus fruit

A total of 13 supervised trials were carried out on oranges in Brazil during 2015-2018 (Ref: M-571855-02-1, M-627001-01-1 and M-764414-01-1). Each trial consisted of 2 plots; a control untreated plot and a plot treated with a single broadcast spray application of an SC (suspension concentrate formulation) containing 230–240 g ai/L. Each treated plot received a single application at a nominal rate of 144 g ai/ha. No adjuvants were added to the formulation prior to application.

Samples of fruit were collected from all trials 21 days after the last application. Samples were also collected from 11 trials at 7, 14, 27-28 and 33-37 days after the last application, to provide residue decline data. Whole fruits were collected and placed into frozen storage.

Samples were maintained frozen at <-20 °C for a maximum of 302 days (*ca* 10 months) prior to analysis. The storage stability of spiromesifen residues has been sufficiently investigated (see Section 4.0), therefore, it can be assumed that residues (sum of spiromesifen and its metabolites) were stable when analysed.

Residues of spiromesifen and its metabolite spiromesifen-enol were determined by LC- MS/MS, using the validated analytical method 00631 (study M-571855-02-1 and M-764414-01-1) or method BS001-P09-01 (M-627001-01-1). These methods have been validated with an LOQ of 0.01 mg/kg for both analytes (expressed as analyte, resulting in an LOQ of 0.014 mg/kg for spiromesifen-enol, expressed as spiromesifen). Procedural recoveries carried out concurrently with the analyses of trial samples showed recoveries were within the acceptable range of 70–120 percent, with relative standard deviation <20 percent. The results of the trials are presented in Table 12 below. Two of the trials were co-located, in these cases the highest value has been selected for MRL calculations.

Table 12 Residue concentration of spiromesifen from residue trials on oranges in Brazil

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen-enol enol expressed as spiromesifen	total residue spiromesifen calc. as spiromesifen(1)	
GAP, Brazil	SC 240	0.144			1	21					
I15-094-01 Brazil, 2016 Paulinia America, South (Pera Rio)	240 g/L SC	0.144	0.007	1994	1	7	Whole fruit	0.068, 0.086 (0.077)	<0.014, <0.014 (<0.014)	0.082, 0.010 (0.046)	M-571855-02-1
						14		0.042, 0.055 (0.049)	<0.014, <0.014 (<0.014)	0.056, 0.069 (0.063)	
						21		0.038, 0.027 (0.033)	<0.014, <0.014 (<0.014)	0.052, 0.041 (0.047)	
						27		0.022, 0.022 (0.022)	<0.014, <0.014 (<0.014)	0.036, 0.036 (0.036)	
						35		0.012, 0.011 (0.012)	<0.014, <0.014 (<0.014)	0.026, 0.025 (0.026)	
I15-094-02 Brazil, 2016 Ribeirao Preto America, South (Pera Rio)	240 g/L SC	0.144	0.007	2000	1	7	Whole fruit	0.071, 0.085 (0.078)	<0.014, <0.014 (<0.014)	0.085, 0.099 (0.092)	M-571855-02-1
						14		0.055, 0.048 (0.052)	<0.014, <0.014 (<0.014)	0.069, 0.062 (0.066)	
						21		0.028, 0.027 (0.028)	<0.014, <0.014 (<0.014)	0.042, 0.041 (0.042)	
						27		0.013, 0.017 (0.015)	<0.014, <0.014 (<0.014)	0.027, 0.031 (0.029)	
						37		<0.010, 0.011 (0.011)	<0.014, <0.014 (<0.014)	<0.024, 0.025 (<0.025)	
I15-094-03 Brazil, 2016 Sao Carlos America, South (Valencia)	240 g/L SC	0.147	0.007	2042	1	7	Whole fruit	0.024, 0.029 (0.027)	<0.014, <0.014 (<0.014)	0.038, 0.043 (0.041)	M-571855-02-1
						14		0.012, <0.010 (0.011)	<0.014, <0.014 (<0.014)	0.026, <0.024 (<0.025)	
						21		0.011, <0.010 (0.011)	<0.014, <0.014 (<0.014)	0.025, <0.024 (0.025)	
						28		<0.010,<0.010 (<0.010)	<0.014, <0.014 (<0.014)	<0.024, <0.024 (<0.024)	
						37		<0.010, <0.010 (<0.010)	<0.014, <0.014 (<0.014)	<0.024, <0.024 (<0.024)	
I15-094-04 Brazil, 2016 Holambra America, South (Taiti)	240 g/L SC	0.141	0.007	1964	1	21	Whole fruit	0.035, 0.027 (0.031)	<0.014, <0.014 (<0.014)	0.049, 0.041 (0.045)	M-571855-02-1
I15-094-05 Brazil, 2016 Ituverava America, South (Pera Rio)	240 g/L SC	0.149	0.007	2074	1	21	Whole fruit	0.023, 0.031 (0.027)	<0.014, <0.014 (<0.014)	0.037, 0.045 (0.041)	M-571855-02-1
BS001-17DA BS001-17DA-TRTD Brazil, 2017 Paranavaí America, South (Folha Murchar)	240 g/L SC	0.140	0.010	1441	1	7	Whole fruit	0.058, 0.043 (0.051)	<0.010, <0.010 (<0.010)	0.068, 0.053 (0.061)	M-627001-01-1
						14		0.072, 0.038 (0.055)	<0.010, <0.010 (<0.010)	0.082, 0.048 (0.065)	
						21		0.011, <0.010 (0.011)	<0.010, <0.010 (<0.010)	0.021, <0.020 (0.021)	
						28		<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
						34		<0.010, <0.010 (<0.010)	<0.010, <0.010 (<0.010)	<0.020, <0.020 (<0.020)	
BS002-17DA BS002-17DA-TRTD Brazil, 2017 Nova Esperança America, South	240 g/L SC	0.140	0.010	1444	1	7	Whole fruit	0.069, 0.062 (0.066)	<0.010, <0.010 (<0.010)	0.079, 0.072 (0.076)	M-627001-01-1
						14		0.120, 0.043 (0.082)	<0.010, <0.010 (<0.010)	0.130, 0.053 (0.092)	
						21		0.041, 0.024	<0.010, <0.010	0.051, 0.034	

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen-enol enol expressed as spiromesifen	total residue spiromesifen calc. as spiromesifen(1)	
GAP, Brazil	SC 240	0.144			1	21					
(Valencia)								(0.033)	(<0.010)	(0.043)	
						28		0.030, 0.023 (0.027)	<0.010, <0.010 (<0.010)	0.040, 0.033 (0.037)	
						34		0.033, 0.019 (0.026)	<0.010, <0.010 (<0.010)	0.043, 0.029 (0.036)	
BS003-17DA BS003-17DA-TRTD Brazil, 2017 Conchal America, South (Pera)	240 g/L SC	0.140	0.010	1464	1	7	Whole fruit	0.207, 0.242 (0.225)	<0.010, <0.010 (<0.010)	0.217, 0.252 (0.235)	M-627001-01-1
						14		0.107, 0.116 (0.112)	<0.010, <0.010 (<0.010)	0.117, 0.126 (0.122)	
						21		0.105, 0.080 (0.093)	<0.010, <0.010 (<0.010)	0.115, 0.090 (0.103)	
						28		0.057, 0.060 (0.059)	<0.010, <0.010 (<0.010)	0.067, 0.070 (0.069)	
						35		0.052, 0.050 (0.051)	<0.010, <0.010 (<0.010)	0.062, 0.060 (0.061)	
BS004-17DA BS004-17DA-TRTD Brazil, 2017 Aguaí America, South (Folha Murcha)	240 g/L SC	0.140	0.010	1467	1	7	Whole fruit	0.102, 0.076 (0.089)	<0.010, <0.010 (<0.010)	0.112, 0.086 (0.099)	M-627001-01-1
						14		0.062, 0.038 (0.050)	<0.010, <0.010 (<0.010)	0.072, 0.048 (0.060)	
						21		0.041, 0.032 (0.037)	<0.010, <0.010 (<0.010)	0.051, 0.042 (0.047)	
						28		0.029, 0.023 (0.026)	<0.010, <0.010 (<0.010)	0.039, 0.033 (0.036)	
						35		0.026, 0.022 (0.024)	<0.010, <0.010 (<0.010)	0.036, 0.032 (0.034)	
I16-056-01 Brazil, 2017 Paulinia, SP, America, South (Péra Rio)	230 g/L SC	0.143	0.007	2076	1	7	Whole fruit	0.10	<0.014	0.11	M-764414-01-1
						14		0.092	<0.014	0.11	
						21		0.065	<0.014	0.079	
						28		0.062	<0.014	0.076	
						33		0.089	<0.014	0.10	
I16-056-02 Brazil, 2017 Ribeirão Preto, SP, America, South (Péra Rio)	230 g/L SC	0.137	0.007	2012	1	7	Whole fruit	0.047	<0.014	0.061	M-764414-01-1
						14		0.014	<0.014	0.028	
						21		<0.010	<0.014	<0.024	
						28		<0.010	<0.014	<0.024	
						35		<0.010	<0.014	<0.024	
I16-056-04 Brazil, 2017 Paulinia, SP, America, South (Valencia)	230 g/L SC	0.138	0.007	2013	1	7	Whole fruit	0.035	<0.014	0.049	M-764414-01-1
						14		0.034	<0.014	0.048	
						21		0.035	<0.014	0.049	
						26		0.013	<0.014	0.027	
						35		0.016	<0.014	0.030	
I16-056-05 Brazil, 2017 São Carlos, SP, America, South (Valencia)	230 g/L SC	0.139	0.007	2030	1	7	Whole fruit	0.031	<0.014	0.045	M-764414-01-1
						14		0.012	<0.014	0.026	
						21		<0.010	<0.014	<0.024	
						28		<0.010	<0.014	<0.024	
						35		<0.010	<0.014	<0.024	

Notes:

(1) Mean residue values from duplicate samples presented in parenthesis.

Mango

A total of five supervised trials were carried out on mango in Brazil during 2019 (M-675094-01-1). Each trial consisted of 2 plots; a control untreated plot and a plot treated with broadcast spray applications of

Oberon® 2 SC (a suspension concentrate formulation containing 240 g ai/L). Each plot received 3 applications at a nominal rate of 144 g ai/ha, applications were made with an interval of 7 days. No adjuvants were added to the formulation prior to application. For the five trials, two were conducted in Petrolina region (007SRBR18R03-01 and 007SRBR18R03-02) and two in Juazeiro region (007SRBR18R03-04 and 007SRBR18R03-05), however trials are more than 20 km from one another and different varieties were used, thus can be considered as independent.

Samples of mango were collected from all trials 5 days after the 1st application, 5 days after the 2nd application and then 3, 5, 7, 10 and 14 days after the last application. After collection of whole fruit, the stones were removed and samples separated into peel and pulp fractions, before being placed into frozen storage.

Samples were maintained frozen at <-20 °C for a maximum of 150 days (*ca* 5 months) prior to analysis. The storage stability of spiromesifen residues has been sufficiently investigated (see Stability section), therefore, it can be assumed that residues (sum of spiromesifen and its metabolites) were stable when analysed.

Residues of spiromesifen and its metabolite spiromesifen-enol were determined by LC- MS/MS, using the validated analytical method 00631. The method has been validated with an LOQ of 0.01 mg/kg for both analytes (expressed as analyte, resulting in an LOQ of 0.014 mg/kg for spiromesifen-enol, expressed as spiromesifen). Procedural recoveries carried out concurrently with the analyses of trial samples showed recoveries were within the acceptable range of 70–120 percent, with relative standard deviation <20 percent. The results of the trials are presented in Table 13 below.

Table 13 Residue concentration of spiromesifen from residue trials on mangoes in Brazil

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol expressed as spiromesifen	total residue spiromesifen calc. as spiromesifen[(1)]	
GAP, Brazil	SC 240	0.144			3	5					
RABS0162, 007SRBR18R03-01-TRTD Brazil, 2019 Petrolina America, South (Palmer)	240 g/L SC	0.140 0.148 0.149	0.018 0.018 0.018	729 821 827	3	5DAA1	Fruit	0.057	<0.014	0.071	M-675094-01-1
						5DAA2		0.062	<0.014	0.076	
						3		0.094, 0.075 (0.085)	<0.014, <0.014 (<0.014)	0.108, 0.089 (0.099)	
						5		0.050, 0.062 (0.056)	<0.014, <0.014 (<0.014)	0.064, 0.076 (0.070)	
						7		0.023, 0.053 (0.038)	<0.014, <0.014 (<0.014)	0.037, 0.067 (0.052)	
						10		0.044, 0.085 (0.065)	<0.014, <0.014 (<0.014)	0.058, 0.099 (0.079)	
						14		0.024, 0.018 (0.021)	<0.014, <0.014 (<0.014)	0.038, 0.032 (0.035)	
						5DAA1	Peel	0.23	<0.014	0.24	
						5DAA2		0.37	<0.014	0.38	
						3		0.55	<0.014	0.56	
						5		0.45, 0.25 (0.35)	<0.014, <0.014 (<0.014)	0.46, 0.26 (0.36)	
						7		0.38	<0.014	0.39	
						10		0.21	<0.014	0.22	
						14		0.32	<0.014	0.33	
						5DAA1	Pulp	<0.01	<0.014	<0.024	
						5DAA2		<0.01	<0.014	<0.024	
						3		<0.01	<0.014	<0.024	
						5		<0.01, <0.01 (<0.01)	<0.014, <0.014 (<0.014)	<0.024, <0.024 (<0.024)	

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol expressed as spiromesifen	total residue spiromesifen calc. as spiromesifen[(1)]	
GAP, Brazil	SC 240	0.144			3	5					
						7		<0.01	<0.014	<0.024	
						10		<0.01	<0.014	<0.024	
						14		<0.01	<0.014	<0.024	
007SRBR18R03-02-TRTD Brazil, 2019 Petrolina America, South (Palmer)	240 g/L SC	0.148 0.142 0.143	0.018 0.018 0.018	820 791 795	3	5DAA1	Fruit	0.044	<0.014	0.058	M-675094-01-1
						5DAA2		0.098	<0.014	0.11	
						3		0.14, 0.15 (0.145)	<0.014, <0.014 (<0.014)	0.15, 0.16 (0.155)	
						5		0.11, 0.14 (0.125)	<0.014, <0.014 (<0.014)	0.12, 0.15 (0.135)	
						7		0.088, 0.084 (0.086)	<0.014, <0.014 (<0.014)	0.10, 0.098 (0.099)	
						10		0.059, 0.080 (0.070)	<0.014, <0.014 (<0.014)	0.073, 0.094 (0.084)	
						14		0.072, 0.070 (0.071)	<0.014, <0.014 (<0.014)	0.086, 0.084 (0.085)	
						5DAA1	Peel	0.25	<0.014	0.26	
						5DAA2		0.41	<0.014	0.42	
						3		0.62	<0.014	0.63	
						5		0.83, 0.55 (0.690)	<0.014, <0.014 (<0.014)	0.84, 0.56 (0.700)	
						7		0.50	<0.014	0.51	
						10		0.36	<0.014	0.37	
						14		0.29	<0.014	0.30	
						5DAA1	Pulp	<0.01	<0.014	<0.024	
						5DAA2		<0.01	<0.014	<0.024	
						3		<0.01	<0.014	<0.024	
						5		<0.01, <0.01 (<0.01)	<0.014, <0.014 (<0.014)	<0.024, <0.024 (<0.024)	
						7		<0.01	<0.014	<0.024	
						10		<0.01	<0.014	<0.024	
						14		<0.01	<0.014	<0.024	
007SRBR18R03-03-TRTD Brazil, 2019 Casa Nova America, South (Palmer)	240 g/L SC	0.150 0.144 0.147	0.0018 0.0018 0.0018	833 80 819	3	5DAA1	Fruit	0.030	<0.014	0.044	M-675094-01-1
						5DAA2		0.064	<0.014	0.078	
						3		0.13, 0.10 (0.115)	<0.014, <0.014 (<0.014)	0.14, 0.11 (0.125)	
						5		0.10, 0.11 (0.105)	<0.014, <0.014 (<0.014)	0.11, 0.12 (0.115)	
						7		0.081, 0.010 (0.046)	<0.014, <0.014 (<0.014)	0.095, 0.11 (0.103)	
						10		0.098, 0.090 (0.094)	<0.014, <0.014 (<0.014)	0.11, 0.10 (0.105)	
						14		0.086, 0.037 (0.062)	<0.014, <0.014 (<0.014)	0.10, 0.051 (0.076)	
						5DAA1	Peel	0.11	<0.014	0.12	
						5DAA2		0.37	<0.014	0.38	
						3		0.62	<0.014	0.63	
						5		0.36, 0.41 (0.385)	<0.014, <0.014 (<0.014)	0.37, 0.42 (0.395)	
						7		0.44	<0.014	0.45	
						10		0.46	<0.014	0.47	
						14		0.18	<0.014	0.19	
						5DAA1	Pulp	<0.01	<0.014	<0.024	
						5DAA2		<0.01	<0.014	<0.024	
						3		<0.01	<0.014	<0.024	
						5		<0.01, <0.01	<0.014, <0.014	<0.024, <0.024	

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol expressed as spiromesifen	total residue spiromesifen calc. as spiromesifen[(1)]	
GAP, Brazil	SC 240	0.144			3	5					
								(<0.01)	(<0.014)	(<0.024)	
						7		<0.01	<0.014	<0.024	
						10		<0.01	<0.014	<0.024	
						14		<0.01	<0.014	<0.024	
007SRBR18R03-04-TRTD Brazil, 2019 Juazeiro America, South (Kent)	240 g/L SC	0.150 0.145 0.148	0.018 0.018 0.018	833 805 826	3	5DAA1	Fruit	0.56	<0.014	0.070	M-675094-01-1
						5DAA2		0.096	<0.014	0.11	
						3		0.13, 0.15 (0.140)	<0.014, <0.014 (<0.014)	0.14, 0.16 (0.150)	
						5		0.26, 0.12 (0.190)	<0.014, <0.014 (<0.014)	0.27, 0.13 (0.200)	
						7		0.10, 0.14 (0.120)	<0.014, <0.014 (<0.014)	0.11, 0.15 (0.130)	
						10		0.098, 0.13 (0.114)	<0.014, <0.014 (<0.014)	0.11, 0.14 (0.125)	
						14		0.062, 0.13 (0.096)	<0.014, <0.014 (<0.014)	0.076, 0.014 (0.045)	
						5DAA1	Peel	0.17	<0.014	0.18	
						5DAA2		0.68	<0.014	0.69	
						3		0.59	<0.014	0.60	
						5		0.56, 0.48 (0.520)	<0.014, <0.014 (<0.014)	0.57, 0.49 (0.530)	
						7		0.76	<0.014	0.77	
						10		0.42	<0.014	0.43	
						14		0.29	<0.014	0.30	
						5DAA1	Pulp	<0.01	<0.014	<0.024	
						5DAA2		<0.01	<0.014	<0.024	
						3		<0.01	<0.014	<0.024	
						5		<0.01, <0.01 (<0.01)	<0.014, <0.014 (<0.014)	<0.024,<0.024 (<0.024)	
						7		<0.01	<0.014	<0.024	
						10		<0.01	<0.014	<0.024	
						14		<0.01	<0.014	<0.024	
007SRBR18R03-05-TRTD Brazil, 2019 Juazeiro America, South (Palmer)	240 g/L SC	0.146 0.145 0.146	0.018 0.018 0.018	813 805 812	3	5DAA1	Fruit	0.077	<0.014	0.091	M-675094-01-1
						5DAA2		<0.01	<0.014	<0.024	
						3		0.094, 0.12 (0.107)	<0.014, <0.014 (<0.014)	0.11, 0.13 (0.120)	
						5		0.20, 0.23 (0.215)	<0.014, <0.014 (<0.014)	0.21, 0.24 (0.225)	
						7		0.074, 0.12 (0.097)	<0.014, <0.014 (<0.014)	0.088, 0.13 (0.109)	
						10		0.11, 0.073 (0.092)	<0.014, <0.014 (<0.014)	0.12, 0.087 (0.104)	
						14		0.042, 0.041 (0.042)	<0.014, <0.014 (<0.014)	0.056, 0.055 (0.056)	
						5DAA1	Peel	0.24	<0.014	0.25	
						5		0.54	<0.014	0.55	
						3		0.69	<0.014	0.70	
						5		1.1, 0.79 (0.945)	<0.014, <0.014 (<0.014)	1.1, 0.80 (0.950)	
						7		0.60	<0.014	0.61	

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol expressed as spiromesifen	total residue spiromesifen calc. as spiromesifen[(1)]	
GAP, Brazil	SC 240	0.144			3	5					
						10		0.37	<0.014	0.38	
						14		0.37	<0.014	0.38	
						5DAA1	Pulp	<0.01	<0.014	<0.024	
						5DAA2		<0.01	<0.014	<0.024	
						3		<0.01	<0.014	<0.024	
						5		<0.01, <0.01 (<0.01)	<0.014, <0.014 (<0.014)	<0.024, <0.024 (<0.024)	
						7		<0.01	<0.014	<0.024	
						10		<0.01	<0.014	<0.024	
						14		<0.01	<0.014	<0.024	

Notes:

(1) – mean residue values from duplicate samples presented in parenthesis

(2) - For whole fruit samples, the stones were not analysed but the residue level is calculated assuming that they are included but contain no residue. For this, a conversion factor is calculated from the mass of the whole fruit and the mass of fruit without stone according to the equation that follows: CF = Mass without stone/Mass without fruit; The concentration of the whole fruit is calculated using the conversion factor as follows: Reportable values (mg/kg) = concentration x CF

DAA1 – Days after application 1

DAA2 – Days after application 2

Papaya

A total of five supervised trials were carried out on papaya in Brazil during 2017 (M-632116-01-1). Each trial consisted of 2 plots, a control untreated plot and a plot treated with broadcast spray applications of Oberon (a suspension concentrate formulation containing 240 g ai/L). Each plot received 3 applications at a nominal rate of 144 g ai/ha, applications were made with an interval of 7 days. No adjuvants were added to the formulation prior to application.

Samples of papaya were collected from all trials 3, 5, 7, 10 and 14 days after the last application. After collection, samples of fruit were placed into frozen storage.

Samples were maintained frozen at <-20 °C for a maximum of 212 days (*ca* 7 months) prior to analysis. The storage stability of spiromesifen residues has been sufficiently investigated (see Stability section), therefore, it can be assumed that residues (sum of spiromesifen and its metabolites) were stable when analysed.

Residues of spiromesifen and its metabolite spiromesifen-enol were determined by LC- MS/MS, using the validated analytical method 00631. The method has been validated with an LOQ of 0.01 mg/kg for both analytes (expressed as analyte, resulting in an LOQ of 0.014 mg/kg for spiromesifen-enol, expressed as spiromesifen). Procedural recoveries carried out concurrently with the analyses of trial samples showed recoveries were within the acceptable range of 70–120 percent, with relative standard deviation <20 percent. The results of the trials are presented in Table 14 below.

Table 14 Spiromesifen residues in papaya resulting from supervised trials in Brazil

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol expressed as spiromesifen	total residue spiromesifen calc. as spiromesifen	
GAP, Brazil	SC 240	0.144			3	5					
I16-046-01 Brazil, 2017 Anhumas, SP (Papaya)	240 g/L SC	0.145 0.145 0.139	0.014 0.014 0.014	1010 1010 969	3	3	Fruit	0.075	0.015	0.090	M-632116-01-1
						5		0.013	<0.014	0.027	
						7		0.085	<0.014	0.099	
						10		0.031	<0.014	0.045	
						14		0.017	<0.014	0.031	
I16-046-02 Brazil, 2017 Oswaldo Crus, SP (Formosa)	240 g/L SC	0.142 0.141 0.141	0.014 0.014 0.014	992 986 984	3	3	Fruit	0.31	0.022	0.33	M-632116-01-1
						5		0.17	0.023	0.19	
						7		0.21	0.020	0.23	
						10		0.21	0.020	0.23	
						14		0.13	0.015	0.15	
I16-042-03 Brazil, 2017 Martinópolis, SP (Formosa)	240 g/L SC	0.139 0.145 0.145	0.014 0.014 0.014	970 1008 1014	3	3	Fruit	0.018	<0.014	0.032	M-632116-01-1
						5		0.032	<0.014	0.046	
						7		0.022	<0.014	0.036	
						10		<0.01	<0.014	<0.024	
						14		<0.01	<0.014	<0.024	
I16-042-04 Brazil, 2017 Rinópolis, SP (Formosa)	240 g/L SC	0.143 0.145 0.145	0.014 0.014 0.014	1003 1008 1010	3	3	Fruit	0.20	0.020	0.22	M-632116-01-1
						5		0.30	0.029	0.33	
						7		0.17	0.018	0.19	
						10		0.098	<0.014	0.11	
						14		0.100	0.014	0.11	
I16/042-05 Brazil, 2017 Rancharia, SP (Formosa)	240 g/L SC	0.138 0.140 0.140	0.014 0.014 0.014	967 978 981	3	3	Fruit	0.15	<0.014	0.16	M-632116-01-1
						5		0.12	<0.014	0.13	
						7		0.12	<0.014	0.13	
						10		0.063	<0.014	0.077	
						14		0.034	<0.014	0.048	

Beans with pods

A total of 8 supervised trials were carried out on beans with pods in the United States during 2005 (Ref: M-282963-01-1). Each trial consisted of 2 plots; a control untreated plot and a plot treated with a broadcast spray application of Oberon® 2 SC (a suspension concentrate formulation containing 2 lb ai/gal, equivalent to 240 g ai/L). Each treated plot received 3 applications at a nominal rate of 0.19 lb ai/A (equivalent to 213 g ai/ha), applications were made with an interval of 6–14 days. In one trial, two additional applications were made at approximately 213 g ai/ha. No adjuvants were added to the formulation prior to application.

Samples of beans with pods and bean foliage (vines) were collected from all trials 1 day after the last application. Foliage samples were shaken to minimize the inclusion of loose soil in the samples. Additional samples of beans in pods were collected from 1 trial 3, 7, 10 and 14 days after the last application to provide residue decline data. All samples were then placed into frozen storage.

Samples were maintained frozen at <-20 °C for a maximum of 498 days (*ca* 16 months) prior to analysis. The storage stability of spiromesifen residues has been sufficiently investigated (see Section 4.0), therefore, it can be assumed that residues (sum of spiromesifen and its metabolites) were stable when analysed.

Residues of spiromesifen and its metabolite spiromesifen-enol were determined by LC-MS/MS, using the validated analytical method 00631. The method has been validated with an LOQ of 0.05 mg/kg for both analytes (expressed as analyte, resulting in an LOQ of 0.07 mg/kg for spiromesifen-enol, expressed as spiromesifen). Procedural recoveries carried out concurrently with the analyses of trial samples showed recoveries were generally within the acceptable range of 70–120 percent, with relative standard deviation <20 percent. The results of the trials are presented in Table 15 below. Two of the trials were co-located in Wooster, OH, therefore the highest residue value from this location has been selected for MRL calculations.

Table 15 Spiromesifen residues in beans with pods resulting from supervised trials in the United States

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)				Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol as spiromesifen	total residue spiromesifen calc. as spiromesifen(1)	*mean total residues scaled to GAP(2)*	
GAP, Canada	SC 240	0.144			3	1						
09410.05-FL42 United States, 2005 Citra America, North (Leon snap bean)	240 g/L SC	0.222 0.219 0.220	0.066 0.066 0.066	339 334 335	3	1	Seed with pod	0.07, 0.06	<0.07, <0.07	0.14, 0.13 (0.14)	*0.091*	M-282963-01-1
							Foliage	2.37, 1.57	0.62, 0.57	2.99, 2.14 (2.57)	*1.68*	
09410.05-GA*15 United States, 2005 Tifton America, North (Roma II snap bean)	240 g/L SC	0.214 0.215 0.216	0.048 0.048 0.048	450 452 454	3	1	Seed with pod	0.26, 0.39	0.11, 0.11	0.37, 0.50 (0.44)	*0.248, 0.335 (0.295)*	M-282963-01-1
							Foliage	14.9, 14.7	1.48, 1.55	16.38, 16.25 (16.32)	*10.93*	
09410.05-MD17 United States, 2005 Salisbury, Wicomico County America, North (E – Z Pick snap bean)	240 g/L SC	0.215 0.219 0.214	0.063 0.063 0.063	341 347 339	3	1	Seed with pod	0.16, 0.18	<0.07, <0.07	0.23, 0.25 (0.24)	*0.160*	M-282963-01-1
						3		0.06, 0.07	<0.07, <0.07	0.13, 0.14 (0.14)	-	
						7		<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	
						10		<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	
						14		<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	
						1	Foliage	0.77, 3.63	0.35, 0.54	1.12, 4.17 (2.65)	*1.77*	
09410.05-NY17 United States, 2005 Freeville, Tompkins County America, North (Cabernet dry bean)	240 g/L SC	0.219 0.216 0.225	0.076 0.076 0.077	289 284 295	3	1	Seed with pod	0.14, 0.24	<0.07, 0.11	0.21, 0.35 (0.28)	*0.183*	M-282963-01-1
							Foliage	3.70, 2.72	0.65, 0.50	4.35, 3.22 (3.79)	*2.48*	
09410.05-OH*14 United States, 2005 Wooster America, North (Concessa bush bean) (co-located with trial OH*16)	240 g/L SC	0.213 0.214 0.212	0.034 0.033 0.035	636 648 604	3	1	Seed with pod	<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	M-282963-01-1
							Foliage	1.45, 1.27	0.45, 0.49	1.90, 1.76 (1.83)	*1.24*	
09410.05-OH*16 United States, 2005	240 g/L SC	0.213 0.211 0.213	0.033 0.033 0.035	638 640 606	3	1	Seed with pod	<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	M-282963-01-1
							Foliage	1.16, 10.3	0.38, 0.35	1.54, 1.38	-	

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)				Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol as spiromesifen	total residue spiromesifen calc. as spiromesifen(1)	*mean total residues scaled to GAP(2)*	
GAP, Canada	SC 240	0.144			3	1						
Wooster America, North (Carson Way bean) (Co-located with trial OH*14)										(1.46)		
09410.05-SC*04 United States, 2005 Charleston America, North (Blue Lake 274 bush bean)	240 g/L SC	0.219 0.208 0.210	0.110 0.110 0.110	198 189 191	3	1	Seed with pod	0.27, 0.19	<0.07, <0.07	0.34, 0.26 (0.30)	*0.203*	M-282963-01-1
							Foliage	4.07, 7.25	0.57, 0.95	4.64, 8.20 (6.42)	*4.35*	
09410.05-WA19 United States, 2005 Prosser America, North (Blue Lake 274 edible podded bean)(3)	240 g/L SC	0.214 0.211 0.215 0.211 0.213	0.099 0.096 0.097 0.083 0.082	216 220 223 253 259	3	1	Seed with pod	0.06, <0.05	<0.07, <0.07	0.13, <0.12 (0.13)	*0.053*	M-282963-01-1
							Foliage	7.46, 9.10	0.84, 0.99	8.30, 10.09 (9.20)	*3.74*	

Notes:

(1) – mean residue values from duplicate samples presented in parenthesis

(2) – where applicable

(3) – Trial received an additional 2 applications to allow the crop time to mature – since the terminal residue in seed with pod is less than in all other trials it is clear that the additional 2 applications do not affect the terminal residue – the trial is considered acceptable and residues have been scaled according to total application rate

Beans without pods

A total of seven supervised trials were carried out on beans without pods in the United States during 2005 (Ref: M-282963-01-1). Each trial consisted of 2 plots; a control untreated plot and a plot treated with a broadcast spray application of Oberon® 2 SC (a suspension concentrate formulation containing 2 lb ai/gal, equivalent to 240 g ai/L). Each treated plot received 3 applications at a nominal rate of 0.19 lb ai/A (equivalent to 213 g ai/ha), applications were made with an interval of 6–14 days. No adjuvants were added to the formulation prior to application.

Samples of beans and bean foliage (vines) were collected from all trials 1 day after the last application. Foliage samples were shaken to minimise the inclusion of loose soil in the samples. All samples were then placed into frozen storage.

Samples were maintained frozen at <-20 °C for a maximum of 468 days (*ca* 15 months) prior to analysis. The storage stability of spiromesifen residues has been sufficiently investigated (see Section 4.0), therefore, it can be assumed that residues (sum of spiromesifen and its metabolites) were stable when analysed.

Residues of spiromesifen and its metabolite spiromesifen-enol were determined by LC-MS/MS, using the validated analytical method 00631. The method has been validated with an LOQ of 0.05 mg/kg for both analytes (expressed as analyte, resulting in an LOQ of 0.07 mg/kg for spiromesifen-enol, expressed as spiromesifen). Procedural recoveries carried out concurrently with the analyses of trial samples showed

recoveries were generally within the acceptable range of 70–120 percent, with relative standard deviation <20 percent. The results of the trials are presented in Table 16 below.

Table 16 Spiromesifen residues in beans without pods resulting from supervised trials in the United States

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)				Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No			spiromesifen	spiromesifen enol as spiromesifen	total residue spiromesifen calc. as spiromesifen [1]	*mean total residues scaled to GAP[2]*	
GAP, Canada	SC 240	0.144			3	1						
09410.05-CA135 United States, 2005 Riverside America, North (Fordhook 242 lima bean)	240 g/L SC	0.214 0.217 0.217	0.091 0.094 0.094	236.1 231.7 231.3	3	1	Seed, green	<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	M-282963-01-1
							Foliage	2.95, 1.74	0.49, 0.37	3.44, 2.11 (2.78)	*1.85*	
09410.05-MD18 United States, 2005 Salisbury America, North (Burpee Improved lima bean)	240 g/L SC	0.214 0.220 0.214	0.063 0.063 0.063	338.6 347.7 339.1	3	1	Seed, green	<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	M-282963-01-1
							Foliage	3.44, 3.70	0.56, 0.52	4.00, 4.22 (4.11)	*2.74*	
09410.05-NC22 United States, 2005 Clinton America, North (Fordhook lima bean)	240 g/L SC	0.214 0.213 0.215	0.057 0.057 0.057	375.6 373.8 377.0	3	1	Seed, green	<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	M-282963-01-1
							Foliage	1.58, 2.59	0.35, 0.41	1.93, 3.00 (2.47)	*1.66*	
09410.05-NJ28 United States, 2005 Bridgeton America, North (Baby lima bean)	240 g/L SC	0.215 0.214 0.217	0.067 0.067 0.067	322.5 320.5 326.8	3	1	Seed, green	<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	M-282963-01-1
							Foliage	6.26, 6.76	0.52, 0.65	6.78, 7.41 (7.10)	*4.75*	
09410.05-OH*15 United States, 2005 Fremont America, North (Flagrand French shell bean)	240 g/L SC	0.215 0.210 0.212	0.037 0.037 0.043	804.9 780.9 494.3	3	1	Seed, green	<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	M-282963-01-1
							Foliage	0.62, 0.51	0.24, 0.20	0.86, 0.71 (0.79)	*0.54*	
09410.05-WA20 United States, 2005 Prosser America, North (Fordhook 242 succulent bean)	240 g/L SC	0.213 0.216 0.214	0.083 0.083 0.083	256.1 259.4 258.6	3	1	Seed, green	<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	M-282963-01-1
							Foliage	10.9, 10.2	0.84, 0.88	11.74, 11.08 (11.41)	*7.67*	
09410.05-WI23 United States, 2005 Arlington America, North (Lima – 909 Cyprus succulent bean)	240 g/L SC	0.214 0.217 0.217	0.091 0.094 0.094	236.1 231.7 231.3	3	1	Seed, green	<0.05, <0.05	<0.07, <0.07	<0.12, <0.12 (<0.12)	-	M-282963-01-1
							Foliage	4.84, 3.77	0.81, 0.84	5.65, 4.61 (5.13)	*3.42*	

Notes:

(1) – mean residue values from duplicate samples presented in parenthesis

(2) – where applicable

Dry beans

A total of 10 supervised trials were carried out on dry shelled beans in the United States during 2005 (Ref: M-282963-01-1). Each trial consisted of 2 plots; a control untreated plot and a plot treated with a broadcast spray application of Oberon® 2 SC (a suspension concentrate formulation containing 2 lb ai/gal, equivalent to 240 g ai/L). Each treated plot received 3 applications at a nominal rate of 0.19 lb ai/A (equivalent to 213 g ai/ha), applications were made with an interval of 6–14 days. No adjuvants were added to the formulation prior to application. One trial (05-ID15) received an additional 4th application in error, and another trial (05-CO16) received 3 applications that were approximately 13–14 percent over the target rate.

Samples of beans were collected from all trials 9–10 days after the last application. The beans were dried on the vine or in the field after the plants were pulled. Dry beans were then shelled by hand or *via* a mechanical thresher or sheller, and then the dry, shelled beans were placed into frozen storage.

Samples were maintained frozen at <-20 °C for a maximum of 346 days (*ca* 11 months) prior to analysis. The storage stability of spiromesifen residues has been sufficiently investigated (see Section 4.0), therefore, it can be assumed that residues (sum of residues of spiromesifen and its metabolites) were stable when analysed.

Residues of spiromesifen and its metabolite spiromesifen-enol were determined by LC- MS/MS, using the validated analytical method 00631. The method has been validated with an LOQ of 0.01 mg/kg for both analytes (expressed as analyte, resulting in an LOQ of 0.014 mg/kg for spiromesifen-enol, expressed as spiromesifen). Procedural recoveries carried out concurrently with the analyses of trial samples showed recoveries were generally within the acceptable range of 70–120 percent, with relative standard deviation <20 percent. These low recoveries for parent spiromesifen are consistent with the conversion of parent into the enol metabolite occurring in dry bean, as demonstrated in the storage stability study of Sarti (2016).

The results of the trials are presented in Table 17 below. Eight of the trials were co- located, in 4 different locations, in these cases the highest value from each location has been selected for MRL calculations.

Table 17 Spiromesifen residues in dry shelled beans resulting from supervised trials in the United States

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol as spiromesifen	total residue spiromesifen calc. as spiromesifen(1)	
GAP, Canada	SC 240	0.144			3	10					
09410.05-CA134 United States, 2005 Parlier America, North (Henderson bush lima bean)	240 g/L SC	0.212 0.216 0.214	376.3 430.1 379.0	56.3 50.3 56.5	3	10	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.024, <0.024 (<0.024)	M-282963-01-1
09410.05-CO15 United States, 2005 Fort Collins America, North	240 g/L SC	0.222 0.220 0.219	0.114 0.114 0.114	194 192 191	3	9	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.024, <0.024 (<0.024)	M-282963-01-1

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol as spiromesifen	total residue spiromesifen calc. as spiromesifen(1)	
GAP, Canada	SC 240	0.144			3	10					
(Grand Mesa pinto dry bean) *co-located with trial C016											
09410.05-CO16 United States, 2005 Fort Collins America, North (Grand Mesa pinto bean) *co-located with trial C015	240 g/L SC	0.243 0.242 0.241	0.114 0.114 0.114	213 212 212	3	9	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.024, <0.024 (<0.024)	M-282963-01-1
09410.05-ID15 United States, 2005 Kimberly, Twins Falls County America, North (Othello Pinto bean)	240 g/L SC	0.224 0.221 0.225 0.233	0.078 0.078 0.078 0.078	287 283 288 298	4	10	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.024, <0.024 (<0.024)	M-282963-01-1
09410.05-ND11 United States, 2005 Velva America, North (Norstar navy dry bean) *co-located with trial ND12	240 g/L SC	0.216 0.215 0.214	0.192 0.192 0.192	112 112 111	3	10	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.024, <0.024 (<0.024)	M-282963-01-1
09410.05-ND12 United States, 2005 Velva America, North (Maverick pinto dry bean) *co-located with trial ND11	240 g/L SC	0.217 0.212 0.214	0.192 0.192 0.192	113 110 112	3	10	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.024, <0.024 (<0.024)	M-282963-01-1
09410.05-ND13 United States, 2005 Fargo, Cass County America, North (Navigator navy dry bean) *co-located with trial ND14	240 g/L SC	0.215 0.211 0.213	0.127 0.127 0.127	169 166 167	3	9	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.024, <0.024 (<0.024)	M-282963-01-1
09410.05-ND14 United States, 2005 Fargo, Cass County America, North (Eclipse black dry bean) *co-located with trial ND13	240 g/L SC	0.213 0.211 0.215	0.127 0.127 0.127	167 165 169	3	9	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.024, <0.024 (<0.024)	M-282963-01-1
09410.05-WI21 United States, 2005 Arlington America, North (Dark red kidney dry bean) *co-located with trial WI22	240 g/L SC	0.210 0.215 0.210	0.092 0.096 0.094	227 225 224	3	10	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.024, <0.024 (<0.024)	M-282963-01-1
09410.05-WI22	240 g/L SC	0.216	0.092	235	3	10	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.024, <0.024	M-282963-01-1

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol as spiromesifen	total residue spiromesifen calc. as spiromesifen(1)	
GAP, Canada	SC 240	0.144			3	10					
United States, 2005 Arlington America, North (Light red kidney dry bean) *co-located with trial WI21		0.223 0.221	0.097 0.094	230 235						(<0.024)	

Notes:

(1) – Mean residue values from duplicate samples presented in parenthesis.

Soya bean

A total of nine supervised trials were carried out on soybeans (dry) in Brazil during 2016 (Ref: M-600100-01-1). Each trial consisted of 2 plots; a control untreated plot and a plot treated with a broadcast spray application of Oberon 240 SC (a suspension concentrate formulation containing 240 g ai/L). Each treated plot received 2 applications at a nominal rate of 144 g ai/ha, applications were made with an interval of 5 days. No adjuvants were added to the formulation prior to application.

Samples of soybeans (dry seed) were collected from all trials 21 days after the last application. Additionally, at five trial sites samples were collected 6–7, 13–14, 26–28 and 34–35 days after the last application. All samples were placed into frozen storage.

Samples were maintained frozen at <-20 °C for a maximum of 263 days (*ca* 9 months) prior to analysis. The storage stability of spiromesifen residues has been sufficiently investigated (see Section 4.0), therefore, it can be assumed that residues (sum of spiromesifen and its metabolites) were stable when analysed.

Residues of spiromesifen and its metabolite spiromesifen-enol were determined by LC- MS/MS, using the validated analytical method BS001-P09-01. The method has been validated with an LOQ of 0.01 mg/kg for both analytes. Procedural recoveries carried out concurrently with the analyses of trial samples showed recoveries were within the acceptable range of 70–120 percent, with relative standard deviation <20 percent. The results of the trials are presented in Table 18 below.

Table 18 Spiromesifen residues in soybeans resulting from supervised trials in Brazil

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol as spiromesifen	total residue spiromesifen calc. as spiromesifen(1)	
GAP, Brazil	SC 240	0.144			2	21					
BS001-16DA Brazil, 2016 Primavera do Leste, Mato Grosso (M7739 Ipro)	240 g/L SC	0.141 0.142	0.067 0.063	212 224	2	7	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	M-600100-01-1
						14		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						21		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						28		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						35		<0.01, <0.01	<0.014,	<0.02, <0.02	

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol as spiromesifen	total residue spiromesifen calc. as spiromesifen[1]	
GAP, Brazil	SC 240	0.144			2	21					
									<0.014	(<0.024)	
BS002-16DA Brazil, 2016 Tamarana, Paraná (Monsoy 5947)	240 g/L SC	0.142 0.144	0.066 0.066	216 219	2	7	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	M-600100-01-1
						14		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						21		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						28		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						35		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
BS003-16DA Brazil, 2016 Restinga Seca, Rio Grande do Sul (NA 5909 RG)	240 g/L SC	0.147 0.142	0.067 0.066	218 216	2	7	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	M-600100-01-1
						14		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						21		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						28		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						35		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
BS004-16DA Brazil, 2016	240 g/L SC	0.145 0.143	0.066 0.066	220 216	2	7	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	M-600100-01-1
Montividiu, Goías (Anta 82 RR)						14		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						21		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						28		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						35		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
BS005-16DA Brazil, 2016 Barreiras, Bahia (M 8808 IPRO)	240 g/L SC	0.150 0.151	0.066 0.065	229 231	2	6	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	M-600100-01-1
						13		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						20		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						26		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
						34		<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	
BS006-16HA Brazil, 2016 Primavera do Leste, Mato Grosso (M7739 Ipro)	240 g/L SC	0.144 0.144	0.064 0.064	225 224	2	21	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	M-600100-01-1
BS007-16HA Brazil, 2016 Londrina, Paraná (Monsoy 5917)	240 g/L SC	0.150 0.150	0.065 0.065	230 232	2	21	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	M-600100-01-1
BS008-16HA Brazil, 2016 Uberlândia, Minas Gerais (SYN1163 RR)	240 g/L SC	0.143 0.144	0.064 0.064	224 226	2	21	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	M-600100-01-1
BS009-16HA Brazil, 2016	240 g/L SC	0.143 0.148	0.065 0.065	221 227	2	21	Seed, dry	<0.01, <0.01	<0.014, <0.014	<0.02, <0.02 (<0.024)	M-600100-01-1

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Residues (mg/kg)			Reference
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.			spiromesifen	spiromesifen enol as spiromesifen	total residue spiromesifen calc. as spiromesifen[(1)]	
GAP, Brazil	SC 240	0.144			2	21					
Conchal, São Paulo (BMX Potencia)											

Notes:

[(1)] – Mean residue values from duplicate samples presented in parenthesis.

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

In Storage

Further data were not provided.

In Processing

Three processing studies for orange and one for soybeans were provided, investigating the fate of residues in processed commodities.

Oranges Study 1

The processing of oranges to dried pulp, oil, juice, raw juice and marmalade was performed in two field residues trials carried out in the United States (Ref: M-635467-01-1).

Each trial consisted of two plots; a control untreated plot, and a plot treated with a single airblast application of Oberon 240 SC (a suspension concentrate formulation containing 240 g spiromesifen/L). The test substance was applied to orange trees (BBCH 83–89) at an exaggerated rate of approximately 720 g ai/ha, with an application volume of approximately 1800–2200 L/ha. Samples of mature oranges were harvested 2 days after the last application, samples of RAC (oranges whole fruit, unwashed) were placed in frozen storage and an additional bulk sample was shipped at ambient temperature to the processing facility. Oranges were processed using processes representative of industrial practices into dried pulp, oil, juice, raw juice and marmalade. Samples of the RAC and processed commodities were stored frozen prior to analysis for a maximum of 253 days (*ca* 8 months).

Residues of spiromesifen and spiromesifen-enol were determined by LC-MS/MS using the validated analytical method BS001-P09-02. The method was validated with an LOQ of 0.01 mg/kg for all matrices, with the exception of orange oil, which has an LOQ of 0.1 mg/kg (expressed as spiromesifen). Procedural recoveries carried out concurrently with the analyses of trial samples were within the range of 70–120 percent, with relative standard deviation of <20 percent.

The results of the trials are presented in Table 19 below. These results indicate that residues of total spiromesifen only concentrate when oranges are processed into orange oil, in all other processed commodities a dilution of residues was observed.

Table 19 Summary of spiromesifen residues in processed oranges from study M-635467-01-1

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Spiromesifen (mg/kg)	BSN 2060 enol (expressed as spiromesifen) (mg/kg)	Total spiromesifen calc. as spiromesifen	Processing factor for total spiromesifen
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.						
GAP, Brazil	SC 240 g/L	0.144			1	21					
RABS0089	240 g/L SC	0.729	0.039	1881	1	2	Fruit (RAC)	0.33, 0.64,	0.01, 0.014,	0.34, 0.66, 0.74	-

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Spiromesifen (mg/kg)	BSN 2060 enol (expressed as spiromesifen) (mg/kg)	Total spiromesifen calc. as spiromesifen	Processing factor for total spiromesifen
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.						
GAP, Brazil	SC 240 g/L	0.144			1	21					
BS009-17PA United States, 2017 Howey in the Hills, Florida, United States (typical to region)								0.73 (0.57)	0.012 (0.012)	(0.58)	
							Pulp, dry	0.17, 0.16, 0.16 (0.17)	0.26, 0.27, 0.24 (0.26)	0.43, 0.44, 0.41 (0.42)	0.7
							Raw juice	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	<0.03
							Pasteurized Juice	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	<0.03
							Marmalade	0.012, 0.013, 0.015 (0.0136)	<0.01, <0.01, <0.01 (<0.01)	0.023, 0.023, 0.025 (0.024)	0.04
							Oil	128, 128, 128 (128)	<0.1, <0.1, <0.1 (<0.1)	129, 128, 128 (128)	221
RABS0089 BS010-17PA United States, 2017 Sanger, California, United States (Valencia)	240 g/L SC	0.721	0.039	1873	1	2	Fruit (RAC)	0.14, 0.25, 0.12 (0.17)	<0.01, <0.01, <0.01 (<0.01)	0.15, 0.26, 0.12 (0.18)	-
							Pulp, dry	0.06, 0.056, 0.057 (0.058)	0.14, 0.13, 0.12 (0.13)	0.20, 0.19, 0.18 (0.19)	1.05
							Raw juice	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	<0.11
							Pasteurized Juice	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	<0.11
							Marmalade	0.012, 0.013, <0.01 (0.012)	<0.01, <0.01, <0.01 (<0.01)	0.022, 0.023, <0.02 (0.022)	0.12
							Oil	31.8, 33.4, 32.2 (32)	<0.1, <0.1, <0.1 (<0.1)	32.9, 33.5, 32.3 (33.1)	183

Notes:

Values are reported to 3 significant figures where possible.

Mean values are presented in parenthesis.

For orange whole fruit RAC, the results are individual analyses of three separate samples. For the other processed commodities, the results are three analyses of a single sample.

Oranges Study 2

The processing of oranges to peel (washed), pulp, juice, oil, pomace (wet and dry), dry pulp and marmalade was performed in two field residues trials carried out in the United States (Ref: M-762109-03-1).

Each trial consisted of three plots; a control untreated plot, and two plots treated with two airblast applications of Oberon Speed (an SC formulation containing 228.6 g spiromesifen/L). The test substance was applied at an exaggerated rate of approximately 875 g ai/ha (plot TRTHI) or 525 g ai/ha (plot TRTLO), applications were made to orange trees at BBCH 83 with an interval of 10 days, with an application volume of approximately 2000–2500 L/ha. An adjuvant, crop oil concentrate (COC), was used in all applications at a rate of 1.0 percent v/v. The plot TRTLO was conducted as a reserve, however this was later not needed and therefore fruit were not harvested from this plot.

Samples of mature oranges (BBCH 83–85) were harvested from in each trial 7 days after the last application, samples of RAC (oranges whole fruit, unwashed) were placed in frozen storage and an additional bulk sample was shipped at ambient temperature to the processing facility. Oranges were processed using processes representative of industrial practices into washed fruit, peel (unwashed and washed), pulp, pasteurized juice, oil, pomace (wet and dry), dried pulp and marmalade. Samples of the

RAC and processed commodities were stored frozen prior to analysis for a maximum of 391 days (*ca* 13 months).

Residues of spiromesifen and spiromesifen-enol were determined by LC-MS/MS using the validated analytical method BS001-P09-02. The method was validated with an LOQ of 0.01 mg/kg for all matrices, with the exception of orange oil, which had an LOQ of 0.5 mg/kg in this study (expressed as spiromesifen). Procedural recoveries carried out concurrently with the analyses of trial samples were within the range of 70–120 percent, with relative standard deviation of <20 percent.

The results of the trials are presented in Table 20 below. These results indicate that residues of total spiromesifen concentrate when oranges are processed into orange oil, peel (washed and unwashed), dry pulp and pomace (wet and dry). In all other processed commodities, a dilution of residues was observed.

Table 20 Summary of spiromesifen residues in processed oranges from study M-762109-03-1

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Spiromesifen (mg/kg)	BSN 2060 enol (expressed as spiromesifen) (mg/kg)	Total spiromesifen calc. as spiromesifen	Processing factor for total spiromesifen
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.						
GAP, Brazil	SC 240 g/L	0.144			1	21					
RABS0044 BS071-18PA United States, 2018 San Luis Obispo, California, United States (Valencia)	228.6 g/L SC	0.897	0.044	2026	2	7	Fruit (RAC)	0.19, 0.35, 0.32 (0.29)	0.023, 0.032, 0.043 (0.033)	0.21, 0.38, 0.37 (0.32)	-
		0.877	0.04	2183							
							Fruit, washed	0.20, 0.37, 0.13	0.011, 0.024,	0.21, 0.40,	0.8
								-0.24	<0.01 (0.015)	0.14 (0.25)	
							Peel, unwashed	0.92, 0.94, 0.92	0.069, 0.072,	0.99, 1.02,	3.1
								-0.93	0.068 (0.070)	0.99 (1.0)	
							Peel, washed	0.71, 0.68, 0.7	0.023, 0.024,	0.73, 0.71,	2.3
								-0.7	0.022 (0.023)	0.72 (0.72)	
							Fruit, peeled	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	0.06
							Pasteurized juice	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	0.06
							Oil	67.9, 69.1, 70.2	<0.5, <0.5, <0.5	68.4, 69.6, 70.7	218
								-69.1	(<0.5)	-69.6	
							Pomace, wet	0.17, 0.18, 0.17	<0.01, <0.01,	0.18, 0.19,	0.6
								-0.18	<0.01 (<0.01)	0.18 (0.19)	
							Pomace, dry	0.49, 0.61,	0.13, 0.15,	0.62, 0.76,	2.2
								0.55 (0.55)	0.14 (0.14)	0.69 (0.69)	
							Pulp, dry	0.34, 0.35,	0.56, 0.59,	0.91, 0.94,	2.9
								0.34 (0.34)	0.56 (0.57)	0.9 (0.92)	
							Marmalade	0.024, 0.021, 0.023 (0.023)	0.010, 0.010, <0.01 (0.010)	0.034, 0.031, 0.032 (0.033)	0.1
RABS0044 BS072-18PA United States,	228.6 g/L SC	0.872	0.034	2535	2	7	Fruit (RAC)	0.14, 0.16, 0.12 (0.14)	0.012, 0.010, 0.011 (0.011)	0.15, 0.17, 0.13 (0.15)	-
		0.875	0.035	2508							

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Spiromesifen (mg/kg)	BSN 2060 enol (expressed as spiromesifen) (mg/kg)	Total spiromesifen calc. as spiromesifen	Processing factor for total spiromesifen
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.						
2018 Sanger, California, United States (Valencia)											
							Fruit, washed	0.12, 0.072,	<0.01, <0.01,	0.131, 0.082,	0.7
								0.1 (0.099)	<0.01 (<0.01)	0.112 (0.11)	
							Peel, unwashed	0.76, 0.78, 0.77	0.49, 0.49,	0.81, 0.83,	5.5
								-0.77	0.51 (0.49)	0.82 (0.82)	
							Peel, washed	0.49, 0.47, 0.48	0.034, 0.032,	0.52, 0.51,	3.5
								-0.48	0.033 (0.033)	0.52 (0.52)	
							Fruit, peeled	<0.01, <0.01,	<0.01, <0.01,	<0.02, <0.02,	0.1
								<0.01 (<0.01)	<0.01 (<0.01)	<0.02 (<0.02)	
							Pasteurized juice	<0.01, <0.01,	<0.01, <0.01,	<0.02, <0.02,	0.1
								<0.01 (<0.01)	<0.01 (<0.01)	<0.02 (<0.02)	
							Oil	19.5, 19.0, 19.8	<0.5, <0.5, <0.5	20.0, 19.5, 20.4	33
								-19.4	(<0.5)	-19.9	
							Pomace, wet	0.19, 0.19, 0.19	0.016, 0.017,	0.21, 0.20,	1.4
								-0.19	0.017 (0.017)	0.21 (0.21)	
							Pomace, dry	0.7, 0.64,	0.21, 0.2,	0.91, 0.83,	5.7
								0.64 (0.66)	0.2 (0.20)	0.84 (0.86)	
							Pulp, dry	0.68, 0.63,	0.48, 0.42,	1.2, 1.1, 1.1	7.3
								0.64 (0.65)	0.43 (0.44)	-1.1	
							Marmalade	<0.01, <0.01,	<0.01, <0.01,	<0.02, <0.02,	0.1
								<0.01 (<0.01)	<0.01 (<0.01)	<0.02 (<0.02)	

Notes:

Values are reported to 3 significant figures where possible

Mean values are presented in parenthesis

For whole fruit samples (RAC and washed), the results are individual analyses of three separate samples. For the other processed commodities, the results are three analyses of a single sample.

Oranges Study 3

The processing of oranges to juice was performed in four field residues trials carried out in Brazil (Ref: M-754191-01-1).

Each trial consisted of two plots; a control untreated plot, and a plot treated with a single airblast application of Oberon 240 SC (a suspension concentrate formulation containing 240 g spiromesifen/L). The test substance was applied to orange trees (BBCH 83–89) at a nominal rate of approximately 144 g ai/ha, with an application volume of approximately 2000 L/ha. Samples of mature oranges were harvested 1, 3, 7, 14 and 21 days after the last application. Analysis of both the RAC and the processed juice was only carried out on days 1 and 3, therefore the processing values derived from these have been presented here. Samples of RAC (oranges whole fruit) were processed into raw juice at the test site and stored frozen (<-20 °C) prior to analysis.

Samples of the RAC and processed commodities were stored frozen prior to analysis for a maximum of 98 days (*ca* 3 months).

Residues of spiromesifen and spiromesifen-enol were determined by LC-MS/MS using the validated analytical method 00631, as described in Section 3.1 of this document. The method was validated with an LOQ of 0.01 mg/kg for all matrices (expressed as analyte, resulting in an LOQ of 0.014 mg/kg for spiromesifen-enol, expressed as spiromesifen). Procedural recoveries carried out concurrently with the analyses of trial samples were within the range of 70–120 percent, with relative standard deviation of <20 percent.

The results of the trials are presented in Table 21 below. These results indicate that residues of total spiromesifen are diluted when oranges are processed into orange juice.

Table 21 Summary of spiromesifen residues in processed oranges from study M-754191-01-1

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Spiromesifen (mg/kg)	BSN 2060 enol (expressed as spiromesifen) (mg/kg)	Total spiromesifen calc. as spiromesifen	Processing factor for total spiromesifen
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.						
GAP, Brazil	SC 240 g/L	0.144			1	21					
RABS0168 L20RP018-01 Brazil, 2020 Fernando Prestes/SP, Brazil	240 g/L SC	0.142	0.007	1980	1	1	Fruit (RAC)	0.032	<0.014	0.046	-
							Raw juice	<0.01	<0.014	<0.024	<0.5
						3	Fruit (RAC)	0.020	<0.014	0.034	-
							Raw juice	<0.01	<0.014	<0.024	<0.7
RABS0168 L20RP018-02 Brazil, 2020 Baretos/SP, Brazil	240 g/L SC	0.147	0.007	2034	1	1	Fruit (RAC)	0.025	<0.014	0.039	-
							Raw juice	<0.01	<0.014	<0.024	<0.6
						3	Fruit (RAC)	0.022	<0.014	0.036	-
							Raw juice	<0.01	<0.014	<0.024	<0.6
RABS0168 L20RP018-03 Brazil, 2020 Paulinia/SP, Brazil	240 g/L SC	0.143	0.007	1977	1	1	Fruit (RAC)	0.11	<0.014	0.12	-
							Raw juice	<0.01	<0.014	<0.024	<0.2
						3	Fruit (RAC)	0.064	<0.014	0.078	-
							Raw juice	<0.01	<0.014	<0.024	<0.3
RABS0168 L20RP018-04 Brazil, 2020 Uberlandia/MG, Brazil	240 g/L SC	0.139	0.007	1937	1	1	Fruit (RAC)	0.088	<0.014	0.10	-
							Raw juice	<0.01	<0.014	<0.024	<0.2
						3	Fruit (RAC)	0.083	<0.014	0.097	-
							Raw juice	<0.01	<0.014	<0.024	<0.2

Notes:

Values are reported to 2 significant figures where possible.

Soya beans

The processing of soya beans to aspirated grain fractions, meal, hulls, flour, milk, solvent extracted RBD (refined, bleached and deodorized) oil and cold-pressed RBD oil was performed in two field residues trials carried out in the United States (Ref: M-631397-01-1).

Each trial consisted of two plots; a control untreated plot, and a plot treated with two spray applications of Oberon 240 SC (a suspension concentrate formulation containing 240 g spiromesifen/L). The test substance was applied to soya bean crop at BBCH 78–79 at an exaggerated rate of approximately 720 g ai/ha, with an application volume of approximately 200–300 L/ha. Samples of mature soya beans were harvested 21 days after the last application and shipped frozen to the processing facility. Soya beans were processed using processes representative of industrial practices into aspirated grain fractions, flour, hulls, meal, milk, solvent extracted RBD oil and cold-pressed RBD oil.

Samples of the RAC and processed commodities were stored frozen prior to analysis for a maximum of 339 days (*ca* 11 months).

Residues of spiromesifen and spiromesifen-enol were determined by LC-MS/MS using the validated analytical method BS001-P09-01. The method was validated with an LOQ of 0.01 mg/kg for all matrices, with the exception of aspirated grain fractions, which has an LOQ of 1.0 mg/kg (expressed as spiromesifen). Procedural recoveries carried out concurrently with the analyses of trial samples were within the range of 70–120 percent, with relative standard deviation of <20 percent.

The results of the trials are presented in Table 22 below. These results indicate that residues of total spiromesifen concentrate when soya beans are processed into aspirated grain fractions and hulls. In all other processed commodities, a dilution of residues was observed.

Table 22 Summary of spiromesifen residues in processed soya beans

Study, Trial Country, Year, Location (Variety)	Application					PHI (days)	Commodity	Spiromesifen (mg/kg)	BSN 2060 enol (expressed as spiromesifen) (mg/kg)	Total spiromesifen calc. as spiromesifen	Processing factor for total spiromesifen
	Formulation (g ai/L)	kg/ha (a.s.)	kg/hL (a.s.)	Water (L/ha)	No.						
GAP, Brazil	SC 240 g/L	0.288			2	21					
RABSN033 BS010-16PA United States, 2016 Henderson, Nebraska, United States (Dekalb)	240 g/L SC	0.70 0.71	0.314 0.314	223 226	2	21	Soybean seed (RAC)	0.037, 0.032, 0.057 (0.042)	0.018, 0.017, 0.047 (0.027)	0.055, 0.049, 0.10 (0.069)	-
							Aspirated grain fractions	10(a)	4.1(a)	14(a)	203
							Flour	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	<0.3
							Hulls	0.04, 0.038, 0.038 (0.039)	0.039, 0.038, 0.039 (0.038)	0.079, 0.075, 0.077 (0.077)	1.1
							Meal	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	<0.3
							Milk	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	<0.3
							Solvent extracted RBD oil	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	<0.3
							Cold pressed RBD oil	0.013, 0.016, 0.013 (0.014)	<0.01, <0.01, <0.01 (<0.01)	0.023, 0.026, 0.023 (0.024)	<0.3
RABSN033 BS011-16PA United States, 2016 Springfield, Nebraska, United States (Channel 2808)	240 g/L SC	0.73 0.72	0.370 0.367	197 198	2	21	Soybean seed (RAC)	0.091, 0.14, 0.1 (0.11)	0.05, 0.062, 0.053 (0.055)	0.14, 0.2, 0.15 (0.16)	-
							Aspirated grain fractions	17(a)	14(a)	31(a)	194
							Flour	<0.01, <0.01, <0.01 (<0.01)	0.014, 0.012, 0.12 (0.013)	0.024, 0.022, 0.022 (0.023)	0.14
							Hulls	0.08, 0.077, 0.075 (0.077)	0.15, 0.14, 0.14 (0.14)	0.23, 0.22, 0.22 (0.22)	1.4
							Meal	<0.01, <0.01, <0.01 (<0.01)	0.013, 0.013, 0.012 (0.013)	0.023, 0.023, 0.022 (0.023)	0.1
							Milk	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	<0.1
							Solvent extracted RBD oil	<0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02 (<0.02)	<0.1
							Cold pressed RBD oil	0.018, 0.017, 0.017 (0.017)	<0.01, <0.01, <0.01 (<0.01)	0.027, 0.027, 0.027 (0.027)	0.2

Notes:

(a) – Mean of 6 analysis.

Values are reported to 3 significant figures where possible.

Mean values are presented in parenthesis.

For soya bean seed samples (RAC), the results are individual analyses of three separate samples. For the processed commodities, the results are three (or six) analyses of a single sample.

APPRAISAL

Spiromesifen is a contact insecticide-acaricide belonging to the titronic acid class of compounds. The mode of action is inhibition of lipid biosynthesis, especially triglycerides and free fatty acids.

Spiromesifen was first evaluated by the 2016 JMPR where an ADI of 0–0.03 mg/kg bw was established and an ARfD was determined to be unnecessary. The residue definition for compliance with MRLs for plant and animal commodities and for dietary risk assessment for animal commodities is *sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen*. For dietary risk assessment for plant commodities, the residue definition is *sum of spiromesifen, spiromesifen-enol and 4-hydroxymethyl-spiromesifen-enol (free and conjugated), expressed as spiromesifen*. The residue is fat-soluble.

Spiromesifen was scheduled at the Fifty-second Session of the CCPR for evaluation of additional uses by the 2022 JMPR. The Meeting received information on GAP, analytical methods, storage stability data, processing studies and residue trials on oranges, mango, papaya, legume vegetables and pulses.

Methods of analysis

The Meeting received additional information on analytical methods for spiromesifen and spiromesifen-enol in plant commodities for data gathering.

LC-MS/MS method 00631 was evaluated by the 2016 JMPR and successfully validated in high water content (broccoli, cucumber, pepper, melon, beans, tomato), high starch content (corn/maize, sugar beet), high oil content (cotton), high acid content (strawberry) commodities and tea. Additional method validation data for the modified method 00631/M001 are available as part of the supervised residue trials relied upon in this submission. The Meeting concluded that this method is valid for the determination of spiromesifen and spiromesifen-enol with LOQ values at 0.01 mg/kg in mango, dry beans and at 0.05 mg/kg in succulent shelled beans and edible podded beans for each compound individually.

Another LC-MS/MS method BS001-P09-01, a modification of analytical method 00631, was evaluated by the 2016 JMPR and successfully validated in high starch content commodities (wheat: grain, aspirated grain fractions, bran, flour, germ, middling's, shorts; and sorghum: grain and aspirated grain fractions). Additional method validation data for the modified method BS001-P09-02 are available as part of the supervised residue trials relied upon in this submission. The Meeting concluded that this method is valid for the determination of spiromesifen and spiromesifen-enol with LOQ values at 0.01 mg/kg in orange whole fruit, pomace, dry pulp, juice, marmalade, peel, wet pulp and at 0.10 mg/kg in orange oil for each compound individually.

Stability of pesticide residues in stored analytical samples

The stability of spiromesifen spiromesifen-enol and metabolite 4-hydroxymethyl-Sp-enol residues under frozen conditions was investigated in high protein, high oil, high acid, high water and high starch content commodities by the 2016 JMPR. The total residues of spiromesifen and spiromesifen-enol were stable for at least 24 months in all matrices tested.

The current Meeting received additional storage stability data for spiromesifen and spiromesifen-enol under freezer storage conditions for dry beans, coffee beans and citrus fruits The total

residue (sum of spiromesifen and spiromesifen-enol) and spiromesifen-enol (per se) were demonstrated to be stable for at least 24 months in all matrices investigated. Parent spiromesifen (per se) was stable for at least 24 months in coffee beans and citrus fruits, but degraded significantly in dry beans within 30 days, resulting from the formation of spiromesifen-enol.

The samples analysed in the supervised residue trials included in this submission were stored for up to a maximum of 498 days (ca. 16 months) prior to analysis, therefore, the available data for the sum of spiromesifen and spiromesifen-enol is sufficient to cover these frozen storage intervals.

Results of supervised residue trials on crops

Oranges sweet, sour (subgroup)

The critical GAP for the use of spiromesifen on citrus fruits in Brazil is a single foliar spraying with 0.144 kg ai/ha and 21 days PHI.

Thirteen supervised field trials on oranges conducted in Brazil, matching the cGAP were provided. In whole fruits, residues for the sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen were (n = 13): 0.021, < 0.024(2), 0.025, 0.041, 0.042, 0.043, 0.045, 0.047(2), 0.049, 0.100 and 0.103 mg/kg.

As oranges covered by the registered use correspond to the Codex subgroups for subgroup 1C (Oranges, Sweet, Sour) the Meeting decided to extrapolate the estimates from oranges to subgroup 1C (Oranges, Sweet, Sour).

The Meeting estimated a maximum residue level of 0.15 mg/kg and an STMR of 0.043 mg/kg for Subgroup of Oranges, Sweet, Sour.

Mango

The critical GAP for mango is from registrations in Brazil with three applications (minimum application interval of 7 days) at 0.144 kg ai/ha and a 5-day PHI.

Five supervised field trials conducted in Brazil, matching the cGAP were provided. In whole fruits, residues for the sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen were (n = 5): 0.079, 0.115, 0.135, 0.200 and 0.225 mg/kg. All residue values in pulp were (n = 5): < LOQ (< 0.024(5) mg/kg).

The Meeting estimated a maximum residue level of 0.5 mg/kg for mango and an STMR of 0.024 mg/kg based on the residue values in mango pulp.

Papaya

The critical GAP for papaya is from registrations in Brazil with three applications (minimum application interval of 7 days) at 0.144 kg ai/ha and a 5-day PHI.

Five supervised field trials conducted in Brazil, matching the cGAP were provided. In whole fruits, residues for the sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen were (n = 5): 0.046, 0.099, 0.13, 0.23, 0.33 mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg and an STMR of 0.13 mg/kg for papayas.

Beans with pods

The critical GAP for beans with pods is from registrations in Canada with three applications (minimum application interval of 7 days) at 0.144 kg ai/ha and a 1-day PHI.

Seven supervised field trials conducted in United States using a higher application rate (3 × 0.213 kg ai/ha) were provided. The Meeting agreed to use proportionality to scale down residues to the cGAP.

In beans with pods, unscaled residues of sum of spiromesifen and spiromesifen-enol expressed as spiromesifen were (n = 7): < 0.12, < 0.13, 0.14, 0.24, 0.28, 0.3, 0.44 mg/kg.

In beans with pods, scaled residues of sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen were (n = 7): < 0.053, 0.091, < 0.12, 0.16, 0.18, 0.20 and 0.30 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.16 mg/kg for beans with pods.

Beans without pods

The critical GAP for beans without pods is from registrations in Canada with three applications (minimum application interval of 7 days) at 0.144 kg ai/ha and a 1-day PHI.

Seven supervised field trials conducted in United States using a higher application rate (3 × 0.213 kg ai/ha) were provided.

In beans without pods, residues of sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen were (n = 7): < 0.12 (7)mg/kg.

The Meeting estimated a maximum residue level of 0.15(*) mg/kg and an STMR of 0.12(*) mg/kg for beans with pods.

Dry beans and soya beans

For dry beans, the critical GAP for dry beans is from registrations in Canada with three applications (minimum application interval of 7 days) at 0.144 kg ai/ha and a 10-day PHI.

Six supervised field trials conducted in United States using a higher application rate (3 × 0.213 kg) were provided. In dry seeds residues for the sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen were (n = 6): < 0.024 (6)mg/kg.

For soya beans, the critical GAP for soya beans is from registrations in Brazil with two applications (minimum application interval of 5 days) at 0.144 kg ai/ha and a 21-day PHI.

Nine supervised field trials conducted in Brazil, matching the cGAP were provided. In dry seeds residues for the sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen were (n = 9): < 0.024 (9)mg/kg.

The Meeting noted that all residues in dry beans and soya beans were below the LOQ (0.024 mg/kg) and decided to propose estimates for the whole subgroup of dry beans.

The Meeting estimated a maximum residue level of 0.03(*) mg/kg and an STMR of 0.024 mg/kg for the subgroup of dry beans.

Animal feed

Bean forage (green)

The critical GAP for beans is from registrations in Canada with three applications (minimum application interval of 7 days) at 0.144 kg ai/ha and a 1-day PHI.

Seven supervised field trials conducted in United States at a higher application rate GAP (3 × 0.213 kg ai/ha, same treatment regime) were provided.

In foliage, unscaled residues of sum of spiromesifen and spiromesifen-enol expressed as spiromesifen (n = 7): 0.38, 0.43, 0.54, 0.58, 0.60, 0.82 and 0.86 mg/kg.

In foliage, scaled residues of sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen were (n = 7): 0.26, 0.29, 0.37, 0.39, 0.41, 0.55 and 0.58 mg/kg.

The Meeting estimated a median residue of 0.39 mg/kg and a highest residue of 0.58 mg/kg for bean forage (as received) based on the scaled data set.

Fate of residues during processing

The Meeting received new processing studies on oranges and soya bean. Processed commodities from oranges (dry pulp, juice, marmalade, oil, dry pomace) and soya bean (flour, hulls, meal, milk, oil) were derived using simulated commercial practices. Processing factors and residue estimates are summarized below.

Table 23 Processing factors, STMR-Ps and HR-Ps for the sum of spiromesifen and spiromesifen-enol expressed as spiromesifen and used for dietary risk assessment and livestock dietary burden calculation

Raw commodity	Processed commodity	Individual processing factors	Median or best estimate processing factor	STMR-P (Median-P) = STMR RAC × PF (mg/kg)	Maximum residue level for RAC (mg/kg)	Maximum residue level for processed commodity = maximum residue level × PF (mg/kg)[a]
Oranges	Pulp, dry	0.7, 1.05, 2.9, 7.3	2	0.043 × 2 = 0.086	0.15	0.15 × 2 = 0.3
	Raw juice	< 0.03,< 0.11, < 0.2, < 0.2, < 0.2, < 0.3, 0.3, < 0.5, < 0.6, < 0.6, < 0.7	0.3	-		-
	Pasteurize Juice	< 0.03, < 0.06, < 0.1, < 0.11	< 0.08	0.043 × 0.08 = 0.0034		-
	Marmalade	0.04, < 0.1, 0.1, 0.12,	0.1	0.043 × 0.1 = 0.0043		-
	Orange oil, edible	133, 183, 218, 221	201	0.043 × 201 = 8.6	0.15	0.15 × 201 = 30
	Fruit, raw, without peel	< 0.06, < 0.1	< 0.08	0.043 × 0.08 = 0.0034		-
	Pomace, dry	2.9, 7.3	5.1	0.043 × 5.1 = 0.22		-
Soya bean	Milk	0.1, < 0.3	0.2	0.024 × 0.2 = 0.005		-
	flour	0.14, < 0.3	0.22	0.024 × 0.22 = 0,0053		-
	Oil (refined)[b]	< 0.1, < 0.3	0.2	0.024 × 0.2 = 0.005		-
	Oil (crude)[c]	0.2, < 0.3	0.25	0.024 × 0.25 = 0.006	0.03(*)	0.03(*)
	aspirated grain	194, 203	199	0.024 × 199 = 4.8		-

Raw commodity	Processed commodity	Individual processing factors	Median or best estimate processing factor	STMR-P (Median-P) = STMR RAC × PF (mg/kg)	Maximum residue level for RAC (mg/kg)	Maximum residue level for processed commodity = maximum residue level × PF (mg/kg)[a]
	Meal	< 0.1, < 0.3	< 0.2	0.024 × 0.2 = 0.005	0.03(*)	0.03(*)
	Hulls	1.1, 1.4	1.25	0.024 × 1.25 = 0.03	0.03(*)	0.03(*)

Notes:

[a] calculated values are rounded according to the OECD rounding classes.

[b] Solvent extracted RBD.

[c] Cold pressed RBD.

The Meeting estimated a maximum residue level of 30 mg/kg for orange oil edible, 0.3 mg/kg for citrus pulp, dry and of 0.03(*) mg/kg for soya bean oil (crude), hulls, and meal.

Residues in animal commodities

Farm animal feeding studies

No additional information on transfer of residues to livestock was provided to the current Meeting. Please refer to the 2016 JMPR Report.

Estimated maximum and mean dietary burdens of livestock and animal commodities maximum residue levels

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the Meeting. The dietary burdens, estimated using the most recent version of the OECD livestock dietary burden calculator, are presented in Annex 6 and summarised below. In the 2016 JMPR the calculations were made according to the animal diets listed in Appendix IX of the 2016 edition of the FAO manual. Results of the estimated maximum and mean dietary burdens are summarised in Table 24.

Table 24 Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden: spiromesifen, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	2.6	1.3	25.5	6.8	9.6	4.7	0.01	0.01
Dairy cattle	5.0	2.3	23.6	6	39.9❶	8.6❷	5.50	2.50
Poultry – broiler	0.01	0.01	0.02	0.02	0.07	0.07	0.002	0.002
Poultry – layer	0.01	0.01	0.62❸❺	0.1❹	0.07	0.07	0.001	0.001

Notes:

❶ Suitable for estimation of maximum residue levels in meat and milk.

❷ Suitable for estimation of median residue levels in meat and milk

❸Suitable for estimation of maximum residue levels in poultry meat

❹ Suitable for estimation of median residue levels in poultry meat and eggs

❺ Suitable for estimation of maximum residue levels in eggs

The spiromesifen dietary burden reached a maximum level of 25.5 ppm of dry matter diet in beef cattle, 39.9 ppm diet in dairy cattle and 0.62 mg/kg diet in poultry. The mean dietary burdens were

6.8 mg/kg in beef cattle, 8.6 mg/kg diet in dairy cattle and 0.1 mg/kg in poultry. These results are similar to the previous livestock dietary burden calculations performed by the 2016 JMPR (highest maximum dietary burden was 25 ppm of dry matter diet in beef cattle, 40 ppm diet in dairy cattle and 5.3 mg/kg diet in poultry). The meeting confirmed its previous recommendations for animal commodities.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

The definition of the residue for compliance with the MRL for plant and animal commodities and for dietary risk assessment for animal commodities: *sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen*.

The definition of the residue for dietary risk assessment for plant commodities: *sum of spiromesifen, spiromesifen-enol and 4-hydroxymethyl-spiromesifen-enol (free and conjugated), expressed as spiromesifen*.

The residue is fat-soluble.

Table 25 Residue levels suitable for establishing maximum residue limits and for IEDI assessment

CCN	Commodity name	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg
		New	Previous	
FC 0004	Subgroup of oranges, Sweet, Sour	0.15	-	0.043
FI 0345	Mango	0.5	-	0.024
FI 0350	Papaya	0.7	-	0.13
VP 0061	Beans with pods (Phaseolus spp.) immature pods and succulent seeds)	0.5	-	0.16
VP 0062	Beans without pods (Phaseolus spp.) (succulent seeds)	0.15*	-	0.12
VD 2065	Dry beans, subgroup	0.03*	-	0.024
JF 0004	Orange Juice	-	-	0.0034
OR 0004	Orange oil, edible	30	-	8.6[a]
OC 0541	Soya bean oil, crude	0.03*	-	0.006
OR 0541	Soya bean oil, refined	-	-	0.005 [a]
	Soya bean milk	-	-	0.005 [a]
DM 0541	Soya bean flour	-	-	0.0053
AL 1030	Bean forage (green)	-	-	0.39 (ar)
AB 0001	Citrus pulp, dried	0.3	-	0.086
AL 3538	Soya bean, hulls	0.03*	-	0.03
AL 3539	Soya bean meal	0.03*	-	0.005

[a] Value not relevant for IEDI assessment calculations.

(ar) as received..

Table 26 Additional values used in estimating livestock dietary burdens

CCN	Commodity name	Median residue (-P) mg/kg	highest residue (-P) mg/kg
	Soybean aspirated grain	4.8	-

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for spiromesifen is 0–0.03 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for spiromesifen were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs were 3–20 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of spiromesifen from uses considered by the JMPR is unlikely to present a public health concern.

Short-term dietary exposure

The Meeting determined that an ARfD is not necessary for spiromesifen. The Meeting therefore concluded that the short-term dietary exposure to residues of spiromesifen resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

REFERENCES

Report Code	Author	Year	Study title, Institute
M-553911-01-2.	Sarti, A	2016	Storage stability of spiromesifen (BSN 2060) and its metabolite spiromesifen-enol (BSN 2060-enol) in/on dry bean, coffee and citrus during freezer storage for up to 24 months.
M-571855-02-1, I15-094	Fernandes, J.	2021	Amendment nº 01 to the final report - Determination of the residues of spiromesifen and its metabolite spiromesifen-enol in/on citrus (fruits) after spraying of Oberon in the field in Brazil.
M-627001-01-1, RABS0090.	Li, Y.	2018	Oberon 240 SC - Magnitude of the residues in/on citrus; import tolerances.
M-588894-01-1, BS-001-P09-02	Gould, T.; Murphy, I. A.	2017	An analytical method for the determination of residues of BSN2060 and BSN2060-enol in crop matrices using LC/MS/MS.
M-764414-01-1, 16-056	Silva, M	2020	Determination of the residues of abamectin and spiromesifen in/on orange (fruit) after spraying of FTB:102000031748 in the field in Brazil
M-675094-01-1, RABS0162	Oliveira, R. C.	2019	Oberon 240 SC - Magnitude of the residues of spiromesifen in/on mango (fruit, peel and pulp) after spraying of Oberon 240 SC in Brazil.
M-632116-01-1, I16-046	Carvalho	2018	Determination of the residues of spiromesifen in/on papaya (fruit) after spraying of FTB: 102000026994 in the field in Brazil.
M-282963-01-1, 09410	Dorschner, K. W.	2007	Spiromesifen: Magnitude of the residue on bean (dry shelled, succulent shelled, edible podded)
M-600100-01-1, RABSN031	Harbin, A. M.	2017	Oberon 240 SC - Magnitude of residues in/on soybean; Import tolerances
M-635467-01-1, RABS0089	Brungardt, J	2018	Oberon 240 SC (Spiromesifen) - Magnitude of the residue in/on citrus processed commodities.
M-762109-03-1, RABS0044	Chase, D.	2021	Amendment number 2 to magnitude of the residues of spiromesifen in/on orange processed commodities after spray application of abamectin & spiromesifen SC 240 (11.4+228.6 g/L).
M-754191-01-1, RABS0168.	Alves, F. M.	2020	Determination of the residues of spiromesifen in/on orange (fruit and juice) after spraying of FTB: 102000026994 in the field in Brazil.
M-631397-01-1, RABSN033	Li, Y.	2018	Oberon 240 SC (spiromesifen) - Magnitude of the residue in/on soybean processed commodities.

SULFOXAFLOR (252)

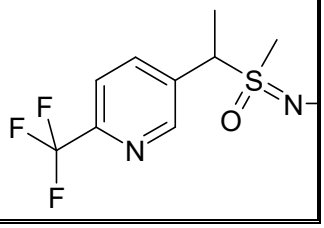
EXPLANATION

Sulfoxaflor was evaluated for the first time by JMPR 2011 when an acceptable daily intake (ADI) of 0–0.05 mg/kg bw and an acute reference dose (ARfD) of 0.3 mg/kg bw were established. Sulfoxaflor underwent subsequent evaluations by the JMPR in 2014, 2016, and 2021 (Extra).

The definition of the residue in plants and animals for both compliance with MRLs and for dietary assessment is sulfoxaflor. The residue is not fat-soluble.

The current Meeting received information on residues in globe artichoke and sunflower to supplement trials evaluated at the 2021 Extra Meeting.

Table 1 Metabolites of sulfoxaflor referenced in this document

Common or code name	Chemical name	Structure
X11719474	1-[methyl(oxido){1-[6-(trifluoromethyl)pyridin-3-yl]ethyl}-λ^6-sulfanylidene]urea	

METHODS OF RESIDUE ANALYSIS

QuEChERS

Field trial samples of globe artichoke flower heads and sunflower seeds were analysed for residues of sulfoxaflor and X11719474 using a standard QuEChERS method. Briefly, residues of sulfoxaflor and X11719474 were extracted from homogenized samples by shaking for 30 minutes with water + acetonitrile + QuEChERS salts. An aliquot of the acetonitrile layer was cleaned up by dispersive SPE (globe artichoke only). The extract was diluted with 0.1 percent formic acid and then analysed for residues by LC-MS/MS. Concurrent recovery data are summarized below.

Table 2 Summary of concurrent recovery of sulfoxaflor and its main metabolite from avocado, blueberry, caneberry, globe artichoke, asparagus, and sunflower commodities

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Globe artichoke flower heads	Sulfoxaflor	0.01	86, 93, 95, 106, 107, 109, 124	103	12	210115
		0.1	91, 93, 96, 104, 111	99	8.4	
		1	77, 79, 84, 101, 108	90	15	
	X11719474	0.01	89, 95, 98, 99, 99, 101, 117	100	8.6	
		0.1	91, 93, 93, 98, 110	97	8.0	
		1	73, 78, 84, 100, 104	88	15	
Sunflower seed	Sulfoxaflor	0.01	87, 93, 94, 96, 98, 103, 110	97	7.6	210116
		0.1	104, 104, 105, 108, 112	107	3.2	
		1	96, 101, 101, 105, 108	102	4.5	
	X11719474	0.01	93, 96, 96, 98, 101, 106, 111	100	6.4	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Mean recovery [%]	RSD [%]	Reference
		0.1	98, 100, 100, 100, 106	101	3.0	
		1	96, 99, 100, 100, 102	99	2.2	

STABILITY OF RESIDUES IN STORED SAMPLES

New data on the stability of sulfoxaflor and its two main metabolites in frozen stored samples were not provided. Field trial samples were stored at ca. -20 °C for up to 104 days for globe artichoke and up to 133 days for sunflower seed.

USE PATTERN

Registered labels describing the use of sulfoxaflor were submitted to the present Meeting for globe artichoke and sunflower (Table 3). For both uses, the timing of application is triggered by pest pressure, and applications are broadcast spray.

Table 3 Registered uses of sulfoxaflor submitted to the 2020/2022 JMPR

Use site	Country	Formulation		Application					PHI, days
		Conc.	Type	Rate, g/ha/applic	Rate, g/ha/year	Water, L/ha	Max No.	Interval, days	
Artichoke (globe)	United States	50%	WG	101	298	ns	4	7	3
Sunflower subgroup [a)]	United States	50%	WG	96	193	ns	2	7	14

Notes:

[a)] Calendula, castor oil plant, Chinese tallowtree, euphorbia, evening primrose, jojoba, niger seed, rose hip, safflower, stokes aster, sunflower, tallowwood, tea oil plant, vernonia.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on globe artichoke and sunflower.

The field trial reports included method validation data, as recoveries from spiked samples at levels reflecting those observed in the field trial samples; dates from critical events during the study, including application, harvest, storage, and analysis; as well as detailed information on the field site and treatment parameters. Analytical reports were sufficiently detailed and included example chromatograms and example calculations. Samples were analysed by the method described above for plant commodities.

The field trial study designs included control plots. Measured residues from control plots were <LOQ and are not included in the summary tables in this evaluation.

When calculating average residues, values below the LOQ were assumed to be at the LOQ. In the summary tables, residue values leading to maximum residue estimations and used for long-term dietary risk assessment are underlined. The highest individual values selected for estimating acute dietary risks are bolded.

Although the submitted study reports include analysis of X11719474, that compound is not part of the residue definitions for sulfoxaflor. Therefore, field trial results for X11719474 are not included in this evaluation.

Table 4 Supervised trials for sulfoxaflor

Category	Crop	Table
Stalk and stem vegetables	Globe artichoke	Table 5
Oilseeds	Sunflower	Table 5

Globe artichoke

One trial was conducted in the United States during the 2021 season (Shepard, E. 2022, Report 210115). Treatment consisted of three foliar broadcast applications of ca. 100 g ai/ha, on a 7-day interval. Harvest occurred 0, 1, 3, 8, 14, and 21 days after the last application (DALA).

Following harvest, samples (12–14 flower heads, *ca*. 4–6 kg) were placed into frozen storage within 4 hours of collection and shipped frozen to the analytical facility. Upon arrive at the facility, samples were put into frozen storage. Prior to analysis, samples were homogenized in the presence of dry ice and then returned to frozen storage. Samples were stored for a maximum of 3.4 months prior to analysis. This duration is supported by available storage stability data reviewed by the 2021 Extra JMPR.

Samples were analysed for residues of sulfoxaflor using a QuEChERS analytical method. Concurrent recovery data indicate that the method is suitable.

Table 5 Results of sulfoxaflor residue trials in globe artichoke in the United States in 2021

Location; year (Trial ID)	Crop Variety	Application			Matrix	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Sulfoxaflor	X11719474 (sulfoxaflor equivalents)	
Critical GAP (United States)	--	3 [7]	101 (298/crop)	ns	--	3	--		--
Castroville, CA (Trial 01)	Madrigal	1 [--] 2 [7] 3 [6]	103 101 104	320 313 321	Flower head	0	0.69, 0.71 [0.70]	<0.015, <0.015 [<0.015]	210115
						1	0.57, 0.60 [0.58]	<0.015, <0.015 [<0.015]	
						3	0.36, 0.45 [0.41]	<0.015, <0.015 [<0.015]	
						8	0.17, 0.23 [0.20]	<0.015, <0.015 [<0.015]	
						14	0.097, 0.11 [0.10]	<0.015, <0.015 [<0.015]	
						21	0.049, 0.037 [0.043]	0.017, 0.016 [0.017]	
Field trials below evaluated by the 2021 Extra JMPR									
Salinas, CA, United States; 2014 (CA64)[A)]	$F_1$41 annual	1 [--] 2 [7] 3 [6]	101.7 101.6 101.2	94 94 94	Flower head	1	0.291, 0.251 [0.271]	<0.01, <0.01 [<0.01]	PR 10858
						3	0.254, 0.197 [0.226]	<0.01, <0.01 [<0.01]	
						7	0.127, 0.258 [0.192]	<0.01, <0.01 [<0.01]	
						14	0.0421, 0.0628 [0.0524]	<0.01, <0.01 [<0.01]	
						20	0.0179,	<0.01, <0.01	

Location; year (Trial ID)	Crop Variety	Application			Matrix	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Sulfoxaflor	X11719474 (sulfoxaflor equivalents)	
							0.0128 [0.0154]	[<0.01]	
Salinas, CA, United States; 2014 (CA66)[A)]	$F_1$41 annual	1 [--] 2 [7] 3 [9]	100.5 100.4 100.3	931 930 929	Flower head	3	0.149, 0.114 [0.132]	<0.01, <0.01 [<0.01]	
Castroville, CA, United States; 2014 (CA65)	Green globe perennial	1 [--] 2 [9] 3 [8]	101.4 101.3 103.7	705 704 721	Flower head	3	0.293, 0.227 [0.260]	<0.01, <0.01 [<0.01]	
L'Acadie, QC, Canada; 2014 (QC419)	Imperial star	1 [--] 2 [8] 3 [7]	101.6 98.2 102.6	493 477 498	Flower head	3	0.234, 0.199 [0.216]	<0.01, <0.01 [<0.01]	

Note:

[A)] Side-by-side trials using different spray volume/ha. Trials are not independent.

Sunflower seed

Five field trials were conducted in United States during the 2021 season (Shepard, E. 2022, Report 210116). Treatment consisted of two broadcast applications of sulfoxaflor each at *ca* 102 g ai/ha. The retreatment interval was 6–8 days. Harvest occurred 0 to 21 DALA.

Following harvest, samples (1–2.7 kg) were placed into frozen storage within 3 hours of collection and shipped frozen the analytical facility. Upon arrive at the facility, samples were put into frozen storage. Prior to analysis, samples were homogenized in the presence of dry ice and then returned to frozen storage. Samples were stored for a maximum of 4.4 months prior to analysis. This duration is supported by available storage stability data reviewed by the 2021 Extra JMPR.

Samples were analysed for residues of sulfoxaflor using a QuEChERS analytical method. Concurrent recovery data indicate that the method is suitable. Residues of X11719474 were not detectable in any sample of sunflower seed.

Table 6 Results of sulfoxaflor residue trials in sunflower in the United States in 2021

Location; year (Trial ID)	Crop Variety	Application			Matrix	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Sulfoxaflor	
Critical GAP (US)	--	2 [7]	96 (193/year)	--	--	14	--	--
Carlyle, IL (Trial 01)	SW1020CL	1 [--] 2 [6]	101 101	315 313	Seeds	0	0.16, 0.20 [0.18]	210116
						3	0.082, 0.17 [0.12]	
						7	0.096, 0.085 [0.090]	
						13	0.090, 0.093 [0.092]	
						21	0.058, 0.064 [0.061]	
Northwood, ND (Trial 02)	Cobalt II	1 [--] 2 [8]	102 103	280 280	Seeds	13	0.022, 0.026 [0.024]	
Velva, ND (Trial 03)	8N270CLDM	1 [--] 2 [7]	101 100	196 196	Seeds	14	0.22, 0.16 [0.19]	
Prosser, NE (Trial 04)	Peredovik	1 [--] 2 [7]	104 102	224 226	Seeds	14	0.056, 0.038 [0.047]	

Location; year (Trial ID)	Crop Variety	Application			Matrix	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Sulfoxaflor	
Great Bend, KS (Trial 05)	M88H477CLC473	1 [--] 2 [7]	103 103	195 196	Seeds	0	0.15, 0.28 [0.21]	
						3	0.096, 0.084 [0.090]	
						7	0.076, 0.10 [0.089]	
						14	0.075, 0.068 [0.071]	
						21	0.081, 0.072 [0.076]	
Field trials below evaluated by the 2021 Extra JMPR								
Fargo, ND, US; 2013 (ND14)	8N270CLDM	1 [-] 2 [6]	100 102	309 318	Seeds	16	0.0132, 0.0131 [0.013]	PR 11095
Minot, ND, US; 2013 (ND15)	8N270CLDM	1 [-] 2 [7]	102 99	215 206	Seeds	15	0.0164, 0.0205 [0.018]	
Las Cruces, NM, US; 2013 (NM12)	S678	1 [-] 2 [7]	108 102	299 290	Seeds	14	<0.01, <0.01 [<0.01]	
Aurora, SD, US; 2013 (SD08)	Durango	1 [-] 2 [6]	105 100	421 402	Seeds	15	0.146, 0.151 [0.15]	

APPRAISAL

Sulfoxaflor (ISO common name) is a broad-spectrum, sulfoximine insecticide with registered uses on multiple crops. It was evaluated for the first time by JMPR 2011, which established an acceptable daily intake (ADI) of 0–0.05 mg/kg bw and an acute reference dose (ARfD) of 0.3 mg/kg bw. Sulfoxaflor underwent subsequent evaluations by the JMPR in 2014, 2016, and 2021 (Extra). The 2022 JMPR agreed to consider additional residue data provided to support uses on globe artichoke and sunflower, for which the 2021 Extra Meeting was unable to make recommendations.

The definition of the residue in plants and animals for both compliance with MRLs and for dietary risk assessment is sulfoxaflor. The residue is not fat-soluble.

The current Meeting received information on residues in globe artichoke and sunflower to supplement trials evaluated at the 2021 Extra Meeting.

Methods of analysis

The Meeting received concurrent recovery data for use of a standard QuEChERS multiresidue method. The method was demonstrated to have adequate performance for sulfoxaflor from concurrent recovery samples of globe artichoke and sunflower seed, with an LOQ of 0.01 mg/kg. Metabolism studies evaluated by the 2011 JMPR showed high extraction efficiency (73–97 percent TRR) with similar solvent as that used in the QuEChERS method.

Stability of pesticide residues in stored analytical samples

Based on data provided to it, the 2021 Extra Meeting concluded that residues of sulfoxaflor are stable for at least 24 months in globe artichoke and in sunflower seeds during frozen storage. In the studies provided to the current Meeting, samples were stored at -20 °C for up to 3.4 months (globe artichoke) and 4.4 months (sunflower seed).

Results of supervised residue trials on crops

The Meeting received data from supervised residue trials in globe artichoke and sunflower. These data were to supplement studies reviewed by the 2021 Extra Meeting, which concluded that the numbers of trials for those crops were insufficient to make recommendations.

Globe artichoke

The critical GAP for globe artichoke is from the United States. The label provides for up to four applications, each at 101 g ai/ha, on a 7-day interval, with a 3-day PHI. The label also specifies an annual limit of 298 g ai/ha; thus, the critical GAP is three applications at the maximum rate.

Residue of sulfoxaflor in globe artichoke from the additional trial provided to the meeting was (n=1): 0.41 mg/kg. In independent trials evaluated by the 2021 Extra JMPR, residues were (n=3): 0.22, 0.23, and 0.26 mg/kg. Altogether, residues of sulfoxaflor in globe artichoke from independent trials matching the critical GAP were (n=4): 0.22, 0.23, 0.26, and 0.41 mg/kg.

The Meeting estimated a maximum residue level of 0.9 mg/kg, an STMR of 0.245 mg/kg, and an HR of 0.45 mg/kg (from a single sample) for sulfoxaflor in globe artichoke.

Subgroup of sunflower seeds

Sunflower seed

The critical GAP is from the United States on the sunflower subgroup of oilseeds and consists of two applications, each at 96 g ai/ha, with a 7-day re-treatment interval and a 14-day PHI.

Residues of sulfoxaflor in sunflower seeds from independent trials approximating the critical GAP were (n=5): 0.024, 0.047, 0.076, 0.092, and 0.19 mg/kg. In independent trials approximating the critical GAP and evaluated by the 2021 Extra JMPR, residues were (n=4): < 0.01, 0.013, 0.018, and 0.15 mg/kg. Altogether, residues of sulfoxaflor in sunflower seeds from independent trials approximating the critical GAP were (n=9): < 0.01, 0.013, 0.018, 0.024, 0.047, 0.076, 0.092, 0.15, and 0.19 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and a STMR of 0.047 mg/kg for residues of sulfoxaflor in sunflower seed. Noting that the registered use includes all commodities in the Codex Subgroup of sunflower seeds (SO 2091) and that sunflower seed is the recommended representative commodity, the Meeting agreed to extrapolate the recommendations to Subgroup 023B Sunflower Seeds.

Fate of residues during processing

The 2021 Extra Meeting evaluated a sunflower seed processing study and derived best-estimate processing factors of 0.71 for both sunflower meal and refined oil. Based on the STMR of 0.047 mg/kg in seed, the STMR-Ps for both meal and refined oil were 0.033 mg/kg.

Residues in animal commodities

The only animal feed item considered by the current Meeting was sunflower meal. Inclusion of sunflower meal in the animal dietary calculations did not change the maximum and mean burdens for cattle or poultry from those calculated by the 2014 JMPR (3.22 and 1.26 ppm respectively for cattle and 0.93 and 0.31 ppm respectively for poultry); therefore, the Meeting confirmed is previous recommendations for residues in animal commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL and for dietary risk assessment for plant and animal commodities: *sulfoxaflor.*

The residue is not fat-soluble.

Table 7 Recommendations for residues of sulfoxaflor from the 2022 JMPR

CCN	Crop/Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
VS 0620	Globe artichoke	0.9		0.245	0.45
SO 2091	Subgroup of sunflower seeds	0.4	--	0.047	--
For dietary risk assessment and/or dietary burden calculations					
AM 0702	Sunflower seed, meal	--	--	0.033	--
OR 0702	Sunflower seed oil, edible	--	--	0.033	--

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for sulfoxaflor is 0–0.05 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for sulfoxaflor were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 1–7 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of sulfoxaflor from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for sulfoxaflor is 0.3 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for sulfoxaflor were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2022 JMPR Report.

The IESTIs varied from 0–2 percent of the ARfD for children and 0–1 percent for the general population. The Meeting concluded that acute dietary exposure to residues of sulfoxaflor from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Report Code	Author(s)	Year	Title
210115	Shepard, E.	2022	Magnitude of the Residues of Sulfoxaflor and X11719474 in or on Globe Artichoke Raw Agricultural Commodities Following Three Applications with GF-2032 (2021)
210116	Shepard, E.	2022	Magnitude of the Residues of Sulfoxaflor and X11719474 in or on Sunflower Raw Agricultural Commodities Following Two Applications with GF-2372 (2021)

TETRANILIPROLE (324)

First draft prepared by K. Mahieu and T. van der Velde-Koerts, Centre for Nutrition, Prevention and Health Services (VPZ), National Institute for Public Health and the Environment (RIVM), The Netherlands

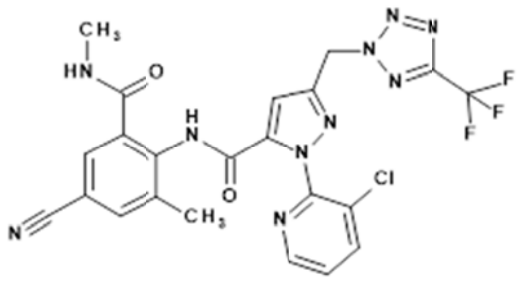
EXPLANATION

Tetraniliprole (ISO name) is a broad spectrum fast acting insecticide that belongs to the anthranilic diamide chemical class. The mode of action of anthranilic diamides involves activating ryanodine receptors, which play a critical role in muscle function.

Tetraniliprole was scheduled at the Fifty-first Session of the CCPR (2019) for evaluation as a new compound by the 2020 JMPR, which was postponed to the 2021 JMPR for toxicology and to the 2022 JMPR for residues. The 2021 JMPR estimated and ADI of 0–2 mg/kg bw and concluded that an ARfD was not necessary.

The Meeting received information on identity, physical chemical properties, plant and animal metabolism, aerobic soil degradation, residue analysis, storage stability, use patterns, supervised trials on citrus fruits, pome fruits, stone fruits, grapes, head and stem brassica vegetables, fruiting vegetables, leafy vegetables, soya beans, root and tuber vegetables, cereal grains, tree nuts, and rice, fate of residues during processing, and livestock feeding studies.

IDENTITY

ISO common name:	Tetraniliprole
IUPAC name:	1-(3-chloropyridin-2-yl)-N-[4-cyano-2-methyl-6-(methyl¬carbamoyl) phenyl]-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide
CAS name:	1H-pyrazole-5-carboxamide, 1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-methyl-6-[(methylamino)carbonyl]phenyl]-3-[[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl]-(9CI)
CAS Registry No:	1229654-66-3
CIPAC No:	not allocated
Structural formula:	
Molecular formula	$C_{22}H_{16}ClF_3N_{10}O_2$
Molecular weight:	544.88 g/mol

PHYSICAL AND CHEMICAL PROPERTIES

Table 1a Physical and chemical properties of the pure active ingredient

Parameter	Result	References	Guidelines/method
Appearance: Batch: 2012-003401	Purity: 98.3% w/w Colour: beige	Eyrich& Ziemer, 2013a, M-464861-01-1, Report	OPPTS 830.6302/ visual, 830.6303/ visual and

Parameter	Result	References	Guidelines/method
	Physical state: powder (24 °C) Odour: Acetous (24 °C)	PA13/064	830.6304/ olfactory determination
Vapour pressure: Batch: 2012-003401 (light grey powder)	Purity: 98.3% w/w 3.2×10^{-6} mPa at 20 °C 4.6×10^{-6} mPa at 25 °C 2.3×10^{-5} mPa at 50 °C	Dreisch, 2013, M-467652-01-1, Report CSL-13-0703.01	OECD 104 and 113 and OPPTS 830.7950 Extrapolation from the experimental data (DCS for determination of thermal stability followed by vapour pressure balance method)
Melting point: Batch: 2012-003401 (light grey powder)	Purity: 98.3% w/w 226.9-229.6 °C	Winkler, 2013, M-462084-01-1, Report 20130189.01	OECD 102 (1995) Melting point / melting range and OPPTS 830.7200/ DCS: Differential Scanning Calorimetry
Boiling point/Decomposition temperature: Batch: 2012-003401 (light grey powder)	Purity: 98.3% w/w Decomposition: starting at 230 °C	Winkler, 2013, M-462084-01-1, Report 20130189.01	OECD 103 (1995) Boiling point / boiling range OECD 113 (1981) Screening test for thermal stability and stability in air and OPPTS 830.7220/ DCS: Differential Scanning Calorimetry
Octanol/water partition coefficient (25 °C): Batch: 2012-003401	Purity: 98.3% w/w 2.6 (pH = 4) 2.6 (pH = 7) 1.9 (pH = 9)	Eyrich&Ziemer, 2013b, M-472127-01-1, Report PA13/062	OECD 117, EC Guideline L383A, Method A8 and OPPTS 830.7550/ HPLC method
Solubility in water (20 °C): Batch: 2012-003401	Purity: 98.3% w/w 1.2 mg/L (distilled water with final pH of 6.31)) 1.0 mg/L (pH 4) 1.0 mg/L (pH 7) 1.3 mg/L (pH 9)	Wiche&Ziemer, 2013a, M-470608-01-1, Report PA13/078	OECD 105, EC Guideline L383A, Method A6 and OPPTS 830.7840.SUPP/ flask method
Solubility in organic solvents (20 °C): Batch: 2012-003401	Purity: 98.3 % w/w <<0.001 g/L in heptane 0.17 g/L in toluene 2.9 g/L in methanol 5.3 g/L in dichloromethane 6.4 g/L in ethyl acetate 21.8 g/L in acetone >280 in dimethyl sulfoxide	Eyrich&Ziemer, 2014a, M-476259-01-1, Report PA13/103	OECD 105, EC Guideline L383A, Method A6 and OPPTS 830.7840/flask method + HPLC analyses
Relative density (D_4^{20}): Batch: 2012-003401	Purity: 98.3% w/w 1.52 (20 °C), compared to water at 4 °C	Ziemer&Strunk, 2013, M-463068-01-1, Report PA13/090	OPPTS 830.730/ Air comparison pycnometer (solids)
Specific gravity:	1.52 g/cm^3 at 20 °C Derived from relative density based on density for water of 1.0 g/cm^3	Ziemer&Strunk, 2013, M-463068-01-1, Report PA13/090	OPPTS 830.730/ Air comparison pycnometer (solids)
Hydrolysis in sterile water in the dark: Sample ID: KML 9394 Radiolabel: [pyrazole-carboxamide-^{14}C]	(Radio)chemical purity: >99% DT_{50} (days) pH 4 265 days at 20 °C pH 7 58.0 days at 20 °C pH 9 1.27 days at 20 °C DT_{50} (days) pH 4 287 days at 25 °C pH 7 38.8 days at 25 °C pH 9 0.75 days at 25 °C DT_{50} (days) pH 4 10.9 days at 50 °C pH 7 3.74 days at 50 °C	Hein & Kasel, 2016, M-565616-01-1, Report M1112152-3	OECD 111, OPPTS 835.2120 and 835.2130

Parameter	Result	References	Guidelines/method
	pH 9 0.04 days at 50 °C At normal temperatures tetraniliprole is stable at pH 4, hydrolyses slowly at pH 7 and rapidly at pH 9. One degradation product was identified as tetraniliprole-N-methyl-quinazolinone with a maximum amount of 99.6% AR. At higher temperatures tetraniliprole degrades more rapidly.		
Photolysis in sterile water: Sample ID: KML 9532 Radiolabel: [pyrazole-carboxamide-^{14}C]	(Radio)chemical purity: >98% Photodegradation tetraniliprole was studied under simulated sunlight in sterile aqueous acetate buffer solution (pH 4) at 25 ºC for 11 days with 0.48 mg ai/L, with a mean irradiance of 694 Watts/m^2. Under these conditions tetraniliprole was rapidly degraded with an experimental DT_{50} value of 3.4 days and DT_{90} value of 11.3 days. The predicted environmental DT_{50} value is calculated to be e.g. 10.5 solar summer days at Phoenix, Arizona, United States. Tetraniliprole was stable under dark conditions (a DT_{50} of 188.5 days and a DT_{90} of 626.2 days). One major degradation product was identified as tetraniliprole-deschloro-oxazine with a maximum amount of 72.7% AR. The sum of five minor unidentified metabolites increased to 12.5% AR at day 11, with max individual levels of 1.9-6.6% AR. The mean material balances were 98.9% AR for irradiated samples and 102.6% AR for dark samples	Heinemann & Kasel, 2014, M-484185-01-1, Report EnSa-13-0320	OECD 316, OPPTS 835.2240
Dissociation constant in water: Batch: 2012-003401	Purity: 98.3% w/w pKa = 9.1 (n=3)	Wiche & Ziemer, 2013b, M-471896-01-1, Report PA13/146	OECD 112 and OPPTS 830.7370.SUPP/ spectrophotometry
Volatility at 20 °C: (Henry's law constants) Batch: 2012-003401	Purity: not reported 1.5×10^{-3} Pa m³/mol (distilled water pH 6.3) 1.7×10^{-3} Pa m³/mol (pH 4) 1.7×10^{-3} Pa m³/mol (pH 7) 1.3×10^{-3} Pa m³/mol (pH 9)	Ziemer, 2013, M-471687-01-2, Report AF13/035	Calculated based on vapour pressure and solubility in water (Report CSL-13-0703.01 and Report PA13/078)
Surface tension: Batch: 2012-003401	Purity: 98.3% w/w 72.1 mN/m at 20 °C	Eyrich & Ziemer, 2014b, M-485602-01-2, Report PA14/024	OECD 115 OPPTS 830.SUPP/ harmonised ring method

Table 1b Physical and chemical properties of the Technical material

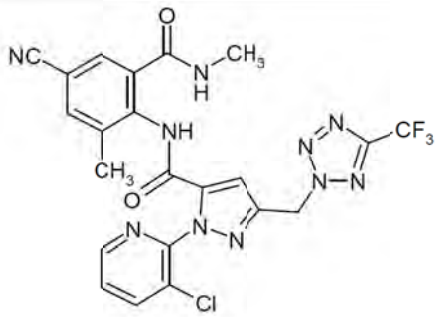
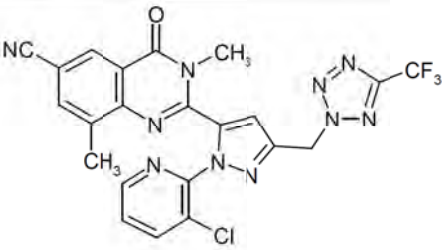
Parameter	Result	References	Guidelines
Appearance: Batch: PFV15AK006	Purity: 96.6% w/w Colour: light yellow Physical state: powder (23 °C) No odour (23 °C)	Ziemer & Strunk, 2015a, M-541574-01-1, Report PA15/126	OPPTS 830.6302/ visual, OPPTS 830.6303/ visual and OPPTS 830.6304/ olfactory determination
Melting range: Batch: PFV15AK006	Purity: 96.6% w/w 228.4-230.1°C under decomposition	Nau, 2016, M-548001-01-1, Report 20150415.01	OECD 102 and OPPTS 830.7200/ DCS
pH in distilled water: Batch: PFV15AK006	Purity: 96.6% w/w 5.2 (23°C), n=2 1% suspension in water	Ziemer & Strunk, 2015b, M-541564-01-1, Report PA15/125	OPPTS 830.7000, CIPAC MT 75/ pH meter
Thermal stability: Batch: PFV15AK006	Decomposition: starting at 235 °C	Nau, 2016, M-548001-01-1, Report 20150415.01	OECD 103 and 113 and OPPTS 830.7220/ DCS

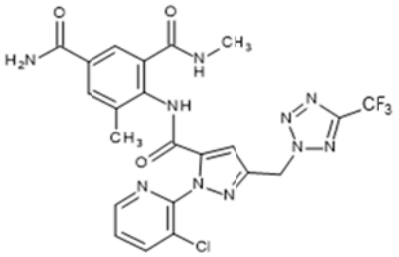
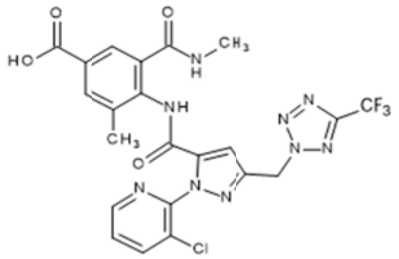
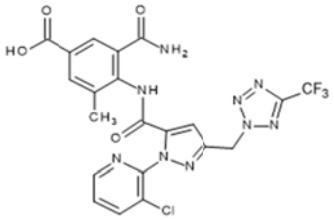
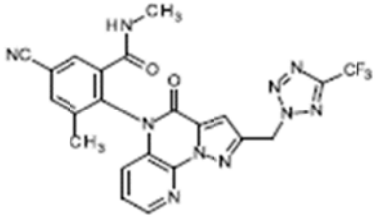
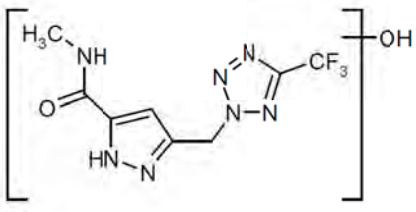
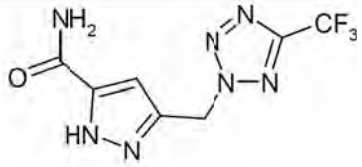
Formulations

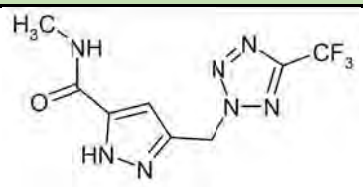
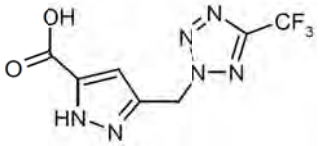
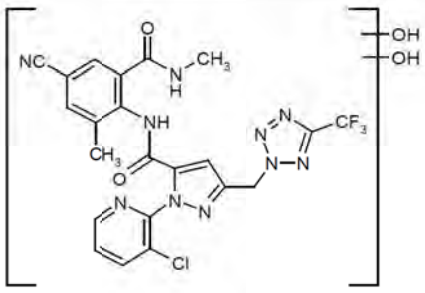
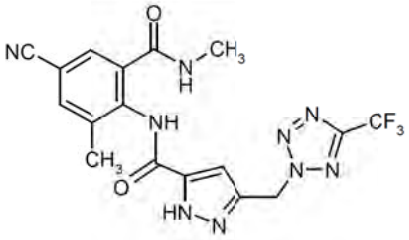
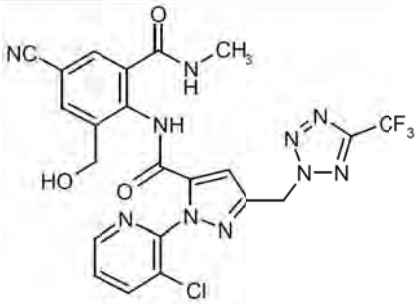
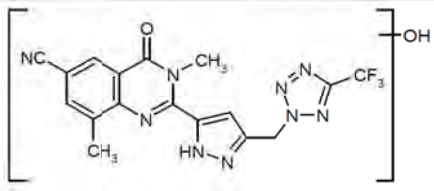
Tetraniliprole has not been evaluated by JMPS and therefore no FAO specifications for technical and formulated tetraniliprole have been published.

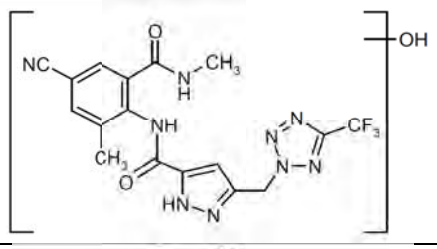
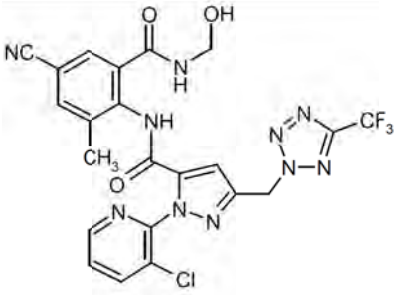
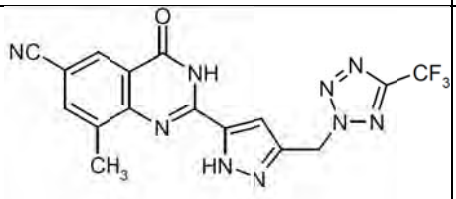
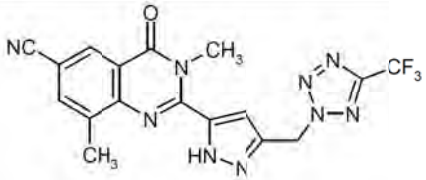
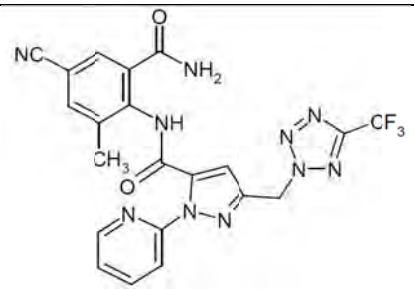
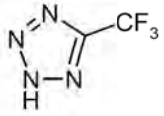
A suspension concentrate (SC) formulation containing 200 g ai/L is commercially available in Australia, Canada, Guatemala, Korea, New Zealand, Peru and the United States. A flowable concentrate for seed treatment (FS) formulation containing 480 g ai/L is commercially available in the Australia, Canada, Japan and the United States as well as a granule box 15 g/kg in Japan.

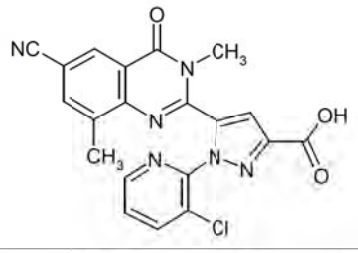
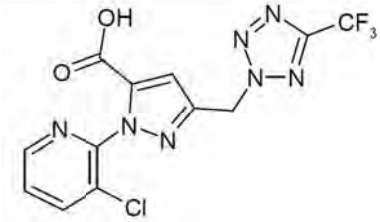
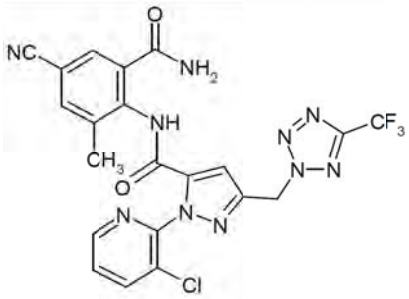
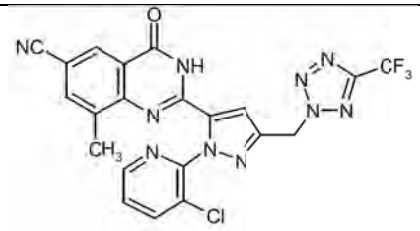
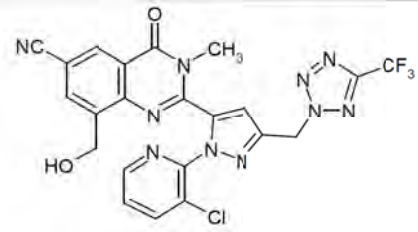
Abbreviations

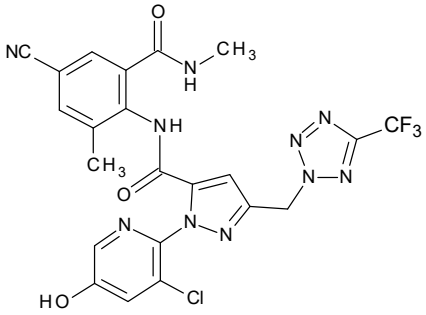
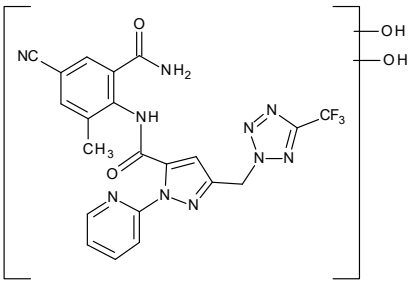
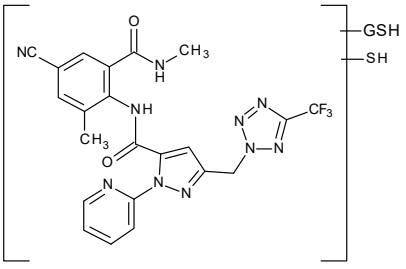
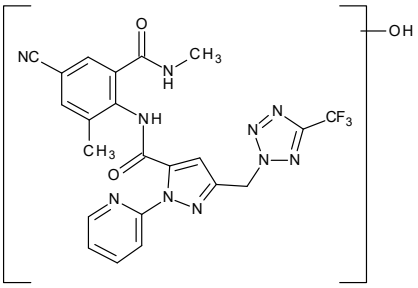
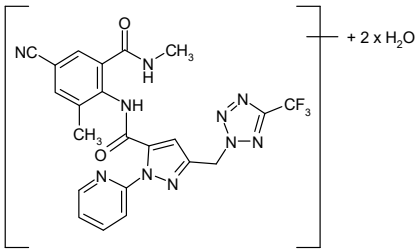
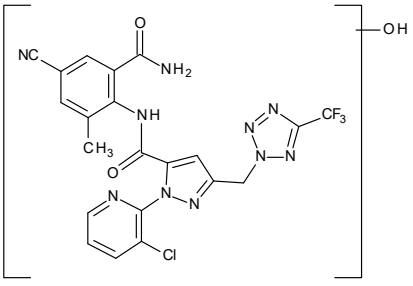
Name/Code	Chemical structure	Chemical name	Found in
Tetraniliprole Code: BCS-CL73507		1-(3-chloropyridin-2-yl)-N-[4-cyano-2-methyl-6-(methylcarbamoyl)phenyl]-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide Molecular formula: $C_{22}H_{16}ClF_3N_{10}O_2$ Molecular weight: 544.88 g/mol	Primary crops (tomato, apple, potato, lettuce, paddy rice, maize), poultry, ruminant, rat
Tetraniliprole-N-methyl-quinazolinone Code: BCS-CQ63359		2-[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]-3,8-dimethyl-4-oxo-3,4-dihydroquinazoline-6-carbonitrile Molecular formula: $C_{22}H_{14}ClF_3N_{10}O$ Molecular weight: 526.86 g/mol	Primary crops (tomato, potato, paddy rice, maize), poultry, ruminant, rat

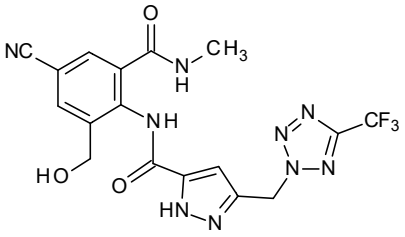
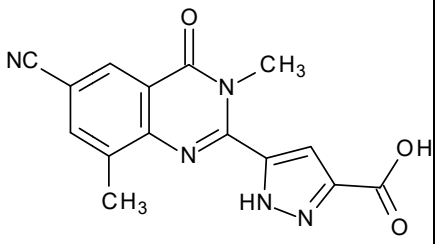
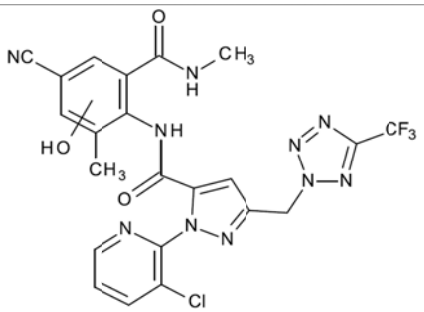
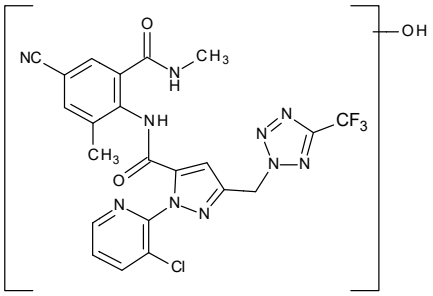
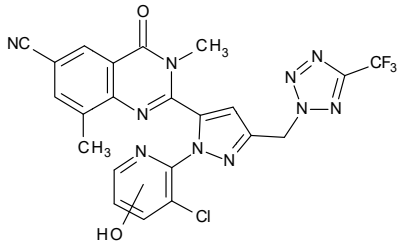
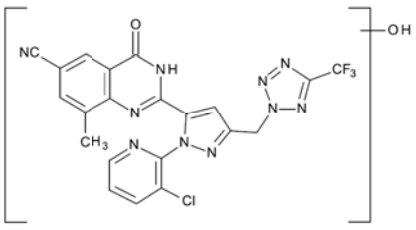
Name/Code	Chemical structure	Chemical name	Found in
Tetraniliprole-amide Code: BCS-CR60014		4-({[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]carbonyl}amino)-N3,5-dimethylisophthalamide Molecular formula: $C_{22}H_{18}ClF_3N_{10}O_3$ Molecular weight: 562.9 g/mol	Confined rotational crops, soil degradation,
Tetraniliprole-carboxylic acid Code: BCS-CR74541		4-({[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]carbonyl}amino)-3-methyl-5-(methylcarbamoyl) benzoic acid Molecular formula: $C_{22}H_{17}ClF_3N_9O_4$ Molecular weight: 563.9 g/mol	Confined rotational crops, Soil degradation studies
Tetraniliprole-desmethyl-amide-carboxylic acid Code: BCS-CU81055		3-carbamoyl-4-({[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]carbonyl}amino)-5-methylbenzoic acid Molecular Formula: $C_{21}H_{15}ClF_3N_9O_4$ Molecular Weight: 549.9 g/mol	Confined rotational crops, Soil degradation studies
Tetraniliprole-deschloro-pyrazine Code: BCS-CY28897		5-cyano-N,3-dimethyl-2-[4-oxo-2-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}pyrazolo[1,5-a]pyrido[3,2-e]pyrazin-5(4H)-yl]benzamide Molecular formula: $C_{22}H_{15}F_3N_{10}O_2$ Molecular weight: 508.43 g/mol	Photodegradation in natural water
Tetraniliprole-pyrazole-5-N-methylamide-hydroxy		Not available	Poultry, rat
Tetraniliprole-pyrazole-5-amide Code: BCS-CY28908		3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide Molecular formula: $C_7H_6F_3N_7O$ Molecular weight: 261.2 g/mol	Poultry, ruminant, rat

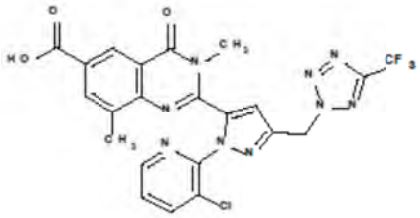
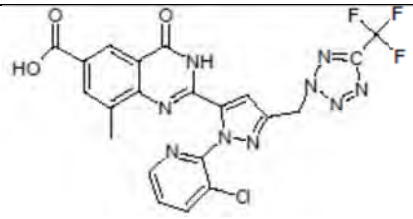
Name/Code	Chemical structure	Chemical name	Found in
Tetraniliprole-pyrazole-5-N-methyl-amide Code: BCS-CZ84317		N-methyl-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide Molecular formula: C8H8F3N7O Molecular weight:: 275.2 g/mol	Poultry, ruminant, rat
Tetraniliprole-pyrazole-5-carboxylic acid Code: BCS-CY28906		3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxylic acid Molecular formula: $C_7H_5F_3N_6O_2$ Molecular weight:: 262.2 g/mol	Poultry, ruminant, rat
Tetraniliprole-dihydroxy		Not available	Poultry, rat
Tetraniliprole-despyridyl Code: BCS-CT27799		N-[4-cyano-2-methyl-6-(methylcarbamoyl)phenyl]3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide Molecular formula: $C_{17}H_{14}F_3N_9O_2$ Molecular weight:: 433.35 g/mol	Poultry, rat
Tetraniliprole-benzylalcohol BCS-CZ91631		1-(3-chloropyridin-2-yl)-N-[4-cyano-2-(hydroxymethyl)-6-(methylcarbamoyl)phenyl]-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide Molecular formula: $C_{22}H_{16}ClF_3N_{10}O_3$ Molecular weight: 560.88 g/mol	Poultry, ruminant, rat
Tetraniliprole-despyridyl-N-methyl-quinazolinone-hydroxy		Not available	Poultry

Name/Code	Chemical structure	Chemical name	Found in
Tetraniliprole-despryridyl-hydroxy		Not available	Poultry
Tetraniliprole-hydroxy-N-methyl Code: BCS-CZ91629		1-(3-chloropyridin-2-yl)-N-{4-cyano-2-[(hydroxymethyl)carbamoyl]-6-methylphenyl}-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide Molecular formula: $C_{22}H_{16}ClF_3N_{10}O_3$ Molecular weight:: 560.9 g/mol	Poultry, ruminant, rat
Tetraniliprole-despyridyl-quinazolinone		8-methyl-4-oxo-2-(3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl)-3,4-dihydroquinazoline-6-carbonitrile Molecular formula: $C_{16}H_{10}F_3N_9O$ Molecular weight;; 401.1 g/mol	Poultry
Tetraniliprole-despyridyl-N-methyl-quinazolinone Code: BCS-CY28894		3,8-dimethyl-4-oxo-2-(3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl)-3,4-dihydroquinazoline-6-carbonitrile Molecular formula: $C_{17}H_{12}F_3N_9O$ Molecular weight:: 415.3 g/mol	Poultry, rat Photodegradation in natural water
Tetraniliprole-deschloro-desmethyl-amide		N-(2-carbamoyl-4-cyano-6-methylphenyl)-1-(pyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide Molecular formula: $C_{21}H_{15}F_3N_{10}O_2$ Molecular weight;; 496.4 g/mol	Poultry, rat
Tetraniliprole-tetrazole Code: BCS-BS22071		5-(trifluoromethyl)-2H-tetrazole Molecular formula: $C_2HF_3N_4$ Molecular weight:: 138.1 g/mol	Poultry, ruminant, rat

Name/Code	Chemical structure	Chemical name	Found in
Tetraniliprole-N-methyl-quinazolinone-pyrazole-3-carboxylic acid		1-(3-chloropyridin-2-yl)-5-(6-cyano-3,8-dimethyl-4-oxo-3,4-dihydroquinazolin-2-yl)-1H-pyrazole-3-carboxylic acid Molecular formula: $C_{20}H_{13}ClN_6O_8$ Molecular weight:: 500.8 g/mol	Ruminant
Tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid		1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxylic acid Molecular formula: $C_{12}H_7ClF_3N_7O_2$ Molecular weight:373.7 g/mol	Ruminant, rat
Tetraniliprole-desmethyl-amide Code: BCS-CN42374		N-(2-carbamoyl-4-cyano-6-methylphenyl)-1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide Molecular formula: $C_{21}H_{14}ClF_3N_{10}O_2$ Molecular weight:: 530.9 g/mol	Ruminant, rat
Tetraniliprole-quinazolinone		2-[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]-8-methyl-4-oxo-3,4-dihydroquinazoline-6-carbonitrile Molecular formula: $C_{21}H_{12}ClF_3N_{10}O$ Molecular weight:: 512.8 g/mol	Ruminant, rat
Tetraniliprole-N-methyl-quinazolinone-benzylalcohol		2-[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazol-5-yl]-8-(hydroxymethyl)-3-methyl-4-oxo-3,4-dihydroquinazoline-6-carbonitrile Molecular formula: $C_{22}H_{14}ClF_3N_{10}O_2$ Molecular weight:: 542.9 g/mol	Ruminant, rat

Name/Code	Chemical structure	Chemical name	Found in
Tetraniliprole-5-hydroxypyridine		1-(3-chloro-5-hydroxypyridin-2-yl)-N-[4-cyano-2-methyl-6-(methylcarbamoyl)phenyl]-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide Molecular formula: $C_{22}H_{16}ClF_3N_{10}O_3$ Molecular weight: 560.9 g/mol	Rat
Tetraniliprole-deschloro-desmethyl-amide-dihydroxy		Not available	Rat
Tetraniliprole-deschloro-GSH-thio-conjugate		Not available	Rat
Tetraniliprole-deschloro-hydroxy		Not available	Rat
Tetraniliprole-deshydrochloro-dihydrate		Not available	Rat
Tetraniliprole-desmethyl-amide-hydroxy		Not available	Rat

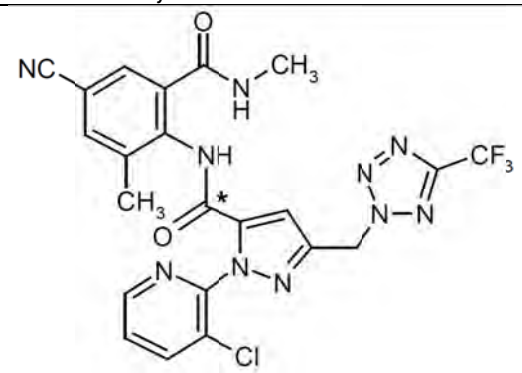
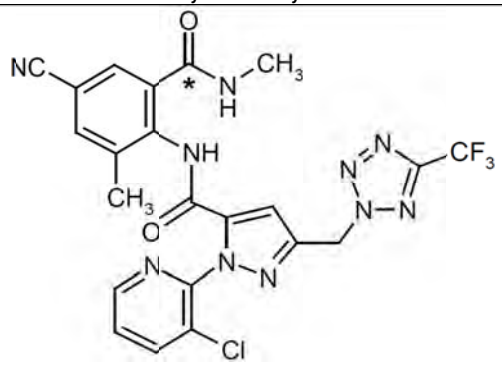
Name/Code	Chemical structure	Chemical name	Found in
Tetraniliprole-despyridyl-benzylalcohol		N-[4-cyano-2-(hydroxymethyl)-6-(methylcarbamoyl)phenyl]-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1H-pyrazole-5-carboxamide Molecular formula: $C_{17}H_{14}F_3N_9O_3$ Molecular weight:: 449.4 g/mol	Rat
Tetraniliprole-despyridyl-N-methyl-quinazolinone-pyrazole-3-carboxylic acid		5-(6-cyano-3,8-dimethyl-4-oxo-3,4-dihydroquinazolin-2-yl)-1H-pyrazole-3-carboxylic acid Molecular formula: $C_{15}H_{11}N_5O_3$ Molecular weight:: 309.3 g/mol	Confined rotational crops, rat
Tetraniliprole-phenylhydroxy		Not available	Rat
Tetraniliprole-hydroxy		Not available	Ruminant, rat
Tetraniliprole-N-methyl-quinazolinone-hydroxypyridyl		Not available	Rat
Tetraniliprole-quinazolinone-hydroxy		Not available	Ruminant

Name/Code	Chemical structure	Chemical name	Found in
Tetraniliprole-N-methyl-quinazolinone-carboxylic acid Code: BCS-CT30673		2-[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1Hpyrazol-5-yl]-3,8-dimethyl-4-oxo-3,4-dihydroquinazoline-6-carboxylic acid Molecular formula: $C_{22}H_{16}ClF_3N_9O_3$ Molecular weight: 545.9 g/mol	soil
Tetraniliprole - quinazolinone-carboxylic acid Code: BCS-CU81056		2-[1-(3-chloropyridin-2-yl)-3-{[5-(trifluoromethyl)-2H-tetrazol-2-yl]methyl}-1Hpyrazol-5-yl]-8-methyl-4-oxo-3,4-dihydroquinazoline-6-carboxylic acid Molecular formula: $C_{21}H_{13}ClF_3N_9O$ Molecular weight: 531 g/mol	soil

METABOLISM AND ENVIRONMENTAL FATE

Plant metabolism

The meeting received plant metabolism studies for tetraniliprole after application on tomatoes (soil drench), apples (foliar), potatoes (foliar applications or seed treatment in furrow), lettuce (foliar a), paddy rice (foliar application or granular treatment in planting holes) and maize (seed treatment) using either [pyrazole-carboxamide-^{14}C]- or [phenyl-carbamoyl-^{14}C]-labelled tetraniliprole, as shown below.

Pyrazole-carboxamide label	Phenyl-carbamoyl label
	
[pyrazole-carboxamide-^{14}C]-tetraniliprole	[phenyl-carbamoyl-^{14}C]-tetraniliprole

Notes:

* = Position of ^{14}C-radiolabel.

Seed treatments and soil treatments (soil drench, in-furrow, granular)

Tomato–Indoor soil drench application

The metabolic fate of tetraniliprole after a single soil drench application was investigated in tomato plants (*Lycopersicon esculentum* Mill., variety: Philona) in two studies (Bongartz and Schmeling, 2014a, M-495009-01-1, Report EnSa-14-0485 and Bongartz and Schmeling, 2014b, M-495019-01-1, Report EnSa-14-0484). Plants were grown individually in pots in a greenhouse in Germany (Monheim am Rhein). Radiolabelled tetraniliprole was formulated as SC 200 and applied to two tomato plants at growth stage BBCH 15–16 (fifth to sixth leaf on main shoot unfolded) as a single drench application to Einheitserde T soil (characteristics not reported). The application rate with pyrazole-carboxamide label was 7.81 mg ai/plant, equivalent to 156.3 g ai/ha based on a plant density of 20,000 plants/ha (Report EnSa-14-0485). The application rate with phenyl-carbamoyl labelwas 7.65 mg ai/plant, equivalent to 153.1 g ai/ha based on a plant density of 20,000 plants/ha (Report EnSa-14-0484).

Tomatoes were picked from the plants during the whole ripening period (BBCH 81–89), between 83 to 99 days after application. In intervals of 2–3 days the fruits which appeared fully ripe were picked and stored. At the end of the ripening period, after all fruits were harvested (BBCH 89) a leaf sample was taken from each plant. The tomato and leaf samples were extracted three times with a mixture of acetonitrile/water/formic acid (8/2/0.1, pH determined but not reported) followed by a partitioning step of the combined extracts against dichloromethane.

The total radioactive residue (TRR) values of the extracted samples were quantified by LSC and were calculated based on the radioactivity determined in the extracted phases and the remaining solids.

Pyrazole-carboxamide label: The extraction efficiency was >90 percent TRR for both fruit and leaf samples. For tomatoes, the main portion of the radioactivity (67.8 percent TRR) partitioned into the organic phase and 22.9 percent TRR into the aqueous phase. Similar to the fruit, the main portion of the radioactivity (75.7 percent TRR) in the leaf samples partitioned into the organic phase and 18.3 percent TRR into the aqueous phase.

Phenyl-carbamoyl label: The extraction efficiency was >86 percent TRR for both fruit and leaf samples. For tomatoes, the main portion of the radioactivity (55.5 percent TRR) partitioned into the organic phase and 31.0 percent TRR into the aqueous phase. Similarly in leaves, the main portion of the radioactivity (77.9 percent TRR) partitioned into the organic phase and 16.6 percent TRR into the aqueous phase.

The distribution of radioactivity in the tomato fruit and leaf samples is presented in Table 2.

Table 2 Distribution of radioactivity in the extracts of tomato fruits and leaves following indoor drench application of pyrazole-carboxamide and of phenyl-carbamoyl labelled tetraniliprole

	Pyrazole-carboxamide label				Phenyl-carbamoyl label			
Study/ report number	M-495009-01-1, EnSa-14-0485				M-495019-01-1, EnSa-14-0484			
Application	1 × 156 g ai/ha, drench application				1 × 153 g ai/ha drench application			
Sample	Tomato fruits		Tomato leaves		Tomato fruits		Tomato leaves	
Days after treatment	83-99		99		83-99		99	
TRR (mg eq/kg)	<0.001		0.005		<0.001		0.006	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total extracted	90.7	<0.001	93.9	0.005	86.5	<0.001	94.4	0.005
Partition organic phase total	67.8	<0.002	75.7	0.004	55.5	<0.002	77.9	0.004
Partition aqueous phase	22.9	<0.001	18.3	0.001	31.0	<0.001	16.6	0.001
PES	9.3	<0.001	6.1	<0.001	13.5	<0.001	5.6	<0.001

	Pyrazole-carboxamide label				Phenyl-carbamoyl label			
Study/ report number	M-495009-01-1, EnSa-14-0485				M-495019-01-1, EnSa-14-0484			
Application	1 × 156 g ai/ha, drench application				1 × 153 g ai/ha drench application			
Sample	Tomato fruits		Tomato leaves		Tomato fruits		Tomato leaves	
Days after treatment	83-99		99		83-99		99	
Accountability	100	<0.001	100	0.005	100	<0.001	100	0.006

The organic phases of both leaf and fruit samples were cleaned up by SPE. The measurement of the radioactivity in liquid samples was carried out by liquid scintillation counting (LSC). All solid samples were combusted in an oxygen atmosphere using an oxidiser. The released $^{14}CO_2$ was trapped in an alkaline scintillation cocktail and the radioactivity was determined by LSC. HPLC-radioactivity analysis was performed with radiometric and UV-detection. Non-radiolabelled reference compounds (tetraniliprole and tetraniliprole-N-methyl-quinazolinone) were used.

Parent tetraniliprole was a major compound in both fruit and leaves and amounted to 22 percent TRR (<0.001 mg eq/kg) and 24 percent TRR (0.001 mg eq/kg), respectively with the pyrazole-carboxamide label and amounted to 34 percent (<0.001 mg eq/kg) and 27 percent (0.002 mg eq/kg) TRR, respectively with the phenyl-carbamoyl label. Tetraniliprole-N-methyl-quinazolinone was a major metabolite and amounted to 11 percent TRR (<0.001 mg eq/kg) for fruit and 34 percent TRR (0.002 mg eq/kg) for leaves with the pyrazole-carboxamide label and amounted to 20 percent (<0.001 mg eq/kg) TRR for fruit and 37 percent (0.002 mg eq/kg) TRR for leaves with the phenyl-carbamoyl label. All other detected metabolites in leaf extracts each amounted to ≤ 4.1–4.4 percent TRR (0.001 mg eq/kg) with both labels. The metabolic profile of tetraniliprole in tomato fruit and leaf samples is presented in Table 3.

Table 3 Distribution of parent compound and metabolites in the extracts of tomato fruits and leaves following in door drench application of pyrazole-carboxamide and of phenyl-carbamoyl labelled tetraniliprole

Label	Pyrazole-carboxamide label				Phenyl-carbamoyl label			
Study / report number	M-495009-01-1, EnSa-14-0485				M-495019-01-1, EnSa-14-0484			
Application	1 × 156 g ai/ha, drench application				1 × 153 g ai/ha drench application			
Component / Sample	Tomato fruits		Tomato leaves		Tomato fruits		Tomato leaves	
Days after treatment	83-99		99		83-99		99	
TRR (mg eq/kg)	<0.001		0.005		<0.001		0.006	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Organic phase of the acetonitrile/water/formic acid extract								
Tetraniliprole	22.4	<0.001	24.5	0.001	34.0	<0.001	27.1	0.002
Tetraniliprole-N-methyl-quinazolinone	10.7	<0.001	33.7	0.002	20.0	<0.001	37.2	0.002
Total identified	33.1	<0.001	58.2	0.003	54.1	<0.001	64.3	0.004
Unknown 1	14.5	<0.001	-	-	-	-	-	-
Unknown 2	15.5	<0.001	-	-	-	-	-	-
Unknown 3	-	-	1.8	<0.001	-	-	-	-
Unknown 4	-	-	3.4	<0.001	-	-	3.3	<0.001
Unknown 5	-	-	3.9	<0.001	-	-	2.4	<0.001
Unknown 6	-	-	2.1	<0.001	-	-	4.1	<0.001
Unknown 7	-	-	1.9	<0.001	-	-	1.2	<0.001
Unknown 8	-	-	4.4	<0.001	-	-	2.6	<0.001
Total characterised [a]	30.0	<0.001	17.5	<0.001	-	-	13.5	<0.001
Losses of the organic phase during SPE	4.6	<0.001	-	-	1.4	<0.001	-	-
Aqueous phase of the	22.9	<0.001	18.3	0.001	31.0	<0.001	16.6	0.001

Label	Pyrazole-carboxamide label				Phenyl-carbamoyl label			
Study / report number	M-495009-01-1, EnSa-14-0485				M-495019-01-1, EnSa-14-0484			
Application	1 × 156 g ai/ha, drench application				1 × 153 g ai/ha drench application			
Component / Sample	Tomato fruits		Tomato leaves		Tomato fruits		Tomato leaves	
Days after treatment	83-99		99		83-99		99	
TRR (mg eq/kg)	<0.001		0.005		<0.001		0.006	
extract [b]								
Total extracted [c]	90.7	<0.001	93.9	0.005	86.5	<0.001	94.4	0.005
PES	9.3	<0.001	6.1	<0.001	13.5	<0.001	5.6	<0.001
Accountability	100	<0.001	100	<0.001	100	<0.001	100	0.006

Notes:
[a] Characterised by extraction, partitioning and chromatographic behaviour.
[b] Characterised by extraction and partitioning (most likely polar compounds).
[c] Total extracted = total identified + total characterised + losses of the organic phase during SPE + aqueous phase of the extract.

Only little radioactivity was taken up in the plants, indicating little translocation from soil to the plants. Pyrazole-carboxamide and phenyl-carbamoyl labelled tetraniliprole were moderately metabolised in tomatoes following soil application. The only observed metabolic reaction was an intra-molecular condensation (cyclisation) leading to tetraniliprole-N-methyl-quinazolinone.

Potato – outdoor seed treatment in furrow

A study was conducted to investigate the distribution and metabolism of pyrazole-carboxamide labeled tetraniliprole in potatoes after an in furrow seed treatment (Piskorski, 2015a, M-508350-01-1, Report EnSa-14-1306). The study was carried out in a Swiss (Witterswil) outdoor testing facility. Potato plants (*Solanum tuberosum* L, variety: Agri Bio) were cultivated under natural temperature and light conditions in the outdoor facility. Treated potato plants were grown in a single compartment surrounded with control potato plants. The soil used for the study was Spaniergrund (sandy loam). Radiolabelled pyrazole-carboxamide tetraniliprole was formulated as FS380P and applied to the seed in the furrow directly before covering the seed tubers with soil at an actual application rate of 199.8 g ai/ha.

Tubers from all the potato plants were collected 151 days after the application (BBCH 99). Aliquots of homogenized potato tubers were combusted and TRR amounted to only 0.001 mg eq/kg, indicating little translocation from soil to tubers. The investigation of the metabolic pathway was not possible due to the very low amount of radioactivity in the samples and the high matrix content.

Rice–Indoor granular soil application

The metabolism of tetraniliprole in paddy rice after a single granular treatment was investigated, using two different radiolabels in two different studies (Bongartz & Schallau, 2014a, M-496790-01-1, Report EnSa-14-0487 and Bongartz & Schallau, 2014b, M-496783-01-1, Report EnSa-14-0486). The studies were carried out in the greenhouse in Monheim am Rhein in Germany. Paddy rice (*Oryza sativa L.* var. Balilla) was cultivated under artificial temperature and light conditions in the greenhouse. Plants were pre-grown in a Japanese nursery box. The pre-grown seedlings (BBCH13-14, three to four leaves unfolded) were transplanted into a plant container (0.5 m^2) after addition of granular test compound in 11 planting holes. The containers were filled with sandy loam (Monheim 4). The pyrazole-carboxamide and phenyl-carbamoyl labelled tetraniliprole were applied onto Sepiolite carrier granules, at rates of was 205 and 211 g ai/ha, , respectively.

Samples of rice forage (BBCH 34–35, PHI 64 days), kernels, husks and straw (BBCH 89–92, PHI 150 days) were collected for analysis. All samples were extracted three times with a mixture of acetonitrile/water/formic acid (8/2/0.1, pH 3.5–3.8). The combined extracts were concentrated by rotary evaporation (40 °C bath temperature).

The measurement of the radioactivity in the liquid samples was carried out by LSC. All solid samples were combusted in an oxygen atmosphere using an oxidiser. The released $^{14}CO_2$ was trapped in an alkaline scintillation cocktail and the radioactivity was determined by LSC. The TRRs were low for all commodities with both labels (0.003–0.004 mg eq/kg for kernels, 0.008–0.0011 mg eq/kg for forage, 0.018–0.026 mg eq/kg for husks and 0.069–0.098 mg eq/kg for straw).

The extraction efficiency for straw, husks and forage was >75 percent for all commodities with the pyrazole-carboxamide label. The extraction efficiency with the phenyl-carbamoyl label was >87percent for all commodities except for kernels (49 percent). The post extraction solids (PES) of all commodities amounted to ≤0.009 mg eq/kg and were not further investigated. The distribution of radioactivity in rice samples is presented in Table 4.

Table 4 Distribution of radioactivity in the extracts of paddy rice following granular application of 205–211 g ai/ha of pyrazole-carboxamide or phenyl-carbamoyl labelled tetraniliprole

Component/sample	Forage		Kernels		Husks		Straw	
	% TRR (mg eq/kg)		% TRR (mg eq/kg)		% TRR (mg eq/kg)		% TRR (mg eq/kg)	
Report number	EnSa-14-0487	EnSa-14-0486	EnSa-14-0487	EnSa-14-0486	EnSa-14-0487	EnSa-14-0486	EnSa-14-0487	EnSa-14-0486
Days after treatment	64		150		150		150	
Label [a]	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car
TRR (mg/kg)	0.011	0.008	0.003	0.004	0.026	0.018	0.098	0.069
Solvent extract	89.1 (0.010)	91.2 (0.008)	75.8 (0.002)	49.0 (0.002)	84.8 (0.022)	87.1 (0.016)	90.8 (0.089)	91.0 (0.063)
Total extracted	89.1 (0.010)	91.2 (0.008)	75.8 (0.002)	49.0 (0.002)	84.8 (0.022)	87.1 (0.016)	90.8 (0.089)	91.0 (0.063)
PES	10.9 (0.001)	8.8 (0.001)	24.2 (0.001)	51.0 (0.002)	15.2 (0.004)	12.9 (0.002)	9.2 (0.009)	9.0 (0.006)
Accountability	100 (0.011)	100 (0.008)	100 (0.003)	100 (0.004)	100 (0.026)	100 (0.018)	100 (0.098)	100 (0.069)

Notes:
[a] Pyr-car = [pyrazole-carboxamide-^{14}C]-label; Phen-car= [phenyl-carbamoyl-^{14}C]-label.

Forage, husk and straw extracts were analysed by HPLC with radiometric- and UV-detection. HPLC method ANTAM was used for metabolite profiling and quantitation. Kernel extracts were analysed by one-dimensional thin-layer chromatography (TLC). The identification of parent compound and metabolites was performed by TLC co-chromatography with non-labelled reference compounds that were visualised by UV. The main compound found with the pyrazole-carboxamide label in the extracts was parent tetraniliprole (81.0 percent TRR (0.009 mg/kg) for forage, 48.4 percent TRR (0.001 mg/kg) for kernels, 77.9 percent TRR (0.020 mg/kg) for husks and 76.9 percent TRR(0.075 mg/kg) for straw). A minor metabolite was tetraniliprole-N-methyl-quinazolinone and amounted to 5.2 percent TRR (0.001 mg eq/kg) for forage, 9.9 percent TRR (<0.001 mg eq/kg) for kernels and 13.9 percent TRR (0.014 mg eq/kg) for straw. Tetraniliprole-N-methyl-quinazolinone was not detected in the kernel extracts. Similarly, the main compound found with the [phenyl-carbamoyl-^{14}C]-label in the extracts was parent tetraniliprole (78.9 percent TRR (0.007 mg/kg) for forage, 21.8 percent TRR (0.001 mg/kg) for kernels, 83.2 percent TRR (0.015 mg/kg) for husks and 77.3 percent TRR (0.054 mg/kg) for straw). A minor

metabolite was tetraniliprole-N-methyl-quinazolinone, which amounted to 12.3 percent TRR (0.001 mg eq/kg) for forage, 6.2 percent TRR (<0.001 mg eq/kg) for kernels, 3.9 percent TRR (0.001 mg eq/kg) for husks and 10.8 percent TRR (0.007 mg eq/kg) for straw. The metabolic profile of tetraniliprole in rice is presented in Table 5.

Table 5 Distribution of parent compound and metabolites in the extracts of paddy rice following granular application of 205 g ai/ha pyrazole-carboxamide or 211 g ai/ha phenyl-carbamoyl labelled tetraniliprole

Component / Sample	Forage		Kernels		Husks		Straw	
	% TRR (mg eq/kg)		% TRR (mg eq/kg)		% TRR (mg eq/kg)		% TRR (mg eq/kg)	
Report number	EnSa-14-0487	EnSa-14-0486	EnSa-14-0487	EnSa-14-0486	EnSa-14-0487	EnSa-14-0486	EnSa-14-0487	EnSa-14-0486
Days after treatment	64		150		150		150	
Label [a]	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car
TRR (mg/kg)	0.011	0.008	0.003	0.004	0.026	0.018	0.098	0.069
Tetraniliprole	81.0 (0.009)	78.9 (0.007)	48.4 (0.001)	21.8 (0.001)	77.9 (0.020)	83.2 (0.015)	76.9 (0.075)	77.3 (0.054)
N-methyl-quinazolinone	5.2 (0.001)	12.3 (0.001)	9.9 (<0.001)	6.2 (<0.001)	-	3.9 (0.001)	13.9 (0.014)	10.8 (0.007)
Total identified	86.2 (0.010)	91.2 (0.008)	58.3 (0.001)	28.0 (0.001)	77.9 (0.020)	87.1 (0.016)	90.8 (0.089)	88.1 (0.061)
Unknown 1	-	-	-	-	4.6 (0.001)	-	-	-
Unknown 3	-	-	-	-	2.3 (0.001)	-	-	-
Unknown 6	-	-	-	-	-	-	2.0 (0.001)	-
Unknown 8	-	-	-	-	-	-	1.0 (0.001)	-
Unknown 13	-	-	17.4 (0.001)	21.0 (0.001)	-	-	-	-
Total characterised	-	-	17.4 (0.001)	21.0 (0.001)	6.9 (0.002)	-	-	-
PES	10.9 (0.001)	8.8 (0.001)	24.2 (0.001)	51.0 (0.002)	15.2 (0.004)	12.9 (0.002)	9.2 (0.009)	9.0 (0.006)
Accountability	97.1 (0.011)	100 (0.008)	100 (0.003)	100 (0.004)	100 (0.026)	100 (0.018)	100 (0.098)	97.1 (0.067)

Notes:
[a] Pyr-car = [pyrazole-carboxamide-^{14}C]-label; Phen-car= [phenyl-carbamoyl-^{14}C]-label.

Maize–Outdoor seed treatment

The distribution and metabolism of pyrazole-carboxamide label formulated as FS 380P with maize after seed treatment was investigated in an outdoor facility in Witterswil in Switzerland. Maize plants (*Zea mays*, variety: Mezdi) were cultivated under natural temperature and light conditions in the outdoor facility of the test facility. The soil used for the study was Spaniergrund (soil type: sandy loam) (Piskorski, 2015b, Report EnSA-15-0013, M-525419-01-2). The seeds were placed in the soil and volumes of 72 and 172 µL application solution were carefully loaded onto each seed to achieve actual application rates of 62.8 and 150.1 g ai/ha, respectively (two separate treated plots with individual rates), based on a seed density of 80000 seeds/ha. Directly after the application, the seeds were covered with soil.

For each treatment plot, forage was sampled at BBCH 79–83 and mature plants at BBCH 89, corresponding with a PHI of 98 and 145 days, respectively. The mature plants were separated into kernel and stover samples. TRR values for forage, stover and kernels; determined by combustion/LSC analysis, were low for both treatment plots (<0.001 mg eq/kg for kernels, ≤0.006 mg eq/kg for forage, and 0.004 mg eq/kg for stover (62.8 g ai/ha plot)). As an exception, the TRR for the stover high rate (150 g ai/ha) sample was calculated as a sum of radioactivity found in extracts and PES (0.008 mg eq/kg). Due to the low TRRs, the stover sample from the high dose treatment plot was the only sample subjected to further analysis.

For analysis, the stover sample was homogenized and repeatedly (three times) extracted by with a mixture of acetonitrile/water/ formic acid (8/2/0.1). The extraction efficiency was determined as 75.8 percent TRR. The extracts were combined and concentrated before partition against ethyl acetate. The organic phase amounted to 70.1 percent TRR (0.005 mg eq/kg) and the aqueous phase 5.7 percent TRR (<0.001 mg eq/kg). The aqueous phase was not further investigated. The PES accounted for 0.002 mg eq/kg and was not investigated further. The distribution of radioactivity in maize stover is presented in Table 6.

Table 6 Distribution of radioactivity in the extracts of maize stover following seed treatment with [pyrazole-carboxamide -^{14}C]-tetraniliprole

Sample	Maize stover	
Application	150 g ai/ha as seed treatment applied just after seeding, before coverage by soil	
TRR (mg/kg)	0.008	
Days after treatment	145	
	% TRR	mg eq/kg
Solvent extract	75.8	0.006
water phase	*5.7*	*<0.001*
organic phase	*70.1*	*0.005*
Total extracted	75.8	0.006
Water phase	5.7	<0.001
Organic phase	70.1	0.005
PES	24.2	0.002
Accountability	100	0.008

The partition organic phase of the stover extract was analysed by one-dimensional TLC with UV-detection. The extracted residues were identified as parent tetraniliprole and tetraniliprole-N-methyl-quinazolinone representing 26.1 percent TRR (0.002 mg eq/kg) and 17.4 percent TRR (0.001 mg eq/kg), respectively. All other metabolite peaks represented ≤0.001 mg eq/kg. The main observed metabolic reaction was the cyclisation in the parent molecule leading to tetraniliprole-N-methyl-quinazolinone (Table 7).

Table 7 Distribution of parent compound and metabolites in the extracts of maize stover following seed treatment with [pyrazole-carboxamide-^{14}C]-tetraniliprole

Application: 150 g ai/ha as seed treatment applied just after seeding, before coverage by soil		
TRR (mg/kg)	0.008	
Days after treatment	145	
	% TRR	mg eq/kg
Organic phase	70.1	0.005
Tetraniliprole (parent compound)	26.1	0.002
Tetraniliprole-N-methyl-quinazolinone	17.4	0.001
Total identified	*43.5*	*0.003*

Application: 150 g ai/ha as seed treatment applied just after seeding, before coverage by soil		
Unknown 1	15.4	0.001
Unknown 2	2.5	<0.001
Unknown 3	2.7	<0.001
Unknown 4	2.5	<0.001
Unknown 5	3.4	<0.001
Total characterised	*26.5*	*0.001*
Post extracted solids (PES)	24.2	0.002
Aqueous phase (not further analysed)	5.7	<0.001
Accountability	99.9	0.007

Foliar treatments

Apples–Indoor foliar application

The metabolic fate of tetraniliprole was investigated in apple fruit and leaves after two spray applications in apple trees (*Malus domesticus*, variety: James Grieve) in two studies (Bongartz & Kluxen, 2015a, M-514517-01-1, Report EnSa-14-0492 and Bongartz & Kluxen, 2015b, M-514503-01-1, Report EnSa-14-0490). The apple trees were cultivated under natural temperature and light conditions in a greenhouse building (6682) in Germany (Monheim am Rhein), each tree in its own planting container in sandy soil (Monheim 4). Radiolabelled tetraniliprole was formulated as an SC 200 and applied twice to two apple trees at growth stages BBCH 71 (start of fruit development) and BBCH 73 (after the second fruit fall) with a retreatment interval of 33 days. The actual individual spray applications ranged from 86–88 g ai/ha with the pyrazole-carboxamide label, resulting in a total application rate of approximately 53 mg ai/tree, equivalent to a total application rate of 159.1 g ai/ha based on a plant density of 3,000 trees/ha (Report EnSa-14-0492). The individual spray applications with the phenyl-carbamoyl label were 85 and 86 g ai/ha, resulting in a total application rate of approximately 54 mg ai/tree, equivalent to a total application rate of 161.4 g ai/ha based on a plant density of 3,000 trees/ha (Report EnSa-14-0490).

Apple fruit (both labels) and leaves (pyrazole-carboxamide label only) were harvested from the trees at the end of the fruit ripening period (BBCH 89) at PHI of 64 days (fruits) and 66 days (leaves). A subset of apple fruits were dipped and subsequently rinsed with dichloromethane (=surface washing), diced, and homogenized under nitrogen and stored frozen until extraction. For determination of the extraction efficiency of the residue method, another subset of the fruits (without surface wash) was homogenised as described above. Leaves were not surface washed and stored in the freezer until homogenisation and extraction. Samples were extracted three times with a mixture of acetonitrile/water/formic acid (8/2/0.1; pH not reported). The surface wash solution and the first extract of the fruit samples were concentrated. The extracts of the leaves were combined and concentrated.

The measurement of radioactivity in liquid samples was carried out by LSC. All solid samples were combusted in an oxygen atmosphere using an oxidiser. The released $^{14}CO_2$ was trapped in an alkaline scintillation cocktail and the radioactivity was determined by LSC.

TRR in apple fruits was calculated based on the radioactivity in the surface wash solution and the fruit sample and amounted to 0.18 mg eq/kg in total (pyrazole-carboxamide label) to 0.25 mg eq/kg in total (phenyl-carbamoyl label).

Pyrazole-carboxamide label: The extraction efficiency (sum of surface wash and solvent extract) in the fruit samples was 98 percent TRR. The largest portion TRR was found in the surface wash solution (0.18 mg eq/kg, 97 percent TRR). The distribution of radioactivity in apple fruit and leaves is presented in Table 8.

Phenyl-carbamoyl-^{14}C] label: The extraction efficiency (sum of surface wash and solvent extract) in the fruit samples was 99.6 percent.

Table 8 Distribution of radioactivity at PHI 64-66 days in the extracts of apple fruit and leaves following 2 foliar indoor applications each of pyrazole-carboxamide or phenyl-carbamoyl labelled tetraniliprole, with a retreatment interval of 33 days

Label	[pyrazole-carboxamide-^{14}C]				[phenyl-carbamoyl-^{14}C]	
Study/ report number	M-514517-01-1, EnSa-14-0492				M-514503-01-1, EnSa-14-0490	
Application	2 x 86-88 g ai/ha				2 x 84-86 g ai/ha	
Sample	Apple fruit		Apple leaves		Apple fruit	
Days after the last treatment	64		66		64 d	
TRR (mg eq/kg)	0.183		99.4		0.525	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Dichloromethane surface wash	96.7	0.177	-	-	92.1	0.232
Acetonitrile/water/formic acid extraction	3.0	0.005	99.5	98.9	7.5	0.019
Total extracted	99.6	0.183	99.5	98.9	99.6	0.251
PES	0.4	0.001	0.5	0.541	0.4	0.001
Accountability	100	0.183	100	99.4	100	0.252

All sample extracts were analysed by HPLC with radiometric and UV-detection. The identification of the compounds detected in the fruit extract was performed by HPLC co-chromatography with the non-radiolabelled reference compound. Parent tetraniliprole was the main compound in the apple fruit sample. The highest amount of parent compound (0.18 mg/kg eq, 99.2 percent TRR with the pyrazole-carboxamide label and 0.25 mg/kg eq, 99.3 percent TRR with the phenyl-carbamoyl label) was found in apple fruits (surface wash and extract combined). Besides parent compound, there were no other metabolites detected in the fruit extract with either label. Two very minor compounds (<0.001 mg eq/kg, ≤ 0.3 percent TRR) were found in the surface wash solution of the pyrazole-carbamoyl label and only one minor compound (0.001 mg eq/kg, 0.2 percent TRR) was detected in the surface wash solution of the phenyl-carbomoyl label.

Parent tetraniliprole was the main compound in the apple leaves sample and amounted to 98 mg eq/kg, 98.6 percent TRR. Six minor compounds were detected in the leaf extract and ranged from 0.051 to 0.32 mg eq/kg (0.1 to 0.3 percent TRR).

These results show that minimal metabolism of [pyrazole-carboxamide-^{14}C] and [phenylcarbamoyl-^{14}C] tetraniliprole occurs in apple fruit and leaves after foliar application of tetraniliprole. Only parent compound was identified (Table 9).

Table 9 Distribution of parent compound and metabolites in the extracts of apple fruit and leaves following 2 foliar indoor applications each of 85–88 g [pyrazole-carboxamide and of phenyl-carbamoyl label

Label	[pyrazole-carboxamide-^{14}C]				[phenyl-carbamoyl-^{14}C]	
Study / report number	M-514517-01-1, EnSa-14-0492				M-514503-01-1, EnSa-14-0490	
Application	2 x 86-88 g ai/ha, RTI 33 days				2 x 84-86 g ai/ha, RTI 33 days	
Component / Sample	Apple fruit		Apple leaves		Apple fruit	
Days after treatment	64		66		64	
TRR (mg eq/kg)	0.183		99.4		0.252	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Dichloromethane surface wash solution						
Tetraniliprole	96.2	0.177	-	-	91.8	0.231

Label	[pyrazole-carboxamide-^{14}C]			[phenyl-carbamoyl-^{14}C]		
Study / report number	M-514517-01-1, EnSa-14-0492			M-514503-01-1, EnSa-14-0490		
Application	2 x 86-88 g ai/ha, RTI 33 days			2 x 84-86 g ai/ha, RTI 33 days		
Component / Sample	Apple fruit		Apple leaves		Apple fruit	
Unknown	0.2	<0.001	-	-	0.2	0.001
Unknown	0.3	<0.001	-	-	-	-
Acetonitrile/water/formic acid extract						
Tetraniliprole	3.0	0.005	98.6	98.0	7.5	0.019
Unknown	-	-	0.1	0.051	-	-
Unknown	-	-	0.1	0.081	-	-
Unknown	-	-	0.1	0.086	-	-
Unknown	-	-	0.2	0.176	-	-
Unknown	-	-	0.2	0.163	-	-
Unknown	-	-	0.3	0.323	-	-
Total identified [a]	99.2	0.182	98.6	98.0	99.3	0.25
Tetraniliprole [a]	*99.2*	*0.182*	*98.6*	*98.0*	*99.3*	*0.25*
Total characterised	0.4	<0.001	0.9	0.881	0.2	0.001

Notes:

[a] Results from dichloromethane surface wash + acetonitrile/water/formic acid extract.

Lettuce – indoor foliar application

The distribution and metabolism of pyrazole-carboxamide label (Piskorski, 2014a, M-496411-01-1, Report EnSa-14-0613) or phenyl-carbamoyl label (Piskorski, 2014b, M-496407-01-1, Report EnSa-14-0612) in lettuce after two foliar spray applications was investigated. Lettuce plants (*Lactuca sativa* L, variety: Reine de Mai) were cultivated under artificial temperature and light conditions in the greenhouse of a Swiss (Witterswil) test facility. Plants were grown in three plastic containers using Spaniergrund (sandy loam soil). Radiolabelled tetraniliprole was formulated as SC 200 and applied to the plants. The two applications were performed as a spray foliar treatment at approximately BBCH 44–45 (40–50 percent of the expected head size reached), 14 days before harvest and again 7 days later, 7 days before harvest. The actual application rates were 2 × 59–60 g ai/ha (total rate 119 g ai/ha in both studies).

A total of six lettuce heads (leaves) were collected 7 days after the second application (BBCH 49). Aliquots of homogenized lettuce leaves were repeatedly (three times) extracted with acetonitrile/water/formic acid (8/2/0.1). Extracts were combined and concentrated.

The measurement of the radioactivity in liquid samples was carried out by LSC. All solid samples were combusted using an oxidiser, the released $^{14}CO_2$ was trapped in an alkaline scintillation cocktail and the radioactivity was determined by LSC.

In the lettuce leaf samples, the TRR amounted to 4.0 mg eq/kg and 4.1 mg eq/kg with the respective labels. The PES amounted to 0.020 mg eq/kg (0.5 percent TRR) and 0.038 mg eq/kg (0.9 percent TRR) with both labels respectively. The distribution of radioactivity in lettuce leaves is presented in Table 10.

Table 10 Distribution of radioactivity in the extracts of lettuce leaves following two foliar application of pyrazole-carboxamide and of phenyl-carbamoyl labelled tetraniliprole

Label	[pyrazole-carboxamide-^{14}C]		[phenyl-carbamoyl-^{14}C]	
Study/ report number	M-496411-01-1, EnSa-14-0613		M-496407-01-1, EnSa-14-0612	
Application	2 x 59-60 g ai/ha, RTI 7 days		2 x 59-60 g ai/ha, RTI 7 days	
Days after the last treatment	7		7	
TRR (mg eq/kg)	4.063		4.122	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Total extracted	99.5	4.04	99.1	4.08
PES	0.5	0.020	0.9	0.038
Accountability	100	4.06	100	4.12

Extracts were analysed by HPLC with radiometric and UV-detection and one-dimensional TLC with UV detection (at 254 nm). The identification of the compounds was performed with non-radiolabelled test item. Nearly all radioactivity (99.5 percent TRR) could be extracted and all extracted residues were identified as parent tetraniliprole. No metabolic degradation was observed, see Table 11.

Table 11 Distribution of parent compound and metabolites in the extracts of lettuce leaves following 2 foliar indoor application of pyrazole-carboxamide- and of phenyl-carbamoyl labelled tetraniliprole. Samples were collected 7 days after the last treatment

Label	[pyrazole-carboxamide-^{14}C]		[phenyl-carbamoyl-^{14}C]	
Study/ report number	M-496411-01-1, EnSa-14-0613		M-496407-01-1, EnSa-14-0612	
Application	2 x 59-60 g ai/ha, RTI 7 days		2 x 59-60 g ai/ha, RTI 7 days	
TRR (mg eq/kg)	4.063		4.122	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Tetraniliprole (parent)	99.5	4.04	99.1	4.08
Total identified	99.5	4.04	99.1	4.08
Total characterised	0	0	0	0
PES	0.5	0.020	0.9	0.038
Accountability	100	4.06	100	4.12

Potato–Indoor foliar application

The metabolic fate of tetraniliprole in potatoes was investigated after two foliar applications with pyrazole-carboxamide (Bongartz & Junge, 2015a, M-508626-01-1, Report EnSa-14-0493) or with phenyl-carbamoyl (Bongartz & Junge, 2015b, M-508624-01-1, Report EnSa-14-0491) labelled tetraniliprole. Potato plants (*Solanum tuberosum* L, cv. Cilena) were cultivated under artificial temperature and light conditions in a greenhouse in Germany (Monheim am Rhein). Plants were grown in planting containers filled with Monheim 4 soil (sandy loam). Two foliar applications ranging from 101-105 g ai/ha each were applied at a 49 day interval at growth stages BBCH 38 and BBCH 97–99 (last application 14 days before harvest). The total application rate corresponded to 207 g ai/ha and 206 g ai/ha, respectively, based on an average seed density of 55,000 seed potatoes/ha.

Potato plants were harvested at maturity (BBCH 99) and separated into tubers and leaves. The leaves were not further processed/analysed. Soil was removed by hand and the potato tubers were washed with water. The radioactivity in the washing water was determined by LSC. The potato tubers were air dried, cut into four aliquots and mixed thoroughly. Potato tubers were homogenized and extracted three times with a mixture of acetonitrile/water (8/2) plus 0.1 percent formic acid (pH was determined but not reported). The solids were separated from the extract by centrifugation. The extracts

were combined and the radioactivity was determined by LSC (three aliquots) after volume measurement. PES were air-dried and homogenized. Aliquots were radio assayed by combustion followed by LSC.

TRR for potato tubers was very low (0.001 mg eq/kg). The tuber solvent extract and PES amounted to 74.6–79.3 percent TRR (0.001 mg eq/kg) and 20.7–25.4 percent TRR (< 0.001 mg eq/kg), respectively with both labels. The distribution of radioactivity in potato tubers is presented in Table 12.

Table 12 Distribution of radioactivity in the extracts of potato following indoor foliar applications of [pyrazole-carboxamide-^{14}C]- and of [phenyl-carbamoyl-^{14}C]-labelled tetraniliprole.

Label	[pyrazole-carboxamide-^{14}C]		[phenyl-carbamoyl-^{14}C]	
Study/ report number	M-508626-01-1, EnSa-14-0493		M-508624-01-1, EnSa-14-0491	
Application	2 x 103-104 g ai/ha, RTI 49 days		2 x 101-105 g ai/ha, RTI 49 days	
Days after the last application	14		14	
TRR (mg eq/kg)	0.001		0.001	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Total extracted	79.3	0.001	74.6	0.001
Partition organic phase total	59	0.001	64.6	<0.001
Partition aqueous phase	20.3	<0.001	10.1	<0.001
PES	20.7	<0.001	25.4	<0.001
Accountability	100	0.001	100	0.001

Parent compound and metabolites were quantified after multiple purification (partitioning 3 × ethyl acetate) and concentration steps in the organic phase (59 percent TRR, 64.6 percent TRR) of the potato tubers extract by TLC. The aqueous phase (20.3 percent TRR and 10.1 percent TRR, respectively) was not further explored. Potato tuber extracts were analysed by TLC with UV_{254} -detection using non-radiolabelled reference compounds (tetraniliprole and tetraniliprole-N-methylquinazolinone). Parent tetraniliprole was the main compound in the extract and represented 29 percent TRR (< 0.001 mg eq/kg) with the pyrazole-carboxamide label and 42 percent TRR (< 0.001 mg eq/kg) with the phenyl-carbamoyl label. Tetraniliprole-N-methyl-quinazolinone was the only identified metabolite in the extract and represented 9.0 percent TRR (<0.001 mg eq/kg) and 13 percent TRR (<0.001 mg eq/kg) for both labels, respectively. All other metabolite peaks represented <0.001 mg eq/kg. The metabolic profile of tetraniliprole in potato is presented in Table 13.

Table 13 Distribution of parent compound and metabolites in the extracts of potato tubers following two indoor foliar application of [pyrazole-carboxamide-^{14}C]- and of [phenyl-carbamoyl-^{14}C]-labelled tetraniliprole

Label	[pyrazole-carboxamide-^{14}C]		[phenyl-carbamoyl-^{14}C]	
Study reference	M-508626-01-1, EnSa-14-0493		M-508624-01-1, EnSa-14-0491	
Application	2 x 103-104 g ai/ha, RTI 49 days		2 x 101-105 g ai/ha, RTI 49 days	
Days after the last treatment	14		14	
TRR (mg eq/kg)	0.001		0.001	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Tetraniliprole (parent compound)	29.4	<0.001	42.3	<0.001
Tetraniliprole-N-methyl-quinazolinone	9.0	<0.001	13.0	<0.001
Total identified in organic fraction	38.4	<0.001	55.3	<0.001
Unknown	11.8	<0.001	5.3	<0.001
Unknown	3.6	<0.001	2.4	<0.001
Unknown	1.6	<0.001	1.6	<0.001
Unknown	3.6	<0.001	-	<0.001
Total characterised in organic fraction	20.6	<0.001	9.3	<0.001

Label	[pyrazole-carboxamide-^{14}C]		[phenyl-carbamoyl-^{14}C]	
Study reference	M-508626-01-1, EnSa-14-0493		M-508624-01-1, EnSa-14-0491	
Application	2 x 103-104 g ai/ha, RTI 49 days		2 x 101-105 g ai/ha, RTI 49 days	
Total identified & characterised in organic fraction	*59*	*<0.001*	*64.6*	*<0.001*
Aqueous phase (not investigated)	*20.3*	*<0.001*	*10.1*	*<0.001*
Total extracted	79.3	0.001	74.6	0.001
PES	20.7	<0.001	25.4	<0.001
Accountability	100	0.001	100	0.001

The only observed metabolic reaction was the intra-molecular condensation (cyclisation) of parent compound leading to tetraniliprole-N-methyl-quinazolinone.

Paddy rice–Indoor foliar treatment

The metabolism of tetraniliprole in paddy rice after two foliar treatments was investigated in two studies using either pyrazole-carboxamide label (Bongartz and Schallau, 2015a, M-496793-02-1, Report EnSa-14-0489) or phenyl-carbamoyl label (Bongartz and Schallau, 2015b, M-496787-02-1, Report EnSa-14-0488). Paddy rice (*Oryza sativa*, variety: Balilla) was cultivated under artificial temperature and light conditions in a greenhouse in Germany (Monheim am Rhein). Plants (11 plants/pot) were grown in planting containers filled with Monheim 4 soil (soil type: sandy loam). Two foliar applications ranging from 101–105 g ai/ha were applied at a 42-day interval. The radiolabelled tetraniliprole was formulated as SC 200 and applied onto the plants at BBCH 14 (four leaves unfolded) and BBCH 73–77 (early to late milk stage). Actual application rates were 52.3 g ai/ha for the first and 50.9 g ai/ha for the second treatment with the pyrazole-carboxamide label and 50.6 g ai/ha for the first and 49.9 g ai/ha for the second treatment with the phenyl-carbamoyl label.

Samples of rice forage, kernels, husks and straw were collected for analysis at BBCH 34–35 (rice forage) and BBCH 89–92 (husks, kernels and straw including panicles), corresponding with PHI of 13 and 56 days, respectively. All samples were extracted three times with a mixture of acetonitrile/water/formic acid (8/2/0.1; pH 3.5–3.8). The solids were separated from the extract by suction through a filter and the extracts were combined. The solids of husks and straw were additionally extracted twice with acetonitrile/water/formic acid (8/2/0.1) using microwave assistance and the extracts combined. The volume of each extract was determined and three aliquots radio-assayed by LSC. The extraction solids were air-dried and aliquots were radio assayed by LSC following combustion. The combined extracts were concentrated by rotary evaporation (40°C bath temperature).

TRR was found to be low in kernels (0.040 mg eq/kg and 0.024 mg eq/kg, for the respective labels). The TRRs of forage, husks and straw were higher due to their exposed surfaces during foliar treatment (1.3, 2.5 and 4.3 mg eq/kg, respectively for the pyrazole-carboxamide label and 2.6, 2.1 and 4.6 mg eq/kg, respectively for the phenyl-carbamoyl label).

The extraction efficiency for all commodities was >92 percent with both labels. PES of husks and straw were further extracted using microwave assistance; releasing 1.8 percent TRR (0.047 mg eq/kg) and 1.2 percent TRR (0.050 mg eq/kg), respectively with the pyrazole-carboxamide label and releasing 5.0 percent (0.106 mg/kg) and 2.1 percent (0.094 mg/kg), respectively with the phenyl-carbamoyl label. The PES of forage and kernels amounted to ≤0.022 mg eq/kg with both labels and were not further investigated. The distribution of radioactivity in forage, kernel, husk and straw samples is presented in Table 14.

Table 14 Distribution of radioactivity in the extracts of paddy rice following foliar application of [pyrazole-carboxamide-^{14}C]- and of [phenyl-carbamoyl-^{14}C]-labelled tetraniliprole

Component/sample	Forage		Kernels		Husks		Straw	
Application	2 x 50-52 g ai/ha, RTI 42 days, foliar, indoor							
	% TRR (mg eq/kg)		% TRR (mg eq/kg)		% TRR (mg eq/kg)		% TRR (mg eq/kg)	
Report number	EnSa-14-0489	EnSa-14-0488	EnSa-14-0489	EnSa-14-0488	EnSa-14-0489	EnSa-14-0488	EnSa-14-0489	EnSa-14-0488
PHI	13 days		56 days		56 days		56 days	
Label [a]	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car
TRR (mg/kg)	1.31	2.58	0.040	0.024	2.52	2.11	4.32	4.57
Solvent extract (initial)	98.3 (1.28)	99.1 (2.56)	93.7 (0.037)	92.7 (0.022)	97.6 (2.46)	94.2 (1.99)	98.5 (4.25)	97.4 (4.45)
Losses during concentration	-	0.1 (0.002)	-	-	-	-	-	0.1 (0.006)
PES	1.7 (0.022)	0.8 (0.022)	6.3 (0.002)	7.3 (0.002)	-	-	-	-
Microwave assisted extract	-	-	-	-	1.8 (0.047)	5.0 (0.106)	1.2 (0.050)	2.1 (0.094)
Total extracted	98.3 (1.28)	99.2 (2.56)	93.7 (0.037)	92.7 (0.022)	99.4 (2.50)	99.2 (2.09)	99.6 (4.30)	99.6 (4.55)
Remaining solids	-	-	-	-	0.6 (0.015)	0.7 (0.016)	0.4 (0.016)	0.4 (0.019)
Accountability	100 (1.31)	100 (2.58)	100 (0.040)	100 (0.024)	100 (2.52)	100 (2.12)	100 (4.32)	100 (4.57)

Notes:
[a] Pyr-car = [pyrazaole-carboxamide-^{14}C]-label; Phen-car= [phenyl-carbamoyl-^{14}C]-label

The concentrated extracts were used for HPLC analysis with radiometric- and UV-detection. HPLC method ANTAM was used for metabolite profiling and quantitation. The main compound found in the extracts was parent tetraniliprole (97 percent TRR (1.27 mg/kg) TRR for forage, 92 percent (0.037 mg/kg) for kernels, 96 percent (2.42 mg/kg) for husks and 95.3 percent (4.11 mg/kg) for straw). A minor metabolite was tetraniliprole-N-methyl-quinazolinone and amounted to 1.1 percent (0.014 mg eq/kg) TRR for forage, 1.5 percent (0.001 mg eq/kg) for kernels, 2.1 percent (0.052 mg eq/kg) for husks and 2.6 percent (0.112 mg eq/kg) for straw. All other metabolite peaks represented $\underline{<}$ 0.7 percent TRR. With the [phenyl-carbamoyl-^{14}C]-label a similar profile was observed. The main compound detected in the extracts was parent tetraniliprole (98 percent (2.54 mg/kg) TRR for forage, 91 percent (0.022 mg/kg) for kernels, 93 percent (1.95 mg/kg) for husks and 94.4 percent (4.31 mg/kg) for straw). A minor metabolite was tetraniliprole-N-methyl-quinazolinone and amounted to 0.7 percent (0.018 mg eq/kg) TRR for forage, 1.8 percent (<0.001 mg eq/kg) for kernels, 3.7 percent (0.078 mg eq/kg) for husks and 3.3 percent (0.151 mg eq/kg) for straw. All other metabolite peaks represented ≤ 0.8 percent TRR. Tetraniliprole was hardly metabolised in the rice after foliar treatment. The only observed metabolic reaction was an intra-molecular condensation (cyclisation) leading to tetraniliprole-N-methyl-quinazolinone (Table 15).

Table 15 Distribution of parent compound and metabolites in the extracts of paddy rice following indoor foliar application of [pyrazole-carboxamide-^{14}C]- and of [phenyl-carbamoyl-^{14}C]-labelled tetraniliprole (2 × 50–52 g ai/ha, RTI 42 days)

Component / Sample	Forage		Kernels		Husks		Straw	
	% TRR (mg eq/kg)		% TRR (mg eq/kg)		% TRR (mg eq/kg)		% TRR (mg eq/kg)	
Report number	EnSa-14-0489	EnSa-14-0488	EnSa-14-0489	EnSa-14-0488	EnSa-14-0489	EnSa-14-0488	EnSa-14-0489	EnSa-14-0488
Days after the last application	13		56		56		56	
Label [a]	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car
TRR (mg/kg)	1.31	2.58	0.040	0.024	2.52	2.11	4.32	4.57
Tetraniliprole	97.1 (1.27)	98.4 (2.537)	92.2 (0.037)	90.9 (0.022)	95.9 (2.42) [b]	92.6 (1.95) [d]	95.2 (4.11) [f]	94.4 (4.31) [h]
Tetraniliprole-N-methyl-quinazolinone	1.1 (0.014)	0.7 (0.018)	1.5 (0.001)	1.8 (<0.001)	2.0 (0.052) [c]	3.7 (0.078) [e]	2.6 (0.11) [g]	3.3 (0.15) [i]
Total identified	98.2 (1.28)	99.1 (2.56)	93.7 (0.037)	92.7 (0.022)	98.0 (2.47)	96.3 (2.03)	97.8 (4.22)	97.7 (4.46)
Unknown 1 [j]	-		-	-	0.4 (0.009)	-	0.2 (0.007)	-
Unknown 2 [j]	-	-	-	-	0.1 (0.001)	-	-	-
Unknown 3 [j]	-	-	-	-	-	-	0.2 (0.009)	-
Unknown 4 [j]	-	-	-	-	-	-	0.3 (0.014)	-
Unknown 5 [j]	-	-	-	-	0.7 (0.017)	0.8 (0.017)	0.3 (0.011)	-
Unknown 6 [j]	-	-	-	-	-	-	0.3 (0.015)	0.5 (0.023)
Unknown 7 [j]	-	-	-	-	-	0.5 (0.010)	0.3 (0.013)	0.6 (0.027)
Unknown 8 [k]	-	-	-	-	0.1 (0.003)	0.1 (0.003)	<0.1 (0.002)	0.1 (0.004)
Unknown 9 [j]	-	-	-	-	-	0.5 (0.011)	-	-
Unknown 10 [j]	-	-	-	-	-	0.4 (0.009)	0.1 (0.006)	0.3 (0.014)
Unknown 11 [k]	-	-	-	-	-	0.4 (0.008)	-	0.2 (0.010)
Unknown 12 [k]	-	-	-	-	0.1 (0.002)	0.2 (0.005)	-	0.1 (0.004)
Total characterised [l]	-	-	-	-	1.3 (0.032)	2.9 (0.062)	1.9 (0.077)	1.8 (0.081)
Total extracted [l]	98.3 (1.28)	99.2 (2.56)	93.7 (0.037)	92.7 (0.022)	99.4 (2.50)	99.2 (2.09)	99.6 (4.30)	99.6 (4.55)
Solids remaining	1.7 (0.022)	0.8 (0.022)	6.3 (0.002)	7.3 (0.002)	0.6 (0.015)	0.7 (0.016)	0.4 (0.016)	0.4 (0.019)
Accountability	100 (1.31)	100 (2.58)	100 (0.040)	100 (0.024)	100 (2.52)	100 (2.12)	100 (4.32)	100 (4.57)

Notes:

[a] Pyr-car = [pyrazole-carboxamide-^{14}C]-label; Phen-car= [phenyl-carbamoyl-^{14}C]-label.

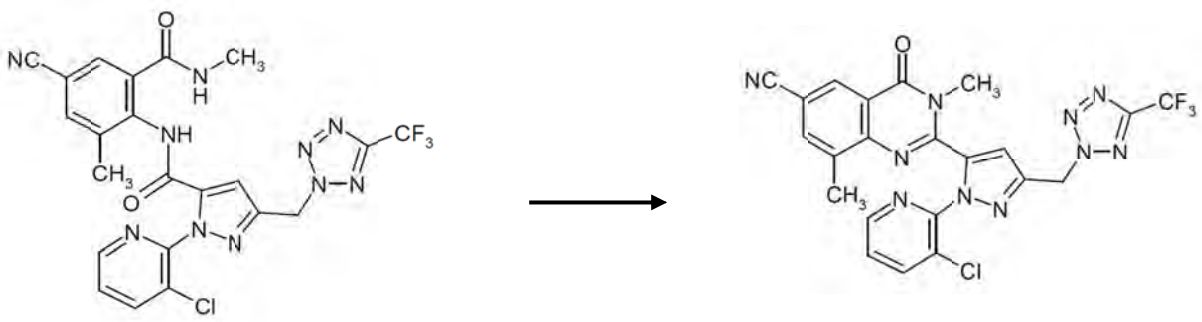
[b] 95.2% TRR (2.4 mg eq/kg) after first extraction + 0.7% TRR (0.018 mg eq/kg) after microwave assisted extraction).

[c] 1.52% TRR (0.039 mg eq/kg) after first extraction + 0.5% TRR (0.013 mg eq/kg after microwave assisted extraction).

[d] 89.7% TRR (1.89 mg eq/kg) after first extraction + 2.9% TRR (0.061 mg eq/kg) after microwave assisted extraction).
[e] 2.3% TRR (0.048 mg eq/kg) after first extraction + 1.4% TRR (0.030 mg eq/kg after microwave assisted extraction).
[f] 94.5% TRR (4.08 mg eq/kg) after first extraction + 0.7% TRR (0.032 mg eq/kg) after microwave assisted extraction).
[g] 2.4% TRR (0.103 mg eq/kg) after first extraction + 0.2% TRR (0.009 mg eq/kg after microwave assisted extraction).
[h] 93.2% TRR (4.26 mg eq/kg) after first extraction + 1.2% TRR (0.054 mg eq/kg) after microwave assisted extraction).
[i] 2.8% TRR (0.13 mg eq/kg) after first extraction + 0.5% TRR (0.023 mg eq/kg after microwave assisted extraction).
[j] Characterised in first series of extractions.
[k] Characterised after microwave assisted extraction.
[l] Including microwave assisted extraction..

Overview of the metabolic pathway in plants

No metabolism in apples (foliar application) or lettuce (foliar application) was observed. One metabolism step could be identified in the metabolism studies in other crops, including tomato (soil drench application), potato (foliar application), paddy rice (foliar application as well as granular soil application), and maize (seed treatment) was the cyclisation in the parent molecule leading to tetraniliprole-N-methyl-quinazolinone (Figure 1).



tetraniliprole tetraniliprole-N-methyl-quinazolinone

Figure 1 Metabolism of tetraniliprole observed in tomato, potato, rice and maize

Environmental fate in water/sediment systems

Hydrolysis in sterile water buffers

The abiotic hydrolytic degradation of [pyrazole-carboxamide-^{14}C]-tetraniliprole was studied at 20, 25 and 50 °C in sterile buffer solutions with pH values of 4, 7 or 9 in the dark in the laboratory (Hein&Kasel, 2016, M-565616-01-1, Report EnSa-14-0308).

A study application rate of 0.3 mg/L was applied, considering the low water solubility of the test item and its degradation products.

Duplicate samples were processed and analysed. The radioactivity in the test solutions was determined by LSC, and HPLC-ESI-MS/MS and radio-detection was used for confirmation of the test item identity and for structure elucidation of the degradation product. Additionally, test item identity was confirmed by NMR.

At 50 °C and pH 4 and pH 7 samples were processed and analysed after 0.25, 1.04, 2.25, 7, 14, 21 and 30 days. The pH 9 samples were processed 0.04, 0.08, 0.17, 0.25, 1.04 and 2.04 days after treatment (DAT) corresponding to 1, 2, 4, 6, 25 and 49 hours. At 25 °C and pH 4 and pH 7 samples were processed after 1.04, 2.25, 7, 14, 21 and 30 days. The pH 9 samples were processed after 0.125, 0.25, 1.04, 2.25, 7,

14, 21 and 30 days. At 20 °C and pH 4 and pH 7 samples were processed after 1.04, 2.25, 7, 14, 21 and 30 days. The pH 9 samples were processed after 0.25, 1.04, 2.25, 7, 14, 21 and 30 days.

On incubation at 50 °C the amount of tetraniliprole in test solution decreased at pH 4 from 100 percent ofapplied radioactivily (AR) at DAT-0 to 16.3 percent AR at DAT-30. At pH 7, the values were 100 and 1.6 percent AR, and at pH 9 were 97.2 and 2.9 percent AR, respectively. At 25 °C, tetraniliprole decreased at pH 4 from 100 to 89.6 percent AR, at pH 7 from 100.0 to 56.5 percent AR, and at pH 9 from 97.2 to 1.8 percent AR. At 20 °C, the amount decreased at pH 4 from 100.0 to 91.5 percent AR, at pH 7 from 100 to 68.1 percent AR, and at pH 9 from 97.2 to 1.5 percent AR at DAT-30.

One degradation product was identified as tetraniliprole-N-methyl-quinazolinone with a maximum amount of 99.6 percent AR at DAT-30 (pH 9, optional test, 20 °C). The total unidentified residues amounted to a maximum of 3.3 percent AR and no single unidentified component exceeded 3.3 percent AR at any sampling interval of all conducted tests.

Following incubation at 50 °C, the material balances at pH 4 ranged from 92.5 to 100 percent AR, at pH 7 from 92.5 to 100.9 percent AR, and at pH 9 from 93.2 to 101.1 percent AR. Following incubation at 25 °C, material balances at pH 4 ranged from 93.0 to 100 percent AR, at pH 7 from 92.2 to 100 percent AR, and at pH 9 from 98.7 to 103.1 percent AR. Following incubation at 20 °C, material balances at pH 4 ranged from 93.7 to 101.8 percent AR, at pH 7 from 93.4 to 100 percent AR, and at pH 9 from 97.1 to 103.9 percent AR.

The degradation rate of tetraniliprole for all pH values at all temperatures was calculated using first order kinetics (SFO). The hydrolytic degradation of tetraniliprole was observed to be pH dependent. Results are summarised in Table 16.

Table 16 Kinetic evaluation of hydrolysis of tetraniliprole at different pH values and at different temperatures

Temperature (^{o}C)	pH	SFO		
		DT_{50} (d)	DT_{90} (d)	χ^2 error ( )
50	4	10.9	36.0	2.7
50	7	3.74	12.4	3.1
50	9	0.04	0.13	5.5
25	4	287	953	1.7
25	7	38.8	129	2.3
25	9	0.75	2.48	3.8
20	4	265	882	1.8
20	7	58.0	193	1.7
20	9	1.27	4.21	4.2

In summary, the hydrolytic degradation of tetraniliprole strongly depended on the temperature and the pH conditions. Tetraniliprole degraded rapidly at pH 9 and well at pH 7 at all temperatures in the laboratory. For pH 4, it was degraded rapidly at 50 °C and slowly at 20 and 25 °C. The calculated half-lives of tetraniliprole were between 0.04 and 10.9 days at 50 °C, between 0.75 and 287 days at 25 °C and between 1.27 and 265 days at 20 °C. One degradation product was identified as tetraniliprole-N-methyl-quinazolinone with a maximum amount of 99.6 percent AR (at DAT-30; pH 9, optional test, 20 °C).

Phototransformation in natural water

The photolytic route and rate of degradation of [pyridinyl-2-^{14}C]-tetraniliprole was studied in sterile natural water (pH 8.5) from the river Rhine under exposure to simulated sunlight in the laboratory for 11 days at 25 ± 2 °C (Heinemann&Kasel, 2016a, M-568022-01-1, Report EnSA-16-0158).

A study application rate of 0.5 mg/L was applied due to the low water solubility of the test item. 11 days of incubation under exposure to simulated sunlight were equivalent to 69.6 days of solar summer days in Japan. Control samples were incubated in the dark.

Mean material balances were 101 and 103 percent AR for irradiated and dark samples, respectively. The maximum amounts of carbon dioxide were 38.9 and ≤0.1 percent AR in irradiated and dark samples, respectively. Formation of volatile organic compounds (VOC) was insignificant as demonstrated by values of ≤0.2 percent AR at all sampling intervals for both, irradiated and dark samples.

The amount of tetraniliprole in the test solution decreased from 97.7 percent AR at study start (DAT-0) to non-detectable amounts and 4.8 percent AR at DAT-11 in irradiated and dark samples, respectively. Besides the formation of carbon dioxide two degradation products were identified in irradiated samples with the following maximum occurrences: tetraniliprole-N-methyl-quinazolinone with 39.2 percent AR at DAT-1 and tetraniliprole-deschloro-pyrazine with 38.8 percent AR at DAT-2. The total unidentified residues amounted to a maximum of 62.4 percent AR and no single component exceeded 7.3 percent AR at any sampling interval. In dark samples tetraniliprole was degraded to tetraniliprole-N-methyl-quinazolinone with the maximum occurrence of 99.0 percent AR at DAT-11. A summary of the degradation results is presented in Table 17.

Table 17 Photodegradation of [pyridinyl-2-^{14}C]-tetraniliprole in natural water with mean values expressed as percent AR (± SD)

Compound	Sample	Days exposed to simulated sunlight						
		0 [a]	0.25	1	2	4	7	11
Tetraniliprole	Irradiated	97.7 (0.1)	88.0 (1.9)	43.4 (6.9)	13.2 (0.0)	1.2 (0.0)	ND	ND
	Dark		88.6 (1.2)	42.2 (3.1)	10.8 (0.2)	3.8 (0.3)	4.9 (0.3)	4.8 (0.3)
Tetraniliprole-N-methyl-quinazolinone	Irradiated	2.3 (0.1)	13.1 (0.1)	39.2 (5.5)	22.4 (0.6)	2.1 (0.3)	ND	ND
	Dark		17.4 (0.9)	61.1 (2.2)	92.6 (0.0)	97.9 (1.7)	98.8 (0.7)	99.0 (0.8)
Tetraniliprole-deschloro-pyrazine	Irradiated	ND	2.2 (0.1)	15.9 (2.7)	38.8 (1.8)	23.0 (1.7)	5.7 (1.4)	0.8 (0.1)
	Dark		ND	ND	ND	ND	ND	ND
Others [b]	Irradiated	ND	2.4 (0.3)	3.5 (0.1)	22.7 (2.2)	56.3 (0.4)	62.4 (1.0)	59.0 (0.5)
	Dark		ND	ND	ND	ND	ND	ND
Total residues in solution	Irradiated	100 (0.0)	105.6 (2.5)	102.0 (1.2)	97.1 (1.0)	82.7 (2.6)	68.1 (2.4)	59.8 (0.4)
	Dark		105.9 (0.0)	103.3 (0.9)	103.5 (0.2)	101.7 (1.9)	103.7 (0.9)	104.9 (1.3)
Carbon Dioxide	Irradiated	NA	0.2 (0.0)	0.8 (0.0)	4.4 (0.0)	15.5 (0.1)	27.1 (0.1)	38.9 (0.5)
	Dark		<0.1 (0.0)	<0.1 (0.0)	<0.1 (0.0)	<0.1 (0.0)	<0.1 (0.0)	<0.1 (0.0)
Volatile organic compounds	Irradiated	NA	<0.1 (0.0)	<0.1 (0.0)	0.2 (0.1)	0.1 (0.0)	0.1 (0.0)	0.2 (0.1)
	Dark		<0.1 (0.0)	0.2 (0.2)	<0.1 (0.0)	0.1 (0.0)	<0.1 (0.0)	0.1 (0.0)
Material balance	Irradiated	100 (0.0)	105.9 (2.5)	102.9 (1.1)	101.7 (0.7)	98.3 (0.3)	95.5 (0.6)	99.2 (1.1)
	Dark		106.0 (2.1)	103.5 (0.7)	103.5 (0.3)	101.8 (2.0)	103.8 (0.9)	105.5 (1.3)

Notes:
ND = Not Detected; NA = Not Analysed.
[a] Time zero analysis for both irradiated and non-irradiated experiments.
[b] Refers to radioactivity not associated with specific compounds, the maximum amount of a single degradation product was 7.3% applied radioactivity.

The experimental data could be well described by a SFO kinetic model. The half-life of tetraniliprole in irradiated samples was 0.77 days with a predicted environmental DT_{50} calculated values of 4.9 solar summer days at Tokyo, Japan. Under dark conditions, DT_{50} was 0.75 days. A summary of the kinetics data is presented in Table 18

Table 18 Kinetic parameters for the photodegradation of [pyridinyl-2-^{14}C]-tetraniliprole in sterile natural water (SFO model)

Test system	DT_{50} (days)	DT_{90} (days)	χ^2 Error(%)	Rate Constant (day^{-1})	DT_{50} under natural conditions(days)	Net Photodegradation Rate Constant[a] /DT^{50} ($days^{-1}$ / days)
Irradiated	0.77	2.6	6.1	0.90	4.9 (Tokyo, Japan)[b]	Degradation under dark conditions was faster than under irradiated conditions
Dark	0.75	2.5	9.5	0.93	No conversion	

Notes:

[a] Net rate constant = rate constant of irradiated samples – rate constant of dark samples.

[b] Since the experimental degradation rates of tetraniliprole were identical under irradiated and dark conditions, it is assumed that the DT_{50} value determined for dark samples also applies for natural conditions.

In another study, the photolytic route and rate of degradation of [pyrazole-carboxamide-^{14}C]-tetraniliprole was studied in sterile natural water (pH 8.0) from the river Rhine under exposure to simulated sunlight in the laboratory for 10 days at 25 ± 2 °C (final study temperature 24.9 °C (mean irradiated) and 24.4 °C (mean dark)) (Heinemann&Junge, 2014, M-489424-01-1, Report EnSA-13-0321).

A study application rate of 0.45 mg/L was applied due to the low water solubility of the test item. 10 days of incubation under exposure to simulated sunlight were equivalent to 69.1 days of solar summer days in Japan. Control samples were incubated in the dark.

Mean material balances were 95.6 and 97.6 percent AR for irradiated and dark samples, respectively. The maximum amounts of carbon dioxide were 10.9 and ≤0.1 percent AR in irradiated and dark samples, respectively. Formation of VO was insignificant as demonstrated by values of ≤0.1 percent AR at all sampling intervals for both, irradiated and dark samples. The amount of tetraniliprole in the test solution decreased from 97.2 percent AR at study start (DAT-0) to 1.2 and 4.1 percent AR at DAT-10 in irradiated and dark samples, respectively.

Besides the formation of carbon dioxide four degradation products were identified in irradiated samples with the following maximum occurrences: tetraniliprole-N-methyl-quinazolinone with 34.5 percent AR at DAT-0.25, tetraniliprole-despyridyl-N-methyl-quinazolinone with 7.2 percent AR at DAT-2, tetraniliprole -deschloro-pyrazine with 37.2 percent AR at DAT-2 and tetraniliprole -pyrazole-5-carboxylic acid with 18.0 percent AR at DAT-10. The total unidentified residues amounted to a maximum of 56.3 percent AR and no single component exceeded 9.4 percent AR at any sampling interval. In dark samples tetraniliprole was degraded to tetraniliprole -N-methyl-quinazolinone with the maximum occurrence of 95.0 percent AR at DAT-10 (Table 19).

Table 19 Photodegradation of [pyrazolecarboxamide-^{14}C]-tetraniliprole in natural water with mean values expressed as percent AR (± standard deviatin)

Compound	Sample	Days exposed to simulated sunlight						
		0 [a]	0.25	1	2	4	7	10
Tetraniliprole	Irradiated	97.2 (0.7)	55.7 (4.9)	38.1 (1.2)	15.0 (2.0)	1.9 (0.1)	1.4 (0.1)	1.2 (0.3)
	Dark		49.3 (2.2)	17.6 (6.7)	10.1 (0.7)	3.6 (0.4)	4.2 (0.9)	4.1 (0.8)
Tetraniliprole -N-methyl-quinazolinone	Irradiated	2.5 (0.0)	34.5 (4.2)	28.6 (2.6)	20.1 (1.3)	1.5 (1.5)	ND	ND
	Dark		45.7 (2.7)	73.7 (7.4)	87.3 (2.4)	91.6 (1.5)	94.5 (1.6)	95.0 (2.2)
Tetraniliprole -despyridyl-N-methyl-quinazolinone	Irradiated	ND	0.8 (0.1)	3.7 (0.3)	7.2 (0.8)	6.9 (0.9)	5.8 (0.4)	6.1 (0.2)
	Dark		ND	ND	ND	ND	ND	ND

Compound	Sample	Days exposed to simulated sunlight						
		0 [a]	0.25	1	2	4	7	10
Tetraniliprole -deschloro-pyrazine	Irradiated	ND	1.8 (0.4)	17.6 (0.4)	37.2 (1.7)	18.0 (0.1)	4.6 (0.9)	1.3 (0.2)
	Dark		ND	ND	ND	ND	ND	ND
Tetraniliprole -pyrazole-5-carboxylic acid	Irradiated	ND	ND	ND	1.9 (0.6)	11.4 (1.2)	17.9 (0.0)	18.0 (1.8)
	Dark		ND	ND	ND	ND	ND	ND
Others [b]	Irradiated	ND	3.3 (0.1)	7.0 (0.3)	15.6 (0.2)	50.7 (0.3)	56.3 (1.0)	51.4 (1.2)
	Dark		1.0 (0.0)	0.8 (0.1)	0.9 (0.0)	0.7 (0.1)	1.4 (0.4)	1.3 (0.2)
Total residues in solution	Irradiated	99.7 (0.7)	96.1 (0.4)	95.1 (1.1)	97.0 (0.2)	90.4 (0.2)	86.0 (2.4)	78.0 (0.3)
	Dark		96.0 (0.6)	92.2 (0.8)	98.3 (3.0)	96.0 (1.2)	100.1 (0.3)	100.4 (1.6)
Carbon Dioxide	Irradiated	NA	<0.1	0.1 (0.0)	0.7 (0.0)	5.3 (0.3)	8.9 (0.0)	10.9 (0.1)
	Dark		<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Volatile organic compounds	Irradiated	NA	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Dark		<0.1	<0.1	0.1 (0.1)	<0.1	<0.1	<0.1
Material balance	Irradiated	100.0 (0.4)	96.1 (0.4)	95.2 (1.1)	98.0 (0.1)	96.0 (0.3)	94.9 (0.4)	89.2 (0.3)
	Dark		96.1 (0.5)	92.2 (0.8)	98.5 (3.2)	96.1 (1.2)	100.1 (0.3)	100.5 (1.6)

Notes:
ND – Not Detected; NA – Not Analysed.
[a] Time zero analysis for both irradiated and non-irradiated experiments.
[b] 'Others' refers to radioactivity not associated with specific compounds, the maximum amount of a single degradation product was 9.4% applied radioactivity.

The experimental data could be well described by a SFO kinetic model. The half-life of tetraniliprole in irradiated samples was 0.7 days, with a predicted environmental DT_{50} calculated values to be e.g. 4.7 solar summer days at Tokyo, Japan. Under dark conditions, a DT_{50} was 0.3 days. A summary of the kinetics is presented in Table 20.

Table 20 Kinetic parameters for the photodegradation of [pyrazolecarboxamide-^{14}C]-tetraniliprole in sterile natural water (SFO model)

Test system	DT_{50} (days)	DT_{90} (days)	Chi^2 Error (%)	Rate Constant (day^{-1})	DT_{50} under natural conditions(days)	Net Photodegradation Rate Constant [a] /DT_{50} [$days^{-1}$ / days]
Irradiated	0.7	2.2	18.2	1.0	4.7 (Tokyo, Japan)	Degradation under dark was faster than under irradiated conditions
Dark	0.3	1.1	16.8	2.2	No conversion	

Notes:
[a] Net rate constant = rate constant of irradiated samples – rate constant of dark samples.

It is concluded that photodegradation contributes to the overall degradation of tetraniliprole under aqueous conditions in natural water.

Environmental fate in soil

Photodegradation on the soil surface

The photodegradation of [pyrazole-carboxamide-^{14}C]-tetraniliprole under exposure to simulated sunlight and aerobic conditions was studied in one German soil in the laboratory for 11 days at 20 ± 2 °C (König& Beckmann, 2014, M-493228-01-1, Report EnSa-14-0217). Soil characteristics are reported in Table 21. Soil was kept in a Suntest® unit containing a Xenon lamp simulating natural sunlight. The light emission was filtered with a 290 nm cut-off UV-filter, which eliminated all wavelengths <290 nm. The intensity of the Xenon lamp was determined at the beginning and the end of the overall test period using a radiometer and photodetector assembly and was calculated as 1089 W/m2 for 300 to 2450 nm. At this light intensity, it takes 8.0 and 5.1 hours in the Suntest® unit to equal one solar summer day at Phoenix and Athens, respectively.

[Pyrazole-carboxamide-^{14}C]-tetraniliprole was applied to thin layers of soil at an application rate of 7.3 mg/kg dry soil weight (equivalent to a field application rate of 200 g tetraniliprole/ha) in individual photolysis vessels. The test was performed in static systems consisting of quartz glass vessels each containing 3 g soil (dry weight equivalents) and equipped with traps for the collection of carbon dioxide and volatile organic compounds. The test systems were continuously exposed to irradiation with artificial sunlight (Xenon lamp with a < 290 nm cut-off filter). 11 days of continuous irradiation were equivalent to 33.4 and 51.8 solar summer days in Phoenix (Arizona, United States) and Athens (Greece), respectively. Control samples were incubated in the dark.

Duplicate test systems were processed and analysed 0, 1, 2, 4, 7, 9 and 11 days after treatment (DAT) for both irradiated and dark samples.

Table 21 Soil characteristics of a German soil

Soil and location	Hoefchen am Hohenseh 4a, Burscheid,
Soil texture (USDA) [a]	silt loam
-- Sand (%)	26
-- Silt (%)	57
-- Clay (%)	17
Organic Carbon (%) [b]	1.8
Organic Matter (%)	3.1
CEC (meq/100 g)	11.5
pH (soil/water 1/1)	6.8
pH (soil/1 N KCl 1/1)	6.1
pH ($CaCl_2$)	6.4
Maximum Water Holding Capacity H_2O ad 100 g soil dry weight [%]	53.5
Water Holding Capacity at pF 2.0 (0.1 bar) [%]	39.5
Disturbed bulk density (g/cm^3)	1.12
Microbial biomass (mg microbial C/kg)	888

Notes:
[a] Classification according to United States Department of Agriculture (USDA).
[b] Organic C = organic matter/1.724 based on the certificate values.

At each sampling interval, the soil was extracted twice at ambient temperature using acetonitrile/water (4/1) and once using acetonitrile. Furthermore, two microwave-accelerated extraction steps were performed using acetonitrile/water (1/1) at 70 °C and acetone/water (1/1) at 50 °C. Degradation products in soil extracts were determined by LSC and by HPLC-radiodetection analysis. Vvolatiles and non-extracted residues were determined by LSC and combustion/LSC, respectively. Compound identity was confirmed by HPLC-MS(/MS) including accurate mass determination and by 1H-

NMR. The LOD for the HPLC-radio-detection method was 0.5 percent AR and the corresponding LOQ was 1.6 percent AR. Volatiles and non-extracted residues were determined by LSC and combustion/LSC, respectively.

The identity of the test item and its degradation product was elucidated by LC-MS/MS and/or assigned by comparison of the retention times with those of unlabelled tetraniliprole and tetraniliprole-N-methyl-quinazolinone.

Overall mean material balances were 98.7 percent AR for irradiated samples and 100 percent AR for dark samples. The maximum amount of carbon dioxide was 0.8 and <0.1 percent AR at study end (DAT-11) in irradiated and dark samples, respectively. Formation of organic volatiles was insignificant (< 0.1 percent AR) at all sampling intervals for both irradiated and dark samples.

Extracted residues ranged from 94.7 to 102.1 percent AR in irradiated samples and from 96.1 to 102.6 percent AR in dark samples. Non-extracted residues (NER) increased from <0.1 percent AR at DAT-0 to 1.3 and 0.4 percent AR at DAT-11 in irradiated and dark samples, respectively.

The amount of tetraniliprole decreased from 101.5 percent AR at DAT-0 to 77.4 percent AR at DAT-11 in irradiated samples and from 101.5 percent AR to 86.5 percent AR in dark samples, indicating degradation in irradiated samples. Tetraniliprole-N-methyl-quinazolinone was identified as a degradation product in irradiated samples (7.0 percent AR at DAT-11) and in the dark samples (5.7 percent AR at DAT-11). The unidentified residues amounted to a maximum of 12.9 percent AR in the irradiated samples with no single component exceeding 1.9 percent AR and the total unidentified residue in the dark samples amounted to 7.5 percent AR with no single component exceeding 2.9 percent AR.

The summary of the amount of [pyrazole-carboxamide-^{14}C]-tetraniliprole and its degradation products is presented in Table 22.

Table 22 Degradation of tetraniliprole in Soil Hoefchen am Hohenseh 4a in irradiated and dark samples (expressed AR; mean duplicates±standard deviation)

Compound	Mean± SD	Days after treatment						
		0	1	2	4	7	9	11
Tetraniliprole	irradiated	101.5±0.2	93.3±1.6	93.4±3.3	86±0.2	79.1±0.0	77.7±4.4	77.4±2.8
	dark	101.5±0.2	97.7±0.2	98.1±0.7	89.4±1.0	90.5±2.3	85.2±0.6	86.5±3.1
Tetraniliprole-N-methyl-quinazolinone	irradiated	n.d.	1.3± 0.3	0.9± 0.1	2.4± 0.5	6.2± 0.4	6.7± 1.4	7.0± 0.5
	dark	n.d.	0.9± 0.0	1.3± 0.1	2.2± 0.1	4.5± 0.1	5.0± 0.3	5.7± 0.6
Sum of Unid./Diff. Residues	irradiated	0.6 ± 0.1	2.7± 0.7	4.5± 0.1	6.3± 0.8	12.5±0.1	11.0±1.2	12.9±0.7
	dark	0.6 ± 0.1	1.9± 0.0	3.2± 0.1	4.4± 0.0	5.9± 0.4	6.5± 0.3	7.5± 0.3
Total extracted residues	irradiated	102.1±0.1	97.3±0.5	98.8±3.3	94.7±1.0	97.8±0.5	95.5±1.8	97.3±0.6
	dark	102.1±0.1	100.5±0.2	102.6±0.7	96.1±1.1	100.8±1.8	96.7±1.2	99.7±2.2
$^{14}CO_2$	irradiated	n.a.	<0.1±0.0	<0.1±0.0	0.2±0.0	0.5±0.1	0.7±0.1	0.8±0.1
	dark	n.a.	<0.1±0.0	<0.1±0.0	<0.1±0.0	<0.1±0.0	<0.1±0.0	<0.1±0.0
Organic volatiles	irradiated	n.a.	<0.1±0.0	<0.1±0.0	<0.1±0.0	<0.1±0.0	<0.1±0.0	<0.1±0.0
	dark	n.a.	<0.1±0.0	<0.1±0.0	<0.1±0.0	<0.1±0.0	<0.1±0.0	<0.1±0.0
Non-extracted residues	irradiated	<0.1±0.0	0.3± 0.0	0.3± 0.0	0.6± 0.1	1.1± 0.0	1.2± 0.2	1.3± 0.2
	dark	<0.1±0.0	0.1± 0.0	0.2± 0.0	0.2± 0.0	0.4± 0.0	0.3± 0.0	0.4± 0.0
Total Recovery	irradiated	102.1±0.1	97.6±0.5	99.1±3.2	95.4±1.1	99.5±0.6	97.5±1.6	99.5±1.4
	dark	102.1± 0.1	100.6± 0.2	102.8±0.7	96.3±1.1	101.2± 1.8	97± 1.2	100.1± 2.1

Notes:
n.d.: Not detected.
n.a.: Not analysed.

The experimental DT_{50} values of tetraniliprole in irradiated and dark samples were calculated using single first order (SFO) kinetics and are summarised in Table 23.

Table 23 Photodegradation kinetics of tetraniliprole in soil Hoefchen am Hohenseh 4a (SFO)

Test System	DT_{50} (days)	DT_{90} (days)	χ^2 error (%)	DT_{50} under local conditions
Irradiated	27.13	90.11	2.394	82.4 (Phoenix, United States) 127.7 (Athens, Greece)
Dark	44.31	147	1.916	No conversion

Aerobic degradation in soil – laboratory studies

[Pyrazole-carboxamide-^{14}C] – German soils

The degradation and time-dependence of sorption of [pyrazole-carboxamide-^{14}C]-tetraniliprole under aerobic laboratory conditions was studied in four terrestrial German soils (Hellpointer & Junge, 2015, M-465975-02-1, Report EnSa-113-0244). Soil characteristic are reported in Table 24 and Table 25.

Soil was treated with [pyrazole-carboxamide-^{14}C]-tetraniliprole, at an application rate of 54.9 µg/100 g soil (dry weight), equivalent to a single field application of 200 g ai/ha. The test was performed in static systems consisting of Erlenmeyer flasks each containing 100 g soil dry weight and equipped with traps for the collection of [^{14}C]-carbon dioxide and volatile organic compounds. The active substance was dispensed on the soil surface and mixed with the soil. Soil samples were incubated under aerobic conditions in the dark at 20 ± 2°C for 119 days. Duplicate soil samples were taken and analysed at 0.5 hours, 24 hours, 2, 6, 9, 16, 22, 29, 62, 91and 119 days of incubation. Soil moisture was adjusted to the maximum water holding capacity (WHC) every 30 days.

Table 24 Soil characteristics for the four different German soils

Soil name	Laacher Hoff AXXa (AX)	Dallendorf II (DD)	Hanscheiderhof (HN)	Hoefchen Am Hohenseh 4a (HF)
Location	Monheim	Blankenheim	Monheim,	Burschied
Soil texture (USDA) [a]	Loamy sand	Loam	Silt loam	Silt loam
-- Sand (%)	79	35	31	25
-- Silt (%)	16	40	54	56
-- Clay (%)	5	25	15	19
Organic Carbon (%)	1.8	5.1	2.7	2.7
Organic Matter (%) [b]	3.1	8.89	4.7	4.7
CEC (meq/100 g)	8.4	19.3	9.6	12.7
pH (0.01 M $CaCl_2$)	6.2	7.3	5.3	6.4
Maximum Water Holding Capacity (g H_2O) *ad* 100 g DW) [d]	53.4	82.7	61.3	66.7
Water Holding Capacity at pF 2.0 (0.1 bar) [%]	13.3	38.5	36.7	32.0
Water Holding Capacity at pF 2.5 (0.33 bar) [%]	11.1	33.9	25.6	23.1
Bulk density (g/cm^3)	1.21	0.96	1.04	1.05

Notes:

[a] According to USDA classification.

[b] % organic matter = % organic carbon × 1.724.

[c] Analyses performed within BCS-D-EnSa-Testing.

[d] The soil moisture [g H_2O *ad* 100 g soil dry weight] was determined using an automated halogen moisture analyser by drying three aliquots of approximately 10–20 g of the sieved soils at 105 °C.

Table 25 Soil viability expressed as mg microbial carbon per kg of soil dry weight

Soil name	Laacher Hoff AXXa (AX)	Dallendorf II (DD)	Hanscheiderhof (HN)	Hoefchen Am Hohenseh 4a (HF)
Microbial biomass at DAT=0[a]	627	2349	486	774
Microbial biomass at DAT = 62[a]	301	1885	429	602
Microbial biomass at DAT = 212[a][b]	172	1207	202	283

Notes:

[a] mg microbial carbon/kg soil dry weight.

[b] Scheduled termination date was 119 days, however, the measurements were undertaken later. This had no negative impact on the results.

Soil samples were extracted with aqueous 0.01 M $CaCl_2$ solution. After shaking for 24 hours to gain equilibrium, the suspension was centrifuged and the supernatant was removed. The remaining soil was extracted with organic solvents (two extractions with acetonitrile/water (80/20), followed by 2 extractions with acetonitrile) at room temperature and once under elevated temperature conditions (70 °C with acetonitrile/water and 50 °C with acetone to obtain elevated temperature organic extracts).

After the extraction steps the suspensions were centrifuged for $CaCl_2$ and organic extractions, respectively. The radioactivity content of these extracts was determined by LSC. The elevated temperature extracted soils were air-dried, homogenized by a planetary mill and NER were determined by combustion/LSC.

Soil extracts were characterised by LSC and HPLC/radio-detection. The LOD and the LOQ for the HPLC/radio-detection method were 0.3 and 0.9 percent AR, respectively. The amount of volatiles and non-extracted residues were determined by LSC and combustion/LSC, respectively. Recovery of the applied activity ranged from 97.2–98.5 percent AR.

The identity of the test item and its degradation products was elucidated by HPLC-MS/MS and/or assigned by comparison of the retention times with those of reference items. Reference standards used were FIX12121 (Reg 4-tetraniliprole-carboxylic acid or tetraniliprole-carboxylic acid), FIX11773 (Reg 2 (BCS-CQ63359 or tetraniliprole-N-methyl-quinazolinone), FIX12014 (BCS-CN61675).

Maximum amounts of $^{14}CO_2$ detected at DAT-119 (study end) ranged from 0.6 percent AR to 2.5 percent AR. Formation of volatile organic compounds was not significant (≤0.1 percent AR at all sampling intervals).

Total extracted residues decreased from 94.1–95 percent AR at DAT-0 to 84.3–94.4 percentAR at DAT-119 in the four soils. Non-extracted residues (NER) increased from 0.2, 0.7, 0.3 and 0.3 percent AR at DAT-0 to maximum amounts of 5.2, 13.9, 9.3 and 8.1 percent AR at DAT-119 in soils AX, DD, HN and HF, respectively.

The amount of tetraniliprole in the soil extracts decreased from 91.2 percent AR at DAT-0 to 41.9 percent AR at DAT-119 in soil AX, from 87.7 to 4.9 percent AR in soil DD, from 92.1 to 55.9 percent AR in soil HN and from 88.8 to 17.1 percent AR in soil HF. Six degradation products were identified above 5 percent AR: tetraniliprole-quinazolinone-carboxylic acid (maximum of 6.5 percent AR at DAT-119 in soil DD), tetraniliprole-N-methyl-quinazolinone (maximum of 14.6 percent AR at DAT-91 in soil HF), tetraniliprole-amide (maximum of 6.9 percent AR at DAT-62 in soil HN), tetraniliprole-carboxylic acid (maximum of 47.8 percent AR at DAT-62 in soil DD), tetraniliprole-desmethyl-amidecarboxylic acid (maximum of 12.0 percent AR at DAT-119 in soil HF), and tetraniliprole-N-methyl-quinazolinone-carboxylic acid (maximum of 10.6 percent AR at DAT-119 in soil DD). Furthermore, five degradation products were found with no component exceeding 3.5 percent AR at any sampling interval.

The amount of [pyrazole-carboxamide-^{14}C]-tetraniliprole and its degradation products as percent of applied radioactivity is summarised in Table 26.

Table 26 Degradation of tetraniliprole in under aerobic conditions (percent AR)

	DAT	0/0.5[a]	0/24[b]	2	6	9	16	22	29	62	91	119
Soil Laacher Hof AXXa												
Tetraniliprole	Mean	93.9	91.2	88.9	83.7	81.5	75.1	70.4	64.8	50.9	48	41.9
	± SD	0.8	± 1.5	±0.9	±0.6	±0.9	±0.3	±0.6	±1.0	± 0.2	±0.0	± 0.5
T-quinazolinone-carboxylic acid	Mean	<LOD	<LOD	<LOD	<LOD	<LOD	n.d.	<LOD	<LOD	<LOD	n.d.	n.d.
	± SD	-	-	-	-	-	-	-	-	-	-	-
T-N-methyl-quinazolinone	Mean	0.5	2.9	2.3	4.7	7.2	8.4	10.1	10.8	13.4	12	13.6
	± SD	± 0.1	± 0.1	±0.1	±0.3	±0.1	±0.0	±0.1	±0.5	± 0.6	±1.5	± 0.0
T-amide	Mean	n.d.	<LOD	1.8	2.9	3.3	3.9	3.8	3.4	2.6	2.1	1.2
	± SD	-	-	±0.1	±0.3	±0.1	±0.3	±0.1	±0.0	± 0.2	±0.0	± 0.0
T-carboxylic acid	Mean	n.d.	n.d.	<LOD	2.1	3.7	7.5	10.1	13.2	21.5	25.4	29.1
	± SD	-	-	-	±0.0	±0.1	±0.2	±0.2	± 0.2	± 0.2	±0.5	± 0.1
Reg5	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	<LOD	<LOD	<LOD	n.d.	n.d.
	± SD	-	-	-	-	-	-	-	-	-	-	-
T-desmethyl-amidecarboxylic acid	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	0.6	0.8	1.3	2.9	3.6	4.3
	± SD	-	-	-	-	-	±0.2	±0.2	± 0.2	± 0.1	±0.1	± 0.1
Reg7	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.6	0.6	1.3	2	2.1
	± SD	-	-	-	-	-	-	±0.0	±0.0	± 0.1	±0.4	± 0.1
T-N-methyl-quinazolinone-carboxylic acid	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	<LOD	0.8	1.1	1.4
	± SD	-	-	-	-	-	-	-	-	±0.0	±0.0	±0.0
Reg9	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	± SD	-	-	-	-	-	-	-	-	-	-	-
Reg10	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	0.4
	± SD	-	-	-	-	-	-	-	-	-	-	± 0.0
Reg11	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.4	<LOD	<LOD
	SD	-	-	-	-	-	-	-	-	± 0.0	-	-
Diffuse Residues	Mean	0.2	0.3	0.3	0.4	0.4	0.5	0.3	0.3	0.4	0.3	0.2
	SD	±0.0	± 0.1	±0.0	±0.0	±0.1	±0.0	±0.0	± 0.1	± 0.1	±0.1	±0.0
Total extracted	Mean	95	95	93.9	94	96.2	96.2	96.6	94.7	94.7	94.7	94.4
	± SD	± 0.6	± 1.2	±0.7	±0.7	±0.7	±0.5	±0.8	±1.3	± 0.0	±0.8	± 0.3
$^{14}CO_2$	Mean	n.a.	n.a.	<0.1	<0.1	<0.1	<0.1	0.1	0.2	0.5	0.8	1.0
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0
Organic volatiles	Mean	n.a.	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	< 0.1	< 0.1	<0.1	< 0.1
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0
Non-extracted residues	Mean	0.4	0.2	0.3	0.8	1.0	1.5	1.7	2.1	3.5	4.1	5.2
	± SD	±0.2	±0.0	±0.2	±0.0	±0.0	±0.0	±0.0	±0.0	±0.1	±0.1	±0.2
Material Balance [c]	Mean	95.3	95.2	94.2	94.8	97.2	97.7	98.4	97	98.8	99.6	100.7
	± SD	±0.8	±1.2	±0.9	±0.7	±0.8	±0.6	±0.9	±1.3	±0.1	±0.8	±0.4
Soil Dollendorf II												
Tetraniliprole	Mean	91.8	87.7	83.2	69.9	59.7	48.8	39.2	32.4	13.8	7.2	4.9
	± SD	± 1.3	± 0.7	±0.3	±0.9	±1.0	±0.4	±0.7	± 0.4	± 0.3	±0.4	± 0.1
T-quinazolinone-carboxylic acid	Mean	0.5	<LOD	<LOD	<LOD	<LOD	0.4	0.5	0.6	2.6	4	6.5
	± SD	± 0.1	-	-	-	-	±0.0	±0.0	±0.0	± 0.1	±0.1	± 0.1
T-N-methyl-quinazolinone	Mean	2.5	2.5	2.3	4.4	8	6.7	7.3	8.3	8.2	7.3	4.1
	± SD	± 0.3	± 0.1	±0.0	±0.1	±2.1	±0.1	±0.6	±0.2	± 0.8	±0.1	± 0.7
T-amide	Mean	n.d.	2.6	3.5	3.2	2.9	2.7	2.5	1.9	0.7	0.4	<LOD
	± SD	-	± 0.1	±0.0	±0.1	±0.1	±0.2	±0.2	± 0.1	± 0.0	±0.0	-
T-carboxylic acid	Mean	n.d.	0.9	5.8	16	21.9	32.2	40.2	42.4	47.8	45	43.3
	± SD		±0.0	±0.1	±0.2	±0.4	±0.0	±1.3	± 0.4	± 0.3	±0.3	± 0.1
Reg5	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD

	DAT	0/0.5[a]	0/24[b]	2	6	9	16	22	29	62	91	119
	± SD	-	-	-	-	-	-	-	-	-	-	-
T-desmethyl-	Mean	n.d.	n.d.	n.d.	<LOD	0.8	2.5	4.2	5.4	9.1	10.0	10.3
amidecarboxylic acid	± SD	-	-	-	-	±0.0	±0.0	±0.2	± 0.0	± 0.0	±0.1	± 0.4
Reg7	Mean	n.d.	n.d.	n.d.	<LOD	0.4	<LOD	0.6	0.9	1.5	2.5	2.3
	± SD	-	-	-	-	±0.0	-	±0.0	±0.0	± 0.1	±0.0	± 0.1
T-N-methyl-	Mean	n.d.	n.d.	n.d.	<LOD	0.4	0.7	1.0	1.9	4.9	7.8	10.6
quinazolinone-	± SD	-	-	-	-	±0.1	±0.1	±0.1	± 0.1	± 0.1	±0.3	±0.4
carboxylic acid												
Reg9	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	0.6	0.7	0.5
	± SD	-	-	-	-	-	-	-	-	±0.0	±0.1	±0.0
Reg10	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.7	0.9	1.1
	± SD	-	-	-	-	-	-	-	-	± 0.1	±0.0	± 0.1
Reg11	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	± SD	-	-	-	-	-	-	-	-	-	-	-
Diffuse Residues	Mean	0.2	0.3	0.4	0.3	0.4	0.4	1.0	0.4	0.3	0.3	0.3
	± SD	±0.0	± 0.1	±0.0	±0.0	±0.0	±0.0	±0.8	± 0.0	± 0.0	±0.0	±0.1
Total extracted	Mean	95.0	94.3	95.5	94.6	94.6	94.6	96.5	94.3	90.2	86.1	84.3
residues	± SD	± 1.1	± 0.5	±0.5	±0.8	±0.6	±0.4	±0.3	±0.6	± 0.2	±0.1	± 0.8
$^{14}CO_2$	Mean	n.a.	n.a.	<0.1	<0.1	<0.1	0.1	0.2	0.3	1.2	2.0	2.5
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.1	±0.2
Organic volatiles	Mean	n.a.	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	< 0.1	< 0.1	<0.1	< 0.1
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0
Non-extracted	Mean	0.8	0.7	1.5	2.0	3.0	3.8	4.7	5.6	9.4	11.5	13.9
residues	SD	±0.0	±0.0	±0.1	±0.1	±0.2	±0.1	±0.2	±0.1	±0.1	±0.1	±1.1
Material Balance	Mean	95.8	95	96.9	96.6	97.7	98.5	101.3	100.2	100.7	99.7	100.6
[c]	± SD	±1.1	±0.5	±0.4	±0.9	±0.4	±0.5	±0.1	±0.7	±0.3	±0.1	±0.6
Soil Hanscheiderhof												
Tetraniliprole	Mean	95.5	92.1	92.9	91.0	89.2	85.3	82.8	79.1	67.9	61.8	55.9
	± SD	± 0.2	± 0.2	±0.8	±0.0	±0.5	±1.0	±0.3	± 0.6	± 1.0	±0.0	± 0.1
T-quinazolinone-	Mean	0.4	0.3	0.4	0.5	<LOD	<LOD	<LOD	0.4	0.3	n.d.	n.d.
carboxylic acid	± SD	± 0.1	± 0.1	±0.0	±0.1	-	-	-	±0.0	±0.0	-	-
T-N-methyl-	Mean	<LOD	1.1	0.8	1.6	2.2	2.6	3.3	3.3	4.7	5.0	6.4
quinazolinone	± SD	-	± 0.1	±0.1	±0.0	±0.1	±0.0	±0.2	±0.1	± 0.2	±0.1	± 0.4
T-amide	Mean	n.d.	<LOD	1.3	2.6	3.4	5.0	5.6	6.4	6.9	6.2	6.0
	± SD	-	-	±0.2	±0.1	±0.2	±0.2	±0.1	±0.0	± 0.2	±0.1	± 0.2
T-carboxylic acid	Mean	n.d.	n.d.	n.d.	0.4	0.9	2.4	3.4	4.7	11.5	16.3	20.0
	± SD	-	-	-	±0.1	±0.1	±0.2	±0.1	± 0.2	± 0.0	±0.5	± 0.1
Reg5	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	<LOD	<LOD	0.3	<LOD
	± SD	-	-	-	-	-	-	-	-	-	±0.0	-
T-desmethyl-	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	0.8	1.0	1.0
amidecarboxylic acid	± SD	-	-	-	-	-	-	-	-	± 0.1	±0.1	± 0.0
Reg7	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	0.4	0.7	0.8
	± SD	-	-	-	-	-	-	-	-	±0.0	±0.1	± 0.1
T-N-methyl-	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	n.d.	0.4
quinazolinone-	± SD	-	-	-	-	-	-	-	-	-	-	±0.1
carboxylic acid												
Reg9	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	± SD	-	-	-	-	-	-	-	-	-	-	-
Reg10	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	0.4	0.3
	± SD	-	-	-	-	-	-	-	-	-	±0.0	± 0.0
Reg11	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.5	0.5	0.5
	± SD	-	-	-	-	-	-	-	-	± 0.0	±0.1	± 0.0
Diffuse Residues	Mean	0.2	0.3	0.3	0.4	0.4	0.5	0.3	0.3	0.4	0.4	0.3
	± SD	±0.0	± 0.0	±0.0	±0.0	±0.0	±0.1	±0.1	± 0.0	± 0.0	±0.1	±0.0
Total extracted	Mean	96.2	94.1	95.7	96.5	96.4	96.2	96.0	94.8	93.9	92.6	91.9

	DAT	0/0.5[a]	0/24[b]	2	6	9	16	22	29	62	91	119
residues	SD	± 0.3	± 0.3	±0.8	±0.2	±0.4	±0.6	±0.2	±0.3	± 1.2	±0.1	± 0.6
$^{14}CO_2$	Mean	n.a.	n.a.	<0.1	<0.1	<0.1	<0.1	0.1	0.1	0.4	0.5	0.6
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0
Organic volatiles	Mean	n.a.	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	< 0.1	< 0.1	<0.1	< 0.1
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0
Non-extracted residues	Mean	0.3	0.3	0.8	1.1	1.4	2.0	2.5	3.0	5.6	5.7	9.3
	± SD	±0.0	±0.0	±0.0	±0.0	±0.0	±0.1	±0.0	±0.0	±0.2	±0.2	±0.7
Material Balance [c]	Mean	96.5	94.5	96.4	97.7	97.8	98.2	98.5	97.9	99.8	98.8	101.7
	± SD	±0.3	±0.3	±0.9	±0.1	±0.4	±0.7	±0.2	±0.4	±1.0	±0.9	±0.8
Soil Hoefchen Am Hohenseh 4												
Compound	DAT	0/0.5[a]	0/24[b]	2	6	9	16	22	29	62	91	119
Tetraniliprole	Mean	94.0	88.8	89.6	82.1	77.2	69.2	63.8	56	34.6	23.9	17.1
	± SD	± 0.6	± 0.3	±0.2	±0.5	±0.3	±0.1	±0.1	± 0.4	± 0.4	±0.1	± 0.8
T-quinazolinone-carboxylic acid	Mean	0.4	0.3	<LOD	<LOD	0.4	n.d.	<LOD	n.d.	0.5	0.6	1.0
	± SD	± 0.0	± 0.0	-	-	±0.1	-	-	-	± 0.1	±0.1	±0.0
T-N-methyl-quinazolinone	Mean	1.2	3.3	2.4	4.9	4.5	8.2	9.5	11.1	13.9	14.6	14.2
	± SD	± 0.1	± 0.1	±0.0	±0.2	±3.0	±0.1	±0.4	±0.1	± 0.2	±0.2	± 1.1
T-amide	Mean	n.d.	1.4	3.4	4.4	4.6	4.8	5.2	4.4	3.3	2.5	1.9
	± SD	-	± 0.1	±0.1	±0.2	±0.1	±0.7	± .1	±0.3	± 0.1	±0.0	± 0.1
T-carboxylic acid	Mean	n.d.	n.d.	1.0	3.1	5.6	10.9	14.6	17.1	27.3	32.1	34.7
	± SD	-	-	±0.0	±0.3	±0.1	±0.2	±0.2	± 0.4	± 0.0	±0.7	± 1.3
Reg5	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<LOD	<LOD	n.d.	n.d.	n.d.
	± SD	-	-	-	-	-	-	-	-	-	-	-
T-desmethyl-amidecarboxylic acid	Mean	n.d.	n.d.	n.d.	n.d.	0.4	1.6	2.5	3.9	8.1	10.0	12.0
	± SD	-	-	-	-	±0.1	±0.1	±0.0	± 0.1	± 0.3	±0.1	± 0.3
Reg7	Mean	n.d.	n.d.	n.d.	n.d.	<LOD	0.5	0.9	1.3	2.1	3.5	3.3
	± SD	-	-	-	-	-	±0.1	±0.1	±0.2	±0.2	±0.1	± 0.2
T-N-methyl-quinazolinone-carboxylic acid	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.4	0.7	1.7	3.3	4.6
	± SD	-	-	-	-	-	-	±0.0	± 0.1	± 0.1	±0.1	± 0.1
Reg9	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	± SD	-	-	-	-	-	-	-	-	-	-	-
Reg10	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.4	0.8	1.1
	± SD	-	-	-	-	-	-	-	-	± 0.1	±0.1	± 0.1
Reg11	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.4	n.d.	0.4
	± SD	-	-	-	-	-	-	-	-	± 0.0	-	± 0.0
Diffuse Residues	Mean	0.3	0.3	0.4	0.4	3.2	0.5	0.3	0.3	0.3	0.3	0.3
	± SD	±0.0	± 0.1	±0.1	±0.1	±2.8	±0.0	±0.1	± 0.0	± 0.0	±0.0	±0.0
Total extracted residues	Mean	95.9±0.6	94.1	97.2	95.2	96.1	95.7	97.6	94.9	92.7	91.7	90.6
	± SD		± 0.5	±0.3	±0.9	±0.7	±1.0	±0.0	±0.5	± 0.0	±0.6	± 0.5
$^{14}CO_2$	Mean	n.a.	n.a.	<0.1	<0.1	<0.1	0.1	0.2	0.4	1.2	1.7	2.2
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.2
Organic volatiles	Mean	n.a.	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	< 0.1	< 0.1	<0.1	< 0.1
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0
Non-extracted	Mean	0.2	0.3	0.6	0.9	1.1	1.9	2.4	2.9	5.5	6.6	8.1
	± SD	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.6
Material Balance [c]	Mean	96.1	94.3	97.8	96.1	97.2	97.7	100.2	98.1	99.4	99.9	100.8
	SD	±0.5	±0.6	±0.3	±0.9	±0.7	±1.0	±0.1	±0.5	±0.0	±0.8	±0.6

Notes:

T = Tetraniliprole; n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation.

[a] Samples shaken on mechanical overhead shaker for 0.5 h or 24 h with $CaCl_2$ solution.

[b] Samples shaken on mechanical overhead shaker for 24 h with $CaCl_2$ solution.

[b] Taken from Material Balance, values may differ due to rounding.

The data were evaluated according to the FOCUS guidance document on degradation kinetics (2006) using the software KinGUI 2 to derive the DT_{50} and DT_{90} values of tetraniliprole. The results are summarised in Table 27.

Table 27: Best-fit degradation kinetics of tetraniliprole in soils under aerobic conditions

Soil (soil type)	Kinetic model	DT_{50} (days)	DT_{90} (days)	χ^2 error (%)	Visual assessment
Laacher Hof AXXa (Loamy Sand)	DFOP	94.5	959.1	1.0	Good
Dollendorf II (Loam)	FOMC	18.4	83.4	1.9	Good
Hanscheiderhof (Silt Loam)	FOMC	183	> 1000	0.7	Good
Hoefchen Am Hohenseh 4a (Silt Loam)	FOMC	43.8	176.8	1.3	Good

Notes:
DFOP = Double first order in parallel; FOMC = first order multi compartment

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Additionally, at each sampling interval, the soils were extracted with aqueous $CaCl_2$ solution (24 hours for determination of desorption behaviour) to determine the time-dependent sorption. The sorption of tetraniliprole to soil increased in the course of the study. The calculated RTDS values (Ratio of concentration of test item in soil (µg/g)/ concentration of test item in solution (µg/mL) were 4.61, 11.69, 6.71 and 8.04 mL/g for soils Laacher Hof AXXa, Dollendorf II, Hanscheiderhof and Hoefchen Am Hohenseh 4a, respectively, at the beginning of the study (DAT-0). With time of aging in soil, values increased to 13.91, 38.62, 19.39, and 28.82 mL/g on DAT-119 for the four soils.

United States soils

The degradation and time-dependence of sorption of [pyrazole-carboxamide-^{14}C]-tetraniliprole under aerobic dark laboratory conditions was investigated in six US for 120 days (Mislankar & Haddix, 2016, M-557172-01-1, Report MEFVP098). Soil characteristics are reported in Table 28 and Table 29.

Soil (50 g) was treated at a nominal application rate of 0.5 mg tetraniliprole/kg soil (dry weight), equivalent to a field rate of 200 g ai/ha (2.5 cm depth, 1.5 g/cm^3 bulk density) based on a single field application rate of 200 g tetraniliprole/ha. The test was performed in flow through system consisting of Erlenmeyer flasks each containing 50 g soil dry weight and equipped with traps for the collection of [^{14}C]-carbon dioxide and volatile organic compounds. The active substance was dispensed on the soil surface and mixed with the soil. Soil samples were incubated under aerobic conditions in the dark at 20 ± 2°C for 120 days. Duplicate soil samples were taken and analysed at 0.5 hours, 24 hours and 2, 7, 14, 28, 42, 63, 91, and 120 days after treatment for soils KS, NE, CA and ND. For CAH and HCB soils, duplicate samples were analysed after 0.5 and 24 hours, and 2, 7, 21, 30, 42, 63, 91, and 120 days after treatment. Soil moisture was adjusted to the maximum water holding capacity (WHC) 4-10 times on days 7/13, 18, 28, 37, 49, 56/58/64, 72/73, 81/84/87, 102/111, and/or 116.

Table 28 Soil characteristics of six soils in the United States of America

Soil name	KS	NE	CA	NF	CAH	HCB
Location	Stilwell, Kansas	Louisville, Nebraska	Sanger, California	Grand Forks County, North Dakota	Hughson California	Northwood, North Dakota
Soil texture (USDA) [a]	Silt loam	Silt loam	Sandy loam	Clay loam	Loamy sand	Clay loam
-- Sand (%)	4.7	15.5	68.5	32.0	78.6	22.4
-- Silt (%)	79.3	63.0	28.4	36.7	16.2	49.6
-- Clay (%)	16.0	21.5	3.1	31.3	5.2	28.0
Organic Carbon (%) [b]						
Organic Matter (%)	1.2	1.8	0.90	6.0	0.39	3.7

Soil name	KS	NE	CA	NF	CAH	HCB
Location	Stilwell, Kansas	Louisville, Nebraska	Sanger, California	Grand Forks County, North Dakota	Hughson California	Northwood, North Dakota
Soil texture (USDA) [a]	Silt loam	Silt loam	Sandy loam	Clay loam	Loamy sand	Clay loam
CEC (meq/100 g)	12.6	16.6	6.7	22.8	6.0	24.3
pH (0.01 M $CaCl_2$)	5.8	6.5	6.2	6.4	7.1	7.3
pH (water 1/1)	6.4	7.0	6.7	6.8	7.5	7.6
pH (saturated paste)	6.2	6.8	6.6	6.7	7.3	7.4
Maximum Water Holding Capacity (g H_2O *ad* 100 gram dry weight bar) [%]	39.3	64.4	27.6	55.5	21.9	63.7
Water Holding Capacity at pF 2.0 (0.1 bar) [%]	32.8	38.6	23.4	55.3	17.2	59.1
Water Holding Capacity at pF 2.5 (0.33 bar) [%]	23.0	27.8	10.9	41.1	8.5	39.8
Bulk density (g/cm^3)	0.96	0.96	1.26	0.86	1.45	0.84

Notes:
[a] Classification according to United States Department of Agriculture (USDA).
[b] Organic C = organic matter/1.724 based on the certificate values.

Table 29 Soil viability of six US soils expressed as CFU/g of dry weight soil

Soil	Micro organism	Initial Untreated Control (CFU/g)	Middle Untreated Control (CFU/g)	Final Untreated Control (CFU/g)	Final Solvent Treated Control (CFU/g)
Stilwell, KS (KS)	Actinomycetes	62,000	122,000	41,200	61,500
	Fungi	5,980	8,160	3,860	3,140
	Bacteria	1,380,000	1,110,000	1,370,000	1,290,000
Louisville, NE (NE)	Actinomycetes	85,600	172,000	59,100	81,200
	Fungi	4,460	11,500	4,170	4,800
	Bacteria	879,000	1,300,000	1,180,000	696,000
Sanger, CA (CA)	Actinomycetes	13,100	241,000	15,400	47,600
	Fungi	14,500	134,000	11,200	8,840
	Bacteria	666,000	1,900,000	837,000	1,600,000
Grand Forks County, ND (ND)	Actinomycetes	70,700	180,000	31,500	41,600
	Fungi	6,010	11,400	5,990	5,020
	Bacteria	778,000	968,000	869,000	1,180,000
Hughson, CA (CAH)	Actinomycetes	61,000	6,180	318,000	306,000
	Fungi	1,460	8,240	8,220	11,000
	Bacteria	488,000	2,750,000	651,000	721,000
Northwood, ND (HCB)	Actinomycetes	169,000	41,800	388,000	287,000
	Fungi	260	2,630	4,490	4,190
	Bacteria	109,000	1,540,000	466,000	747,000

At each sampling interval, the soil was extracted first with aqueous 0.01 M $CaCl_2$ solution, followed by four shake extractions at ambient temperature and two microwave- accelerated extractions at elevated temperature. Acetonitrile/water (80/20) was used for the first two ambient extractions and acetonitrile for the third and fourth extractions. The first microwave-accelerated extraction was performed at 70 °C using acetonitrile/water (80/20) and the second was performed at 50° C using acetone. Soil extracts were characterised by LSC and HPLC-radio-detection. The LOQ for the HPLC-radio-detection method was 1 percent AR. The amount of volatiles and non-extracted residues was determined by LSC and combustion/LSC, respectively. The tetraniliprole residues were analysed by HPLC coupled to a

radio-detector. Identification of the degradation products was accomplished by co-chromatography with authentic standards and LC-ESI/MS with exact mass capability. Reference items used were tetraniliprole-N-methyl-quinazolinone, tetraniliprole-N-methyl-quinazolinone-carboxylic acid, tetraniliprole-carboxylic acid, tetraniliprole-desmethyl-amide-carboxylic acid, and tetraniliprole -amide.

Mean material balances were 95.2, 93.4, 97.1, 99.5, 95.0 and 95.8 percent AR for KS, NE, CA, ND, CAH and HCB soils, respectively. The maximum amount of $^{14}CO_2$ formed was 1.3, 0.8, 1.0, 0.7, 0.8 and 2.1 percent AR at study end (DAT-120) in KS, NE, CA, ND, CAH and HCB soils, respectively. Formation of organic volatiles was insignificant (≤ 0.3 percent AR) at all sampling intervals for all soils.

Total extracted residues decreased from DAT-0 to DAT-120 from 99.2–100 percent AR to 75.2–91.4 percent AR in the six soils. Non-extracted residues increased from 0.3, 0.3, 0.3, 0.8, <LOQ and 0.3 percent AR at DAT-0 to 10.4, 8.4, 3.2, 12.3, 3.5 and 18.9 percent of AR at DAT-120 in soils KS, NE, CA, ND, CAH and HCB soils, respectively.

The amount of tetraniliprole in the soil extracts decreased from DAT-0 to DAT-120 from 98.7–98.8 percent to 23.5–50.7 percent AR in the six soils. Two degradation products, tetraniliprole-carboxylic acid and tetraniliprole-N-methylquinazolinone were identified with a maximum of 34.8 percent AR at DAT-120 in HCB soil and 33.4 percent AR at DAT-120 in CAH soil, respectively. Additionally, three degradation products were found, but with a maximum concentration not exceeding 4.9 percent AR at any sampling interval. The amount of [pyrazole-carboxamide-^{14}C]-tetraniliprole and its degradation products as percent of applied radioactivity is summarised in Table 30.

Table 30 Degradation of tetraniliprole in six United States soils under aerobic conditions (percent AR; mean value of duplicates)

Compound	DAT	0/0.5[a]	0/24[b]	2	7	14	28	42	63	91	120
Stilwell, Kansas (KS)											
Tetraniliprole	Mean ± SD	98.7 ± 2.2	94.5 ± 0.8	89.5 ± 2.2	85.9 ± 1.0	80.5 ± 0.3	74.5 ± 0.0	66.1 ± 0.8	57.9 ± 0.4	50.1 ± 0.6	42.8 ± 0.8
T-N-methyl-quinazolinone	Mean ± SD	1.1 ± 1.5	1.7 ± 0.4	1.8 ± 0.1	3.1 ± 0.1	4.5 ± 0.5	7.7 ± 0.4	8.9 ± 0.7	10.4 ± 0.3	11.8 ± 0.3	13.9 ± 0.5
T-N-methyl-quinazolinone carboxylic acid	Mean ± SD	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	0.3 ± 0.5	0.5 ± 0.8
T-carboxylic acid	Mean ± SD	<LOQ -	<LOQ -	0.5 ± 0.7	1.0 ± 0.1	3.0 ± 0.0	5.4 ± 0.8	9.2 ± 0.4	12.1 ±0.0	15.5 ± 0.4	18.7 ± 0.9
T-amide	Mean ± SD	<LOQ -	- -	0.5 ±0.7	1.5 ±0.1	2.5 ± 0.1	2.8 ± 0.1	2.8 ± 0.3	1.7 ± 0.1	2.2 ± 0.6	1.6 ±0.3
T-desmethyl amide carboxylic acid	Mean ± SD	<LOQ -	- -	<LOQ -	<LOQ -	<LOQ -	0.4 ± 0.5	1.8 ± 0.0	3.1 ± 0.1	3.2 ± 0.3	4.3 ± 0.1
Unknown 1	Mean ± SD	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	0.5 ± 0.2	<LOQ -	<LOQ -	<LOQ -	<LOQ -
Total extracted residues	Mean ± SD	99.7 ± 0.7	96.3 ± 0.4	92.4 ± 2.1	91.5 ± 1.4	90.5 ± 0.7	91.1 ± 0.5	88.8 ± 0.1	85.2 ±0.8	83.2 ± 0.0	81.8 ± 0.6
$^{14}CO_2$	Mean ± SD	n.a. -	n.a. -	0.0 ±0.0	0.1 ±0.0	0.1 ±0.0	0.3 ±0.0	0.5 ±0.0	0.4 ±0.6	1.0 ±0.0	1.3 ±0.0
Organic volatiles	Mean ± SD	n.a. -	n.a. -	0.0 ±0.0	0.0 ±0.0	0.0 ±0.0	0.0 ±0.0	0.0 ±0.0	0.3 ±0.4	0.0 ±0.0	0.0 ±0.0
Total volatiles	Mean ± SD	n.a. -	n.a. -	0.0 ±0.0	0.1 ±0.0	0.1 ±0.0	0.3 ±0.0	0.5 ±0.0	0.7 ±0.2	1.0 ±0.0	1.3 ±0.0
Non-extracted residues	Mean ± SD	0.3 ±0.0	1.1 ±0.0	1.6 ±0.0	2.7 ±0.2	4.2 ±0.7	4.3 ±0.1	5.9 ±0.1	6.9 ±0.1	10.5 ±0.2	10.4 ±0.0
Material Balance	Mean	100.	97.4	94.0	94.3	94.8	95.8	95.3	92.8	94.8	93.5

Compound	DAT	0/0.5[a]	0/24[b]	2	7	14	28	42	63	91	120
[c]		0									
	± SD	±0.5	±0.3	±1.4	±1.1	±1.0	±0.5	±0.2	±0.6	±0.2	±0.4
Louisville, Nebraska (NE)											
Tetraniliprole	Mean	98.8	84.4	89.0	83.8	77.5	71.2	55.4	49.0	37.3	32.2
	± SD	± 0.1	± 0.2	± 0.7	± 0.6	± 1.8	± 3.7	± 0.5	-	± 0.7	± 0.1
T-N-methyl-quinazolinone	Mean	0.9	2.9	2.3	3.3	5.3	8.0	10.3	12.4	14.8	17.6
	± SD	± 1.3	± 1.2	± 0.0	± 0.6	± 1.0	± 1.3	± 0.3	-	± 0.6	± 0.8
T-N-methyl-quinazolinone carboxylic acid	Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.8	0.9	2.0	2.8
	± SD	-	-	-	-	-	-	± 0.2	-	± 0.1	± 0.0
T-carboxylic acid	Mean	<LOQ	<LOQ	0.3	3.4	6.1	9.4	14.7	18.8	24.2	26.0
	± SD	-	-	± 0.4	± 0.1	± 0.9	± 1.7	± 0.3	-	± 1.5	± 1.3
T-amide	Mean	<LOQ	0.6	1.7	1.8	2.1	1.9	1.8	1.4	1.2	0.9
	± SD	-	±0.8	±0.0	±0.2	± 0.0	± 0.2	± 0.0	-	± 0.0	±0.0
T-desmethyl amide carboxylic acid	Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1.6	2.2	2.9	3.3
	± SD	-	-	-	-	-	-	± 0.1	-	± 0.5	± 0.2
Total extracted residues	Mean	99.7	88.0	93.3	92.4	91.0	90.5	84.6	84.7	82.5	83.7
	± SD	± 1.1	± 0.2	± 0.3	± 1.1	± 0.0	± 0.5	± 0.2	-	± 2.3	± 0.8
$^{14}CO_2$	Mean	n.a.	n.a.	0.0	0.1	0.1	0.2	0.5	0.7	1.0	0.8
	± SD	-	-	±0.0	±0.0	±0.0	±0.1	±0.0	-	±0.1	±0.5
Organic volatiles	Mean	n.a.	n.a.	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	-	±0.0	±0.0
Total volatiles	Mean	n.a.	n.a.	0.0	0.1	0.1	0.2	0.5	0.7	1.0	0.8
	± SD	-	-	±0.0	±0.0	±0.0	±0.1	±0.0	-	±0.1	±0.5
Non-extracted residues	Mean	0.3	1.7	1.3	2.0	3.2	3.6	5.0	5.9	8.8	8.4
	± SD	±0.0	±0.0	±0.2	±0.0	±0.1	±0.4	±0.2	-	±0.5	±0.5
Material Balance [c]	Mean	100.0	89.7	94.7	94.5	94.4	94.3	90.1	91.3	92.4	92.9
	± SD	±0.8	±0.1	±0.1	±0.8	±0.1	±0.0	±0.2	-	±2.1	±1.3
Sanger, California (CA)											
Tetraniliprole	Mean	98.8	103.8	91.7	87.6	82.2	75.2	74.9	63.6	56.2	50.7
	± SD	± 8.7	± 0.3	± 1.0	-	± 0.6	± 0.8	± 0.7	± 1.1	± 2.8	± 0.0
T-N-methyl-quinazolinone	Mean	0.6	1.5	2.8	5.4	8.1	11.0	13.3	13.6	15.7	18.7
	± SD	± 0.8	± 0.4	± 0.3	-	± 0.6	± 0.0	± 0.4	± 1.0	± 1.7	± 0.0
T-N-methyl-quinazolinone carboxylic acid	Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.4
	± SD	-	-	-	-	-	-	-	-	-	± 0.5
T-carboxylic acid	Mean	0.4	<LOQ	<LOQ	<LOQ	0.8	3.0	5.9	8.4	14.3	16.8
	± SD	± 0.5	-	-	-	± 0.0	± 0.0	± 0.0	± 0.2	± 0.3	± 0.5
T-amide	Mean	<LOQ	<LOQ	<LOQ	1.8	2.4	3.6	4.7	4.1	4.9	4.2
	± SD	-	-	-	-	± 0.3	± 0.1	± 0.1	±0.0	± 0.1	±0.0
desmethyl amide carboxylic acid	Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1.2	0.3	<LOQ	0.5
	± SD	-	-	-	-	-	-	± 0.0	±0.5	-	± 0.1
Unknown 1	Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.6	<LOQ
	± SD	-	-	-	-	-	-	-	-	± 0.9	-
Unknown 2	Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.4	<LOQ
	± SD	-	-	-	-	-	-	-	-	± 0.5	-
Total extracted residues	Mean	99.7	105.3	94.5	94.8	93.5	92.8	99.9	89.9	92.1	91.4
	± SD	± 7.3	± 0.7	± 0.7	-	± 1.0	± 0.7	± 0.4	± 0.6	± 0.9	± 1.0
$^{14}CO_2$	Mean	n.a.	n.a.	<LOQ	<LOQ	0.1	0.2	0.3	0.4	0.6	1.0
	± SD	-	-	-	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.1
Organic volatiles	Mean	n.a.	n.a.	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	± SD	-	-	-	-	-	-	-	-	-	-
Total volatiles	Mean	n.a.	n.a.	<LOQ	<LOQ	0.1	0.2	0.3	0.5	0.6	1.0

Compound	DAT	0/0.5[a]	0/24[b]	2	7	14	28	42	63	91	120
	± SD	-	-	-	-	±0.0	±0.0	±0.1	±0.0	±0.0	±0.1
Non-extracted residues	Mean ± SD	0.3 ±0.0	0.4 ±0.0	0.7 ±0.0	0.9 -	1.2 ±0.1	1.2 ±0.1	1.8 ±0.2	1.8 ±0.1	2.8 ±0.1	3.2 ±0.0
Material Balance [c]	Mean ± SD	100 ±5.2	105.7 ±0.6	95.2 ±0.5	95.7 -	94.8 ±0.8	94.2 ±0.4	102.1 ±0.1	92.3 ±0.5	95.6 ±0.6	95.6 ±0.6
Grand Forks County, North Dakota											
Tetraniliprole	Mean ± SD	98.2 ± 0.0	99.0 ± 0.5	93.6 ± 1.2	88.2 ± 1.4	82.5 ± 0.1	74.4 ± 0.2	67.7 ± 1.6	56.5 ± 0.2	47.9 ± 0.5	42.7 ± 1.0
T-N-methyl-quinazolinone	Mean ± SD	1.0 ± 1.4	2.1 ± 0.9	1.6 ± 0.3	2.7 ± 0.8	3.4 ± 0.1	5.2 ± 0.4	6.7 ± 0.0	8.7 ± 0.5	9.7 ± 3.8	10.2 ± 1.7
T-N-methyl-quinazolinone carboxylic acid	Mean ± SD	<LOQ -	<LOQ -	<LOQ -	1.1 ± 0.3	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	1.2 ± 0.7
T-carboxylic acid	Mean ± SD	<LOQ -	<LOQ -	1.8 ± 0.3	1.0 ± 0.3	5.0 ± 0.5	9.8 ± 0.2	13.2 ± 0.2	18.0 ± 0.4	21.7 ± 0.4	25.2 ± 0.4
T-amide	Mean ± SD	<LOQ -	0.4 ±0.5	0.0 ±0.0	2.4 ±0.4	2.8 ± 0.2	3.0 ± 0.2	3.2 ± 0.4	2.5 ±0.0	2.3 ± 0.2	2.0 ±0.0
T-desmethyl amide carboxylic acid	Mean ± SD	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	1.4 ± 0.3	2.6 ± 0.4	3.1 ± 0.1
Total extracted residues	Mean ± SD	99.2 ± 1.4	101.5 ± 0.8	96.9 ± 0.7	95.5 ± 0.4	93.8 ± 0.2	92.4 ± 0.7	90.7 ± 1.8	87.2 ±0.1	84.2 ± 3.1	84.4 ± 0.5
$^{14}CO_2$	Mean ± SD	n.a. -	n.a. -	<LOQ -	<LOQ -	0.1 ±0.0	0.1 ±0.0	0.2 ±0.0	0.3 ±0.1	0.6 ±0.0	0.7 ±0.0
Organic volatiles	Mean ± SD	n.a. -	n.a. -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -
Total volatiles	Mean ± SD	n.a. -	n.a. -	0.1 ±0.0	0.1 ±0.0	0.1 ±0.0	0.1 ±0.0	0.2 ±0.0	0.3 ±0.1	0.6 ±0.0	0.7 ±0.0
Non-extracted residues	Mean ± SD	0.8 ±0.1	2.6 ±0.1	3.0 ±0.0	4.5 ±0.2	6.2 ±0.0	6.8 ±0.2	8.2 ±0.3	8.9 ±0.1	13.6 ±0.8	12.3 ±0.1
Material Balance [c]	Mean ± SD	100.0 ±0.9	104.1 ±0.6	100.0 ±0.5	100.1 ±0.4	100.1 ±0.1	99.4 ±0.4	99.3 ±1.1	96.4 ±0.3	98.4 ±1.6	97.4 ±0.3
Hughson, California (CAH)											
Tetraniliprole	Mean ± SD	98.1 ± 1.3	95.7 ± 1.6	90.1 ± 1.2	77.9 ± 10.6	72.9 ± 0.2	70.0 ± 2.2	63.7 ± 2.0	56.6 ± 4.0	45.6 ± 1.6	39.8 ± 2.1
T-N-methyl-quinazolinone	Mean ± SD	1.4 ± 1.4	3.3 ± 1.1	2.2 ± 0.2	12.7 ± 1.5	15.4 ± 0.5	16.4 ± 1.4	20.0 ± 1.5	22.5 ± 5.3	30.5 ± 0.7	33.4 ± 4.0
T-N-methyl-quinazolinone carboxylic acid	Mean ± SD	<LOQ -	<LOQ -	<LOQ -	0.2 ± 0.3	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	0.3 ± 0.5
T-carboxylic acid	Mean ± SD	0.2 ± 0.3	0.2 ± 0.3	0.8 ± 0.1	<LOQ -	1.8 ± 0.2	1.8 ± 0.2	3.7 ± 0.5	8.0 ± 0.2	9.2 ± 1.2	9.6 ± 0.7
T-amide	Mean ± SD	<LOQ -	<LOQ -	0.2 ± 0.3	1.7 ± 0.2	2.6 ± 0.1	3.7 ± 0.1	3.3 ± 0.7	4.1 ±0.7	3.5 ± 0.2	2.6 ±0.5
T-desmethyl amide carboxylic acid	Mean ± SD	0.2 ± 0.3	<LOQ -	0.1 ± 0.2	<LOQ -	<LOQ -	0.4 ± 0.5	<LOQ -	<LOQ -	<LOQ -	<LOQ -
Unknown 3	Mean ± SD	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	2.1 ± 0.0	2.7 ± 0.0
Unknown 4	Mean ± SD	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	0.0 ± 0.0	0.3 ± 0.5
Unknown 5	Mean ± SD	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	<LOQ -	0.0 ± 0.0	0.3 ± 0.4
Total extracted residues	Mean ± SD	100.0 ± 0.5	99.2 ± 0.2	93.4 ± 1.1	92.5 ± 0.4	92.7 ± 0.0	92.2 ± 0.0	90.7 ± 0.8	91.1 ± 0.4	90.8 ± 0.1	89.1 ± 1.2

Compound	DAT	0/0.5[a]	0/24[b]	2	7	14	28	42	63	91	120
$^{14}CO_2$	Mean	n.a.	n.a.	0.1	0.1	0.1	0.1	0.3	0.4	0.6	0.8
	± SD	-	-	±0.0	±0.0	±0.0	±0.1	±0.1	±0.0	±0.1	±0.0
Organic volatiles	Mean	n.a.	n.a.	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	± SD	-	-	-	-	-	-	-	-	-	-
Total volatiles	Mean	n.a.	n.a.	0.1	0.1	0.2	0.1	0.3	0.4	0.6	0.8
	± SD	-	-	±0.0	±0.0	±0.0	±0.1	±0.1	±0.0	±0.1	±0.0
Non-extracted residues	Mean	<LOQ	0.4	0.5	1.0	1.7	1.7	2.0	2.7	2.5	3.5
	± SD	-	±0.0	±0.0	±0.0	±0.2	±0.0	±0.0	±0.2	±0.1	±0.3
Material Balance [c]	Mean	100.0	99.6	94.0	93.6	94.5	94.0	93.1	94.2	94.0	93.4
	± SD	±0.4	±0.1	±0.8	±0.3	±0.2	±0.1	±0.6	±0.1	±0.1	±1.0
Northwood, North Dakota											
Tetraniliprole	Mean	98.7	94.6	88.4	80.6	63.8	59.0	48.9	39.9	30.2	23.5
	± SD	± 2.2	± 0.0	± 0.2	± 0.8	± 0.7	± 0.6	-	± 0.0	± 1.7	± 0.1
T-N-methyl-quinazolinone	Mean	1.0	0.6	1.1	2.8	5.0	4.2	5.9	6.0	7.6	8.5
	± SD	± 0.1	± 0.8	± 0.1	± 0.0	± 0.1	± 0.5	-	± 0.1	± 0.9	± 0.7
T-N-methyl-quinazolinone carboxylic acid	Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.8	<LOQ	2.2	2.8
	± SD	-	-	-	-	-	-	-	-	± 0.4	± 0.1
T-carboxylic acid	Mean	<LOQ	0.7	0.3	4.7	15.4	20.7	24.0	30.2	33.5	34.8
	± SD		± 0.1	± 0.4	± 1.3	± 0.1	± 1.0	-	± 0.0	± 0.9	± 0.8
T-amide	Mean	<LOQ	1.5	2.1	2.5	2.6	2.7	2.0	2.1	0.8	1.2
	± SD	-	± 0.1	± 0.1	± 0.0	± 0.0	± 0.2	-	±0.0	± 1.1	±0.3
T-desmethyl amide carboxylic acid	Mean	<LOQ	<LOQ	1.5	0.9	<LOQ	<LOQ	0.7	2.3	2.8	2.8
	± SD	-	-	± 0.6	± 1.2	-	-	-	± 0.3	± 0.1	± 0.2
Unknown 3	Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1.0
	± SD	-	-	-	-	-	-	-	-	-	± 0.1
Unknown 6	Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.5
	± SD	-	-	-	-	-	-	-	-	-	± 0.7
Total extracted residues	Mean	99.7	97.4	93.4	91.4	86.8	86.6	82.3	80.5	77.2	75.2
	± SD	± 2.1	± 0.8	± 0.8	± 0.7	± 0.9	± 2.0	-	± 0.3	± 0.8	± 0.5
$^{14}CO_2$	Mean	n.a.	n.a.	0.1	0.1	0.2	0.1	0.3	0.5	0.8	2.1
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	-	±0.1	±0.1	±1.4
Organic volatiles	Mean	n.a.	n.a.	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	± SD	-	-	-	-	-	-	-	-	-	-
Total volatiles	Mean	n.a.	n.a.	0.1	0.1	0.2	0.1	0.4	0.6	0.8	2.1
	± SD	-	-	±0.0	±0.0	±0.0	±0.0	-	±0.0	±0.1	±1.4
Non-extracted residues	Mean	0.3	1.4	1.9	4.4	7.0	8.9	10.9	13.6	16.3	18.9
	± SD	±0.0	±0.0	±0.2	±0.1	±0.2	±0.5	-	±0.1	±0.5	±0.6
Material Balance [c]	Mean	100.0	98.8	95.4	95.9	94.1	95.7	93.6	94.7	94.2	96.2
	± SD	±1.5	±0.5	±0.7	±0.4	±0.8	±1.9	-	±0.1	±0.3	±0.1

Notes:

T=tetraniliprole; n.a.: not analysed, DAT: days after treatment, ± SD: standard deviation.

[a] Samples shaken on benchtop shaker for 0.5 h with $CaCl_2$ solution.

[b] Samples shaken on benchtop shaker for 24 h with $CaCl_2$ solution.

[c] Taken from Material Balance, values may differ due to rounding

[d] One replicate only, replicate 2 data not used, test system did not have enough moisture for proper metabolism.

[e] One replicate only, centrifuge bottle broke and sample lost for replicate 2.

[f] One replicate only, for replicate 2 flask may not have received correct dose.

The rate of degradation of tetraniliprole was calculated using kinetic modelling. The data were evaluated according to the FOCUS guidance document on degradation kinetics (2006) using the software KinGUI 2 to derive the DT_{50}, DT_{75} and DT_{90} values of tetraniliprole. The best-fit degradation kinetics of tetraniliprole in soil are summarized in Table 31.

Table 31 Best-fit degradation kinetics of tetraniliprole in soils under aerobic conditions

Soil (soil type)	Kinetic model	DT_{50} (d)	DT_{75} (d)	DT_{90} (d)	χ^2 error (%)	Visual assessment
KS (silt loam)	DFOP	91.8	202	348	1.22	Good
NE (silt loam)	DFOP	58.3	134	234	3.09	Good
ND (clay loam)	DFOP	90.1	200	345	1.62	Good
CA (sandy loam)	DFOP	117	267	465	2.99	Good
CAH (loamy sand)	DFOP	82.5	197	346	1.58	Good
HCB (clay loam)	DFOP	45.8	111	198	1.82	Good

Notes:
DFOP = double first order in parallel.

Additionally, at each sampling interval, the soils were extracted with aqueous $CaCl_2$ solution (24 hours for determination of desorption behaviour) to determine the time-dependent sorption. The sorption of tetraniliprole to soil increased in the course of the study. The calculated RTDS values (Ratio of concentration of test item in soil [µg/g]/ concentration of test item in solution [µg/mL]) were 4.67, 6.87, 1.45, 14.86, 0.58 and 14.91 mL/g for soils KS, NE, CA, ND, CAH and HCB, respectively, at the beginning of the study (DAT-0). With time of aging in soil, these values increased throughout the study in all soils to 12.11, 15.12, 4.64, 80.53, 1.86 and 51.24 mL/g for soils KS, NE, CA, ND, CAH and HCB, respectively, on DAT-120.

Italian paddy soil

The route and rate of degradation of [pyrazole-carboxamide-^{14}C]-tetraniliprole under dark laboratory conditions was investigated in an Italian paddy sandy loam soil at 25 ± 2 °C for 181 days (Heinemann & Kasel, 2016b, M-545810-01-1, Report EnSa-14-1369). Soil characteristics are given in Table 32.

Paddy conditions were re-created using static test systems consisting of cylindrical flasks (5 cm diameter) containing 100 g soil (dry weight equivalent) and 100 mL of de-ionised water (water layer approximately 3.5 cm height). Incubation vessels were equipped with traps (permeable for oxygen) for the collection of carbon dioxide and volatile organic compounds. Untreated test vessels were equilibrated to study conditions for 15 days prior to application (to establish reductive conditions in the deeper soil layer, E_H <200 mV).

The application rate used in the study was 58.9 µg/100g soil (dry weight) based on a single field application rate of tetraniliprole at 200 g/ha (assuming a soil mixing depth of 0.1 m and a soil bulk density of 1.0 g/cm^3). Due to analytical reasons, a 3-fold application rate was used resulting in a nominal study application rate of 600 µg/kg (soil dry weight). Applications were made via pipette drops to the water surface of each pre-equilibrated test system prior to the water and soil in each sample being completely mixed.

Duplicate samples were processed and analysed at 0, 3, 7, 14, 30, 62, 100, 140 and 181 days after treatment (DAT). At each sampling interval, the water was decanted and the remaining soil extracted at ambient temperature, twice using acetonitrile/water (4/1) and twice using acetonitrile. Following this, two microwave-assisted extraction steps were performed using acetonitrile/water (4/1) at 70 ˚C and acetone at 50 ˚C.

Table 32 Soil characteristics Italian paddy soil, including microbial activity

Soil and location	Satirana Lomellina
Soil texture (U± SDA) [a]	Sandy loam
-- Sand (%)	65

Soil and location	Satirana Lomellina
-- Silt (%)	25
-- Clay (%)	10
Organic Carbon (%) [b]	1.0
Organic Matter (%)	1.7
CEC (meq/100 g)	5.9
pH (0.01 M $CaCl_2$)	5.3
pH (water 1/1)	5.6
pH (saturated paste)	5.6
pH (soil/1 N KC 1/1)	5.0
Maximum Water Holding Capacity (g H_2O *ad* 100 gram dry weight bar) [%]	38.1
Water Holding Capacity at pF 2.0 (0.1 bar) [%]	21.2
Bulk density (g/cm^3)	1.14
Microbial activity determination	
DAT-1 BIO	306
DAT-122 BIO/BIO +	282/275
DAT -196 BIO/BIO+	239/24

Notes:

BIO samples were left untreated; BIO+ samples were applied with 200 µL methanol.

[a] Classification according to United States Department of Agriculture (U± SDA).

[b] Organic C = organic matter/1.724 based on the certificate values.

The water and soil extracts were analysed by LSC and HPLC-radio-detection analysis. The LOD and the LOQ for the HPLC/radio-detection method were 0.3 and 0.9 percent of applied radioactivity (percent AR), respectively, for the water phase and 0.6 and 1.7 percent AR, respectively, for the combined extract (the maximum LOD and LOQ values for the combined extract were the values taken into account for calculations). The amount of volatiles and non-extracted residues was determined by LSC and combustion/LSC, respectively.

The identity of the test item and its degradation products were identified by HPLC-MS/MS including accurate mass determination. Reference standards used were parent tetraniliprole and tetraniliprole--N-methyl-quinazolinone.

Overall, the material balance ranged from 101 to 109 percent AR with a mean material balance, throughout the study, of 102 percent AR. The maximum amount of $^{14}CO_2$, detected at the end of the study (DAT-181), was only 0.1 percent AR and formation of volatile organic compounds (VOCs) was also insignificant (≤0.2 percent AR at all sampling intervals).

Residues in the water compartment decreased from 54.9 percent AR (0-DAT) to 2.6 percent AR (181-DAT). Extractable residues in the soil increased from 47.4 percent AR at DAT-0 to 94.0 percent AR at DAT-140 and then decreased to 85.9 percent AR at DAT-181. Extracted residues in the total system (water and soil extracts) decreased from 102 percent AR at DAT-0 to 88.5 percent AR at DAT-181. Non-extracted residues increased from 0.1 percent AR (DAT-0) to 12.1 percent AR (DAT-181).

The amount of tetraniliprole in the water decreased from 54.3 percent AR (DAT-0) to 0.9 percent AR (DAT-181). The amount of tetraniliprole in the soil extracts increased from DAT-0 to DAT-30, from 46.9 to 55.9 percent AR, and then decreased to 34.4 percent AR at the end of the study (DAT-181). The amount of tetraniliprole in the total system decreased from 101 (DAT-0) to 35.3 percent AR (DAT-181).

One degradation product was identified. Tetraniliprole-N-methyl-quinazolinone was found to have a maximum occurrence in the total system of 47.6 percent AR at DAT-140. Other unidentified residues in

the total system amounted to a total maximum of 6.3 percent AR and no single component exceeded 3.6 percent AR at any sampling interval.

The mean recoveries of radioactivity in the Satirana Lomellina soil under paddy conditions are presented in Table 33. The amount of [pyrazole-carboxamide-^{14}C]-tetraniliprole and its degradation product as percent of applied radioactivity is summarised in Table 34.

Table 33 Material balance and distribution of radioactivity in the sandy loam soil Satirana Lomellina under paddy (mixed anaerobic/aerobic) conditions (expressed as percent of applied radioactivity; mean value of duplicate samples)

Fraction		Days after treatment								
		0	3	7	14	30	62	100	140	181
Water		54.9	55.3	44.2	43.9	17.0	7.7	4.5	3.1	2.6
Soil Extracted Residues	Ambient Extract	46.4	43.7	51.0	45.9	64.3	71.1	69.5	80.5	73.2
	Microwave Extract 1	0.9	1.5	2.6	4.3	6.8	8.6	12.7	10.4	9.3
	Microwave Extract 2	0.1	0.5	0.9	1.5	2.2	2.6	3.6	3.1	3.4
	Total extracted	47.4	45.6	54.5	51.7	73.4	82.2	85.8	94.0	85.9
Total Extracted Residues		102.4	100.9	98.7	95.6	90.4	89.9	90.3	97.1	88.5
Non-Extracted Residues		0.1	1.0	2.4	6.2	10.1	10.8	12.0	11.5	12.1
Volatiles	CO_2	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	0.1	< 0.1	0.1	0.1
	VOC	n.a.	0.2	0.1	< 0.1	< 0.1	0.1	< 0.1	<0.1	<0.1
	Total Volatiles	n.a.	0.2	0.2	< 0.1	< 0.1	0.1	< 0.1	0.1	0.1
Material Balance (%)		102.5	102.1	101.3	101.8	100.5	100.8	102.3	108.7	100.7

Notes:
n.a.: not analysed.
VOC = Volatile Organic Compounds.

Table 34 Degradation of tetraniliprole in the sandy loam soil Satirana Lomellina under paddy (mixed anaerobic/aerobic) conditions (expressed as percent AR; mean value of duplicate samples)

Compound	Source		Days after treatment								
			0	3	7	14	30	62	100	140	181
Tetraniliprole	Water	Mean	54.3	54.7	43.4	40.7	15.7	5.1	2.5	1.3	0.9
		± SD	±4.2	±5.4	±6.5	±2.1	±1.0	±0.4	±0.1	±0.0	± .1
	Soil	Mean	46.9	42.7	47.2	42.4	55.9	51.7	43.6	41.6	34.4
		± SD	±4.1	±5.1	±6.4	±2.1	±0.1	±1.2	±1.8	±0.1	±0.8
	Total System	Mean [4]	101.2	97.4	90.6	83.0	71.7	56.9	46.1	43.0	35.3
		± SD	±0.1	±0.3	±0.1	±4.2	±0.9	±0.8	±1.9	±0.0	±0.7
tetraniliprole-N-methylquinazolinone	Water	Mean	n.d.	<LOD	0.8	3.2	0.7	1.3	0.8	<LOD	<LOD
		± SD	-	-	±0.2	±0.2	±0.0	±0.2	±0.0	-	-
	Soil	Mean	n.d.	2.5	6.8	8.3	16.3	28.2	38.9	47.6	45.0
		± SD	-	±0.4	±0.1	±0.4	±0.0	±0.5	±0.3	±0.1	±0.5
	Total System	Mean [4]	n.d.	2.5	7.6	11.5	17.0	29.5	39.6	47.6	45.0
		± SD	-	±0.4	±0.1	±0.7	±0.0	±0.7	±0.3	±0.1	±0.5
Sum of Unid./Diff. Residues [a]	Water	Mean	<LOD	<LOD	n.d.	n.d.	<LOD	<LOD	0.6	0.8	0.8
		± SD	-	-	-	-	-	-	±0.0	±0.1	±0.1
	Soil	Mean	<LOD	<LOD	<LOD	0.6	1.1	2.3	3.1	4.8	5.5
		± SD	-	-	-	±0.0	±0.5	±0.0	±0.0	±0.1	±0.1
	Total System	Mean [4]	0.7	<LOD	<LOD	0.6	1.5	2.7	3.7	5.7	6.3
		± SD	±0.0	-	-	±0.0	±0.9	±0.4	±0.1	±0.1	±0.0
Total Extracted Residues [b]	Water	Mean	54.7	54.7	44.2	43.9	16.8	6.8	3.9	2.2	1.7
		± SD	±4.6	±5.4	±6.7	±2.3	±0.6	±1.0	±0.1	±0.1	±0.2
	Soil	Mean	47.2	45.1	54.2	51.3	73.4	82.2	85.6	94.0	84.9
		± SD	±4.4	±5.6	±6.8	±2.5	±0.6	±0.7	±1.4	±0.1	±0.4

Compound	Source		Days after treatment								
			0	3	7	14	30	62	100	140	181
	Total System	Mean [4]	101.9	99.8	98.4	95.2	90.2	89.0	89.5	96.2	86.6
		± SD	±0.2	±0.2	±0.1	±4.8	±0.0	±0.3	±1.5	±0.0	±0.2
$^{14}CO_2$ [c]		Mean	n.a.	<0.1	<0.1	<0.1	<0.1	0.1	<0.1	0.1	0.1
		± SD	-	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0
Volatile Organic Compounds [c]		Mean	n.a.	0.2	0.1	<0.1	<0.1	0.1	<0.1	<0.1	<0.1
		± SD	-	±0.1	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0	±0.0
Non-extracted residues [c]		Mean	0.1	1.0	2.4	6.2	10.1	10.8	12.0	11.5	12.1
		± SD	±0.0	±0.1	±0.4	±0.4	±0.5	±0.0	±0.2	±0.4	±0.7
Total Recovery [b]		Mean	102	101	101	101.4	100.3	99.9	101.4	107.8	98.8
		± SD	±0.0	±0.1	± .4	±0.4	±0.5	±0.3	±1.7	±0.4	±1.0

Notes:

n.d.: not detected, LOD: limit of detection; ± SD: standard deviation.

[a] Minor components are summed up to sum of unidentified / diffuse residues. None of the unidentified minor components individually exceeded 4% AR.

[b] These values may differ to the material balance values due to rounding errors as well as clean up and chromatographic losses.

[c] Values taken from Material Balance.

[d] Mean values of the entire system could be unequal compared to the sum of the mean values of water and soil, because mean values of the entire system were calculated.

The data for the test item were evaluated according to the FOCUS guidance document on degradation kinetics (2006) using the software KinGUI 2 to derive the DT_{50} and DT_{90} values of tetraniliprole. The degradation of tetraniliprole under paddy conditions best followed double first order in parallel (DFOP) kinetics in both the water and the total system based on the lowest chi^2 error values and visual assessments of fits. The best-fit degradation kinetics of tetraniliprole are summarized in Table 35.

Table 35 Best-fit degradation kinetics of tetraniliprole in a Satirana Lomellina paddy soil system under paddy conditions and overlying water using the double first order in parallel model

Satirana Lomellina sandy loam soil	DT_{50} (d)	DT_{90} (d)	χ^2 error (%)	Visual assessment
Water	4.4	46.0	7.3	Good
Entire System	84.5	548	1.3	Good

The proposed degradation pathway for tetraniliprole in soils under aerobic conditions is presented in Figure 2.

Rotational crops

The Meeting received information on confined and field rotational crops.

Confined rotational crop studies

The metabolism of tetraniliprole was investigated in confined rotational crops after one spray application onto bare soil in two studies [Bongartz&Schallau, 2014c, Report EnSA-14-0494, M-500569-01-1 and Bongartz&Schallau, 2014d, Report EnSA-14-0495, M-500576-01-1]. The soil was treated with 213.1 g phenyl-carbamoyl label/ha according to the envisaged use pattern (Report EnSA-14-0494) and in the second study [pyrazole carboxamide-^{14}C]-labelled tetraniliprole at an application rate of 209.4 g ai/ha was applied (EnSa-14-0495).Turnips (root vegetables), Swiss chard (leafy vegetables) and wheat (cereals) were sown 30 days (1st rotation), 168 days (2nd rotation) and 286 days (3rd rotation) after soil treatment. A sample of immature Swiss chard was harvested at BBCH 45. Wheat forage was sampled at BBCH 29

and wheat hay at BBCH 75-83. Turnip leaves, turnip roots, Swiss chard, wheat straw and wheat grain were harvested at maturity.

The TRRs obtained in the different crop commodities are presented in Table 36.

Table 36 TRR values in confined rotational crops after spray application onto bare soil with [phenyl-carbamoyl-^{14}C]- and [pyrazole carboxamide-^{14}C]- tetraniliprole

	TRR (mg eq/kg)					
Matrix	1st rotation (30 day PBI)		2nd rotation (168 day PBI)		3rd rotation (286 day PBI)	
Report number	EnSa-14-0494	EnSa-14-0495	EnSa-14-0494	EnSa-14-0495	EnSa-14-0494	EnSa-14-0495
Label [a]	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car
Wheat forage	0.060	0.057	0.024	0.030	0.007 [b]	0.014
Wheat hay	0.160	0.208	0.063	0.062	0.028	0.064
Wheat straw	0.116	0.256	0.067	0.104	0.035	0.110
Wheat grain	0.001 [b]	0.006 [b]	0.004 [b]	0.007 [b]	0.002 [b]	0.007 [b]
Turnip leaves	0.006	0.007	0.004 [b]	0.002 [b]	0.003 [b]	0.007 [b]
Turnip roots	0.002	0.004	0.001 [b]	0.008 [b]	0.001 [b]	0.002 [b]
Swiss chard (immature)	0.056	0.056	0.016	0.020	0.012	0.014
Swiss chard (mature)	0.047	0.052	0.014	0.023	0.008 [b]	0.016

Notes:

TRR values were determined by combustion/LSC analysis.

[a] Phen-car = [phenyl-carbamoyl-^{14}C]-label and Pyr-car = [pyrazole-carboxamide-^{14}C]-label.

[b] Samples were not further extracted.

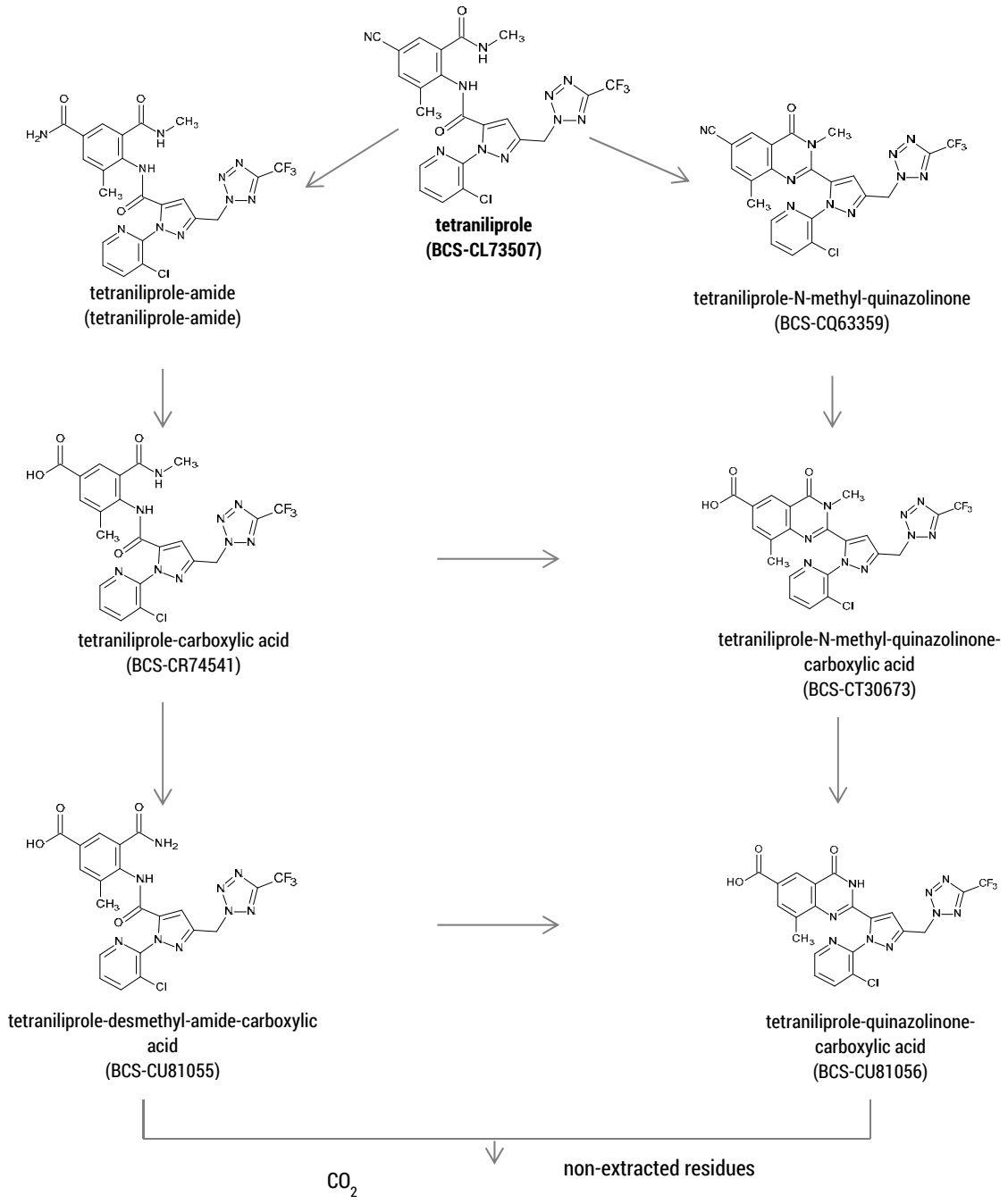


Figure 2 Overview of the degradation pathway of tetraniliprole in aerobic soil

The majority of the radioactive residue of all RACs was extracted with a mixture of acetonitrile/water (8/2) and 10 mL/L formic acid. The residues in the extracts ranged from 77 to 99 percent TRR with [phenyl-carbamoyl-^{14}C]-tetraniliprole and from 79 to 99 percent TRR with [pyrazole-carboxamide-^{14}C]-tetraniliprole. PES of wheat hay of the 1st and 2nd rotation and straw of the 1st rotation were further extracted using microwave assistance with a mixture of acetonitrile/water (1/1) and 3 percent formic acid and, in case of wheat hay of the 2nd rotation, subsequently with 0.1 N hydrochloric acid ([pyrazole-carboxamide-^{14}C]-label only). Microwave assisted solvent extracted residues in the acetonitrile/water mixture were further characterised by partition against ethyl acetate. The radioactive

residues in the organic and aqueous phases in wheat hay and straw amounted to ≤0.010 mg eq/kg (6.4–10.5 percent TRR) and to ≤0.016 mg eq/kg (7.7–15.1 percent TRR) with the respective labels.

Table 37 Distribution of the radioactivity in the extracts of the RACs in the different rotations

	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
[phenyl-carbamoyl-^{14}C]-tetraniliprole								
First rotation								
Crop	Wheat forage		Wheat hay		Wheat straw		Wheat grain	
Initial solvent extract	97.4	0.058	88.1	0.14	84	0.098	-	-
Microwave/acid extract:	-	-	6.4	0.010	10.5	0.012	-	-
- partition against ethyl acetate:								
Aqueous phase	-	-	5.2	0.008	8.9	0.010	-	-
Organic phase	-	-	1.2	0.002	1.6	0.002	-	-
Post extraction solids	2.6	0.002	5.5	0.009	5.7	0.007	-	-
Accountability	100	0.060	100	0.160	100	0.116	-	-
Crop	Turnip leaves		Turnip roots		Swiss chard (immature)		Swiss chard (mature)	
Solvent extract	92.1	0.005	86.2	0.002	99	0.056	98.8	0.046
Post extraction solids	7.9	<0.001	13.8	<0.001	1.0	0.001	1.2	0.001
Accountability	100	0.006	100	0.002	100	0.056	100	0.047
Second rotation								
Crop	Wheat forage		Wheat hay		Wheat straw		Wheat grain	
Initial solvent extract	94.2	0.023	82.9	0.052	93.7	0.063	-	-
Microwave/acid extract:	-	-	10.1	0.006	-	-	-	-
- partition against ethyl acetate:								
Aqueous phase	-	-	4.4	0.003	-	-	-	-
Organic phase	-	-	5.8	0.004	-	-	-	-
Post extraction solids	5.8	0.001	7.0	0.004	6.3	0.004	-	-
Accountability	100	0.024	100	0.063	100	0.067	-	-
Crop	Turnip leaves		Turnip roots		Swiss chard (immature)		Swiss chard (mature)	
Solvent extract	-	-	-	-	97.0	0.016	96.2	0.013
Post extraction solids	-	-	-	-	3.0	<0.001	3.8	0.001
Accountability	-	-	-	-	100	0.016	100	0.014
Third rotation								
Crop	Wheat forage		Wheat hay		Wheat straw		Wheat grain	
Solvent extract	-	-	85.4	0.024	77.4	0.027	-	-
Post extraction solids	-	-	14.6	0.004	22.6	0.008	-	-
Accountability	-	-	100	0.028	100	0.036	-	-
Crop	Turnip leaves		Turnip roots		Swiss chard (immature)		Swiss chard (mature)	
Solvent extract	-	-	-	-	95.6	0.011	-	-
Post extraction solids	-	-	-	-	4.5	0.001	-	-
Accountability	-	-	-	-	100	0.012	-	-
[pyrazole-carboxamide-^{14}C]-tetraniliprole								
First rotation								
Crop	Wheat forage		Wheat hay		Wheat straw		Wheat grain	
Solvent extract	97.7	0.055	85.9	0.178	84.9	0.217	-	-
Microwave/acid extract:	-	-	7.7	0.016	8.7	0.022	-	-
- partition against ethyl acetate:								
Aqueous phase	-	-	3.8	0.008	4.6	0.012	-	-
Organic phase	-	-	3.9	0.008	4.1	0.011	-	-
Post extraction solids	2.3	0.001	6.4	0.013	6.4	0.016	-	-
Accountability	100	0.057	100	0.208	100	0.256	-	-
Crop	Turnip leaves		Turnip roots		Swiss chard		Swiss chard	

	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
					(immature)		(mature)	
Solvent extract	97.5	0.007	93.7	0.003	99.3	0.055	99.1	0.051
Post extraction solids	2.5	<0.001	6.3	<0.001	0.07	<0.001	0.9	<0.001
Accountability	100	0.007	100	0.004	100	0.056	100	0.052
Second rotation								
Crop	Wheat forage		Wheat hay		Wheat straw		Wheat grain	
Solvent extract	95.3	0.028	79.4	0.049	87.9	0.091	-	-
Microwave/acid extract:	-	-	15.1	0.009	-	-	-	-
- partition against ethyl acetate:								
Aqueous phase	-	-	6.5	0.004	-	-	-	-
Organic phase	-	-	8.6	0.005	-	-	-	-
Post extraction solids	4.7	0.001	5.5	0.003	12.1	0.013	-	-
Accountability	100	0.030	100	0.062	100	0.104	-	-
Crop	Turnip leaves		Turnip roots		Swiss chard (immature)		Swiss chard (mature)	
Solvent extract	-	-	-	-	97.8	0.019	97.7	0.022
Post extraction solids	-	-	-	-	2.2	<0.001	2.3	0.001
Accountability	-	-	-	-	100	0.020	100	0.023
Third rotation								
Crop	Wheat forage		Wheat hay		Wheat straw		Wheat grain	
Solvent extract	95.3	0.013	90.4	0.058	80.9	0.089	-	-
Microwave/acid extract:	-	-	-	-	10.5	0.012	-	-
- partition against ethyl acetate:								
Aqueous phase	-	-	-	-	5.8	0.006	-	-
Organic phase	-	-	-	-	4.7	0.005	-	-
- 0.1 N hydrochloric acid	-	-	-	-	3.0	0.003	-	-
Post extraction solids	4.7	0.001	9.6	0.006	5.7	0.006	-	-
Accountability	100	0.014	100	0.064	100	0.110	-	-
Crop	Turnip leaves		Turnip roots		Swiss chard (immature)		Swiss chard (mature)	
Solvent extract	-	-	-	-	96.8	0.014	96.3	0.015
Post extraction solids	-	-	-	-	3.2	<0.001	3.7	0.001
Accountability	-	-	-	-	100	0.014	100	0.016

Extracts were analysed by HPLC. A summary of the rotational crop metabolism data is presented in Table 37. Parent tetraniliprole was the most prominent compound in all RACs. Other identified compounds were tetraniliprole-despyridyl-N-methyl-quinazolinone-pyrazole-3-carboxylic acid, tetraniliprole-dihydroxy, tetraniliprole -amide, tetraniliprole -desmethyl-amide-carboxylic acid, tetraniliprole-carboxylic acid and tetraniliprole-N-methyl-quinazolinone. Low residues of tetraniliprole-pyrazole-5-carboxylic acid (≤0.005 mg/kg) were also detected; this metabolite is specific for the pyrazole-carboxamide label. Each of these identified metabolites amounted to ≤0.023 mg eq/kg (phenyl-carbamoyl label) or to ≤0.028 mg eq/kg (pyrazole-carboxamide label). All unknown metabolites were present in negligible amounts (≤0.002 mg eq/kg and ≤0.006 mg eq/kg with the respective labels).

Table 38 Residues of [phenyl-carbamoyl-^{14}C]-or [pyrazole-carboxamide-^{14}C]-tetraniliprole and metabolites in rotational crop commodities

Metabolite	TRR in mg eq/kg (%TRR)					
	30 day PBI		168 day PBI		286 day PBI	
	EnSa-14-0494	EnSa-14-0495	EnSa-14-0494	EnSa-14-0495	EnSa-14-0494	EnSa-14-0495
	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car
Wheat forage (TRR, mg eq/kg)	0.060	0.057	0.024	0.030	0.007 [a]	0.014
Tetraniliprole (tetraniliprole)	0.053 (88.3%)	0.048 (84.7%)	0.015 (60.4%)	0.014 (45.7%)	NA	0.006 (43.5%)
Tetraniliprole-pyrazole-5-carboxylic acid	-	-	-	0.003 (9.4%)	NA	-
Tetraniliprole-dihydroxy	-	0.001 (2.6%)	0.002 (9.4%)	0.002 (7.3%)	NA	0.002 (17.7%)
Tetraniliprole-amide	0.002 (2.7%)	-	0.001 (5.7%)	-	NA	-
Tetraniliprole-desmethyl-amide-carboxylic acid	-	0.003 (4.8%)	-	0.006 (21.4%)	NA	0.003 (24.5%)
Tetraniliprole-carboxylic acid	-	-	0.002 (9.3%)	0.001 (3.8%)	NA	-
Tetraniliprole-N-methyl-quinazolinone	0.004 (6.3%)	0.003 (5.6%)	0.002 (9.4%)	0.002 (5.8%)	NA	0.001 (9.6%)
Total identified:	0.058 (97.4%)	0.055 (97.7%)	0.023 (94.2%)	0.028 (95.4%)	-	0.013 (95.3%)
Wheat hay (TRR, mg eq/kg)	0.160	0.208	0.063	0.062	0.028	0.064
Tetraniliprole (tetraniliprole)	0.109 (67.9%)	0.123 (59.2%)	0.024 (38.0%)	0.023 (37.1%)	0.015 (52.4%)	0.015 (24.1%)
Tetraniliprole-pyrazole-5-carboxylic acid	-	0.004 (1.9%)	-	0.002 (3.9%)	-	0.005 (7.1%)
Tetraniliprole-dihydroxy	-	0.003 (1.6%)	0.005 (8.1%)	0.004 (6.8%)	-	0.012 (18.6%)
Tetraniliprole-amide	0.006 (3.6%)	0.008 (3.8%)	0.003 (5.0%)	0.004 (6.3%)	-	0.004 (7.0%)
Tetraniliprole-desmethyl-amide-carboxylic acid	-	0.006 (3.1%)	0.007 (11.5%)	0.004 (5.8%)	-	0.013 (19.5%)
Tetraniliprole-carboxylic acid	0.004 (2.7%)	0.006 (2.96%)	0.002 (3.6%)	0.004 (6.7%)	0.005 (16.6%)	0.004 (6.4%)
Tetraniliprole-N-methyl-quinazolinone	0.023 (14%)	0.028 (13.4%)	0.005 (8.1%)	0.008 (12.9%)	0.003 (10.0%)	0.002 (2.9%)
Total identified:	0.141 (88.1%)	0.178 (85.9%)	0.047 (74.3%)	0.049 (79.4%)	0.022 (78.9%)	0.055 (85.7%)
Wheat straw (TRR, mg eq/kg)	0.116	0.256	0.067	0.104	0.035	0.110
Tetraniliprole (tetraniliprole)	0.077 (66.2%)	0.152 (59.5%)	0.038 (57.0%)	0.040 (38.2%)	0.014 (41.0%)	0.017 (15.7%)
Tetraniliprole-pyrazole-5-carboxylic acid	-	0.004 (1.9%)	-	0.004 (4.1%)	-	0.004 (3.8%)
Tetraniliprole-despyridyl-N-methyl-quinazolinone-pyrazole-3-carboxylic acid	-	0.002 (0.9%)	-	-	-	-
Tetraniliprole-dihydroxy	-	0.007 (2.6%)	-	0.011 (10.8%)	-	0.024 (21.6%)
Tetraniliprole-amide	0.005 (4.3%)	0.011 (4.4%)	-	0.005 (4.8%)	-	0.008 (7.1%)
Tetraniliprole-desmethyl-amide-carboxylic acid	-	0.010 (4.0%)	-	0.006 (6.2%)_	0.002 (4.3%)	0.021 (18.8%)
Tetraniliprole-carboxylic acid	0.007 (6.1%)	0.011 (4.2%)	0.010 (14.2%)	0.006 (5.6%)	0.004 (10.5%)	0.005 (4.9%)

Metabolite	TRR in mg eq/kg (%TRR)					
	30 day PBI		168 day PBI		286 day PBI	
	EnSa-14-0494	EnSa-14-0495	EnSa-14-0494	EnSa-14-0495	EnSa-14-0494	EnSa-14-0495
	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car
Tetraniliprole-N-methyl-quinazolinone	0.008 (7.2%)	0.017 (6.6%)	0.015 (2.4%)	0.014 (13.0%)	0.008 (21.6%)	0.002 (1.8%)
Total identified:	0.098 (83.8%)	0.215 (83.8%)	0.063 (93.7%)	0.086 (82.7%)	0.027 (77.4%)	0.081 (73.6%)
Swiss chard (immature) (TRR, mg eq/kg)	0.056	0.056	0.016	0.020	0.012	0.014
Tetraniliprole (tetraniliprole)	0.044 (78.5%)	0.039 (70.6%)	0.006 (39.2%)	0.007 (34.3%)	0.007 (55.6%)	0.003 (24.0%)
Tetraniliprole-despyridyl-N-methyl-quinazolinone-pyrazole-3-carboxylic acid	0.006 (11.4%)	0.008 (15.1%)	0.001 (9.3%)	0.002 (7.9%)	-	<0.001 (2.9%)
Tetraniliprole-dihydroxy	-	0.002 (4.1%)	0.002 (11.1%)	0.003 (14.0%)	0.001 (7.7%)	0.003 (18.9%)
Tetraniliprole-amide	-	-	-	0.001 (3.2%)	-	-
Tetraniliprole-desmethyl-amide-carboxylic acid	-	0.002 (4.0%)	-	0.004 (18.7%)	0.001 (5.8%)	0.004 (26.0%)
Tetraniliprole-carboxylic acid	0.004 (6.6%)	0.002 (3.0%)	0.004 (27.4%)	0.002 (11.7%)	0.002 (14.4%)	0.002 (10.7%)
Tetraniliprole-N-methyl-quinazolinone	0.001 (2.5%)	0.001 (2.6%)	0.002 (10.0%)	0.002 (7.9%)	0.001 (7.5%)	0.002 (14.4%)
Total identified:	0.056	0.055 (99.3%)	0.016 (97.0%)	0.019 (97.8%)	0.011 (95.5%)	0.014 (96.8%)
Swiss chard (mature) (TRR, mg eq/kg)	0.047	0.052	0.014	0.023	0.008 [a]	0.016
Tetraniliprole (tetraniliprole)	0.035 (74.3%)	0.032 (62.5%)	0.004 (29.6%)	0.006 (25.5%)	NA	0.001 (8.8%)
Tetraniliprole-pyrazole-5-carboxylic acid	-	-	-	-	NA	-
Tetraniliprole-despyridyl-N-methyl-quizazolinone-pyrazole-3-carboxylic acid	0.005 (11.6%)	0.008 (16.1%)	0.002 (12.0%)	0.002 (10.8%)	NA	0.001 (6.5%)
Tetraniliprole-dihydroxy	0.001 (1.9%)	0.002 (4.1%)	0.001 (9.6%)	0.004 (17.5%)	NA	0.005 (30.3%)
Tetraniliprole-amide	0.001 (1.8%)	-	-	-	NA	-
Tetraniliprole-desmethyl-amide-carboxylic acid	-	0.003 (5.6%)	0.001 (8.0%)	0.006 (25.1%)	NA	0.005 (31.1%)
Tetraniliprole-carboxylic acid	0.003 (6.2%)	0.003 (5.1%)	0.004 (28.0%)	0.003 (14.3%)	NA	0.002 (11.8%)
Tetraniliprole-N-methyl-quinazolinone	0.001 (2.9%)	0.003 (5.6%)	0.001 (9.0%)	0.001 (4.5%)	NA	0.001 (7.8%)
Total identified:	0.046 (98.8%)	0.051 (99.1%)	0.013 (96.2%)	0.022 (97.7%)	-	0.015 (96.3%)
Turnip leaves (TRR, mg/kg)	0.006	0.007	0.004 [a]	0.002 [a]	0.003 [a]	0.007 [a]
Tetraniliprole (tetraniliprole)	0.004 (69.4%)	0.003 (40.2%)	NA	NA	NA	NA
Tetraniilprole-pyrazole-5-carboxylic acid	-	<0.001 (6.5%)	NA	NA	NA	NA
Tetraniliprole-dihydroxy	-	0.001 (18.9%)	NA	NA	NA	NA
Tetraniliprole-desmethyl-amide-carboxylic	-	0.001 (21.3%)	NA	NA	NA	NA
Tetraniliprole-carboxylic acid	0.001 (22.7%)	<0.001 (7.3%)	NA	NA	NA	NA
Tetraniliprole-N-methyl-quinazolinone	-	<0.001	NA	NA	NA	NA

Metabolite	TRR in mg eq/kg (%TRR)					
	30 day PBI		168 day PBI		286 day PBI	
	EnSa-14-0494	EnSa-14-0495	EnSa-14-0494	EnSa-14-0495	EnSa-14-0494	EnSa-14-0495
	Phen-car	Pyr-car	Phen-car	Pyr-car	Phen-car	Pyr-car
		(3.2%)				
Total identified:	0.005 (92.1%)	0.007 (97.6%)	-	-	-	-
Turnip roots (TRR, mg/kg)	0.002	0.004	0.001 [a]	0.008 [a]	0.001 [a]	0.002 [a]
Tetraniliprole (tetraniliprole)	0.001 (54.4%)	0.002 (47.7%)	NA	NA	NA	NA
Tetraniliprole-desmethyl-amide-carboxylic	-	0.001 (17.5%)	NA	NA	NA	NA
Tetraniliprole-carboxylic acid	<0.001 (19.1%)	<0.001 (12.5%)	NA	NA	NA	NA
Tetraniliprole-N-methyl-quinazolinone	<0.001 (12.7%)	0.001 (16.0%)	NA	NA	NA	NA
Total identified:	0.002 (86.2%)	0.003 (93.7%)	-	-	-	-

Notes:
PBI: Plant back interval.
NA – Not Analysed.
[a] Samples were not subjected to further extraction and identification due to the low total residue values.
Wheat grain samples contained low (<0.01 mg eq/kg) TRR for all PBIs in both labels, therefore samples were not subjected to further extraction and identification.

Field rotational crop studies

According to the confined rotational crop studies, it is possible that rotational crops could take up residues of tetraniliprole from the soil. The Meeting received field rotational crop studies on onions, cucurbits, alfalfa, legumes, soya bean, wheat, barley, sorghum, rape seed, and sunflowers (23B).

Twelve field rotational crops studies were conducted in the growing seasons 2014 and/or 2015 to measure the magnitude of tetraniliprole residues in onions, cucurbits (melon, summer squash, cucumber), alfalfa, peas and beans (fresh with pods), peas and beans (fresh without pods), beans and peas (dried), wheat, barley , sorghum, rape seed, sunflower, wheat and soya bean.

In all studies, residues were determined in plants planted at a target 30-day plant back interval (actual PBI ranged from 25-31 days) following one broadcast application of tetraniliprole to bare soil. Applications were made at a nominal rate of 200 g ai/ha, with actual rates ranging from 170 (onion) to 220 g ai/ha (garden peas, shelled).

Samples of onion from 11 field trials were collected at normal commercial maturity (BBCH 48-49). Samples were stored for a maximum of 453 days (ca 15 months) (Lam, 2016, Report RAFVN039/M-562968-01-1).

Samples of melons, summer squashes, and cucumbers from twenty-seven field trials (Netzband & Beedle, 2016, Report RAFVP101-01/M-563500-02-1) were collected at commercial maturity (BBCH 71-89). Samples were stored for a maximum of 670 days (*ca* 24 months).

Samples of alfalfa forage and hay from a total of eleven field trials (Veal, 2016, Report RAFVP100/M-563135-01-1) were collected at commercial maturity (BBCH 59-61). Once cut, the alfalfa was allowed to re-grow and samples were again collected a second time at commercial maturity (BBCH

55-61). This was repeated once more for a third cutting (BBCH 51-62). Samples were stored for a maximum of 450 days (ca 15 months).

Samples from snow peas and snap beans, both with pods, from sixteen field trials (Dallstream, 2016, Report RAFVN033/M-560245-01-1) were collected at normal commercial maturity and stored frozen for a maximum of 654 days (ca 22 months).

Samples from lima beans and garden peas, both shelled, from 16 trials (Gould and Jerkins, 2016, Report RAFVN035-01/M-560729-02-1) were collected at normal commercial maturity (BBCH 75-89) and stored frozen for a maximum of 643 days (*ca* 21 months).

Samples from dried shelled peas and beans, forage and hay from 16 trials (Miller & Roberts, 2016, Report RAFVN037-01/M560950-02-1) were collected at maturity. Samples were stored frozen for a maximum of 633 days (*ca* 21 months).

Samples of wheat forage, hay, grain, and straw from twelve field trials (Veal & Jerkins, 2016, Report RAFVP086, M-558449-01-1) were collected at commercial maturity. Samples were stored for a maximum of 467 days (*ca* 15 months).

Samples of barley hay, straw and grain from nine field trials (Dallstream & Jerkins, 2016, Report RAFVP085, M-555094-01-1) were collected at commercial maturity. Samples were stored for a maximum of 414 days (*ca* 14 months).

Samples of sorghum forage, grain and fodder from seven field trials (Murphy & Jerkins, 2016a, Report RAFVN029, M-559018-01-1) were collected at commercial maturity. Samples were stored for a maximum of 405 days (*ca* 13 months).

Seed samples from oilseed rape, from seven field trials (Lam & Jerkins, 2016, Report RAFVP085, M-556294-01-1) were collected at commercial maturity (BBCH 89). Samples were stored for a maximum of 385 days (*ca* 13 months).

Samples of sunflower seed from six field trials (Murphy and Jerkins, 2016b, Report RAFVN030/M-558451-01-1) were collected at commercial maturity (BBCH 89). Samples were stored for a maximum of 323 days (*ca* 11 months).

The last field rotation crop study was designed slightly different. In this study six field trials (3 wheat and 3 soya bean) residues in/on wheat or soya bean planted at targeted 30-, 120-, and 365-day plant-back intervals (PBI), each following one application of tetraniliprole to either a target crop of potatoes or to bare soil (Krolski & Jerkins, 2016, Report RAFVP051, M-568415-01-1). Applications were made at an actual rate of 200–210 g ai/ha and were made in-furrow at planting of the primary crop (potatoes) or to bare soil at actual plant intervals ranging from 22–29 days, 108–119 days, and 334–365 days.

In trials that made applications to a primary crop of potatoes, the potatoes were grown to maturity or until the time of rotational crop planting. For the 4-month and 12-month PBI plots, potatoes were harvested after approximately 4 months or at maturity, respectively; for the 1-month PBI plots, the potato plants were disked or tilled into the plot. Samples of wheat forage, hay, grain and straw, soya bean forage, hay and seed were collected at commercial maturity. Samples were stored for a maximum of 604 days (*ca* 20 months).

Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methyl-quinazolinone using the validated analytical method 01414 (see analytical section). The acceptability of the method was confirmed with procedural recoveries within the acceptable range of 70–120 percent. The LOQ was 0.01 mg/kg for both analytes in all commodities.

Storage periods for the various crops were within the demonstrated stability periods for tetraniliprole and tetraniliprole-N-methyl-quinazolinone in high water content, high protein, high oil content and dry and high starch commodities (see section on storage stability).

Considering all twelve field rotational crop studies, the residues in human consumable commodities of the rotational crops with a 30 day PBI were all below the LOQ of <0.01 mg/kg. Quantified residues were observed in samples of animal feed commodities at plant-back intervals up to 365 days. The results are presented in Table 39 and Table 40.

Table 39 Residues of tetraniliprole in rotated feed commodities, using an application rate of 200 g ai/ha to bare soil and a plant back interval (PBI) of 30-days

Trial	PBI (days)	Application rate (g ai/ha)	Tetraniliprole (mg/kg)	Tetraniliprole-N-methyl-quinazolinone (mg/kg)
Dried peas (forage) (Miller & Roberts, 2016, M560950-02-1, Report RAFVN037-01)				
FV282-14RB, Parkdale, OR	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV283-14RB, Parkdale, OR	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV284-14RB, Ephrata, WA	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV285-14RB, Ephrata, WA	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV286-14RB, Ephrata, WA	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV287-14RB, Saskatoon, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV288-14RB, Rosthern, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Dried peas (hay) (Miller & Roberts, 2016, M560950-02-1, Report RAFVN037-01)				
FV282-14RB, Parkdale, OR	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV283-14RB, Parkdale, OR	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV284-14RB, Ephrata, WA	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV285-14RB, Ephrata, WA	30	200	<0.01, 0.01 (0.01)	<0.01, <0.01 (<0.01)
FV286-14RB, Ephrata, WA	30	200	<0.01, 0.01 (0.01)	<0.01, <0.01 (<0.01)
FV287-14RB, Saskatoon, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV288-14RB, Rosthern, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Dried beans (forage) (Miller & Roberts, 2016, M560950-02-1, Report RAFVN037-01)				
FV289-14RA, Carlyle, IL	25	200	0.013, 0.015 (0.014)	<0.01, <0.01 (<0.01)
FV290-14RA, Geneva, MN	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV291-14RA, Lenexa, KS	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV292-14RA, Grand Island, NE	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV293-14RA, Larned, KS	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV294-14RA, Jerome, ID	31	200	0.016, 0.017 (0.016)	<0.01, <0.01 (<0.01)
FV295-14RA, Kerman, CA	29	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV296-14RA, Parkdale, OR	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV297-14RA, Rosthern, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Dried beans (hay) (Miller & Roberts, 2016, M560950-02-1, Report RAFVN037-01)				
FV289-14RA, Carlyle, IL	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV290-14RA, Geneva, MN	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV291-14RA, Lenexa, KS	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV292-14RA, Grand Island, NE	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV293-14RA, Larned, KS	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV294-14RA, Jerome, ID	31	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV295-14RA, Kerman, CA	29	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV296-14RA, Parkdale, OR	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV297-14RA, Rosthern, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Wheat forage (Veal & Jerkins, 2016, M-558449-01-1, Report RAFVP086)				
FV039-14RA, Elko, SC	25	200	0.011, 0.010 (0.011)	<0.01, <0.01 (<0.01)
FV040-14RA, Greenville, MS	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV041-14RA, Rockwood, ON	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV042-14RA, York, NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV043-14RA, Geneva, MN	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)

Trial	PBI (days)	Application rate (g ai/ha)	Tetraniliprole (mg/kg)	Tetraniliprole-N-methyl-quinazolinone (mg/kg)
FV044-14RA, Hinton, OK	30	200	0.013, 0.012 (0.012)	<0.01, <0.01 (<0.01)
FV045-14RA, Taber, AB	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV046-14RA, Levelland, TX	30	210	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV047-14RA, Wall, TX	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV048-14RA, Ephrata, WA	29	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV049-14RA, Rosthern, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV050-14RA, Minto, MB	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Wheat hay (Veal & Jerkins, 2016, M-558449-01-1, Report RAFVP086)				
FV039-14RA, Elko, SC	25	200	0.024, 0.028 (0.026)	<0.01, <0.01 (<0.01)
FV040-14RA, Greenville, MS	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV041-14RA, Rockwood, ON	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV042-14RA, York, NE	28	200	0.013, 0.015 (0.014)	<0.01, <0.01 (<0.01)
FV043-14RA, Geneva, MN	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV044-14RA, Hinton, OK	30	200	0.035, 0.029 (0.032)	<0.01, <0.01 (<0.01)
FV045-14RA, Taber, AB	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV046-14RA, Levelland, TX	30	210	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV047-14RA, Wall, TX	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV048-14RA, Ephrata, WA	29	200	0.017, 0.024 (0.021)	<0.01, <0.01 (<0.01)
FV049-14RA, Rosthern, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV050-14RA, Minto, MB	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Wheat straw (Veal & Jerkins, 2016, M-558449-01-1, Report RAFVP086)				
FV039-14RA, Elko, SC	25	200	0.036, 0.035 (0.036)	0.013, 0.017 (0.015)
FV040-14RA, Greenville, MS	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV041-14RA, Rockwood, ON	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV042-14RA, York, NE	28	200	0.017, 0.016 (0.017)	0.019, 0.015 (0.017)
FV043-14RA, Geneva, MN	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV044-14RA, Hinton, OK	30	200	0.097, 0.088 (0.092)	0.038, 0.032 (0.035)
FV045-14RA, Taber, AB	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV046-14RA, Levelland, TX	30	210	0.010, 0.012 (0.011)	<0.01, <0.01 (<0.01)
FV047-14RA, Wall, TX	30	200	0.011, 0.012 (0.011)	<0.01, <0.01 (<0.01)
FV048-14RA, Ephrata, WA	29	200	0.021, 0.024 (0.022)	0.018, 0.020 (0.019)
FV049-14RA, Rosthern, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV050-14RA, Minto, MB	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Barley hay (Dallstream & Jerkins, 2016, M-555094-01-1, Report RAFVP085)				
FV019-14RA, Elko, SC	25	200	0.010, 0.011 (0.010)	<0.01, <0.01 (<0.01)
FV020-14RA, Northwood, ND	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV021-14RA, Rockwood, ON	30	210	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV022-14RA, Broderick, SK	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV023-14RA, Jerome, ID	27	200	0.015, 0.017 (0.016)	<0.01, <0.01 (<0.01)
FV024-14RA, Sanger, CA	29	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV025-14RA, Rupert, ID	27	200	0.021, 0.016 (0.018)	<0.01, <0.01 (<0.01)
FV026-14RA, Saskatoon, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV027-14RA, Josephburg, AB	31	190	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Barley straw (Dallstream & Jerkins, 2016, M-555094-01-1, Report RAFVP085)				
FV019-14RA, Elko, SC	25	200	0.018, 0.016 (0.017)	<0.01, <0.01 (<0.01)
FV020-14RA, Northwood, ND	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV021-14RA, Rockwood, ON	30	210	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV022-14RA, Broderick, SK	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV023-14RA, Jerome, ID	27	200	0.014, 0.015 (0.014)	0.011, 0.014 (0.013)
FV024-14RA, Sanger, CA	29	200	<0.010, 0.012 (0.011)	<0.01, <0.01 (<0.01)
FV025-14RA, Rupert, ID	27	200	0.012, 0.010 (0.011)	<0.01, <0.01 (<0.01)
FV026-14RA, Saskatoon, SK	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV027-14RA, Josephburg, AB	31	190	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Sorghum forage (Murphy & Jerkins, 2016, M-559018-01-1, Report RAFVN029)				

Trial	PBI (days)	Application rate (g ai/ha)	Tetraniliprole (mg/kg)	Tetraniliprole-N-methyl-quinazolinone (mg/kg)
FV012-14RA, Proctor, AR	29	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV013-14RA, Richland, IA	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV014-14RA, York, NE	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV015-14RA, Gardner, ND	31	190	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV016-14RA, Uvalde, TX	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV017-14RA, Levelland, TX	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV018-14RA, Wall, TX	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Sorghum fodder, dry (Murphy & Jerkins, 2016a, M-559018-01-1, Report RAFVN029)				
FV012-14RA, Proctor, AR	29	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV013-14RA, Richland, IA	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV014-14RA, York, NE	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV015-14RA, Gardner, ND	31	190	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV016-14RA, Uvalde, TX	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV017-14RA Levelland, TX	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV018-14RA, Wall, TX	26	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Alfalfa forage (1st cutting) (Veal, 2016, M-563135-01-1, Report RAFVP100)				
FV119-14RA, North Rose, NY	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV120-14RA, Athens, GA	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV121-14RA, Carlyle, IL	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV122-14RA, Lenexa, KS	27	210	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV123-14RA, Atlantic, IA	30	190	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV124-14RA, Richland, IA	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV125-14RA, York NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV126-14RA, Springfield, NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV127-14RA, Jerome, ID	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV128-14RA, Sanger, CA	30	200	0.013, 0.011 (0.012)	<0.01, <0.01 (<0.01)
FV129-14RA, Ephrata, WA	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Alfalfa forage (2nd cutting) (Veal, 2016, M-563135-01-1, Report RAFVP100)				
FV119-14RA, North Rose, NY	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV120-14RA, Athens, GA	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV121-14RA, Carlyle, IL	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV122-14RA, Lenexa, KS	27	210	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV123-14RA, Atlantic, IA	30	190	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV124-14RA, Richland, IA	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV125-14RA, York NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV126-14RA, Springfield, NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV127-14RA, Jerome, ID	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV128-14RA, Sanger, CA	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV129-14RA, Ephrata, WA	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Alfalfa forage (3rd cutting) (Veal, 2016, M-563135-01-1, Report RAFVP100)				
FV119-14RA, North Rose, NY	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV120-14RA, Athens, GA	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV121-14RA, Carlyle, IL	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV122-14RA, Lenexa, KS	27	210	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV123-14RA, Atlantic, IA	30	190	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV124-14RA, Richland, IA	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV125-14RA, York NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV126-14RA, Springfield, NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV127-14RA, Jerome, ID	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV128-14RA, Sanger, CA	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV129-14RA, Ephrata, WA	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Alfalfa hay (1st cutting) (Veal, 2016, M-563135-01-1, Report RAFVP100)				
FV119-14RA, North Rose, NY	30	200	0.011, 0.011 (0.011)	<0.01, <0.01 (<0.01)
FV120-14RA, Athens, GA	25	200	0.013, 0.013 (0.013)	<0.01, <0.01 (<0.01)

Trial	PBI (days)	Application rate (g ai/ha)	Tetraniliprole (mg/kg)	Tetraniliprole-N-methyl-quinazolinone (mg/kg)
FV121-14RA, Carlyle, IL	25	200	0.015, 0.013 (0.014)	<0.01, <0.01 (<0.01)
FV122-14RA, Lenexa, KS	27	210	0.010, 0.014 (0.012)	<0.01, <0.01 (<0.01)
FV123-14RA, Atlantic, IA	30	190	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV124-14RA, Richland, IA	25	200	0.020, 0.011 (0.015)	<0.01, <0.01 (<0.01)
FV125-14RA, York NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV126-14RA, Springfield, NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV127-14RA, Jerome, ID	27	200	0.013, 0.013 (0.013)	<0.01, <0.01 (<0.01)
FV128-14RA, Sanger, CA	30	200	0.051, 0.042 (0.046)	0.015, 0.012 (0.014)
FV129-14RA, Ephrata, WA	27	200	0.013, 0.015 (0.014)	<0.01, <0.01 (<0.01)
Alfalfa hay (2nd cutting) (Veal, 2016, M-563135-01-1, Report RAFVP100)				
FV119-14RA, North Rose, NY	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV120-14RA, Athens, GA	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV121-14RA, Carlyle, IL	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV122-14RA, Lenexa, KS	27	210	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV123-14RA, Atlantic, IA	30	190	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV124-14RA, Richland, IA	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV125-14RA, York NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV126-14RA, Springfield, NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV127-14RA, Jerome, ID	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV128-14RA, Sanger, CA	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV129-14RA, Ephrata, WA	27	200	0.012, 0.012 (0.012)	<0.01, <0.01 (<0.01)
Alfalfa hay (3rd cutting) (Veal, 2016, M-563135-01-1, Report RAFVP100)				
FV119-14RA, North Rose, NY	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV120-14RA, Athens, GA	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV121-14RA, Carlyle, IL	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV122-14RA, Lenexa, KS	27	210	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV123-14RA, Atlantic, IA	30	190	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV124-14RA, Richland, IA	25	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV125-14RA, York NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV126-14RA, Springfield, NE	28	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV127-14RA, Jerome, ID	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV128-14RA, Sanger, CA	30	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV129-14RA, Ephrata, WA	27	200	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)

Table 40 Residues of tetraniliprole in rotated feed commodities of wheat and soya bean, using an application rate of 200 g ai/ha to bare soil or in furrow at planting of potatoes as primary crop at anticipated plant back intervals (PBIs) of 30-days, 120 and 365 days.

Trial	Commodity	Application rate (g ai/ha)	Application method (actual PBI)	Tetraniliprole (mg/kg)	Tetraniliprole-N-methyl-quinazolinone (mg/kg)
Wheat (30 day nominal PBI) (Krolski & Jerkins, 2016, M-568415-01-1, Report RAFVP051)					
FV113-14RA Seven Springs, NC	Forage	202	Bare soil (27 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Grain			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Straw			<0.01, 0.011 (0.011)	0.010, <0.01 (0.010)
FV114-14RA Springfield, NE	Forage	201	Bare soil (22 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Grain			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Straw			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV115-14RA Ephrata, WA	Forage	207	Potatoes (29 days)	0.023, 0.030 (0.026)	<0.01, <0.01 (<0.01)
	Hay			0.011, 0.017 (0.014)	<0.01, <0.01 (<0.01)
	Grain			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)

Trial	Commodity	Application rate (g ai/ha)	Application method (actual PBI)	Tetraniliprole (mg/kg)	Tetraniliprole-N-methyl-quinazolinone (mg/kg)
	Straw			0.011, 0.010 (0.011)	<0.01, <0.01 (<0.01)
Wheat (120 day nominal PBI) (Krolski & Jerkins, 2016, M-568415-01-1, Report RAFVP051)					
FV113-14RA Seven Springs, NC	Forage	201	Bare soil (119 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Straw			0.014, <0.01 (0.012)	0.012, <0.01 (0.011)
FV114-14RA Springfield, NE	Forage	198	Bare soil (108 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Straw			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV115-14RA Ephrata, WA	Forage	203	Bare soil (119 days)	0.016, 0.016 (0.016)	<0.01, <0.01 (<0.01)
	Hay			0.023, 0.024 (0.024)	<0.01, <0.01 (<0.01)
	Straw			0.014, 0.012 (0.013)	0.010, <0.01 (0.010)
Wheat (365 day nominal PBI) [Krolski & Jerkins, 2016, M-568415-01-1, Report RAFVP051]					
FV113-14RA Seven Springs, NC	Forage	202	Bare soil (360 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Straw			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV114-14RA Springfield, NE	Forage	206	Potatoes (334 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Straw			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV115-14RA Ephrata, WA	Forage	206	Potatoes (365 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Straw			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Soya bean (30 day PBI) (Krolski & Jerkins, 2016, M-568415-01-1, Report RAFVP051)					
FV116-14RA Seven Springs, NC	Forage	201	Bare soil (27 days)	0.011, <0.01 (0.010)	<0.01, <0.01 (<0.01)
	Hay			0.020, 0.022 (0.021)	<0.01, <0.01 (<0.01)
	Seed			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV117-14RA Springfield, NE	Forage	204	Potatoes (25 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Seed			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV118-14RA Ephrata, WA	Forage	207	Potatoes (29 days)	0.020, 0.028 (0.024)	<0.01, <0.01 (<0.01)
	Hay			0.17, 0.13 (0.15)	0.019, 0.014 (0.017)
	Seed			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
Soya bean (120 day PBI) (Krolski an Jerkins, 2016, M-568415-01-1, Report RAFVP051)					
FV116-14RA Seven Springs, NC	Forage	201	Bare soil (118 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			0.011, 0.013 (0.012)	<0.01, <0.01 (<0.01)
FV117-14RA Springfield, NE	Forage	192	Bare soil (117 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV118-14RA Ephrata, WA	Forage	202	Bare soil (119 days)	0.038, 0.040 (0.039)	<0.01, <0.01 (<0.01)
	Hay			0.19, 0.14 (0.16)	0.026, 0.019 (0.023)
Soya bean (365 day PBI) (Krolski an Jerkins, 2016, M-568415-01-1, Report RAFVP051)					
FV116-14RA Seven Springs, NC	Forage	203	Bare soil (363 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV117-14RA Springfield, NE	Forage	201	Potatoes (348 days)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
	Hay			<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)
FV118-14RA Ephrata, WA	Forage	205	Potatoes (365 days)	0.056, 0.044 (0.050)	<0.01, <0.01 (<0.01)
	Hay			0.078, 0.089 (0.083)	0.011, 0.013 (0.012)

Overview of the metabolic pathway in rotational crops

In the confined rotational crops studies the following metabolic reactions were observed:

- hydroxylation of parent compound leading to tetraniliprole-dihydroxy,

- stepwise oxidation of the cyano group of parent compound leading via tetraniliprole-amide to tetraniliprole-carboxylic acid,
- demethylation of tetraniliprole-carboxylic acid leading to tetraniliprole-desmethylamide-carboxylic acid,
- intra-molecular condensation (cyclisation) of parent compound leading to tetraniliprole-N-methyl-quinazolinone, and
- cleavage of the pyridyl and tetrazolyl moiety and a subsequent oxidation forming tetraniliprole-despyridyl-N-methyl-quinazolinone-pyrazole-3-carboxylic acid.

The potential for uptake of residues from soil in practice seems to be negligible for human food commodities (all <0.01 mg/kg, grain, potatoes etc.) and low in livestock feed items. These metabolic pathways are depicted in Figure 3.

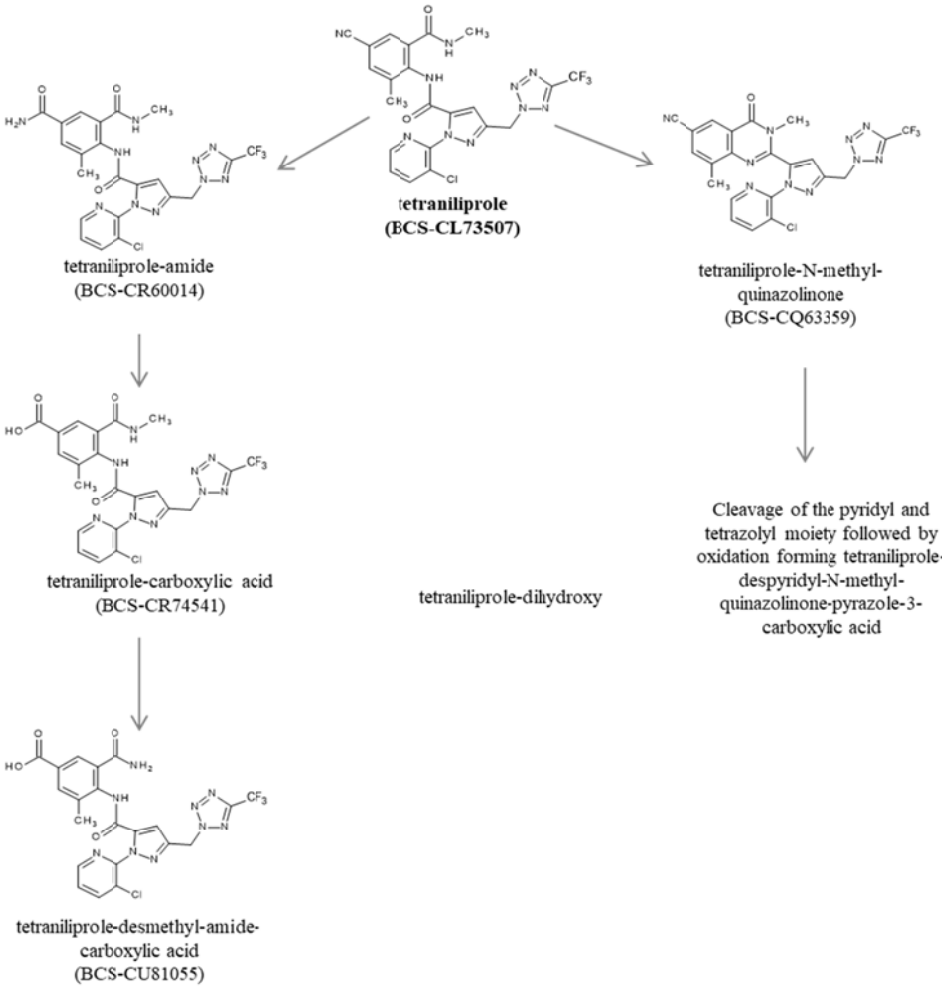
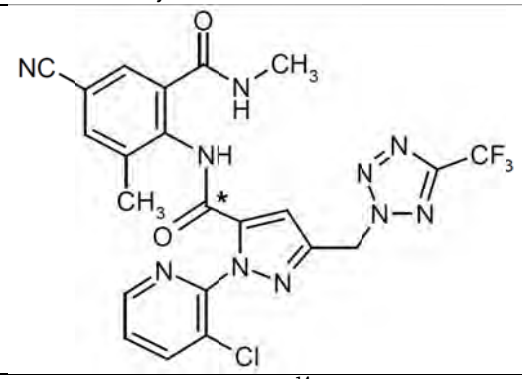
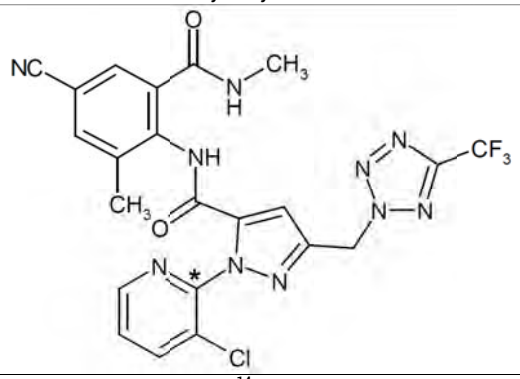
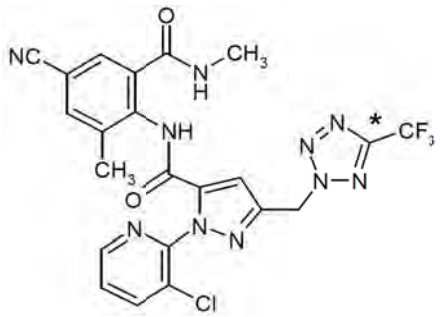


Figure 3 Metabolic pathway of tetraniliprole in rotational crops

Animal metabolism

The Meeting received information on the metabolic fate of tetraniliprole in ruminants (lactating goats) and poultry (laying hens). The metabolism in other mammalian animals was summarized and evaluated by the WHO panel of the JMPR in parallel in 2020.

For livestock metabolism, studies were conducted with either [pyrazole-carboxamide-^{14}C]-, [pyridinyl-2-^{14}C]-, or [tetrazolyl-^{14}C]-labelled tetraniliprole, as shown below.

Pyrazole-carboxamide label	Pyridinyl label
	
[pyrazole-carboxamide-^{14}C]-tetraniliprole	[pyridinyl-2-^{14}C]-tetraniliprole
Tetrazolyl label	
	
[tetrazolyl-^{14}C]-tetraniliprole	

Lactating goat

Pyrazole-carboxamide-label

The metabolism and excretion of [pyrazole-carboxamide-^{14}C]-tetraniliprole was investigated in the lactating goat (Bongartz et al, 2017, M-525616-02-1, Report EnSa-14-0579). The test compound was orally administered in gelatin capsules at a dose of *ca.* 1 mg/kg body weight. Based on the daily feed consumption, the dose level corresponded to 27.04 ppm (mg ai/kg dry feed/day). The goat, Weiße deutsche Edelziege (*Capra Hircus*), received five consecutive doses at 24-hour intervals in the morning after milking and was sacrificed *ca.* 5.5 hours after the last dose.

Throughout the experiment, the goat was housed in a metabolism cage, which permitted separate collection of urine and faeces. The goat was milked in the morning immediately prior to each administration, about eight hours later in the afternoon and *ca.* 1 hour before sacrifice. TRRs were determined in each milk sample and in dissected organs and tissues (muscle, fat, liver and kidney) at sacrifice. The total radioactivity (percent of total dose administered) were additionally determined in each urine and faeces sample. The radioactivity measurement in liquid samples was carried out by LSC. All solid samples were combusted and the radioactivity determined by LSC.

The overall recovery accounted for *ca.* 73 percent of the total applied dose/radioactivity (TAR). Up to the time of sacrifice, *ca*. 2.13 percent TAR was excreted with the urine and *ca.* 67.3 percent TAR with faeces. The daily renal and faecal excretion rates of radioactivity started shortly after the first dosing before plateauing at about 0.6 percent and 13 percent–20 percent TAR for urine and faeces, respectively. Radioactivity in the GI tract and cage washes were not investigated. Approximately 1.24 percent TAR was

secreted with the milk. At sacrifice, radioactive residues in the organs and tissues were *ca.* 2.36 percent TAR (0.42 percent TAR in liver, 0.01 percent TAR in kidney, 0.56 percent TAR in muscle, and 1.36 percent TAR in fat).

The TRR values in milk samples ranged from 0.16 mg eq/kg at 24 hours after the first administration to 0.51 mg eq/kg *ca*. 0.5 hours before sacrifice. The radioactive residues increased significantly during the eight-hour period after each administration followed by a small decrease measured prior to the delivery of the next dose, except for the period from 60 – 96 h, at which the plateau-level (0.42 mg eq/kg) was reached. Regarding organs and tissues, the TRR values amounted to 0.099 mg eq/kg for muscle (composite of round and loin muscle), 0.60 mg eq/kg for fat (composite of perirenal and omental fat), 1.0 mg eq/kg for liver and 0.25 mg/kg for kidney.

The majority of the residues in the milk, organs and tissues were efficiently (90.3–100 percent TRR) extracted three times with a mixture of acetonitrile/water (8/2), using 1 mL formic acid in the first extraction step. In case of fat, 150 mL n-heptane were added to every extraction step and subsequently separated from the aqueous phases. All n-heptane phases and aqueous phases were unified before further processing/ analysis. Solids after extraction of the liver were further extracted using two microwave assisted extractions with mixtures of acetonitrile/water (1/1) using microwave assisted solvent extraction. Formic acid was added to the last extraction step. An additional 9.7 percent TRR was released. Low amounts of radioactivity (≤0.7 percent TRR) remained in the PES of all commodities.

Most of the metabolites were isolated from urine and faeces by HPLC using the methods “ANTAM”, “antam1” and “MI9916”. They were identified in the isolated fractions by LC-MS and LC-MS/MS. Following structure elucidation, the identified metabolites were re-assigned in the profile by HPLC co-chromatography using the isolated fractions. Other metabolites were identified by HPLC co-chromatography with radiolabelled reference compounds taken from the rat ADME study: tetraniliprole-pyrazole-5-N-methylamide, tetraniliprole-hydroxypyridyl-glucuronide, tetraniliprole-desmethyl-amide, and tetraniliprole-hydroxy. Furthermore, the assignment and identification of parent compound and metabolites were achieved by comparison of HPLC metabolite profiles of the analysed samples among each other. Unknown metabolites were characterised based on their extraction and chromatographic behaviour.

Identification rates were very high and ranged between 86 and 96 percent of the TRR in milk, and edible organs and tissues. Parent tetraniliprole was the predominant residue in milk, muscle, liver and kidney and ranged from 55 to 71 percent TRR. For fat, 28 percent TRR was quantified as parent compound. Tetraniliprole-N-methyl-quinazolinone was the main metabolite in fat (67 percent TRR) and a major metabolite in muscle (27.9 percent TRR). A prominent metabolite was tetraniliprole-benzylalcohol (11 percent TRR in milk, 8.0 percent TRR in liver and 6.2 percent TRR in kidney). Metabolite tetraniliprole-hydroxy-N-methyl was found in milk (5.0 percent TRR), liver (8.9 percent TRR) and kidney (3.7 percent TRR).

All other minor metabolites, such as tetraniliprole-N-methyl-quinazolinone-pyrazole-3-carboxylic acid, tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid, tetraniliprole-N-methyl quinazolinone-benzylalcohol, tetraniliprole-desmethyl-amide and tetraniliprole-quinazolinone accounted for ≤2.6 percent TRR (≤0.007 mg eq/kg).

Pyrazole-carboxamide label specific metabolites were tetraniliprole-pyrazole-5-amide, tetraniliprole-pyrazole-5-N-methyl-amide and tetraniliprole-5-carboxylic acid. They were found at low amounts in milk and liver (≤1.8 percent TRR, ≤0.018 mg eq/kg). Several unknown metabolites were characterised by extraction and chromatographic behaviour. And since the exact position of the hydroxy group or the glucuronic acid group could not be determined, due to the general low amount of the

metabolites tetraniliprole-hydroxypyridyl-Gluc, tetraniliprole-N-methyl-quinazolinonehydroxy-Gluc, tetraniliprole-quinazolinone-hydroxy, and tetraniliprole- hydroxy, these metabolites were considered as characterised, only. The metabolic profile of tetraniliprole in milk and edible tissues is presented in Table 41.

Table 41 Distribution of parent compound and metabolites in the extracts of milk and edible tissues of goats following oral administration of [pyrazole-carboxamide-^{14}C]-tetraniliprole for 5 consecutive days at 27 ppm ai in feed.

Component / Sample	Milk (32-101h)		Muscle		Fat		Liver		Kidney	
TRR (mg eq/kg)	0.380		0.099		0.598		0.998		0.253	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Initial solvent extraction (3 ×) with acetonitrile/water 8:2										
Tetraniliprole	70	0.27	64.7	0.064	28	0.16	53.5	0.53	71	0.18
Tetraniliprole-pyrazole-5-carboxylic acid	0.3	0.001	-	-	-	-	1.8	0.018	-	-
Tetraniliprole-N-methyl-quinazolinone-pyrazole-3-carboxylic acid	-	-	-	-	-	-	0.9	0.009	-	-
Tetraniliprole-benzylalcohol	11	0.042	1.9	0.002	0.4	0.002	8.4	0.084	6.2	0.016
Tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid	0.3	0.001	-	-	-	-	0.8	0.008	2.6	0.007
Tetraniliprole-hydroxy-N-methyl	5.0	0.019	0.9	0.001	-	-	8.5	0.085	3.7	0.009
Tetraniliprole-desmethyl-amide	2.1	0.008	1.6	0.002	0.3	0.002	2.1	0.021	2.3	0.006
Tetraniliprole-quinazolinone	0.5	0.002	0.2	<0.001	0.2	0.001	0.4	0.004	0.3	0.001
Tetraniliprole-N-methyl-quinazolinone	1.9	0.007	28	0.028	66.8	0.40	2.2	0.022	5.0	0.013
Unknowns [b]	8.2 [c]	0.031 [c]	2.6 [d]	0.003 [d]	4.9 [e]	0.029 [e]	13.5 [f]	0.14 [f]	8.4 [g]	0.051 [g]
Total identified in the first extract series	91.1	0.35	97.3	0.096	95.1	0.57	76.9	0.77	91.1	0.23
Microwave assisted solvent extraction (2 ×) with acetonitrile/water (1:1) and microwave assistance										
Tetraniliprole (parent)	-	-	-	-	-	-	1.4	0.014	-	-
Tetraniliprole-pyrazole-5-amide	-	-	-	-	-	-	0.8	0.008	-	-
Tetraniliprole-pyrazole-5-N-methyl-amide	-	-	-	-	-	-	1.5	0.015	-	-
Tetraniliprole-pyrazole-5-carboxylic acid	-	-	-	-	-	-	1.8	0.018	-	-
Tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid	-	-	-	-	-	-	1.7	0.017	-	-
Tetraniliprole-hydroxy-N-methyl	-	-	-	-	-	-	0.4	0.004	-	-
Tetraniliprole-quinazolinone	-	-	-	-	-	-	0.3	0.003	-	-
Unknowns	-	-	-	-	-	-	1.7 [f]	0.017 [f]	-	-
Total identified after microwave assisted extraction	-	-	-	-	-	-	8.0	0.080	-	-
Total identified	91.1	0.35	97.1	0.096	95.1	0.57	84.8	0.85	91.1	0.23
Total characterised [h]	8.2	0.031	2.6	0.003	4.9	0.029	15	0.15	8.4	0.051
Total extracted	99.5	0.38	99.7	0.099	100	0.60	100	1.0	99.5	0.25
Post extracted solids	0.5	0.002	0.3	<0.001	<0.1	<0.001	<0.1	<0.001	0.7	0.002

Component / Sample	Milk (32-101h)		Muscle		Fat		Liver		Kidney	
TRR (mg eq/kg)	0.380		0.099		0.598		0.998		0.253	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total	100	0.38	100	0.099	100	0.60	100	1.0	100	0.25

Notes:

[a] Milk collected over 32-101 hours.

[b] Characterised by extraction and partition behaviour.

[c] Comprised of 8 separate regions, the largest of which contained 3.1% TRR, 0.012 mg eq/kg.

[d] Comprised of 3 separate regions, the largest of which contained 1.3% TRR, 0.001 mg eq/kg.

[e] Comprised of 5 separate regions, the largest of which contained 3.3% TRR, 0.020 mg eq/kg.

[f] The initial extract comprised of 14 separate regions, the largest of which contained 6.7% TRR, 0.067 mg eq/kg; in the microwave assisted extract 5 unknown regions were characterised, the largest of which contained 0.5% TRR, 0.05 mg eq/kg.

[g] Comprised of 5 separate regions, the largest of which contained 6.1% TRR, 0.015 mg eq/kg.

Pyridinyl-2-label

The metabolism and excretion of [pyridinyl-2-^{14}C]-tetraniliprole was investigated in the lactating goat (Bongartz *et al*., 2015a, M-525622-01-1, Report EnSa-14-1311). The test compound was orally administered in gelatin capsules at a dose of *ca.* 1 mg/kg body weight. Based on the daily feed consumption, the dose level corresponded to 20.62 ppm (mg ai/kg dry feed/day). The goat, Weiße deutsche Edelziege (*Capra Hircus*), received five consecutive doses at 24-hour intervals in the morning after milking and was sacrificed *ca.* 6 hours after the last dosing.

Throughout the experiment, the goat was housed in a metabolism cage, which permitted separate collection of urine and faeces. The goat was milked in the morning immediately prior to each administration, about eight hours later in the afternoon and *ca.* 20 min before sacrifice. TRRs were determined in each milk sample and in dissected organs and tissues (muscle, fat, liver and kidney) at sacrifice. The total radioactivity (percent TAR) was additionally determined in each urine and faeces sample. The radioactivity measurement in liquid samples was carried out by LSC. All solid samples were combusted and the radioactivity determined by LSC.

The overall recovery accounted for *ca.* 74 percent TAR. Up to the time of sacrifice*, ca.* 2.0 percent of the total dose was excreted with the urine and *ca.* 69 percent TAR with faeces. The daily renal and faecal excretion rates of the radioactivity started shortly after the first dosing before plateauing at about 0.5 percent and 14–18 percent TAR for urine and faeces, respectively. Radioactivity in the GI tract and cage washes were not investigated. Approximately 1.3 percent TAR was secreted with the milk. At sacrifice, radioactive residues in the organs and tissues were *ca*. 1.8 percent TAR (0.44 percent TAR in liver, 0.02 percent TAR in kidney, 0.49 percent TAR in total body muscle, 0.89 percent TAR in total bod fat).

The TRR values in milk samples ranged from 0.14 mg eq/kg at 24 hours to 0.33 mg/kg at 80 hours after the first administration. The radioactive residues increased significantly during the eight-hour period after each administration followed by a decrease prior to the delivery of the next dose. A residue plateau-level in milk of 0.28 mg/kg was reached at about day 4 after the first administration. Regarding organs and tissues, the TRR-values amounted to 0.086 mg eq/kg for muscle (composite of round and loin muscle), 0.39 mg eq/kg for fat (composite of perirenal and omental fat), 0.88 mg eq/kg for liver and 0.24 mg eq/kg for kidney.

The majority of the residues in the milk, organs and tissues were efficiently (91-100 percent TRR) extracted three times with a mixture of acetonitrile/water (8/2), using 1 mL formic acid in the first extraction step. Solids after extraction of the liver were further extracted two times with mixtures of

acetonitrile/water (1/1) using microwave assistance. Formic acid was added to the last extraction step. An additional 8.8 percent TRR was released. Negligible amounts of radioactivity (≤1.0 percent TRR) remained in the PES of all commodities.

Parent compound and metabolites were quantified in the initial and microwave assisted extracts by HPLC. Parent compound and metabolites were identified as in the goat study with the pyrazole-carboxamide label (Bongartz *et al.*, 2017, M-525616-02-1, Report EnSa-14-0579) by spectroscopic methods and co-chromatography with radiolabelled reference compounds. They were assigned to the profiles of the current study based on their retention times and the metabolite pattern in the corresponding extracts.

Identification rates were very high and ranged between 91.6 and 97.6 percent of the TRR in milk, and edible organs and tissues. Parent tetraniliprole was the main residue in milk, muscle, liver and kidney and ranged from 62–69 percent TRR. For fat, 24 percent TRR was quantified as parent compound. Tetraniliprole-N-methyl-quinazolinone was the main metabolite in fat and amounted to 72 percent TRR. Tetraniliprole-N-methyl-quinazolinone was also a major metabolite in milk, muscle and kidney and accounted for 11 percent, 28 percent and 14 percent TRR, respectively. Additionally, metabolite tetraniliprole-benzylalcohol amounted to 9.0 percent TRR in milk and 6.9 percent TRR in liver. Metabolites tetraniliprole-hydroxy-N-methyl and tetraniliprole-desmethyl-amide (both approx. 7 percent TRR) were also found in the liver.

All other minor metabolites, such as tetraniliprole-N-methyl-quinazolinone-pyrazole-3-carboxylic acid, tetraniliprole- pyridinyl-pyrazole-5-carboxylic acid, tetraniliprole-N-methylquinazolinone-benzylalcohol and tetraniliprole-quinazolinone accounted for ≤ 3.6 percent TRR (≤ 0.030 mg eq/kg). Pyridinyl label specific metabolites were not detected in milk or edible organs and tissues. Several unknown metabolites were characterised by extraction and chromatographic behaviour. And since the exact position of the hydroxy group or the glucuronic acid group could not be determined, due to the general low amount of the metabolites tetraniliprole-hydroxypyridyl-Gluc, tetraniliprole-N-methyl-quinazolinonehydroxy-Gluc, tetraniliprole-quinazolinone-hydroxy, and tetraniliprole- hydroxy, these metabolites were considered as characterised, only. The metabolic profile of tetraniliprole in milk and edible tissues is presented in Table 42.

Table 42 Distribution of parent compound and metabolites in the extracts of milk and edible tissues of goats following oral administration of [pyridinyl-2-^{14}C]-tetraniliprole for 5 consecutive days at 21 ppm in feed

Component / Sample	Milk [a]		Muscle		Fat		Liver		Kidney	
TRR (mg eq/kg)	0.243		0.086		0.387		0.878		0.243	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Initial solvent extraction (3 ×) with acetonitrile/water 8:2										
Tetraniliprole	64	0.16	66.4	0.057	24	0.094	59.3	0.52	69	0.17
Tetraniliprole-N-methyl-quinazolinone-pyrazole-3-carboxylic acid	-	-	-	-	-	-	0.3	0.002	-	-
Tetraniliprole-benzylalcohol	9.0	0.022	0.7	0.001	0.5	0.002	6.9	0.061	3.6	0.009
Tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid	0.6	0.002	-	-	-	-	0.5	0.005	1.5	0.004
Tetranilirole-hydroxy-N-methyl	3.5	0.008	0.9	0.001	-	-	6.9	0.061	3.0	0.007
Tetraniliprole-desmethyl-amide	3.5	0.008	0.8	0.001	0.2	0.001	5.3	0.046	2.6	0.006
Tetraniliporole-N-methyl-	0.8	0.002	-	-	0.3	0.001	0.2	0.002	-	-

Component / Sample	Milk [a]		Muscle		Fat		Liver		Kidney	
TRR (mg eq/kg)	0.243		0.086		0.387		0.878		0.243	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
quinazolinone-benzylalcohol										
Tetraniliprole-quinazolinone	1.1	0.003	0.3	<0.001	0.3	0.001	0.4	0.004	0.6	0.002
Tetraniliprole-N-methyl-quinazolinone	11	0.026	28.1	0.024	72	0.279	4.2	0.036	14	0.033
Unknowns [b]	5.7 [c]	0.014 [c]	1.9 [d]	0.002 [d]	2.3 [e]	0.009 [e]	6.9 [f]	0.061 [f]	5.3 [g]	0.013 [g]
Total identified	94	0.23	97	0.083	98	0.38	84	0.74	94	0.23
Microwave assisted solvent extraction (2 ×) with acetonitrile/water (1:1) and microwave assistance										
Tetraniliprole	-	-	-	-	-	-	2.3	0.020	-	-
Tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid	-	-	-	-	-	-	2.9	0.025	-	-
Tetraniliprole-desmethylamide	-	-	-	-	-	-	2.4	0.021	-	-
Total identified after microwave assisted extraction	-	-	-	-	-	-	7.6	0.067	-	-
Unknowns [b]	-	-	-	-	-	-	1.2 [f]	0.011 [f]	-	-
Total identified	94	0.23	97	0.083	98	0.38	92	0.80	94	0.23
Total characterised [h]	5.7	0.014	1.9	0.002	2.3	0.009	8.1	0.071	5.3	0.013
Not analysed [i]	-	-	-	-	-	-	0.3	0.003	-	-
Total extracted	99	0.24	99	0.085	99.9	0.39	100	0.88	99	0.24
Post extracted solids	0.7	0.002	0.8	0.001	0.1	<0.001	<0.1	<0.001	1.0	0.002
Total	100	0.24	100	0.086	100	0.39	100	0.88	100	0.24

Notes:

[a] Milk collected over 32-101 hours.

[b] Characterised by extraction and partition behaviour.

[c] Comprised of 3 separate regions, the largest of which contained 2.2% TRR, 0.005 mg eq/kg.

[d] Comprised of 4 separate regions, the largest of which contained 0.8% TRR 0.001 mg eq/kg.

[e] Comprised of 3 separate regions, the largest of which contained 1.8% TRR, 0.007 mg eq/kg.

[f] Comprised of 8 separate regions, the largest of which contained 3.4% TRR, 0.030 mg eq/kg in the first extraction series and of 1 region in the microwave assisted solvent extract.

[g] Comprised of 4 separate regions, the largest of which contained 3.6% TRR, 0.009 mg eq/kg.

[h] Total characterised is the sum of unknowns from both extraction series.

[i] Losses during concentration procedures or extracts not analysed.

Tetrazolyl-label

The metabolism and excretion of [tetrazolyl-^{14}C]-tetraniliprole was investigated in the lactating goat (Bongartz et al, 2015b, M-525625-02-1, Report EnSa-1321). The test compound was orally administered in gelatin capsules at a dose of *ca.* 1 mg/kg body weight. Based on the daily feed consumption, the dose level corresponded to 37.64 ppm (mg ai/kg dry feed/day). The goat, Weiße deutsche Edelziege (*Capra Hircus*), was given five consecutive doses at 24-hour intervals in the morning after milking and was sacrificed *ca.* 6 hours after the last dosing.

Throughout the experiment, the goat was housed in a metabolism cage, which permitted separate collection of urine and faeces. The goat was milked in the morning immediately prior to each administration, about eight hours later in the afternoon and *ca*. 40 min before sacrifice. TRRs were determined in each milk sample and in dissected organs and tissues (muscle, fat, liver and kidney) at sacrifice. The total radioactivity (percent TAR) was additionally determined in each urine and faeces

sample. The radioactivity measurement in liquid samples was carried out by LSC. All solid samples were combusted and the radioactivity determined by LSC.

The overall recovery accounted for *ca.* 68 percent TAR. Up to the time of sacrifice, *ca.* 3.3 percent TAR was excreted with the urine and ca. 60.9 percent TAR with faeces. The daily renal and faecal excretion rates of the radioactivity started shortly after the first dosing before plateauing at about 0.9 percent and 15-21 percent TAR for urine and faeces, respectively. Radioactivity in the GI tract and cage wash was not investigated. Approximately 1.1 percent TAR was secreted with the milk. At sacrifice, radioactive residues in the organs and tissues were *ca.* 2.4 percent TAR, with 0.57 percent TAR in liver, 0.02 percent TAR in kidney, 0.72 percent TAR in total body muscle, and 1.1 percent TAR in total body fat.

The TRR values in milk samples ranged from 0.12 mg eq/kg at 8 hours to 0.49 mg eq/kg at 72 hours after the first administration. At *ca.* 0.6 hours before sacrifice, the TRR value amounted to 0.43 mg eq/kg. The time course TRR-values of the evening and morning milk samples indicated a more or less steady increase until the fourth dosing, after which a residue plateau-level of 0.43 mg eq/kg was reached.

Regarding organs and tissues, the TRR values amounted to 0.12 mg eq/kg for muscle (composite of round and loin muscle), 0.47 mg eq/kg for fat (composite of perirenal and omental fat), 1.2 mg eq/kg for liver and 0.33 mg eq/kg for kidney.

The majority of the residues in the milk, organs and tissues were efficiently (89.4-100 percent TRR) extracted three times with a mixture of acetonitrile/water (8/2), using 1 mL formic acid in the first extraction step. In case of fat, 150 mL n-heptane were added to every extraction step and subsequently separated from the aqueous phases. All n-heptane phases and aqueous phases were unified before further processing/ analysis. Solids after extraction of the liver were further extracted two times with mixtures of acetonitrile/water (1/1) using microwave assistance. Formic acid was added to the last extraction step. An additional 10.6 percent TRR was released. Low amounts of radioactivity (≤1.0 percent TRR) remained in the PES of all commodities.

Parent compound and metabolites were quantified in the extracts by HPLC. Parent compound and metabolites were identified in the goat study with the pyrazole-carboxamide label (Bongartz et al, 2017, M-525616-02-1, Report EnSa-14-0579) by spectroscopic methods and co-chromatography with radiolabelled reference compounds. They were assigned to the profiles of the current study based on their retention times and the metabolite pattern in the corresponding extracts.

Identification rates were very high and ranged between 86 and 96 percent of the TRR in milk, and edible organs and tissues. In addition tetraniliprole-tetrazole was isolated from urine and identified by spectroscopic methods. Parent tetraniliprole was the main residue in milk (55.4 percent TRR, 0.23 mg/kg), muscle (68 percent TRR, 0.083 mg/kg), liver (53 percent TRR, 0.64 mg/kg, fat (30 percent TRR, 0.14 mg/kg) and kidney (59 percent TRR, 0.20 mg/kg). In fat, the metabolite tetraniliprole-N-methyl-quinazolinone was the main residue and amounted to 0.29 mg eq/kg (62 percent TRR). Tetraniliprole-N-methyl-quinazolinone was also a major metabolite in milk, muscle, kidney and liver and amounted to 0.056 mg eq/kg (13 percent TRR), 0.029 mg eq/kg (23 percent TRR), 0.044 mg eq/kg (13 percent TRR) and 0.067 mg eq/kg (5.6 percent TRR) respectively3.

An additional metabolite in milk, liver and kidney was tetraniliprole-benzylalcohol, and amounted to 0.045 mg eq/kg (11 percent TRR) in milk, 0.11 mg eq/kg (8.9 percent TRR) in liver and 0.020 mg eq/kg (6.0 percent TRR) in kidney. Metabolites tetraniliprole-hydroxy-N-methyl and tetraniliprole-desmethyl-amide were also detected in the liver (0.078 mg eq/kg (6.4 percent TRR) and 0.055 mg eq/kg (4.5 percent TRR), respectively).

All other metabolites, such as tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid, tetraniliprole-N-methyl-quinazolinone-benzylalcohol, tetraniliprole-N-methyl-quinazolinone hydroxypyridyl, tetraniliprole-quinazolinone and tetraniliprole-pyrazole-5-N-methyl-amide were minor and accounted for ≤3.3 percent TRR (≤ 0.040 mg eq/kg). Tetrazolyl label specific metabolites were not identified in milk or edible organs/tissues. Several unknown metabolites were characterised by extraction and chromatographic behaviour. And since the exact position of the hydroxy group or the glucuronic acid group could not be determined, due to the general low amount of the metabolites tetraniliprole-hydroxypyridyl-Gluc, tetraniliprole-N-methyl-quinazolinonehydroxy-Gluc, tetraniliprole-quinazolinone-hydroxy, and tetraniliprole- hydroxy, these metabolites were considered as characterised, only. The metabolic profile of tetraniliprole in milk and edible tissues is presented in Table 43.

Table 43 Distribution of parent compound and metabolites in the extracts of milk and edible tissues of goats following oral administration of [tetrazolyl-^{14}C]-tetraniliprole for 5 consecutive days at 38 ppm ai in feed

Component / Sample	Milk [a]		Muscle		Fat		Liver		Kidney	
TRR (mg/kg)	0.421		0.123		0.473		1.211		0.331	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Initial solvent extraction (3 ×) with acetonitrile/water 8:2										
Tetraniliprole	55	0.23	68	0.083	30	0.14	50.6	0.61	59.4	0.20
Tetraniliprole-pyrazole-5-N-methyl-amide	-	-	-	-	-	-	-	-	-	-
Tetraniliprole-benzylalcohol	11	0.045	1.8	0.002	1.2	0.006	7.9	0.10	6.0	0.020
Tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid	0.8	0.003	-	-	-	-	0.5	0.006	3.6	0.012
Tetraniliprole-hydroxy-N-methyl	3.7	0.016	1.3	0.002	-	-	6.4	0.078	2.3	0.007
Tetraniliprole-desmethyl-amide	3.1	0.013	1.8	0.002	0.5	0.002	3.7	0.045	3.9	0.013
Tetraniliprole-N-methyl-quinazolinone-benzylalcohol	1.0	0.004	-	-	-	-	-	-	-	-
Tetraniliprole-quinazolinone	3.1	0.013	-	-	-	-	2.2	0.026	1.6	0.005
Tetraniliprole-N-methyl-quinazolinone	13	0.056	23	0.029	62	0.29	5.6	0.067	13.4	0.044
Unknowns [b]	8.5 [c]	0.036 [c]	2.8 [d]	0.003 [d]	6.6 [e]	0.031 [e]	12.6 [f]	0.15 [f]	8.8 [g]	0.029 [g]
Identified	91.1	0.38	96.2	0.12	92.9	0.44	76.8	0.93	90.2	0.30
Microwave assisted extraction (2 ×) with acetonitrile/water (1:1)										
Tetraniliprole	-	-	-	-	-	-	2.4	0.030	-	-
Tetraniliprole-pyrazole-5-N-methyl-amide	-	-	-	-	-	-	1.2	0.014	-	-
Tetraniliprole-benzylalcohol	-	-	-	-	-	-	1.0	0.012	-	-
Tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid	-	-	-	-	-	-	2.8	0.034	-	-
Tetraniliprole-desmethyl-amide	-	-	-	-	-	-	0.8	0.010	-	-
Tetraniliprole-quinazolinone	-	-	-	-	-	-	0.6	0.007	-	-
Unknowns	-	-	-	-	-	-	1.7 [f]	0.021 [f]	-	-
Identified after microwave assisted extraction	-	-	-	-	-	-	8.8	0.11	-	-
Total identified	91.1	0.38	96.2	0.12	92.9	0.44	85.7	1.04	90.2	0.30
Total characterised [h]	8.5	0.036	2.8	0.003	6.6	0.03	14	0.17	8.8	0.029
Total extracted	99.6	0.42	99	0.12	99.5	0.47	100	1.2	99.0	0.33
Post Extraction Solid (PES)	0.4	0.002	0.9	0.001	<0.1	<0.001	<0.1	<0.001	1.0	0.003
Not analysed [i]	-	-	-	-	0.5	0.002	-	-	-	-
Total	100	0.421	100	0.12	100	0.47	100	1.2	100	0.33

Notes:

[a] Milk collected over 32-101 hours.

[b] Characterised by extraction and partition behaviour.

[c] Comprised of 5 separate regions, the largest of which contained 3.1% TRR, 0.013 mg eq/kg.

[d] Comprised of 2 separate regions, the largest of which contained 1.8% TRR, 0.002 mg eq/kg.

[e] Comprised of 1 single region.

[f] Comprised of 13 separate regions, the largest of which contained 4.3% TRR, 0.052 mg eq/kg; the microwave assisted extract contained 2 unknown regions, containing 1.0 and 0.7% TRR, 0.012 and 0.009 mg eq/kg, respectively.
[g] Comprised of 3 separate regions, the largest of which contained 5.7% TRR, 0.019 mg eq/kg.
[h] Total characterised is the sum of unknowns from both extraction series.
[i] Losses during concentration procedures or extracts not analysed.

Laying hens

Pyrazole-carboxamide-label

The metabolism and excretion of [pyrazole-carboxamide-^{14}C]-tetraniliprole was investigated in laying hens (Bongartz, 2017, M-535432-02-1, Report EnSA-5-0411). The test compound was orally administered to six hens as an aqueous 0.5 percent tragacanth suspension by gavage using a syringe at a dose of *ca.* 1 mg/kg body weight. Based on the daily feed consumption, the dose level corresponded to *ca.* 18.64 ppm (mg ai/kg dry feed/day). The hens received 14 consecutive doses at 24-hour intervals in the morning and were sacrificed *ca.* 6 hours after the last dose.

The eggs were collected once daily and before sacrifice. TRR were determined in each egg (mixed sample from egg white and yolk) and in dissected organs and tissues (muscle, fat, liver, kidney, skin and eggs from ovary/ oviduct) at sacrifice. The total radioactivity (percent TAR) was additionally determined in each excreta sample. The radioactivity measurement in liquid samples was carried out by LSC. All solid samples were combusted and the radioactivity determined by LSC.

The overall recovery amounted to *ca.* 93 percent TAR. At sacrifice, radioactive residues in the organs and tissues were *ca.* 0.2 percent TAR. Up to the time of sacrifice, *ca.* 92 percent TAR was excreted. After the third administration the daily excretion rate plateaued at 6-7 percent TAR within 24 hours. GI tract and cage wash was not investigated. An average amount of *ca* 0.2 percent TAR was measured in the eggs.

The TRR-values in eggs ranged from 0.005 mg eq/kg at day one to 0.091 mg eq/kg at sacrifice. Following a linear increase a residue plateau-level of 0.088 mg eq/kg was reached approx. at day nine after the first administration. Regarding organs and tissues, the TRR-values amounted to 0.48 m eq/kg in liver, 0.098 mg eq/kg in kidney, 0.046 mg eq/kg in subcutaneous fat, 0.035 mg eq/kg in skin and 0.017 mg eq/kg in skeletal muscle.

Aliquots from eggs, muscle, fat, liver and excreta were extracted three times with a mixture of acetonitrile/ water (8/2). A volume of 1 mL formic acid was added to the first extraction step. In case of fat, 100 mL n-heptane were added to every extraction step and subsequently separated from the aqueous phases. All n-heptane phases and aqueous phases were unified before further processing/ analysis. Solids after initial extraction of eggs and liver were further extracted two times with mixtures of acetonitrile/water (1/1) using microwave assistance (5 min to 120 °C, 20 min at 120 °C). Formic acid was added to the last extraction step. The combined extracts were concentrated by rotary evaporation. Afterwards the distribution and quantities of parent compound and metabolites were determined by HPLC with the profiling method “ANTAM”, except for the microwave assisted extract from eggs, which, due to its very low amount of radioactivity (0.010 mg eq/kg), was further characterised by partition against ethyl acetate and radioactivity measurement of the resulting aqueous and organic phases by LSC.

The majority of the residues in the eggs as well as organs and tissues were efficiently extracted (88.6 to 100 percent TRR). Final PES for all commodities was ≤11.4 percent TRR, 0.002 mg eq/kg. The distribution of radioactivity in eggs and edible tissues is presented in Table 44.

Table 44 Distribution of radioactivity in the extracts of eggs and edible tissues of hens following oral dosing with [pyrazole-carboxamide -^{14}C]-tetraniliprole for 14 consecutive days at 19 ppm in feed

Sample	Eggs [a]		Muscle		Fat		Liver	
TRR (mg/kg)	0.084		0.017		0.046		0.485	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Initial solvent extract	88.1	0.074	88.6	0.015	99.7	0.046	58.7	0.285
Microwave assisted solvent extract	11.9	0.010	-	-	-	-	41.3	0.200
Total extracted	100	0.084	88.6	0.015	99.7	0.046	100	0.485
PES	<0.1	<0.001	11.4	0.002	0.3	<0.001	<0.1	<0.001
Accountability	100	0.084	100	0.017	100	0.046	100	0.485

Notes:
[a] Day 6–13.25.

Parent compound and metabolites were quantified in the extracts by HPLC-chromatography based on the profiling method ANTAM. Metabolites in the extracts were assigned to each other by comparison of the metabolite profiles and their retention times. Parent compound and metabolites were isolated from the extract of eggs (day 6–13.25) and the extract of liver and identified in the isolated fractions either by spectroscopic methods or by HPLC co-chromatography with radiolabelled reference compounds taken from the ADME study with rats using the profiling method "ANTAM". Reference compounds were: tetraniliprole-pyrazole-5-N-methylamide-hydroxy, tetraniliprole-pyrazole-5-carboxylic acid, tetraniliprole-benzylalcohol-Gluc, tetraniliprole-dihydroxy, and tetraniliprole-despiridyl, tetraniliprole-benzylalcohol, and tetraniliprole-hydroxy-N-methyl. Additionally, tetraniliprole-despyridyl and tetraniliprole-pyrazole-5-carboxylic acid were identified by TLC analysis using the corresponding radiolabelled reference compounds.

Parent tetraniliprole was present in eggs, muscle and fat and amounted to 0.008 mg/kg (10 percent TRR) for eggs, 0.002 mg/kg (10 percent TRR) for muscle and to 0.012 mg/kg (26 percent TRR) for fat. Parent compound was also detected in liver and amounted to 0.023 mg/kg (4.8 percent TRR). Metabolite tetraniliprole-despyridyl-N-methyl-quinazolinone was the major compound in eggs, fat and liver and amounted to 0.030 mg eq/kg (36 percent TRR) for eggs, 0.029 mg eq/kg (63 percent TRR) for fat and 0.060 mg eq/kg (12 percent TRR) for liver, while the metabolite amounted only to 0.002 mg eq/kg (8.6 percent TRR) in muscle. In muscle, the metabolite tetraniliprole -pyrazole-5-N-methyl-amide was the main residue and amounted to 0.007 mg eq/kg (40 percent TRR). Tetraniliprole -pyrazole-5-N-methyl-amide was also a prominent portion in liver (0.022 mg eq/kg; 4.6 percent TRR).

Another prominent metabolite was tetraniliprole-pyrazole-5-amide in muscle (0.002 mg eq/kg; 13 percent TRR). In liver, the metabolites were tetraniliprole-pyrazole-5-N-methyl-amide-hydroxy (0.016 mg eq/kg; 3.2 percent TRR), tetraniliprole-pyrazole-5-carboxylic acid (0.037 mg eq/kg; 7.6 percent TRR), tetraniliprole-benzylalcohol-Gluc (0.027 mg eq/kg; 5.6 percent TRR), tetraniliprole-despyridyl (0.042 mg eq/kg; 8.6 percent TRR), tetraniliprole-benzylalcohol (0.024 mg eq/kg; 5.0 percent TRR), tetraniliprole-despyridyl-N-methylquinazolinone-hydroxy and tetraniliprole-despyridyl-hydroxy (0.045 mg eq/kg; 9.3 percent TRR (mixture of both metabolites)), tetraniliprole-hydroxy-N-methyl (0.019 mg eq/kg; 3.8 percent TRR) and tetraniliprole-despyridyl-quinazolinone (0.034 mg eq/kg; 7.0 percent TRR).

All other identified metabolites, such as tetraniliprole-dihydroxy and tetraniliprole-quinazolinone accounted for ≤6.3 percent TRR, only. The metabolic profile of tetraniliprole in eggs and the edible tissues of hens is presented in Table 45.

Table 45 Distribution of parent compound and metabolites in the extracts of eggs and edible tissues of hens following oral administration of [pyrazole-carboxamide-^{14}C]-tetraniliprole for 14 consecutive days at 19 ppm ai in feed

Component / Sample	Eggs [a]		Muscle		Fat		Liver	
TRR (mg/kg)	0.084		0.017		0.046		0.485	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Initial solvent extraction (3 ×) with acetonitrile/water 8:2 + formic acid in the first extraction step								
Extracted	88.1	0.074	88.6	0.015	99.7	0.046	58.7	0.285
Tetraniliprole (parent compound)	10	0.008	10	0.002	26	0.012	4.8	0.023
T-pyrazole-5-N-methyl-amide-hydroxy	0.9	0.001	5.4	0.001	-	-	3.2	0.016
T-pyrazole-5-amide	2.9	0.002	13	0.002	-	-	1.0	0.005
T-pyrazole-5-N-methyl-amide	5.5	0.005	40	0.007	4.5	0.002	4.6	0.022
T-pyrazole-5-carboxylic acid	-	-	-	-	-	-	7.6	0.037
T-benzylalcohol-Gluc	1.3	0.001	-	-	-	-	5.6	0.027
T-dihydroxy	1.8	0.002	2.2	<0.001	-	-	1.4	0.007
T-despyridyl	2.0	0.002	9.1	0.002	3.0	0.001	8.6	0.042
T-benzylalcohol	0.8	0.001	-	-	-	-	5.0	0.024
T-despyridyl-N-methyl-quinazolinone-hydroxy and despyridyl-hydroxy	2.2	0.002	-	-	1.1	<0.001	9.3	0.045
T-hydroxy-N-methyl	2.3	0.002	-	-	-	-	3.8	0.019
T-despyridyl-quinazolinone	4.2	0.004	-	-	1.0	<0.001	7.0	0.034
T-despyridyl-N-methyl-quinazolinone	36	0.030	8.6	0.001	63	0.029	12	0.060
T-N-methyl-quinazolinone	6.3	0.005	-	-	-	-	-	-
Unknowns [b]	12 [c]	0.010 [c]	-	-	1.1 [d]	<0.001 [d]	21 [e]	0.10 [e]
Organic phase (n-heptane)	-	-	-	-	<0.1	<0.001	-	-
Identified	76	0.064	89	0.015	99	0.045	38	0.18
Microwave assisted solvent extraction (2 ×) with acetonitrile/water (1:1) + formic acid in last extraction step								
Extracted	11.9	0.010	-	-	-	-	41.3	0.20
- aqueous phase	5.6	0.005	-	-	-	-	-	-
- organic phase	6.3	0.005	-	-	-	-	-	-
T-pyrazole-5-N-methylamide-hydroxy	-	-	-	-	-	-	2.7	0.013
T-pyrazole-5-N-methylamide	-	-	-	-	-	-	2.1	0.010
T-pyrazole-5-carboxylic acid	-	-	-	-	-	-	6.9	0.034
T-benzylalcohol-Gluc	-	-	-	-	-	-	2.4	0.012
T-despyridyl	-	-	-	-	-	-	3.3	0.016
T-despyridyl-N-methylquinazolinone-hydroxy and T-despyridyl-hydroxy	-	-	-	-	-	-	5.7	0.028
T-despyridyl-quinazolinone	-	-	-	-	-	-	5.2	0.025
T-despyridyl-N-methylquinazolinone	-	-	-	-	-	-	7.9	0.039
Unknowns [b]	-	-	-	-	-	-	5.0 [e]	0.024 [e]
Identified	-	-	-	-	-	-	36.2	0.18
Characterised	11.9	0.010	-	-	-	-	5.0	0.024
Total identified	76	0.064	89	0.015	99	0.045	74.2	0.36
Total characterised [f]	24	0.020	-	-	1.1	<0.001	26	0.12
Total extracted	100	0.084	88.6	0.015	99.7	0.046	100	0.48
PES	<0.1	<0.001	11.4	0.002	0.3	<0.001	<0.1	<0.001
Total	100	0.084	100	0.017	100	0.046	100	0.048

Notes:

T= Tetraniliprole.

[a] Eggs collected at day 6-13.25.

[b] Characterised by extraction and partition behaviour.

[c] Comprised of 12 separate regions, the largest of which contained 6.3% TRR, 0.005 mg/kg.
[d] Comprised of one single region.
[e] Comprised of 17 separate regions, the largest of which contained 5.1% TRR, 0.025 mg/kg; In the microwave assisted extract the unknowns represented only 1 region.
[f] For eggs: sum of aqueous and organic phase and first series of extractions; for liver: sum of unknowns from both extraction series.

Pyridinyl-2-label

The metabolism of [pyridinyl-2-^{14}C]-tetraniliprole was investigated in laying hens (Bongartz *et al*., 2015c, M-539048-01-1, Report M1854600-4). The test compound was orally administered to six hens ("LB Lohmann Brown") as aqueous 0.5 percent Tragacanth suspension by gavage using a syringe at a dose of *ca.* 1 mg/kg body weight. Based on the daily feed consumption, the dose level corresponded to *ca.* 17.94 ppm (mg ai /kg dry feed/day). The hens were given 14 consecutive doses at 24-hour intervals in the morning and were sacrificed *ca*. 6 hours after the last dosing.

The hens were housed in metabolism cages, which permitted separate collection of eggs and excreta. The eggs were collected once daily and before sacrifice. TRRs were determined in each egg (mixed sample from egg white and yolk) and in dissected organs and tissues (muscle, fat, liver, kidney, skin and eggs from ovary/ oviduct) at sacrifice. The total radioactivity (percent TAR) was additionally determined in each excreta sample. The radioactivity measurement in liquid samples was carried out by LSC. All solid samples were combusted and the radioactivity determined by LSC.

The overall recovery amounted to *ca.* 93 percent TAR. Up to the time of sacrifice, the excretion products accounted for *ca.* 92 percent TAR. The daily excretion rate plateaued after the third administration at 6–8 percent TAR within 24 hours. Radioactivity in GI tract and cage wash was not investigated. An average amount of *ca*. 0.2 percent TAR was measured in the eggs. At sacrifice, the test compound-related residues in the organs and tissues dissected from the bodies were calculated or estimated to be about 0.3 percent TAR

The TRR-values in eggs ranged from 0.006 mg eq/kg at day one to 0.10 mg eq/kg at sacrifice. Following a linear increase, a residue plateau-level of 0.084 mg eq/kg was reached approx. at day eight after the first administration. Regarding organs and tissues, the TRR-values amounted to 0.73 mg eq/kg in liver, 0.33 mg eq/kg in kidney, 0.028 mg eq/kg in subcutaneous fat, 0.047 mg eq/kg in the skin and 0.025 mg eq/kg in skeletal muscle.

Aliquots from eggs, muscle, liver and excreta were extracted three times with a mixture of acetonitrile/ water (8/2). Fat was extracted three times with methanol. In all cases, 1 mL formic acid was added to the first extraction step. Solids and extracts were separated after each extraction step by filtration. In case of fat, only a liquid fat phase remained. Solids after extraction of eggs, muscle and liver were additionally extracted two times with mixtures of acetonitrile/water (1/1) using microwave assistance (5 min to 120 °C, 20 min at 120 °C). Formic acid was added to the last extraction step. The combined extracts were concentrated by rotary evaporation. Afterwards, the distribution and quantities of parent compound and metabolites were determined by HPLC with the profiling method "ANTAM".

After extraction of fat, a liquid phase remained. The liquid fat phase and the microwave assisted extract from muscle were not further analysed due to low radioactivity (0.004 mg eq/kg for fat and 0.014 mg eq/kg for muscle). The distribution of radioactivity in eggs and edible tissues is presented in Table 46.

Table 46 Distribution of radioactivity in the extracts of eggs and edible tissues of hens following oral dosing with [pyridinyl-2 -^{14}C]-tetraniliprole for 14 consecutive days at 18 ppm ai in feed

Sample	Eggs [a]		Muscle		Fat		Liver	
TRR (mg/kg)	0.084		0.025		0.028		0.734	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Initial solvent extract	54.3	0.046	43.9	0.011	85.5	0.024	33.8	0.248
Microwave assisted extract	45.7	0.038	56.1	0.014	-	-	66.2	0.486
Liquid phase (fat only)	-	-	-	-	14.5	0.004	-	-
Total extracted	100	0.084	100	0.025	100	0.028	100	0.734
PES	<0.1	<0.001	<0.1	<0.001	<0.1	<0.001	<0.1	<0.001
Accountability	100	0.084	100	0.025	100	0.028	100	0.734

Notes:
[a] Day 6–13.25.

Parent compound and metabolites were quantified in the extracts by HPLC based on the profiling method ANTAM. Parent compound and metabolites were assigned in the profiles of the current study by comparison with the profiles analysed in the study with the pyrazole-carboxamide label (Bongartz, 2017, M-535432-02-1, Report EnSA-5-0411). Unknown metabolites were characterised based on their extraction and chromatographic behaviour. One metabolite was isolated from the extract of liver and identified in the isolated fraction by LC-MS/MS.

Parent tetraniliprole was the main residue in eggs, muscle and fat and amounted to 0.012 mg/kg (14 percent TRR) for eggs, 0.001 mg/kg (3.7 percent TRR) for muscle, 0.015 mg/kg (55 percent TRR) for fat, and 0.012 mg/kg (1.6 percent TRR) for liver. The main compound in liver was metabolite tetraniliprole-benzylalcohol-Gluc, which amounted to 0.047 mg eq/kg (6.5 percent TRR) and was not detected in eggs, muscle or fat. Other prominent metabolites in liver were tetraniliprole-benzylalcohol (0.024 mg eq/kg; 3.3 percent TRR), tetraniliprole-deschloro-desmethyl-amide (0.033 mg eq/kg; 4.5 percent TRR) and tetraniliprole-hydroxy-N-methyl (0.013 mg eq/kg; 1.7 percent TRR). Tetraniliprole-dihydroxy was a prominent metabolite in fat (0.004 mg eq/kg; 15 percent TRR). A minor metabolite was tetraniliprole-N-methyl-quinazolinone and accounted for 7.4 percent TRR (0.006 mg eq/kg) in eggs.

The main part of identified metabolites in the corresponding laying hen studies with the pyrazole-carboxamide label and tetrazolyl label consisted of metabolites formed after cleavage of the pyridinyl moiety. The corresponding pyridine label specific metabolites could not be identified in the current study. Therefore, it can be assumed that the significant amount of characterised radioactivity in the current study can be assigned to metabolites originating from the cleavage of the pyridinyl moiety most likely followed by degradation and/or binding to sample matrix, resulting in a large number of unknown metabolites. For example, a large portion of these unknown metabolites were found in the polar HPLC region of eggs and liver after TLC subquantification. There were no metabolites detected in the microwave assisted solvent extract of muscle (0.014 mg eq/kg, 56 percent TRR) by HPLC analysis, due to the low radioactivity in the sample and the high number of metabolites, as demonstrated in liver. The metabolic profile of tetraniliprole in eggs and edible tissues is presented in Table 47.

Table 47 Distribution of parent compound and metabolites in the extracts of eggs and edible tissues of hens following oral administration of [pyridinyl-2-^{14}C]-tetraniliprole for 14 consecutive days at 18 ppm ai in feed

Component / Sample	Eggs [a]		Muscle		Fat		Liver	
TRR (mg/kg)	0.084		0.025		0.028		0.734	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Initial solvent extraction (3 ×) with acetonitrile/water (8:2) + formic acid in the first extraction step								
Extracted	54.3	0.046	43.9	0.011	85.5	0.024	33.8	0.25
Tetraniliprole (parent compound)	13.8	0.012	3.7	0.001	54.6	0.015	1.6	0.012
T-benzylalcohol-Gluc	-	-	-	-	-	-	2.5	0.018
T-dihydroxy	3.2	0.003	1.5	<0.001	14.9	0.004	-	-
T-benzylalcohol	1.5	0.001	2.4	0.001	-	-	3.3	0.024
T-deschloro-desmethyl-amide	1.7	0.001	1.9	<0.001	4.0	0.001	1.3	0.009
T-hydroxy-N-methyl	3.6	0.003	1.6	<0.001	-	-	1.7	0.013
T-N-methyl-quinazolinone	7.4	0.006	-	-	-	-	-	-
Unknowns [b]	24.9 [c]	0.021 [c]	32.7 [d]	0.008 [d]	12 [e]	0.003 [e]	23.5 [f]	0.17 [f]
Identified	29.4	0.025	11.2	0.003	73.5	0.021	10.4	0.076
Liquid phase	-	-	-	-	14.5	0.004	-	-
Microwave assisted solvent extraction (2 ×) with acetonitrile/water (1:1) + formic acid in last extraction step								
Extracted	45.7	0.038	56.1	0.014	-	-	66.2	0.49
T-benzylalcohol-Gluc	-	-	-	-	-	-	2.6	0.019
T-deschloro-desmethyl-amide							1.9	0.014
Unknowns [b]	45.7 [c]	0.038 [c]	-	-	-	-	59.0 [f]	0.43 [f]
Identified	-	-	-	-	-	-	7.2	0.053
Total identified	29.4	0.025	11.2	0.003	73.5	0.021	17.6	0.13
Total characterised [g]	68.9	0.058	32.7	0.008	12.0	0.003	82.4	0.60
Microwave assisted extract (not analysed)	-	-	56.1	0.014	-	-	-	-
Solids remaining	<0.1	<0.001	<0.1	<0.001	<0.1	<0.001	<0.1	<0.001
Total	99.3	0.083	100	0.025	100	0.028	100	0.73

Notes:
T = Tetraniliprole.
[a] Day 6-13.25.
[b] Characterised by extraction and partition behaviour.
[c] Comprised of 15 separate regions, 9 in the first extraction series and 6 in the second series, the largest of which contained 12.4% TRR, 0.010 mg eq/kg.
[d] Comprised of 5 separate regions, the largest of which contained 19.9% TRR, 0.005 mg eq/kg.
[e] Comprised of 3 separate regions, the largest of which contained 4.8% TRR, 0.001 mg eq/kg.
[f] Comprised of 30 separate regions, the largest of which contained 6.9% TRR, 0.050 mg eq/kg.
[g] Sum of unknowns [b] from both extraction series and organic phase in case of fat.

Tetrazolyl-label

The metabolism of [tetrazolyl-^{14}C]-tetraniliprole was investigated in laying hens (Bongartz *et al*., 2015d, Report M1854608-2, M-539074-01-1). The test compound was orally administered to six hens ("LB Lohmann Brown") as an aqueous 0.5 percent Tragacanth suspension by gavage using a syringe at a dose level of *ca.* 1 mg/kg body weight. Based on the daily feed consumption, the dose level corresponded to *ca.* 18.66 ppm (1.03 mg ai /kg dry feed/day). The hens were given 14 consecutive doses at 24-hour intervals in the morning after and were sacrificed *ca.* 6 hours after the last dosing.

Throughout the experiment, the hens were housed in metabolism cages, which permitted separate collection of eggs and excreta. The eggs were collected once daily and before sacrifice. TRRs

were determined in each egg (mixed sample from egg white and yolk) and in dissected organs and tissues (muscle, fat, liver, kidney, skin and eggs from ovary/ oviduct) at sacrifice. The total radioactivity (percent of TAR) was additionally determined in each excreta sample. The radioactivity measurement in liquid samples was carried out by LSC. All solid samples were combusted and the radioactivity determined by LSC.

The overall recovery amounted to *ca.* 92 percent TAR. The remaining amount of radioactivity (Approx. 8 percent TAR) was expected to still be present in the gastro-intestinal tract at sacrifice, likely due to the short period of time between last administered dose and sacrifice (Approx. 6 hours). An average amount of *ca.* 0.2 percent TAR was measured in the eggs. At sacrifice, residues in the organs and tissues were about 0.4 percent TAR. Up to the time of sacrifice, the excretion products accounted for *ca.* 91.3 percent TAR. The daily excretion rate plateaued after third dose to 6-7 percent TAR within 24 hours.

The TRR-values in eggs ranged from 0.011 mg eq/kg at day one to 0.100 mg eq/kg at sacrifice. Following a linear increase a residue plateau-level of 0.089 mg eq/kg was reached approximately at day nine after the first administration. Regarding organs and tissues, the TRR-values amounted to 0.766 mg eq/kg in liver, 0.172 mg eq/kg in kidney, 0.095 mg eq/kg in subcutaneous fat, 0.078 mg eq/kg in skin and 0.031 mg eq/kg in skeletal muscle.

Aliquots from eggs, muscle, liver and excreta were extracted three times with a mixture of acetonitrile/ water (8/2). In all cases, 1 mL formic acid was added to the first extraction step. In case of fat, 100 mL n-heptane were added to every extraction step and subsequently separated from the aqueous phases. All n-heptane phases and aqueous phases were unified before further processing/ analysis. Solids after the first extraction series of liver were further extracted two times with mixtures of acetonitrile/water (1/1) using microwave assistance (5 min to 120 °C, 20 min at 120 °C). Formic acid was added to the last extraction step. The combined extracts were concentrated by rotary evaporation. Afterwards, the distribution and quantities of parent compound and metabolites were determined by HPLC with the profiling method “ANTAM”.

The majority of the residues in the eggs as well as organs and tissues were efficiently extracted (90.3 to 100 percent TRR) using acetonitrile/water mixtures. In case of liver, post extracted solids were further extracted using microwave treatment. Only up to 9.7 percent (0.008 mg eq/kg) TRR of the residues remained in the PES from all commodities. The distribution of radioactivity in eggs and edible tissues is presented in Table 48.

Table 48 Distribution of radioactivity in the extracts of eggs and edible tissues of hens following oral dosing with [tetrazolyl -^{14}C]-tetraniliprole for 14 consecutive days at 19 ppm ai in feed

Sample	Eggs [a]		Muscle		Fat		Liver	
TRR (mg/kg)	0.086		0.031		0.095		0.766	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Initial solvent extract	90.3	0.078	94.1	0.029	99.7	0.095	54.0	0.414
Microwave assisted extract	-	-	-	-	-	-	46.0	0.352
Organic phase (fat only)	-	-	-	-	<0.1	<0.001	-	-
Total extracted	90.3	0.078	94.1	0.029	99.7	0.095	100	0.766
PES	9.7	0.008	5.9	0.002	0.3	<0.001	<0.1	<0.001
Accountability	100	0.086	100	0.031	100	0.095	100	0.766

Notes:
[a] Day 6 – 13.25.

Parent compound and metabolites were quantified in the extracts by HPLC- based on the profiling method ANTAM. Parent compound and metabolites were assigned in the profiles of the current study by comparison with the profiles analysed in the study with the pyrazole-carboxamide label (Bongartz, 2017, M-535432-02-1, Report EnSA-5-0411). Unknown metabolites were characterised based on their extraction and chromatographic behaviour.

For the isolation of label specific conjugates of tetraniliprole-tetrazole, an additional amount of 100 g egg pool and 50 g liver pool were extracted with acetonitrile/water mixture (8/2). The conjugates were isolated from concentrated extracts by HPLC using the profiling method ANTAM. Due to acidic conditions, the conjugates were cleaved during sample preparation. The common exocon tetraniliprole-tetrazole was identified in the isolated fractions by LC-MS/MS analysis.

To evaluate the impact of acidic conditions on the stability of the conjugates in the extracts during isolation and sample preparation, 500 µL of the initial extracts from eggs and liver were treated with 200 µL 1N HCl and stirred at 70 °C for 30 min and analysed by HPLC.

Parent tetraniliprole was a major compound in fat (0.025 mg/kg, 26 percent TRR). Tetraniliprole was also detected in eggs, muscle and liver (0.004 mg/kg (4.2 percent TRR) in eggs, 0.003 mg/kg (9.4 percent TRR) in muscle and 0.032 mg/kg (4.2 percent TRR) in liver). Metabolite tetraniliprole-despyridyl-N-methyl-quinazolinone was the predominant residue in eggs and fat (0.023 mg eq/kg (27 percent TRR) for eggs and 0.059 mg eq/kg (62 percent TRR) for fat), while the metabolite amounted to 0.002 mg eq/kg (6.8 percent TRR) in muscle and 0.065 mg eq/kg (8.5 percent TRR) in liver. In muscle, the metabolite tetraniliprole-pyrazole-5-N-methyl-amide was the main residue and amounted to 0.005 mg eq/kg (18 percent TRR). Tetraniliprole-pyrazole-5-N-methyl-amide was also a prominent portion in the liver (0.044 mg eq/kg; 5.8 percent TRR). The main compound in liver was metabolite tetraniliprole-despyridyl (0.074 mg eq/kg, 9.6 percent TRR) and was detected in minor amounts in eggs, muscle and fat.

Other metabolites in liver were tetraniliprole-pyrazole-5-carboxylic acid (0.039 mg eq/kg; 5.0 percent TRR), tetraniliprole-benzylalcohol (0.062 mg eq/kg; 8.1 percent TRR), tetraniliprole-despyridyl-N-methyl-quinazolinone-hydroxy and tetraniliprole-despyridyl-hydroxy (0.069 mg eq/kg; 9.0 percent TRR) and tetraniliprole-despyridyl-quinazolinone (0.044 mg eq/kg; 5.8 percent TRR).

The label specific metabolite tetraniliprole-tetrazole and its conjugates were detected in eggs, muscle, fat and liver with levels ranging from (0.8-15 percent TRR). The sum of tetraniliprole-tetrazole and its conjugates amounted to 0.019 mg eq/kg (23 percent TRR) for eggs, 0.009 mg eq/kg (29 percent TRR) for muscle, 0.005 mg eq/kg (5.5 percent TRR) for fat and 0.025 mg qe/kg (3.3 percent TRR) for liver.

All other minor identified metabolites, such as tetraniliprole-pyrazole-5-amide and tetraniliprole-N-methyl-quinazolinone accounted for ≤0.027 mg eq/kg. The metabolic profile of tetraniliprole in eggs and edible tissues is presented in Table 49.

Table 49 Distribution of parent compound and metabolites in the extracts of eggs and edible tissues of hens following oral administration of [tetrazolyl-^{14}C]-tetraniliprole for 14 consecutive days at 19 ppm ai in feed

Component / Sample	Eggs (day 6-13.25)		Muscle		Fat		Liver	
TRR (mg/kg)	0.086		0.031		0.095		0.766	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Initial solvent extraction (3 ×) with acetonitrile/water 8:2 + formic acid in the first extraction step								
Extracted								
Tetraniliprole (parent compound)	4.2	0.004	9.4	0.003	26	0.025	4.2	0.032
Tetrazole + tetrazole conjugates [a]	22.7	0.019	28.9	0.009	5.5	0.005	3.3	0.025
T-pyrazole-5-N-methyl-amide-hydroxy	1.4	0.001	-	-	-	-	0.5	0.004

Component / Sample	Eggs (day 6-13.25)		Muscle		Fat		Liver	
TRR (mg/kg)	0.086		0.031		0.095		0.766	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
T-pyrazole-5-amide	2.7	0.002	9.7	0.003	2.5	0.002	0.9	0.007
T-pyrazole-5-N-methyl-amide	6.0	0.005	17.6	0.005	-	-	2.1	0.016
T-benzylalcohol-Gluc	1.5	0.001	-	-	-	-	3.0	0.023
T-dihydroxy	1.7	0.001	1.4	<0.001	-	-	1.1	0.009
T-despyridyl	3.4	0.003	3.0	0.001	1.9	0.002	5.5	0.042
T-benzylalcohol	0.5	<0.001	-	-	-	-	5.8	0.045
T-despyridyl-N-methyl-quinazolinone-hydroxy and despyridyl-hydroxy	1.5	0.001	-	-	-	-	3.9	0.030
T-hydroxy-N-methyl	1.5	0.001	1.3	<0.001	-	-	3.9	0.030
T-despyridyl-quinazolinone	2.6	0.002	-	-	1.5	0.001	1.5	0.011
T-despyridyl-N-methyl-quinazolinone	27	0.023	6.8	0.002	62	0.059	2.2	0.017
T-N-methyl-quinazolinone	3.1	0.003	-	-	-	-	-	-
Unknowns [b]	10.9 [c]	0.010 [c]	15.9 [d]	0.005 [d]	<0.1	<0.001	16.1 [e]	0.12 [e]
Identified	79.8	0.066	78.1	0.023	99.4	0.094	37.9	0.29
Organic phase (n-heptane)	-	-	-	-	<0.1	<0.001	-	-
Microwave assisted solvent extraction (2 ×) with acetonitrile/water (1:1) + formic acid in last extraction step								
Extracted	-	-	-	-	-	-	46.0	0.35
T-pyrazole-5-amide	-	-	-	-	-	-	2.6	0.020
T-pyrazole-5-N-methyl-amide	-	-	-	-	-	-	3.6	0.028
T-pyrazole-5-carboxylic acid	-	-	-	-	-	-	5.0	0.039
T-despyridyl	-	-	-	-	-	-	4.2	0.032
T-benzylalcohol	-	-	-	-	-	-	2.3	0.018
T-despyridyl-N-methyl-quinazolinone-hydroxy and despyridyl-hydroxy	-	-	-	-	-	-	5.1	0.039
T-despyridyl-quinazolinone	-	-	-	-	-	-	4.3	0.033
T-despyridyl-N-methyl-quinazolinone	-	-	-	-	-	-	6.2	0.048
Unknowns	-	-	-	-	-	-	12.6	0.097
Identified	-	-	-	-	-	-	33.3	0.26
Total identified	79.8	0.066	78.1	0.023	99.4	0.094	71.2	0.55
Total characterised	11	0.010	16	0.005	<0.1	<0.001	29	0.22
Total extracted	90.3	0.078	94.1	0.029	99.7	0.095	100	0.766
Solids remaining	9.7	0.008	5.9	0.002	0.3	<0.001	<0.1	<0.001
Total	100	0.086	100	0.031	100	0.095	100	0.766

Notes:

[a] Sum of tetraniliprole-tetrazole and its 3 conjugates.

[b] Characterised by extraction and partition behaviour.

[c] Comprised of 9 separate regions, the largest of which contained 2.2% TRR, 0.002 mg/kg.

[d] Comprised of 2 separate regions, the largest of which contained 13.4% TRR, 0.004 mg/kg.

[e] Comprised of 13 separate regions, the largest of which contained 4.8% TRR, 0.037 mg/kg.

Overview of the metabolic pathway in livestock

The principal metabolic reactions of tetraniliprole in the lactating goat are listed below:

- intra-molecular condensation (cyclisation) of parent compound leading to quinazolinone compounds;
- hydroxylation in the methyl group of the phenyl moiety leading to tetraniliprole-benzylalcohol and the N-methyl moiety leading to tetraniliprole hydroxy-N-methyl;

- demethylation of the N-methyl group to form tetraniliprole-quinazolinone and tetraniliprole-desmethyl-amide;
- cleavage of the pyridine ring leading to tetraniliprole-pyrazole-5-amide (PC- and tetra-label) followed by further oxidation to a tetraniliprole-pyrazole-5-carboxylic acid or by methylation leading to tetraniliprole-N-methyl-amide;
- cleavage of the phenyl ring to form tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid;
- cleavage of the tetrazole ring followed by oxidation leading to tetraniliprole-N-methyl-quinazolinone-3-carboxylic acid.

A proposed metabolic pathway for tetraniliprole in the lactating goat is presented in Figure 4.

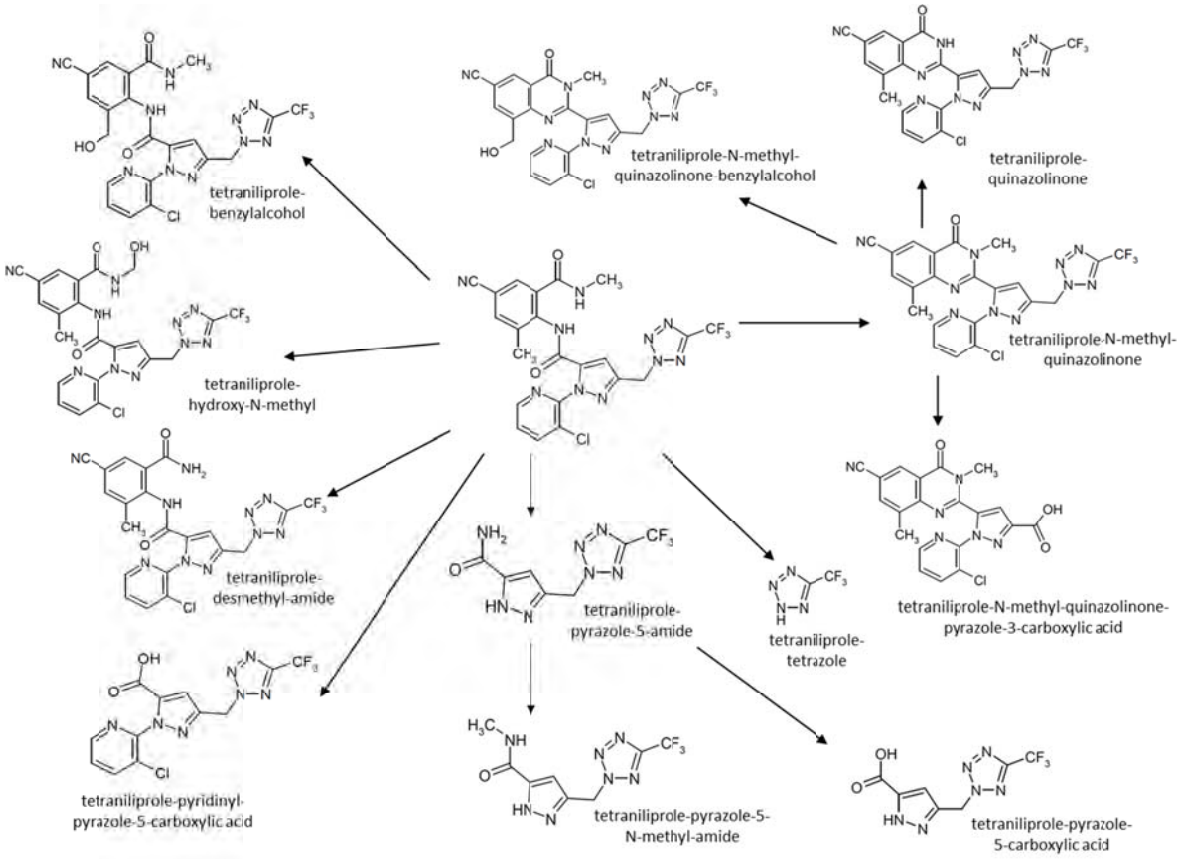


Figure 4 Proposed metabolic pathway for tetraniliprole in the lactating goat

The principal metabolic reactions of tetraniliprole in the laying hen are:

- cleavage of the pyridine ring, leading to despyridyl compounds;
- intra-molecular condensation of despyridyl compounds and parent compound leading to the quinazolinone compounds;
- cleavage of the tetrazole ring, followed by conjugation (tetra-label only);
- hydroxylations, leading to mono- and/or dihydroxy compounds such as tetraniliprole-dihydroxy, tetraniliprole-despyridyl-hydroxy, tetraniliprole-despyridyl-N-methyl-quinazolinone-hydroxy, and tetraniliprole-pyrazole-5-N-methyl-amide-hydroxy;
- conjugation with glucuronic acid after hydroxylation to tetraniliprole-benzylalcohol;

- cleavage of the phenyl moiety, leading to tetraniliprole-5-amide followed by methylation into tetraniliprole-pyrazole-5-N-methyl-amide followed by hydroxylation (tetraniliprole-pyrazole-5-N-methyl-amide-hydroxy);
- demethylation of the N-methyl group after intra-molecular condensation (cyclisation) of tetraniliprole-despyridyl to form tetraniliprole-despyridyl-quinazolinone.

A proposed metabolic pathway for tetraniliprole in the laying hen is presented in Table 5 and Figure 6 shows the positions involved in the metabolic pathway of tetraniliprole in livestock.

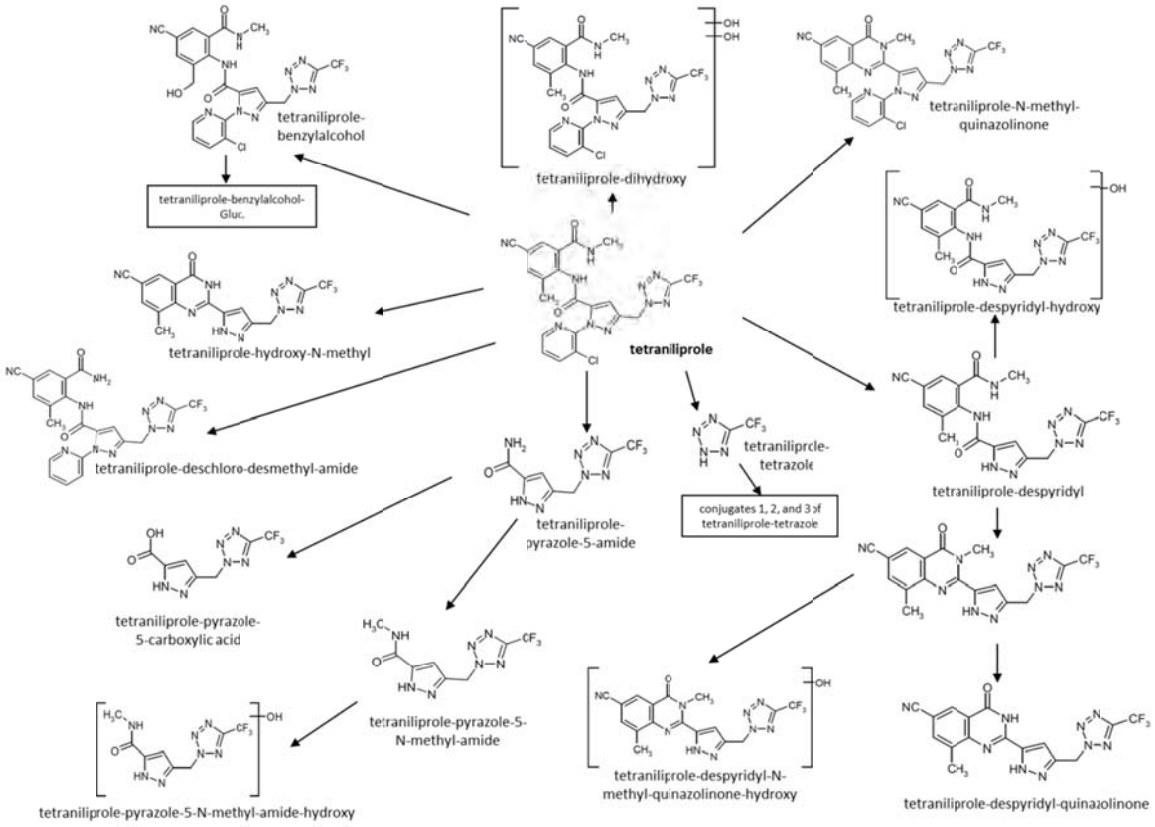


Figure 5 Proposed metabolic pathway for tetraniliprole in the laying hen

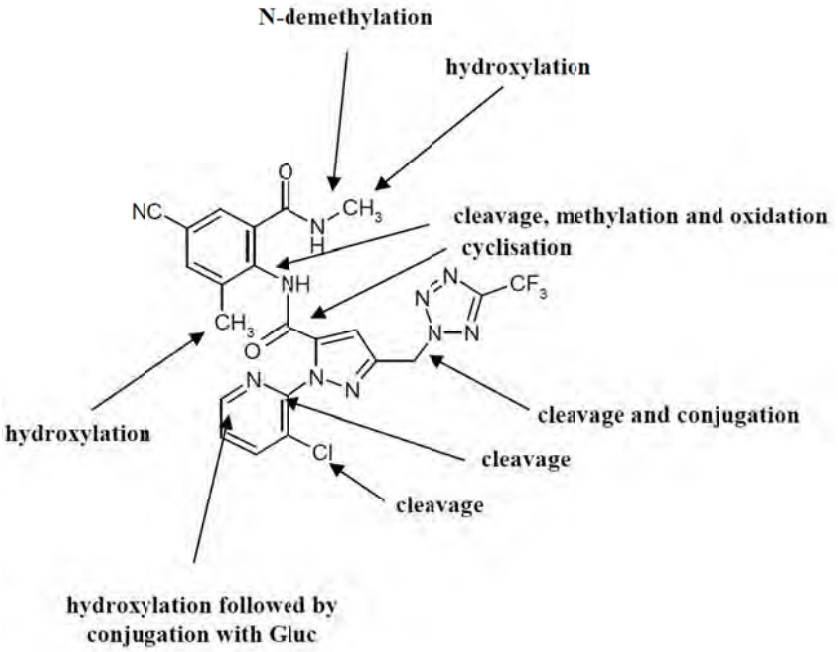


Figure 6 Positions involved in the metabolic pathway of tetraniliprole in livestock

RESIDUE ANALYSIS

Plant commodities

Enforcement methods

LC-MS/MS multi-residue method QuEChERS

The method 01463 (modification RTP-01) was validated for the determination of tetraniliprole and its metabolites tetraniliprole-N-methylquinazolinone and tetraniliprole-benzylalcohol in tomato (watery) and rapeseed (oilseed) (Reed, 2016a, M-564377-01-1, Report 035053).

The tomato and rapeseed seed were dry ice ground and stored frozen before use. Rapeseed (5 g) or tomato (10 g) of the homogenized and deep frozen samples were taken. Depending on the sample 10 mL of water was added as well as acetonitrile. The tube was shaken (15 minutes), 4 g of magnesium sulfate anhydrous, 1 g of sodium chloride, 1 g of trisodium citrate dihydrate and 0.5 g of disodium hydrogencitrate sesquihydrate was added, as well as a mixed internal standard solution and the tube was shaken strongly by hand for 1 minute. After centrifugation (4750 rpm for 5 minutes) about 1 mL supernatant was transferred into a 96 well plate and filtered through a 0.45 µm single syringe filter and analysed for tetraniliprole and both metabolites by LC-MS/MS, operated in electrospray positive mode without any further clean-up step. Transition for tetraniliprole in the primary method used was m/z 545.1 to 356.1 and for the confirmatory method m/z 545.1 to 376.1. For tetraniliprole-N- methylquinazolinone transitions m/z 527.0 to 389.1 and 527.0 to 374.0 (confirmation) were used. Similarly, the two transitions used for metabolite tetraniliprole-benzylalcohol were m/z 561.1 to 356.1 (primary) and 561.1–392.0 (confirmation). Calibration was applied with internal or matrix matched external standards solvent or matrix and using linear regression. The reported LOQ of the method was 0.01 mg/kg for parent and both metabolites.

A validation study was also carried out for tomato, grape (bunches of grapes), wheat (grain), dry bean (seed) and rape (seed) using matrix-matched external standards (tetraniliprole and tetraniliprole-N-methylquinazolinone only) (Stuke&van Berkum, 2016, M-544119-01-2, Report MR-15/091). An independent method validation (ILV) for method 01463 for tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone in food of plant origin by QuEChERS was performed in tomato and rapeseed (seed) (Lakaschus& Lau, 2016, M-554622-01-1, Report S16-00229). The extraction method was performed as described by Reed (2016, M-564377-01-1, Report 035053).

Validation results are shown in Table 50 and Table 52.

Table 50 Validation results for tetraniliprole with HPLC-MS/MS multi residue method QuEChERS (method 01463) n= 5

Commodity	Spike level mg/kg	% Recovery mean	% Recovery range	RSD_r, %	Control samples mg/kg (n)	Calibration	Reference, method
Quantification at m/z 545.1 to 356.1							
Tomato 1st extraction	0.01 0.1	89 101	82-94 97-104	5.6 2.6	<0.3LOQ (2)	Internal standard 10-100 ng/mL $1/x^2$ weighted, r=0.9982	035053 Validation
Tomato 2nd extraction	0.01 0.1	99 102	96-104 97109	3.3 4.8	<0.3LOQ (2)	Internal standard 10-100 ng/mL $1/x^2$ weighted, r=0.9998	035053 Validation
Tomato 1st extraction	0.01 0.1	83 88	58-106 5.6-123	21 40	<0.3LOQ (2)	Matrix matched 5.6-123 ng/mL $1/x^2$ weighted, r=0.9907	035053 Validation
Tomato 2nd extraction	0.01 0.1	102 106	100-103 106-107	1.2 0.4	<0.3LOQ (2)	Matrix matched 10-107 ng/mL $1/x^2$ weighted, r=0.9979	035053 Validation
Tomato	0.01 0.1	97 95	96-99 92-97	1.2 2.2	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Tomato	0.01 0.1	91 96	90-94 93-97	1.9 1.8	<0.3LOQ (2)	Matrix matched 1.25-150 ng/mL 1/x weighted, R=0.9998	S16-00229 ILV
Rapeseed (seed)	0.01 0.1	103 100	95-120 94-105	9.5 3.9	<0.3LOQ (2)	Internal standard 5-50 ng/mL $1/x^2$ weighted, r=0.9992	035053 Validation
Rapeseed (seed)	0.01 0.1	77 67	72-86 76-74	7.5 2.0	<0.3LOQ (2)	Matrix matched 3.7-39 ng/mL $1/x^2$ weighted, r=0.9907	035053 Validation
Rapeseed (seed)	0.01 0.1	102 96	97-105 93-100	3.1 3.4	<0.3LOQ (2)	Matrix matched	MR-15/091 Validation
Rapeseed (seed)	0.01 0.1	82 86	80-87 83-90	3.4 3.2	<0.3LOQ (2)	Matrix matched 1.25-150 ng/mL 1/x weighted, R=0.9995	S16-00229 ILV
Grapes (bunches)	0.01 0.1	106 100	104-109 98-104	2.0 2.3	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Wheat (grain)	0.01 0.1	99 105	92-107 91-114	5.7 9.2	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 validation
Dry bean (seed)	0.01 0.1	98 97	92-101 95-99	3.1 1.8	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Tomato	0.01	88	87-100	9.1	<0.3LOQ	Internal standard	035053

Commodity	Spike level mg/kg	% Recovery mean	% Recovery range	RSD_r, %	Control samples mg/kg (n)	Calibration	Reference, method
1st extraction	0.1	97	90-100	4.1	(2)	10-100 ng/mL $1/x^2$ weighted, r=0.9997	Validation
Tomato 2nd extraction	0.01 0.1	97 102	86-101 99-109	6.6 4.5	<0.3LOQ (2)	Internal standard 10-100 ng/mL $1/x^2$ weighted, r=0.9997	035053 Validation
Tomato 1st extraction	0.01 0.1	84 87	82-106 5.3-116	19 54	<0.3LOQ (2)	Matrix matched 5.6-116 ng/mL $1/x^2$ weighted, r=0.9893	035053 Validation
Tomato 2nd extraction	0.01 0.1	102 106	94-107 105-107	5.4 1.0	<0.3LOQ (2)	Matrix matched 9.4-107 ng/mL $1/x^2$ weighted, r=0.9978	035053 Validation
Tomato	0.01 0.1	98 95	95-100 92-98	1.9 2.4	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Tomato	0.01 0.1	94 98	91-97 94-101	2.5 2.7	<0.3LOQ (2)	Matrix matched 1.25-150 ng/mL 1/x weighted, R=0.9999	S16-00229 ILV
Rapeseed (seed)	0.01 0.1	102 99	96-108 94-102	5.2 3.3	<0.3LOQ (2)	Internal standard 5-50 ng/mL $1/x^2$ weighted, r=0.9996	035053 Validation
Rapeseed (seed)	0.01 0.1	77 76	73-83 73-78	5.2 2.7	<0.3LOQ (2)	Matrix matched 3.9-39 ng/mL $1/x^2$ weighted, r=0.9913	035053 Validation
Rapeseed (seed)	0.01 0.1	102 96	97-105 93-100	3.1 3.2	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Rapeseed (seed)	0.01 0.1	80 85	76-84 83-90	3.6 3.5	<0.3LOQ (2)	Matrix matched 1.25-150 ng/mL 1/x weighted, r=0.9999	S16-00229 ILV
Grapes (bunches)	0.01 0.1	106 101	103-108 99-103	2.0 1.5	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Wheat (grain)	0.01 0.1	99 103	91-103 87-114	5.0 10	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Dry bean (seed)	0.01 0.1	96 96	89-98 94-100	4.1 2.3	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation

Table 51 Validation results for tetraniliprole-N-methylquinazolinone with HPLC-MS/MS multi residue method QuEChERS (method 01463), n=5

Commodity	Spike level mg/kg	% Recovery mean	% Recovery range	RSD_r, %	Control samples (n)	Calibration	Reference, method
Quantification at m/z 527.0 to 389.1							
Tomato 1st extraction	0.01 0.1	92 102	86-99 98-113	6.2 9.2	<0.3LOQ (2)	Internal standard 10-100 ng/mL $1/x^2$ weighted, r=0.9987	035053 Validation
Tomato 2nd extraction	0.01 0.1	97 97	81-106 94-103	9.9 3.7	<0.3LOQ (2)	Internal standard 10-100 ng/mL $1/x^2$ weighted, r=0.9973	035053 Validation
Tomato	0.01	69	52-87	20	<0.3LOQ	Matrix matched	035053

Commodity	Spike level mg/kg	% Recovery mean	% Recovery range	RSD_r, %	Control samples (n)	Calibration	Reference, method
1st extraction	0.1	77	5.6-108	54	(2)	5.2-108 ng/mL $1/x^2$ weighted r=0.9897	Validation
Tomato 2nd extraction	0.01 0.1	91 93	85-95 90-96	4.8 2.1	<0.3LOQ (2)	Matrix matched 8.5-96 ng/mL $1/x^2$ weighted r=0.9975	035053 Validation
Tomato	0.01 0.1	96 95	89-101 92-98	4.8 2.3	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Tomato	0.01 0.1	101 99	98-102 96-102	1.9 2.9	<0.3LOQ (2)	Matrix matched 1.25-150 ng/mL 1/x weighted, R=0.9996	S16-00229 ILV
Rapeseed (seed)	0.01 0.1	94 95	85-106 90-105	10 6.6	<0.3LOQ (2)	Internal standard 5-50 ng/mL $1/x^2$ weighted r=0.9932	035053 Validation
Rapeseed (seed)	0.01 0.1	78 83	71-86 82-85	8.0 1.5	<0.3LOQ (2)	Matrix matched 4.0-43 ng/mL $1/x^2$ weighted, r=0.9909	035053 Validation
Rapeseed (seed)	0.01 0.1	94 86	91-98 83-90	2.9 3.4	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Rapeseed (seed)	0.01 0.1	83 77	76-86 72-84	5.0 7.0	<0.3LOQ (2)	Matrix matched 1.25-150 ng/mL 1/x weighted, r=0.9999	S16-00229 ILV
Grapes (bunches)	0.01 0.1	106 101	105-106 99-103	0.4 1.6	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Wheat (grain)	0.01 0.1	82 94	74-89 80-104	7.9 10	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Dry bean (seed)	0.01 0.1	105 104	100-115 98-110	5.5 4.3	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Tomato 1st extraction	0.01 0.1	84 99	80-89 88-113	4.3 10	<0.3LOQ (2)	Internal standard 10-100 ng/mL $1/x^2$ weighted, r=0.9986	035053 Validation
Tomato 2nd extraction	0.01 0.1	93 92	79-110 83-99	13 7.0	<0.3LOQ (2)	Internal standard 10-100 ng/mL $1/x^2$ weighted, r=0.9971	035053 Validation
Tomato 1st extraction	0.01 0.1	64 75	48-80 5.9-104	20 55	<0.3LOQ (2)	Matrix matched 5.0-104 ng/mL $1/x^2$ weighted, r=0.9962	035053 Validation
Tomato 2nd extraction	0.01 0.1	83 88	74-89 84-91	7.4 3.4	<0.3LOQ (2)	Matrix matched 7.5-91 ng/mL $1/x^2$ weighted, r=0.9955	035053 Validation
Tomato	0.01 0.1	95 95	92-99 93-96	2.9 1.3	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Tomato	0.01 0.1	99 99	94-104 98-101	3.7 1.2	<0.3LOQ (2)	Matrix matched 1.25-150 ng/mL 1/x weighted, R=0.9993	S16-00229 ILV
Rapeseed (seed)	0.01	95	84-106	8.5	<0.3LOQ	Internal standard	035053

Commodity	Spike level mg/kg	% Recovery mean	% Recovery range	RSD_r, %	Control samples (n)	Calibration	Reference, method
	0.1	92	82-98	7.8	(2)	5-50 ng/mL $1/x^2$ weighted, r=0.9962	Validation
Rapeseed (seed)	0.01 0.1	85 81	76-96 77-88	8.8 5.5	<0.3LOQ (2)	Matrix matched 4.4-45 ng/mL $1/x^2$ weighted, r=0.9910	035053 Validation
Rapeseed (seed)	0.01 0.1	92 86	87-102 83-90	7.4 3.6	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Rapeseed (seed)	0.01 0.1	84 80	78-99 75-86	10 6.3	<0.3LOQ (2)	Matrix matched 1.25-150 ng/mL 1/x weighted, R=0.9993	S16-00229 ILV
Grapes (bunches)	0.01 0.1	106 100	102-109 97-102	3.4 2.1	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Wheat (grain)	0.01 0.1	82 92	76-90 79-101	7.7 9.6	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation
Dry bean (seed)	0.01 0.1	100 102	90-114 97-109	9.2 4.3	<0.3LOQ (3)	Matrix matched 1-500 ng/mL 1/x weighted, r≥0.999	MR-15/091 Validation

Table 52 Validation results for tetraniliprole-benzylalcohol with LC-MS/MS multi residue method QuEChERS (method 01463). n=5

Commodity	Spike level mg/kg	% Recovery mean	% Recovery range	RSD_r, %	Control samples mg/kg (n)	Calibration	Reference, method
Quantification at m/z 561.1 to 356.1							
Tomato 1st extraction	0.01 0.1	88 100	81-98 91-110	7.7 8.0	<0.3LOQ (2)	Internal standard 10-100 ng/mL $1/x^2$ weighted, r=0.9982	035053 Validation
Tomato 1st extraction	0.01 0.1	77 84	54-105 3.8-113	25 56	<0.3LOQ (2)	Matrix matched 5.8-113 ng/mL $1/x^2$ weighted, r=0.9931	035053 Validation
Tomato 2nd extraction	0.01 0.1	100 95	97-103 88-106	2.1 8.1	<0.3LOQ (2)	Internal standard 10-100 ng/mL $1/x^2$ weighted, r=0.9980	035053 Validation
Tomato 2nd extraction	0.01 0.1	100 102	96-102 99-104	2.4 1.9	<0.3LOQ (2)	Matrix matched 9.7-104 ng/mL $1/x^2$ weighted, r=0.9979	035053 Validation
Rapeseed (seed)	0.01 0.1	101 98	94-111 93-104	6.4 4.8	<0.3LOQ (2)	Internal standard 5-50 ng/mL $1/x^2$ weighted, r=0.9964	035053 Validation
Rapeseed (seed)	0.01 0.1	64 65	61-70 63-67	5.0 2.4	<0.3LOQ (2)	Matrix matched 3.6-34 ng/mL $1/x^2$ weighted, r=0.9940	035053 Validation
Tomato 1st extraction	0.01 0.1	84 99	78-92 89-106	7.1 7.4	<0.3LOQ (2)	Internal standard 10-100 ng/mL $1/x^2$ weighted, r=0.9981	035053 Validation
Tomato 1st extraction	0.01 0.1	74 82	47-103 3.8-113	28 55	<0.3LOQ (2)	Matrix matched 4.0-113 ng/mL $1/x^2$ weighted, r=0.9964	035053 Validation
Tomato	0.01	99	94-103	3.5	<0.3LOQ (2)	Internal standard	035053

Commodity	Spike level mg/kg	% Recovery mean	% Recovery range	RSD_r, %	Control samples mg/kg (n)	Calibration	Reference, method
2nd extraction	0.1	94	85-102	7.0		10-100 ng/mL $1/x^2$ weighted, r=0.9986	Validation
Tomato 2nd extraction	0.01 0.1	98 101	92-104 100-106	4.5 2.7	<0.3LOQ (2)	Matrix matched 9.9-106 ng/mL $1/x^2$ weighted, r=0.9981	035053 Validation
Rapeseed (seed)	0.01 0.1	102 96	90-113 93-102	9.1 3.6	<0.3LOQ (2)	Internal standard 5-50 ng/mL $1/x^2$ weighted, r=0.9926	035053 Validation
Rapeseed (seed)	0.01 0.1	66 65	59-75 62-66	9.5 2.1	<0.3LOQ (2)	Matrix matched 3.5-34 ng/mL $1/x^2$ weighted, r=0.9935	035053 Validation

Analytical methods used in study reports

HPLC-MS/MS method 1414 determines tetraniliprole and tetraniliprole-methylquinazolinone in various plant matrices. The method was first described by Stuke & Santiago (2014, M-488453-01-2, Report P602145509). An independent laboratory validation of the method for determination of tetraniliprole and tetraniliprole-N-methylquinazolinone was performed in citrus and broccoli (Claussen, 2016, M-564116-01-1, Report 122G1313).

Residues are extracted from the different matrices (2 g of sample) using water (8 mL) and acetonitrile (10 mL) along with automated shaking. After shaking, an internal standard solution is added. The extract is then filtered and the extracted residue levels are determined by LC-MS/MS. Residues of tetraniliprole and tetraniliprole-N-methylquinazolinone (are quantified using external calibration with standards in solvent containing stable isotope labelled standards.

The linearity range of the used detector was determined for tetraniliprole and its metabolite tetraniliprole-N-methylquinazolinone, as well as metabolites tetraniliprole carboxilic acid and tetraniliprole-desmethyl-amide-carboxylic acid using calibration standards containing 0.2, 1 (LOQ), 5, 10 and 25 µg/L of tetraniliprole and its metabolites tetraniliprole-N-methylquinazolinone, tetraniliprole-carboxylic acid, and tetraniliprole-desmethyl-amide-carboxylic acid and 10 µg/L of stable isotope labelled ISTD of each analyte. The correlation coefficients of the 1/× weighted linear regression were in all cases ≥ 0.99. The reported LOQ was 0.01 mg/kg. Validation results are shown in Table 53 and Table 54.

Recovery rates were determined after fortification of control samples with tetraniliprole and tetraniliprole-N-methylquinazolinone at fortification levels (expressed as parent equivalent) of 0.01 mg/kg and 0.1 mg/kg in all sample materials tested. Repeatability (precision) of the method, is given as relative standard deviation (% RSD) for all samples (n = 5) at both fortification levels. Analysis was performed LC-MS/MS.

Analytical Method 01414 for the determination of tetraniliprole and tetraniliprole-N-methylquinazolinone in orange whole fruit and broccoli stems and heads, representing citrus and Brassica crops, respectively, was successfully validated by an independent laboratory (Claussen, 2016, M-564166-01-1, Report 122G1313/RAFVP035).

Additional procedural recoveries obtained from the residue field trials have also been summarised here to support the suitability of the method in all plant matrices included in this evaluation.

Mean recovery values for each matrix were within the range of 70–120 percent, and RSD were generally less than 20 percent for tetraniliprole and tetraniliprole-N-methylquinazolinone at the LOQ and 10 × LOQ fortification levels.

Table 53 Recoveries for the validation of method 01414: tetraniliprole in plant matrices

Matrix	No.	Spike level [mg/kg]	Primary transition m/z 563.0 → 355.9			Confirmatory transition m/z 545.0 → 375.9			Report reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
Alfalfa forage	9	0.01	80-90	85	4	-	-	-	RAFVP100, CR
	3	0.1	96-99	98	2	-	-	-	
Alfalfa hay	9	0.01	76-85	81	4	-	-	-	RAFVP100, CR
	3	0.1	92-92	92	1	-	-	-	
Almond nutmeat	8	0.01	77-94	84	8	77-87	81	6	SARS-15-17, CR&MV
	8	5	79-97	90	7	80-94	89	7	
Almond hulls	8	0.01	79-94	87	6	69-101	81	17	SARS-15-17, CR&MV
	8	5	80-95	87	6	80-91	85	6	
Apple	12	0.01	86-95	91	4	-	-	-	RAFVP104-01
	3	0.25	94-95	94	1	-	-	-	
Apple	4	0.01	90-93	92	1	-	-	-	BCS-0532, CR
	4	1.0	93-96	94	1	-	-	-	
Apple	5	0.01	86-99	90	6	-	-	-	BCS-0531, CR
	5	1.0	92-107	96	7	-	-	-	
Apple juice	3	0.01	91-91	91	0	-	-	-	RAFVP064
	3	2.0	102-104	103	1	-	-	-	
Barley hay	7	0.01	88-102	95	5	-	-	-	RAFVP085, CR
	3	0.1	97-100	98	2	-	-	-	
Barley straw	7	0.01	97-107	100	5	-	-	-	RAFVP085, CR
	3	0.1	98-99	98	1	-	-	-	
Barley grain	8	0.01	96-112	101	5	-	-	-	RAFVP085, CR
	3	0.1	97-98	97	1	-	-	-	
Broccoli	5	0.01	87-101	93	6.3	80-104	91	10	122G1313/RAVP035
	5	0.1	86-97	90	4.9	91-104	96	5.4	
Broccoli	6	0.01	83-108	96	11	-	-	-	RAFVP096, CR&MV
	3	0.1	70–97	83	16	-	-	-	
	3	1.0	94-97	96	2	-	-	-	
Cabbage	11	0.01	64-108	87	14	-	-	-	RAFVP096, CR&MV
	8	0.1	70–100	84	12	-	-	-	
	3	1.0	86-107	97	11	-	-	-	
Cauliflower	6	0.01	77-126	97	19	-	-	-	RAFVP096, CR&MV
	2	0.1	69-82	76	NA	-	-	-	
	4	1.0	94-101	98	3	-	-	-	
Cherry	10	0.01	91-114	99	8	94-121	106	10	SARS-15-15, CR&MV
	5	0.1	94-116	100	9	-	-	-	
	10	5	83-110	96	9	84-96	92	5	
Citrus	5	0.01	83-94	87	5.1	87-111	100	11	122G1313/RAVP035
	5	0.1	79-93	87	7.1	81-93	88	5.0	
Cucumber	5	0.01	90-96	94	3	-	-	-	RAFVP101, CR
	2	0.1	94, 89	92	NA	-	-	-	
	3	1.0	80-103	91	13	-	-	-	
Dry bean seed	5	0.01	108-112	110	1.3	88-116	97	13.6	01414
	5	0.1	98-107	103	3.8	91-108	100	6.3	
Dry bean forage	7	0.01	91-112	100	8	-	-	-	RAFVN037, CR
	5	0.1	95-104	100	4	-	-	-	
Dry bean hay	7	0.01	74-108	92	12	-	-	-	RAFVN037, CR

Matrix	No.	Spike level [mg/kg]	Primary transition m/z 563.0 → 355.9			Confirmatory transition m/z 545.0 → 375.9			Report reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
	5	0.1	93-97	96	2	-	-	-	
Dry bean seed	7	0.01	89-108	99	6	-	-	-	RAFVN037, CR
	5	0.1	93-102	98	4	-	-	-	
Dry pea forage	7	0.01	79-99	87	8	-	-	-	RAFVN037, CR
	5	0.1	93-98	95	2	-	-	-	
Dry pea hay	7	0.01	86-112	100	10	-	-	-	RAFVN037, CR
	5	0.1	95-102	99	3	-	-	-	
Dry pea seed	7	0.01	80-102	92	9	-	-	-	RAFVN037, CR
	5	0.1	86-95	92	5	-	-	-	
Field corn forage	14	0.01	63-100	82	12	77-88	81	6	SARS-15-06, CR&MV
	9	0.1	78-88	83	5	-	-	-	
	14	5	88-122	102	12	88-92	89	2	
Field corn grain	15	0.01	70–96	85	9	68-84	76	9	SARS-15-06, CR&MV
	10	0.1	67-89	80	8	-	-	-	
	14	5	78-117	95	17	76-80	78	2	
Field corn stover	13	0.01	72-104	93	9	99-117	108	7	SARS-15-06, CR&MV
	8	0.1	65-86	77	12	-	-	-	
	13	5	86-118	102	11	83-96	88	6	
Field corn (aspirated grain)	6	0.01	90-104	96	5	75-90	81	7	SARS-15-06, CR&MV
	1	0.1	77	NA	NA	-	-	-	
	6	5	80-93	87	6	83-95	88	5	
Field corn flour	6	0.01	84-100	92	7	86-102	94	7	SARS-15-06, CR&MV
	1	0.1	90	NA	NA	-	-	-	
	6	5	89-103	97	5	92-102	98	4	
Field corn grits	6	0.01	95-117	105	9	92-138	108	18	SARS-15-06, CR&MV
	1	0.1	97	NA	NA	-	-	-	
	6	5	89-99	96	4	91-100	97	4	
Field corn oil	6	0.01	94-114	102	7	94-100	97	3	SARS-15-06, CR&MV
	1	0.1	89	NA	NA	-	-	-	
	6	5	94-105	101	4	98-105	103	3	
Field corn meal	6	0.01	88-98	94	5	78-92	85	7	SARS-15-06, CR&MV
	1	0.1	93	NA	NA	-	-	-	
	6	5	94-112	102	6	96-114	104	7	
Field corn pollen	10	0.001	86-127	102	13	103-125	113	8	SARS-15-06, CR&MV
	10	0.01	81-103	94	9	88-104	97	8	
Field corn starch	6	0.01	89-103	96	5	82-101	96	8	SARS-15-06, CR&MV
	1	0.1	92	NA	NA	-	-	-	
	6	5	87-96	91	3	87-93	90	3	
Garden pea seed (succulent without pods)	7	0.01	82-105	90	8	-	-	-	RAFVN035, CR
	5	0.1	99-104	102	2	-	-	-	
Grape (brunches of grapes)	5	0.01	95-110	101	5.7	97-113	107	5.8	01414
	5	0.1	87-115	102	11	92-114	104	7.8	
Grape, fruit	11	0.01	93-106	98	5	84-109	101	11	SARS-14-07, CR&MV
	6	0.1	94-103	99	3	-	-	-	
	9	5	84-107	94	8	86-101	93	7	
Grape, raisin	6	0.01	93-123	103	13	100-114	103	6	SARS-14-07, CR&MV
	1	0.1	110	110	NA	-	-	-	
	6	5	73-112	89	14	75-88	84	6	

Matrix	No.	Spike level [mg/kg]	Primary transition m/z 563.0 → 355.9			Confirmatory transition m/z 545.0 → 375.9			Report reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
Grape, juice	6	0.01	97-111	104	5	89-110	100	8	SARS-14-07, CR&MV
	1	0.1	101	101	NA	-	-	-	
	6	5	92-114	102	8	98-106	101	3	
Grapefruit	15	0.01	82-93	88	5	-	-	-	RAFVP089, CR
	3	1.0	101-102	102	1	-	-	-	
Lemon	14	0.01	81-92	86	4	-	-	-	RAFVP089, CR
	3	1.0	99-101	100	1	-	-	-	
Lettuce, head	9	0.01	85-104	97	6	89-101	96	5	SARS-15-12, CR & MV
	4	0.1	91-99	95	4	-	-	-	
	9	5	90-103	95	4	90-97	94	3	
Lettuce, leaf	10	0.01	61-108	93	15	85-107	98	9	SARS-14-11, CR & MV
	5	0.1	77-102	91	12	-	-	-	
	10	5	88-125	100	12	98-110	104	4	
Lima bean, seed	7	0.01	93-98	95	2	-	-	-	RAFVN035, CR
	5	0.1	94-100	97	3	-	-	-	
Mandarin	13	0.01	79-92	87	5	-	-	-	RAFVP089, CR
	3	1.0	99-101	100	1	-	-	-	
Melon	6	0.01	80-106	93	11	-	-	-	RAFVP101, CR
	2	0.1	95, 103	99	NA	-	-	-	
	4	1.0	85-104	97	10	-	-	-	
Mustard greens	7	0.01	91-101	96	3	-	-	-	RAFVN036, CR
	3	0.25	99-105	102	3	-	-	-	
	3	8.0	101-103	102	1	-	-	-	
Mustard greens (cooked)	3	0.01	88-96	91	5	-	-	-	RAFVN036, CR
	3	2.5	100-105	102	2	-	-	-	
Mustard greens (washed, cooked)	3	0.01	83-97	90	8	-	-	-	RAFVN036, CR
	3	2.5	100-104	102	2	-	-	-	
Onion, bulb	7	0.010	91-108	99	7	-	-	-	RAFVN039, CR
	3	0.1	91-97	95	3	-	-	-	
Orange fruit	19	0.01	84-93	88	3	-	-	-	RAFVP089, CR
	3	1.0	100-103	102	2	-	-	-	
Orange oil	3	0.01	95-97	96	1	-	-	-	RAFVN026
	3	2.0	106-111	108	3	-	-	-	
	3	4.0	106-108	107	1	-	-	-	
Orange juice	3	0.01	85-88	86	2	-	-	-	
	3	2.0	99-102	100	1	-	-	-	
Peach	11	0.01	67-111	92	15	80-105	94	11	SARS-15-16 CR&MV
	6	0.1	75-93	85	8	-	-	-	
	11	5	89-107	97	5	92-99	95	3	
Pear	15	0.01	77-90	84	4	-	-	-	RAFVP104-01, CR
	3	0.25	90-91	90	1	-	-	-	
	3	0.75	105-106	106	1	-	-	-	
Pear	4	0.01	93-99	96	2	-	-	-	BCS-0532, CR
	4	1.0	94-104	97	5	-	-	-	
Pear	4	0.01	85-92	87	4	-	-	-	BCS-0531, CR
	4	1.0	89-97	92	4	-	-	-	
Pecan nutmeat	8	0.01	71-92	84	11	87-94	92	3	SARS-14-02, CR&MV
	3	0.1	81-88	84	5	-	-	-	
	8	5	70–90	83	8	79-88	84	4	

Matrix	No.	Spike level [mg/kg]	Primary transition m/z 563.0 → 355.9			Confirmatory transition m/z 545.0 → 375.9			Report reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
Pepper	10	0.01	81-101	88	8	77-98	86	10	SARS-14-20, CR&MV
	5	0.1	75-101	93	11	-	-	-	
	10	5	81-101	94	7	90-106	97	6	
Plum	10	0.01	90-114	106	7	96-108	103	4	SARS-14-01, CR&MV
	5	0.1	88-106	98	7	-	-	-	
	10	5	87-106	96	6	92-98	95	3	
Potato starch	3	0.01	82-84	83	1	-	-	-	RAFVP062
	3	2.0	103-106	104	1	-	-	-	
Potato tuber	16	0.01	87-98	92	3	-	-	-	RAFVP074, CR
	3	1.0	96-99	98	2	-	-	-	
Prune	6	0.01	91-102	98	4	85-109	96	11	SARS-14-01, CR&MV
	1	0.1	100	100	NA	-	-	-	
	6	5	96-105	100	3	97-104	100	3	
Rapeseed seed	5	0.01	93-100	96	3.2	82-97	90	6.0	01414
	5	0.1	91-97	95	2.7	82-90	87	3.5	
Rapeseed seed	7	0.01	90-104	99	5	-	-	-	RAFVP084
	3	0.1	102-104	103	1	-	-	-	
Rice, bran	7	0.010	90-104	94	4.9	-	-	-	RAFV0032, CR
	5	0.20	102-104	103	0.8	-	-	-	
Rice, brown rice	17	0.010	92-115	100	5	-	-	-	RAFV00414&RAFV0085, CR
	16	0.1	93-105	98	3	-	-	-	
Rice, hulls	7	0.010	87-101	90	5.5	-	-	-	RAFV0032, CR
	5	3.0	97-98	98	0.5	-	-	-	
	5	3.5	84-95	89	4.7	-	-	-	
Rice, panicles	14	0.010	93-113	101	5	-	-	-	RAFV00414&RAFV0085, CR
	10	0.1	94-102	98	2	-	-	-	
	1	3.0	102	NA	NA	-	-	-	
	1	4.0	97	NA	NA	-	-	-	
Rice, straw	21	0.010	66-140	97	18	-	-	-	RAFV00414&RAFV0085, CR
	20	0.1	70–113	92	13	-	-	-	
	1	6.0	87	NA	NA	-	-	-	
	1	7.0	100	NA	NA	-	-	-	
Rice, polished	7	0.010	90-99	96	3.3	-	-	-	RAFV0032, CR
	5	0.10	92-104	98	5.6	-	-	-	
Rice, whole grain	21	0.010	87-123	98	8	-	-	-	RAFV00414&RAFV0085, CR
	20	0.1	88-115	98	6	-	-	-	
	2	2.0	98, 100	99	NA	-	-	-	
Rice, whole grain	7	0.010	96-101	98	1.7	-	-	-	RAFV0032, CR
	5	0.10	97-99	98	0.5	-	-	-	
Rice, whole grain	8	0.010	77-110	95	12.4	-	-	-	I14-046
	6	1.0	72-11	92	17.7	-	-	-	CR
Rice, whole plant (no roots)	14	0.010	86-108	96	8	-	-	-	RAFV00414&RAFV0085, CR
	11	0.1	92-100	95	3	-	-	-	
	1	4.0	95	NA	NA	-	-	-	
	1	5.0	96	NA	NA	-	-	-	
Rice, whole plant (no roots, panicles removed)	14	0.010	84-106	96	8	-	-	-	RAFV00414&RAFV0085, CR
	10	0.1	86-109	97	6	-	-	-	
	2	2.0	93, 97	95	NA	-	-	-	
Snap bean	7	0.01	93-111	101	6	-	-	-	RAFVN033
	5	0.1	86-103	95	7	-	-	-	

Matrix	No.	Spike level [mg/kg]	Primary transition m/z 563.0 → 355.9			Confirmatory transition m/z 545.0 → 375.9			Report reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
Snow pea	7	0.01	73-114	100	16	-	-	-	RAFVN033
	5	0.1	86-102	97	6	-	-	-	
Sorghum fodder	7	0.01	91-98	95	2	-	-	-	RAFVN029
	3	0.1	97-98	97	1	-	-	-	
Sorghum forage	7	0.01	91-104	95	4	-	-	-	RAFVN029
	3	0.1	97-102	99	2	-	-	-	
Sorghum grain	7	0.01	94-103	98	3	-	-	-	RAFVN029
	3	0.1	98-99	99	1	-	-	-	
Soya bean aspirated grain fraction	6	0.01	85-110	98	9	88-114	100	12	SARS-15-03, CR&MV
	1	0.1	95	-	NA	-	-	-	
	6	5	88-93	90	2	79-93	87	6	
Soya bean forage	6	0.01	83-92	89	4	87-96	92	4	SARS-15-03, CR&MV
	1	0.1	85	85	NA	-	-	-	
	6	5	92-103	94	6	92-98	95	3	
Soya bean forage	7	0.01	84-93	90	3	-	-	-	RAFVP051
	3	0.1	95-101	98	3	-	-	-	
Soya bean hay	6	0.01	80-106	92	11	71-122	103	18	SARS-15-03, CR&MV
	1	0.1	79	NA	NA	-	-	-	
	6	5	80-95	86	7	78-88	84	5	
Soya bean hay	7	0.01	79-85	82	3	-	-	-	RAFVP051
	3	0.1	93-98	96	2	-	-	-	
Soya bean hulls	6	0.01	70–87	81	7	77-86	83	4	SARS-15-03, CR&MV
	1	0.1	73	NA	NA	-	-	-	
	6	5	73-83	80	4	80-85	83	2	
Soya bean meal	6	0.01	86-92	89	3	89-100	94	4	SARS-15-03, CR&MV
	1	0.1	83	NA	NA	-	-	-	
	6	5	81-94	86	6	83-90	86	4	
Soya bean oil, refined	6	0.01	87-101	94	5	86-102	95	6	SARS-15-03, CR&MV
	1	0.1	97	NA	NA	-	-	-	
	6	5	90-103	94	5	93-105	97	5	
Soya bean seed	12	0.01	62-95	79	11	72-89	81	9	SARS-15-03, CR&MV
	7	0.1	64-74	70	6	-	-	-	
	17	5	71-91	78	8	72-79	75	4	
Soya bean seed, dry	7	0.01	85-90	88	2	-	-	-	RAFVP051
	3	0.1	99-103	101	2	-	-	-	
Spinach	11	0.01	70–92	83	8	79-115	93	15	SARS-14-14, CR&MV
	6	0.1	75-94	87	8	-	-	-	
	11	5	82-129	96	17	80-88	83	4	
	5	10	114-120	117	2	103-125	115	7	
Summer squash	5	0.01	87-115	102	11	-	-	-	RAFVP101
	2	0.1	103, 86	95	NA	-	-	-	
	3	1.0	94-107	101	6	-	-	-	
Sunflower seed	7	0.01	94-105	100	5	-	-	-	RAFVP030
	3	0.1	105-109	108	2	-	-	-	
Sweetcorn forage	12	0.01	72-110	95	12	88-119	102	11	SARS-15-05, CR&MV
	7	0.1	70–94	81	11	-	-	-	
	12	5	75-97	91	87	89-94	91	3	
Sweet corn (kernals with husk removed)	12	0.01	83-109	94	9	68-102	88	14	SARS-15-05, CR&MV
	7	0.1	80-105	90	9	-	-	-	
	12	5	82-104	95	7	84-94	89	4	

Matrix	No.	Spike level [mg/kg]	Primary transition m/z 563.0 → 355.9			Confirmatory transition m/z 545.0 → 375.9			Report reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
Sweet corn pollen	5	0.001	89-122	108	12	99-147	123	15	SARS-15-05, MV
	5	0.01	89-110	97	9	91-106	97	6	
Sweet corn stover	14	0.01	62-111	91	14	94-130	104	15	SARS-15-05, CR&MV
	9	0.1	64-98	82	12	-	-	-	
	14	5	70–103	90	11	93-102	97	4	
Tomato / fruit	5	0.01	94-108	101	5.7	86-102	92	6.7	01414
	5	0.1	93-98	96	2.4	86-95	89	4.0	
Tomato fruit	14	0.01	76-102	90	7	84-99	94	6	SARS-14-19, CR&MV
	9	0.1	86-107	93	8	-	-	-	
	14	5	82-124	100	9	95-106	99	5	
Tomato paste	6	0.01	91-99	95	3	87-108	97	9	SARS-14-19, CR&MV
	1	0.1	97	97	NA	-	-	-	
	6	5	96-121	103	9	98-118	106	7	
Tomato puree	6	0.01	91-107	98	6	88-112	100	12	SARS-14-19, CR&MV
	1	0.1	98	98	NA	-	-	-	
	6	5	88-108	101	8	93-104	100	5	
Wheat (grain)	5	0.01	88-108	104	8	88-110	98	8.7	01414
	5	0.1	99-114	105	7	94-112	103	7.9	
Wheat forage	7	0.01	94-106	98	5	-	-	-	RAFVP086
	4	0.1	98-100	99	1	-	-	-	
Wheat forage	9	0.01	88-95	90	2	-	-	-	RAFVP051
	5	0.1	96-106	99	4	-	-	-	
Wheat grain	11	0.01	86-104	97	5	-	-	-	RAFVP086
	5	0.1	99-111	105	5	-	-	-	
Wheat grain	8	0.01	87-97	92	4	-	-	-	RAFVP051
	3	0.1	96-101	99	3	-	-	-	
Wheat hay	7	0.01	96-105	99	4	-	-	-	RAFVP086
	4	0.1	98-101	99	1	-	-	-	
Wheat hay	9	0.01	85-94	89	3	-	-	-	RAFVP051
	5	0.1	95-105	98	4	-	-	-	
Wheat straw	7	0.01	91-116	101	8	-	-	-	RAFVP086
	5	0.1	94-106	99	5	-	-	-	
Wheat straw	9	0.01	87-95	92	3	-	-	-	RAFVP051
	5	0.1	93-103	101	4	-	-	-	

Notes:
NA = Not applicable; CR = Concurrent Recovery; MV = Method Validation

Table 54 Recoveries for the validation of method 01414: tetraniliprole-N-methylquinazolinone in plant matrices

Matrix	N°	Spike level [mg/kg]	Primary transition m/z 527.0 → 389.2			Confirmatory transition m/z 527.0 →373.7			Report Reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
Alfalfa forage	9	0.01	82-99	92	5	-	-	-	RAFVP100, CR
	3	0.1	102-106	104	2	-	-	-	
Alfalfa hay	9	0.01	93-106	97	4	-	-	-	RAFVP100, CR
	3	0.1	96-99	97	2	-	-	-	
Almond nutmeat	8	0.01	76-96	85	8	82-96	88	6	SARS-15-17, CR&MV
	8	5	74-90	84	6	73-87	81	6	
Almond	8	0.01	81-106	92	10	84-126	109	15	SARS-15-17,

Matrix	N°	Spike level [mg/kg]	Primary transition m/z 527.0 → 389.2			Confirmatory transition m/z 527.0 →373.7			Report Reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
hulls	8	5	72-89	81	6	71-89	80	8	CR&MV
Apple	12	0.01	101-117	104	5	-	-	-	RAFVP104-01, CR
	3	0.25	103-110	107	4	-	-	-	
Apple	4	0.01	92-95	94	1	-	-	-	BCS-0532, CR
	4	1.0	82-92	89	6	-	-	-	
Apple	5	0.01	85-108	95	9	-	-	-	BCS-0531, CR
	5	1.0	81-113	91	15	-	-	-	
Apple juice	3	0.01	100-106	104	4	-	-	-	RAFVP064
	3	2.0	96-97	96	1	-	-	-	
Barley hay	7	0.01	98-111	104	5	-	-	-	RAFVP085, CR
	3	0.1	107-111	108	2	-	-	-	
Barley straw	7	0.01	100-110	104	3	-	-	-	RAFVP085, CR
	3	0.1	106-114	109	4	-	-	-	
Barley grain	8	0.01	98-109	105	5	-	-	-	RAFVP085, CR
	3	0.1	104-107	105	2	-	-	-	
Broccoli	5	0.01	87-104	95	7.8	82-126	106	16	RAFVP035/122G1 313, ILV
	5	0.1	106-117	109	4.0	107-115	111	2.7	
Broccoli	6	0.01	81-97	89	7	-	-	-	RAFVP096, CR&MV
	3	0.1	87-95	91	4	-	-	-	
	3	1.0	81-96	91	9	-	-	-	
Cabbage	11	0.01	64-99	85	11	-	-	-	RAFVP096, CR&MV
	8	0.1	69-98	82	13	-	-	-	
	3	1.0	82-86	83	3	-	-	-	
Cauliflower	6	0.01	71-131	92	23	-	-	-	RAFVP096, CR&MV
	2	0.1	65-76	71	NA	-	-	-	
	4	1.0	83-96	88	7	-	-	-	
Cherry	10	0.01	70–108	95	11	93-125	105	11	SARS-15-15, CR&MV
	5	0.1	87-107	94	9	-	-	-	
	10	5	80-109	93	9	82-98	88	7	
Citrus	5	0.01	74-91	85	7.5	68-88	78	9.9	RAFVP035/122G1 313 ILV
	5	0.1	91-107	101	6.0	82-108	101	11	
Cucumber	5	0.01	74-103	88	12	-	-	-	RAFVP101, CR
	2	0.1	84, 96	90	NA	-	-	-	
	3	1.0	82-89	86	4	-	-	-	
Dry bean (seed)	5	0.01	83-97	90	7.6	95-113	106	6.6	01414
	5	0.1	89-104	95	6.4	88-99	96	4.6	
Dry bean forage	7	0.01	86-114	95	10	-	-	-	RAFVN037, CR
	5	0.1	87-107	98	8	-	-	-	
Dry bean hay	7	0.01	82-109	92	10	-	-	-	RAFVN037, CR
	5	0.1	96-105	101	4	-	-	-	
Dry bean seed	7	0.01	83-113	93	11	-	-	-	RAFVN037, CR
	5	0.1	96-109	103	5	-	-	-	
Dry pea forage	7	0.01	90-113	102	9	-	-	-	RAFVN037, CR
	5	0.1	95-100	97	2	-	-	-	
Dry pea hay	7	0.01	80-108	95	10	-	-	-	RAFVN037, CR
	5	0.1	86-101	94	6	-	-	-	
Dry pea seed	7	0.01	73-103	86	10	-	-	-	RAFVN037, CR
	5	0.1	85-94	91	4	-	-	-	
Field corn forage	14	0.01	78-99	88	12	91-114	100	10	SARS-15-06, CR&MV
	9	0.1	77-88	82	4	-	-	-	
	6	5	84-89	87	2	83-88	85	3	
Field corn	15	0.01	82-112	91	8	72-119	92	20	SARS-15-06,

Matrix	N°	Spike level [mg/kg]	Primary transition m/z 527.0 → 389.2			Confirmatory transition m/z 527.0 →373.7			Report Reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
grain	10	0.1	77-106	86	10	-	-	-	CR&MV
	12	5	63-86	73	9	73-80	77	4	
Field corn stover	13	0.01	79-101	88	8	70–96	84	12	SARS-15-06, CR&MV
	8	0.1	76-95	84	7	-	-	-	
	6	5	77-96	86	9	79-88	83	4	
Field corn (aspirated grain)	6	0.01	72-98	81	12	82-125	106	16	SARS-15-06, CR&MV
	1	0.1	74	74	NA	-	-	-	
	6	5	77-88	84	4	81-92	85	5	
Field corn flour	6	0.01	89-109	98	8	82-104	99	10	SARS-15-06, CR&MV
	1	0.1	93	93	NA	-	-	-	
	6	5	90-102	95	5	89-101	94	5	
Field corn grits	6	0.01	84-100	90	6	70–104	84	16	SARS-15-06, CR&MV
	1	0.1	102			-	-	-	
	6	5	83-98	92	6	83-97	91	6	
Field corn oil	6	0.01	77-88	83	5	76-107	89	14	SARS-15-06, CR&MV
	1	0.1	79			-	-	-	
	6	5	75-89	84	6	83-92	85	5	
Field corn meal	6	0.01	82-102	93	9	80-126	105	16	SARS-15-06, CR&MV
	1	0.1	92			-	-	-	
	6	5	91-107	98	6	90-104	96	6	
Field corn pollen	10	0.01	80-109	94	10	76-170	111	34	SARS-15-06, CR&MV
	10	5	74-105	93	13	83-104	96	8	
Field corn starch	6	0.01	91-101	96	4	83-114	96	12	SARS-15-06, CR&MV
	1	0.1	92	92	NA	-	-	-	
	6	5	83-100	89	7	81-92	87	5	
Garden pea seed (succulent without pods)	7	0.01	87-108	100	7	-	-	-	RAFVN035, CR
	5	0.1	92-105	98	6	-	-	-	
Grape (brunches of grapes)	5	0.01	86-103	93	7.7	98-107	104	3.8	01414
	5	0.1	90-102	96	5.4	90-98	95	3.6	
Grape	11	0.01	82-109	98	7	91-112	104	8	SARS-14-07, CR&MV
	6	0.1	93-105	99	5	-	-	-	
	9	5	78-114	85	13	77-85	81	4	
Grape juice	6	0.01	71-100	92	12	93-106	100	5	SARS-14-07, CR&MV
	1	0.1	101	101	NA	-	-	-	
	6	5	82-88	86	3	82-91	86	4	
Grape raisin	6	0.01	97-111	105	5	87-98	93	4	SARS-14-07, CR&MV
	1	0.1	93	93	NA	-	-	-	
	6	5	73-80	78	4	66-72	70	4	
Grapefruit	15	0.01	99-108	102	3	-	-	-	RAFVP089
	3	1.0	101-101	101	0	-	-	-	
Lemon	14	0.01	100-109	104	2	-	-	-	RAFVP089
	3	1.0	99-102	100	1	-	-	-	
Lettuce, head	9	0.01	87-104	92	6	85-101	90	7	SARS-15-12, CR & MV
	4	0.1	91-98	93	3	-	-	-	
	9	5	85-100	90	5	85-91	88	3	
Lettuce, leaf	10	0.01	82-108	96	10	86-128	106	16	SARS-14-11, CR & MV
	5	0.1	83-107	94	11	-	-	-	
	10	5	82-105	95	8	91-111	102	9	

Matrix	N°	Spike level [mg/kg]	Primary transition m/z 527.0 → 389.2			Confirmatory transition m/z 527.0 →373.7			Report Reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
Lima bean, seed	7	0.01	82-105	90	8	-	-	-	RAFVN035, CR
	5	0.1	99-104	102	2	-	-	-	
Mandarin fruit	13	0.01	99-107	102	2	-	-	-	RAFVP089, CR
	3	1.0	99-101	100	1	-	-	-	
Melon	6	0.01	85-91	88	2	-	-	-	RAFVP101, CR
	2	0.1	91, 75	83	NA	-	-	-	
	4	1.0	76-96	85	11	-	-	-	
Mustard greens	7	0.01	83-97	92	5	-	-	-	RAFVN036, CR
	3	0.25	99-101	100	1	-	-	-	
	3	8.0	96-97	96	1	-	-	-	
Mustard greens (cooked)	3	0.01	89-93	91	2	-	-	-	RAFVN036, CR
	3	2.5	101-102	102	1	-	-	-	
Mustard greens (washed, cooked)	3	0.01	85-93	89	4	-	-	-	
	3	2.5	102-105	103	1	-	-	-	
Onion, bulb	7	0.010	97-10	102	3	-	-	-	RAFVN039, CR
	3	0.1	98-101	100	1	-	-	-	
Orange fruit	19	0.01	92-105	101	4	-	-	-	RAFVP089, CR
	3	1.0	97-99	98	1	-	-	-	
Orange oil	3	0.01	105-110	107	3	-	-	-	RAFVN026
	3	2.0	100-103	102	2	-	-	-	
	3	4.0	102-103	103	1	-	-	-	
Orange juice	3	0.01	104-106	106	1	-	-	-	RAFVN026
	3	2.0	97-98	97	1	-	-	-	
Pear	15	0.01	112-119	115	2	-	-	-	RAFVP104-01
	3	0.25	112-114	113	1	-	-	-	
	3	0.75	105-107	106	1	-	-	-	
Pear	4	0.01	92-101	97	4	-	-	-	BCS-0532
	4	1.0	90-101	96	5	-	-	-	
Pear	4	0.01	82-91	89	5	-	-	-	BCS-0531
	4	1.0	83-91	86	5	-	-	-	
Peach	11	0.01	80-108	96	9	95-100	96	5	SARS-15-16, CR&MV
	6	0.1	76-97	90	8	-	-	-	
	11	5	87-112	99	8	88-104	94	7	
Pecan nutmeat	8	0.01	73-93	84	10	84-101	91	7	SARS-14-02, CR&MV
	3	0.1	69-91	77	16	-	-	-	
	8	5	80-95	84	5	81-92	86	5	
Pepper	10	0.01	82-104	95	8	62-106	87	19	SARS-14-20, CR&MV
	5	0.1	84-91	89	5	-	-	-	
	10	5	77-100	93	8	84-105	99	9	
Plum	10	0.01	82-105	96	7	100-123	110	8	SARS-14-01, CR&MV
	5	0.1	98-114	104	7	-	-	-	
	10	5	76-104	93	12	81-85	83	2	
Potato starch	3	0.01	109-114	111	3	-	-	-	RAFVP062
	3	2.0	95-99	97	2	-	-	-	
Potato tuber	16	0.01	74-112	99	10	-	-	-	RAFVP074, CR
	3	1.0	95-96	96	1	-	-	-	
Prune	6	0.01	95-111	101	6	87-121	103	13	SARS-14-01, CR&MV
	1	0.1	109	109	NA	-	-	-	
	6	5	83-103	89	9	84-94	89	4	

Matrix	N°	Spike level [mg/kg]	Primary transition m/z 527.0 → 389.2			Confirmatory transition m/z 527.0 →373.7			Report Reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
Rape (seed)	5	0.01	92-100	95	3.6	70–90	82	9.6	01414
	5	0.1	95-102	98	2.7	85-96	89	4.8	
Rapeseed seed	7	0.01	84-106	98	7	-	-	-	RAFVP084
	3	0.1	99-109	102	6	-	-	-	
Rice, bran	7	0.0103	80-98	84	7.7	-	-	-	RAVF0032, CR
	5	0.207	103-106	104	1.3	-	-	-	
Rice, brown	17	0.010	78-126	97	11	-	-	-	RAFV00414&RAFV0085, CR
	16	0.1	86-108	98	7	-	-	-	
Rice, hulls	7	0.0103	84-96	88	4.6	-	-	-	RAVF0032, CR
	5	3.103	95-98	96	0.7	-	-	-	
	5	3.620	88-95	91	2.8	-	-	-	
Rice, panicles	14	0.010	89-128	102	11	-	-	-	RAFV00414&RAFV0085, CR
	10	0.1	95-107	100	4	-	-	-	
	1	3.0	98	NA	NA	-	-	-	
	1	4.0	100	NA	NA	-	-	-	
Rice, polished	7	0.0103	81-90	86	3.8	-	-	-	RAVF0032, CR
	5	0.1034	105-116	110	3.3	-	-	-	
Rice, straw	21	0.010	69-112	88	13	-	-	-	RAFV00414&RAFV0085, CR
	20	0.1	70–104	91	9	-	-	-	
	1	6.0	93	NA	NA	-	-	-	
	1	7.0	87	NA	NA	-	-	-	
Rice, whole grain	21	0.010	68-119	94	15	-	-	-	RAFV00414&RAFV0085, CR
	20	0.1	88-113	99	8	-	-	-	
	2	2.0	96, 103	100	NA	-	-	-	
Rice, whole grain	7	0.0103	89-95	93	3.5	-	-	-	RAVF0032, CR
	5	1.034	96-100	98	1.8	-	-	-	
Rice, whole grain	8	0.010	74-99	88	12	-	-	-	I14-046 CR
	6	1.0	78-96	85	9.6	-	-	-	
Rice, whole plant (no roots)	14	0.010	70–125	96	15	-	-	-	RAFV00414&RAFV0085, CR
	11	0.1	88-100	93	4	-	-	-	
	1	4.0	104	NA	NA	-	-	-	
	1	5.0	92	NA	NA	-	-	-	
Rice, whole plant (no roots, panicles removed)	14	0.010	70–106	93	10	-	-	-	RAFV00414&RAFV0085, CR
	10	0.1	92-101	96	3	-	-	-	
	2	2.0	98-100	99	NA	-	-	-	
Snap bean	7	0.01	73-115	100	16	-	-	-	RAFVN033
	5	0.1	86-102	97	6	-	-	-	
Snow pea	7	0.01	84-126	98	14	-	-	-	RAFVN033
	5	0.1	95-109	102	5	-	-	-	
Sorghum fodder	7	0.01	90-103	97	5	-	-	-	RAFVN029
	3	0.1	101-103	102	1	-	-	-	
Sorghum forage	7	0.01	92-99	96	3	-	-	-	RAFVN029
	3	0.1	95-101	98	3	-	-	-	
Sorghum grain	7	0.01	96-106	98	4	-	-	-	RAFVN029
	3	0.1	99-100	100	1	-	-	-	
Soya bean aspirated grain fraction	6	0.01	83-92	87	5	62-96	82	16	SARS-15-03, CR&MV
	1	0.1	80	NA	NA	-	-	-	
	6	5	84-90	86	3	85-92	88	3	
Soybean	6	0.01	76-85	82	4	60-83	75	12	SARS-15-03,

Matrix	N°	Spike level [mg/kg]	Primary transition m/z 527.0 → 389.2			Confirmatory transition m/z 527.0 →373.7			Report Reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
forage	1	0.1	83	83	NA	-	-	-	CR&MV
	6	5	85-99	94	6	88-99	94	4	
Soya bean forage	7	0.01	89-98	94	3	-	-	-	RAFVP051
	3	0.1	95-101	98	3	-	-	-	
Soya bean hay	6	0.01	64-109	89	18	88-114	99	12	SARS-15-03, CR&MV
	1	0.1	109	NA	NA	-	-	-	
	6	5	78-95	84	7	78-83	81	3	
Soya bean hay	7	0.01	87-93	90	2	-	-	-	RAFVP051
	3	0.1	93-97	95	2	-	-	-	
Soya bean hulls	6	0.01	71-83	78	5	74-88	80	7	SARS-15-03, CR&MV
	1	0.1	67	NA	NA	-	-	-	
	6	5	73-83	79	5	77-85	82	4	
Soya bean meal	6	0.01	76-92	84	7	71-108	79	22	SARS-15-03, CR&MV
	1	0.1	84	NA	NA	-	-	-	
	6	5	80-87	83	7	78-89	82	5	
Soybean oil (refined)	6	0.01	63-78	73	7	70–83	77	6	SARS-15-03, CR&MV
	1	0.1	80	NA	NA	-	-	-	
	6	5	72-84	77	6	70–78	73	5	
Soybean seed	12	0.01	63-102	81	15	73-94	83	12	SARS-15-03, CR&MV
	7	0.1	65-99	84	13	-	-	-	
	12	5	64-88	76	10	72-73	72	1	
Soya bean seed, dry	7	0.01	89-95	92	2	-	-	-	RAFVP051
	3	0.1	97-100	98	2	-	-	-	
Spinach	11	0.01	75-106	86	12	66-106	88	21	SARS-14-14
	6	0.1	81-102	87	9	-	-	-	
	11	5	74-98	87	7	83-94	88	5	
Summer squash	5	0.01	82-95	89	6	-	-	-	RAFVP101
	2	0.1	97, 96	97	NA	-	-	-	
	3	1.0	92-94	93	1	-	-	-	
Sunflower seed	7	0.01	88-106	98	6	-	-	-	RAFVP030
	3	0.1	92-99	97	4	-	-	-	
Sweetcorn forage	12	0.01	78-124	90	13	85-129	97	19	SARS-15-05, CR&MV
	7	0.1	86-96	90	6	-	-	-	
	12	5	71-94	83	8	85-92	88	4	
Sweetcorn (kernals with husk removed)	12	0.01	73-125	101	14	85-102	88	16	SARS-15-05, CR&MV
	7	0.1	85-120	104	12	-	-	-	
	12	5	80-102	92	8	79-90	85	5	
Sweetcorn pollen	5	0.001	84-108	95	9	44-111	83	32	SARS-15-05, MV
	5	0.01	100-109	103	3	97-116	105	9	
Sweetcorn stover	14	0.01	55-112	87	18	70–103	84	15	SARS-15-05, CR&MV
	9	0.1	64-103	84	14	-	-	-	
	14	5	68-101	87	12	92-99	94	3	
Tomato / fruit	5	0.01	99-104	102	1.8	87-98	94	5.2	01414
	5	0.1	96-105	100	3.6	85-97	90	5.4	
Tomato fruit	14	0.01	87-110	97	8	95-142	114	16	SARS-14-19, CR&MV
	9	0.1	90-116	100	8	-	-	-	
	14	5	74-104	95	10	96-108	101	5	
Tomato paste	6	0.01	87-108	96	8	85-107	99	9	SARS-14-19, CR&MV
	1	0.1	88	88	NA	-	-	-	
	6	5	88-118	103	10	100-112	107	5	
Tomato	6	0.01	97-116	105	7	82-107	98	10	SARS-14-19,

Matrix	N°	Spike level [mg/kg]	Primary transition m/z 527.0 → 389.2			Confirmatory transition m/z 527.0 →373.7			Report Reference
			Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]	
puree	1	0.1	92	-	NA	-	-	-	CR&MV
	6	5	89-97	94	3	90-104	97	5	
Wheat (grain)	5	0.01	84-95	90	5.0	92-112	102	7.8	01414
	5	0.1	92-102	98	3.7	83-94	89	6.1	
Wheat forage	7	0.01	102-106	104	2	-	-	-	RAFVP086
	4	0.1	103-110	106	3	-	-	-	
Wheat forage	9	0.01	92-100	96	3	-	-	-	RAFVP051
	5	0.1	96-100	99	2	-	-	-	
Wheat grain	11	0.01	89-107	99	7	-	-	-	RAFVP086
	5	0.1	102-110	107	3	-	-	-	
Wheat grain	8	0.01	94-108	100	4	-	-	-	RAFVP051
	3	0.1	98-105	102	4	-	-	-	
Wheat hay	7	0.01	97-113	105	5	-	-	-	RAFVP086
	4	0.1	100-105	102	3	-	-	-	
Wheat hay	9	0.01	92-99	96	2	-	-	-	RAFVP051
	5	0.1	96-100	99	2	-	-	-	
Wheat straw	7	0.01	91-115	102	7	-	-	-	RAFVP086
	5	0.1	103-109	105	3	-	-	-	
Wheat straw	9	0.01	94-108	103	5	-	-	-	RAFVP051
	5	0.1	95-109	103	6	-	-	-	

Notes:
NA = Not applicable; CR = Concurrent Recovery; MV = Method Validation.

Animal commodities

Enforcement methods

LC-MS/MS Method FV-002-A16-01 determines tetraniliprole, and its metabolites in animal commodities (Williams, 2016a, M-545487-01-2, Report FV-002-A16-01). The method was described and validated by in house validation studies in ruminant matrices and poultry muscle (Williams, 2016b, M-563847-01-1, Report RAFVP046) and in poultry matrices (Williams & Jerkins, 2016, M-569448-01-1, Report RAFV0033).

Samples of 5 ± 0.05 g were weighed into a 50-mL conical tube and fortified with the appropriate mixed standard solution. A mixture of 4:1 acetonitrile:water (~20 mL) was added to each sample as well as ~0.20 mL of formic acid. The samples were left to macerate for ~2 minutes prior to centrifugation (~5 minutes at ~3000 G). The extract was decanted and filtered with a SPE reservoir, filter paper, or other equivalent. Another ~20 mL of 4:1 acetonitrile:water was added to each sample, followed again by maceration, centrifugation and filtering. A Mixed Internal Standard Solution (0.250 mL of the 1.0 µg/mL) was added and the sample was further diluted to ~50 mL 4:1 v/v acetonitrile:water. For fat samples a slightly different approach was used with the first extraction step. In addition to the acetonitrile:water and formic acid also ~50 mL of hexane was added. After centrifugation at 2000G the hexane layer was discarded and the bottom layer was transferred to the filter.

For further sample preparation a 2 mL aliquot was transferred to a culture tube and dried to completeness in a turbovap at 50 °C, reconstituted in approximately 1 mL of 0.1 percent aqueous formic acid, mixed well and transferred to a C18 cartridge prewashed with about 1 mL of acetonitrile followed by water. The sample was eluted into an HPLC vial with 0.5 mL 4:1 v/v acetonitrile:water and 0.5 mL 0.1 percent aqueous formic acid, capped and mixed.

Samples were analysed by LC-MS/MS at m/z 545.1 to 356.1 (parent) and m/z 527.0 to 389.1 (tetraniliprole-N-methyl-quinazolinone), and 561.1 to 356.1 (tetraniliprole-benzylalcohol). Calibration was applied with internal standards in solvent (20/80/2 (v/v) water/acetonitrile/acetic acid) using linear regression with 1/× weighting. The reported LOQ for each analyte was 0.01 mg/kg. Validation results are shown in Tables 55 to Table 57.

An independent laboratory validation (ILV) was performed on ruminant milk and liver (Reed, 2016b, M-564372-01-1, Report number 034822-1/RAFVP0033). Furthermore, concurrent recoveries for parent and both metabolites tetraniliprole-N-methylquinazoline and tetraniliprole-benzylalcohol in cream, skim milk, milt, fats, kidney, liver and muscle from a dairy feeding study were available (Williams, 2016c, M-569181-01-1, Report RAFVP037). All results are shown in Table 55 to Table 57.

Average recoveries for the confirmation mass transitions at m/z 545.1 to 376.1 (tetraniliprole) and m/z 527.0 to 248.1 (tetraniliprole-N-methyl-quinazoline), and 561.1 to 392.0 (tetraniliprole-benzylalcohol) ranged from 75–107 percent for parent, 76–111 percent for tetraniliprole-benzylalcohol, and 76–107 percent for tetraniliprole-N-methylquinazoline in all animal tissues at 0.01-0.10 mg/kg (n=5 at each level). Control samples at these transitions were below 0.3 LOQ. Linearity at these transitions was confirmed in the range 0.1–100 µg/L.

Table 55 Recoveries for the validation of method FV-002-A16-01 for determination of tetraniliprole in animal matrices with reported LOQ of 0.01 mg/kg

Matrix	Spike level mg/kg	n	Primary transition m/z 545.1 → 356.1		Confirmatory transition m/z 545.1 → 376.1		Control samples ng/g (n)	Calibration internal standards	Report reference
			% Recovery mean range	RSD (%)	% Recovery mean range	RSD (%)			
Poultry Eggs	0.01	7	81 77–82	2.2	83 80 – 85	2.3	0.43 (3), 0.33 (3)	0.1-100 ng/mL, 1/x weighted, r≥0.999	RAFV0033 IHLV
	0.1	5	93 93–94	0.6	96 93–98	2.3			
Poultry Liver	0.01	7	83 80–90	4.0	81 75–88	4.7	0.04 (3), 0.06 (3)		
	0.1	5	88 75–93	8.4	87 74–93	8.6			
Poultry, Muscle [a]	0.01	7	96 95–97	0.8	98 96–100	1.6	0.17 (3), 0.15 (3)		
	0.1	5	98 96–98	0.7	99 98–100	0.8			
Ruminant Milk	0.01	7	98 96–99	0.8	99 97–100	1.1	0.13 (3), 0.14 (3)		
	0.1	5	99 98–100	0.7	100 99–101	0.8			
Ruminant Kidney	0.01	7	96 93–99	2.0	99 97–100	1.2	0.04 (3), 0.08 (3)		
	0.1	5	97 95–99	1.4	99 98–100	0.8			
Ruminant Fat	0.01	7	97 93–99	2.2	96 93–99	1.9	0.00 (3), 0.00 (3)		
	0.1	5	96 95–97	0.7	96 95–97	0.7			
Ruminant Liver	0.01	7	97 95–98	0.8	98 94–104	3.6	0.21 (3), 0.24 (3)		
	0.1	5	96 95–97	1.0	98 97–100	1.4			
Ruminant Milk	0.01	5	102 98–104	2.2	101 96–104	3.1	<0.2 LOQ (2)	0.1-100 ng/mL, r≥0.9998	034822/ RAFVP033 ILV
	0.1	5	85 78–88	5.3	84 76–89	6.3			
Ruminant Liver	0.01	5	98 96–101	2.2	98 95–103	3.3	<0.2 LOQ (2)		
	0.1	5	100 98–103	2.2	102 100–103	1.1			
Ruminant Cream	0.01	3	93 90–97	4.1	-	-	<0.2 LOQ (1/run)	0.10-500 ng/mL, r>0.99	RAFVP037 Dairy feeding study
	0.5	3	97 94–100	3.1	-	-			
Ruminant Omen. fat	0.01	4	84 80–88	4.9	-	-			
	1.1	3	102 101–104	1.7	-	-			
Ruminant Periren. fat	0.01	4	86 84–89	2.5	-	-			
	1.1	3	103 101–104	1.7	-	-			
Ruminant Subcut. fat	0.01	4	84 78–90	6.0	-	-			
	1.1	3	96 95–96	0.6	-	-			
RuminantKidney	0.01	4	87 85–88	1.7	-	-			
	0.03	3	86 85–88	1.8	-	-			
	0.30	5	101 96–107	4.3	-	-			

Matrix	Spike level mg/kg	n	Primary transition m/z 545.1 → 356.1		Confirmatory transition m/z 545.1 → 376.1		Control samples ng/g (n)	Calibration internal standards	Report reference
			% Recovery mean range	RSD (%)	% Recovery mean range	RSD (%)			
Ruminant Liver	0.01	5	86 79–92	6.5	-	-			
	1.6	3	101 101–101	0	-	-			
Ruminant Milk	0.01	14	95 83–102	6.2	-	-			
	0.3	3	104 101–108	3.5	-	-			
Ruminant Muscle	0.01	4	86 83–90	3.5	-	-			
	0.1	3	79 70–84	3.5	-	-			
Skim Milk	0.01	4	96 95–96	0.6	-	-			
	0.2	3	95 90–98	4.6	-	-			

Notes:
IHLV = In House Laboratory Validation.
[a] Results also reported in Williams & Jerkins, 2016, M-569448-01-1, Report RAFV0033.

Table 56 Recoveries for the validation of method FV-002-A16-01 for determination of tetraniliprole-N-methylquinazolinone in animal matrices with reported LOQ of 0.01 mg/kg

Matrix	Spike level (mg/kg)	n	Primary transition m/z 527.0 → 389.1			Confirmatory transition m/z 527.0 → 248.1			Control samples mg/kg (n)	Calibration, internal standards	Report reference
			% recovery mean	range	RSD (%)	% recovery mean	range	RSD (%)			
Poultry muscle [a]	0.01	7	99	97–101	1.5	96	93–98	1.9	0.09 (3), 0.12 (3)	0.1-100 ng/mL, 1/x weighted, r≥0.999	RAFVP046 IHLV
	0.1	5	98	97–99	1.0	96	95–97	0.9			
Ruminant milk	0.01	7	98	96–100	1.2	97	94–99	1.6	0.18 (3), 0.21 (3)		
	0.1	5	98	97–99	1.0	96	96–98	0.9			
Ruminant kidney	0.01	7	104	101–107	2.1	100	96–105	3.3	0.15 (3), 0.00 (3)		
	0.1	5	99	98–101	1.2	94	92–94	1.0			
Ruminant fat	0.01	7	99	98–100	1.0	95	93–97	1.8	0.02 (3), 0.00 (3)		
	0.1	5	94	92–95	1.1	92	90–93	1.4			
Ruminant liver	0.01	7	105	102–107	1.6	98	96–101	1.7	0.21 (3), 0.14 (3)		
	0.1	5	102	99–105	2.2	95	93–97	1.5			
Ruminant milk	0.01	5	105	100–108	3.7	103	96–106	3.9	<0.2 LOQ (2)	0.1-100 ng/mL, r≥0.9996	034822/ RAFVP033 ILV
	0.1	5	82	76–88	5.7	82	75–90	8.0			
Ruminant liver	0.01	5	101	96–107	4.6	97	90–104	5.3	<0.2 LOQ (2)		
	0.1	5	105	101–107	2.2	100	96–105	4.0			
Ruminant Cream	0.01	3	96	94–97	1.6	-		-			RAFVP037 Dairy feeding study
	0.50	3	85	83–86	1.8	-		-			
RuminantOmen. fat	0.01	4	96	91–102	4.7	-		-			
	1.1	3	94	94–95	0.6	-		-			
Ruminant Periren. fat	0.01	4	95	91–98	3.1	-		-			
	1.1	3	95	94–96	1.1	-		-			
Ruminant Subcut. fat	0.01	4	93	90–97	3.5	-		-			
	1.1	3	87	86–88	1.3	-		-			
RuminantKidney	0.01	4	96	92–98	2.8	-		-			
	0.03	3	86	84–88	2.4	-		-			
	0.30	5	90	87–99	5.5	-		-			
Ruminant Liver	0.01	5	95	87–102	5.9	-		-			
	1.6	3	89	88–91	1.7	-		-			
Ruminant Milk	0.01	14	99	91–102	2.8	-		-			
	0.3	3	94	91–96	2.7	-		-			
Ruminant Muscle	0.01	4	99	94–104	4.3	-		-			
	0.1	3	91	82–95	8.3	-		-			
Skim Milk	0.01	4	100	99–100	0.6	-		-			

Matrix	Spike level (mg/kg)	n	Primary transition m/z 527.0 → 389.1		Confirmatory transition m/z 527.0 → 248.1		Control samples mg/kg (n)	Calibration, internal standards	Report reference
			% recovery mean range	RSD (%)	% recovery mean range	RSD (%)			
	0.2	3	81 75–85	6.8	-	-			

Notes:
[a] Results also reported in Williams & Jerkins, 2016, M-569448-01-1, Report RAFV0033.
IHLV = In House Laboratory Validation.

Table 57 Recoveries for the validation of method FV-002-A16-01 for determination of tetraniliprole-benzylacohol in animal matrices with reported LOQ of 0.01 mg/kg

Matrix	Spike level (mg/kg)	n	Primary transition m/z 561.1 →356.1			Confirmatory transition m/z 561.1 → 392.0			Control Samples mg/kg (n)	Calibration Internal standard	Report reference
			% recovery mean	range	RSD (%)	% recovery mean	range	RSD (%)			
Poultry muscle [a]	0.01	7	101	94–107	4.0	101	95–104	3.1	0.12 (3), 0.21 (3)	0.1–100 ng/mL, 1/x weighted, r≥0.999	RAFVP046 IHLV
	0.1	5	102	100–104	1.6	106	103–111	3.0			
Ruminant milk	0.01	7	101	99–103	1.4	103	101–106	1.5	0.20 (3), 0.24 (3)		
	0.1	5	102	97–105	3.2	103	99–105	2.1			
Ruminant kidney	0.01	7	99	95–102	2.1	102	99–106	2.6	0.00 (3), 0.00 (3)		
	0.1	5	99	95–101	3.0	104	102–106	1.7			
Ruminant fat	0.01	7	98	91–105	4.8	101	97–106	3.2	0.00 (3), 0.00 (3)		
	0.1	5	100	98–104	2.3	99	97–102	2.0			
Ruminant liver	0.01	7	92	84–97	4.6	102	94–108	4.9	0.34 (3), 0.00 (3)		
	0.1	5	94	90–99	3.8	100	96–106	3.7			
Ruminant milk	0.01	5	109	106–113	2.5	109	105–113	2.6	<0.2 LOQ (2)	0.1–100 ng/mL, r≥0.9998	034822/ RAFVP033 ILV
	0.1	5	81	76–85	5.4	83	76–88	6.4			
Ruminant liver	0.01	5	95	89–102	7.2	101	96–107	4.3	<0.2 LOQ (2)		
	0.1	5	99	96–101	1.9	101	98–104	2.2			
Ruminant Cream	0.01	3	104	103–105	1.0	-		-	<0.2 LOQ (1/run)	0.10–500 ng/mL, r>0.99	RAFVP037 Dairy feeding study
	0.50	3	109	106–111	2.3	-		-			
RuminantOmen. fat	0.01	4	85	79–94	7.6	-		-			
	1.1	3	97	94–100	3.1	-		-			
Ruminant Periren. fat	0.01	4	87	82–95	6.9	-		-			
	1.1	3	97	93–99	3.3	-		-			
Ruminant Subcut. fat	0.01	4	84	77–97	11	-		-			
	1.1	3	88	87–90	1.7	-		-			
RuminantKidney	0.01	4	91	85–97	5.8	-		-			
	0.03	3	96	89–103	7.3	-		-			
	0.30	5	97	90–103	5.9	-		-			
Ruminant Liver	0.01	5	88	84–93	3.9	-		-			
	1.6	3	100	98–103	2.5	-		-			
Ruminant Milk	0.01	14	101	86–119	6.9	-		-			
	0.3	3	112	108–119	5.4	-		-			
Ruminant Muscle	0.01	4	86	84–87	2.0	-		-			
	0.1	3	77	67–83	11	-		-			
Skim Milk	0.01	4	108	106–11	2.4	-		-			
	0.2	3	104	97–115	9.3	-		-			

Notes:
IHLV = In House Laboratory Validation.
[a] Results also reported in Williams & Jerkins, 2016, M-569448-01-1, Report RAFV0033.

Soil

HPLC-MS/MS method 01373 (Freitag & Koch, 2015, M-486110-02-1, Amendment to Report MR-13/100, Report RAFVP019) determines tetraniliprole and its metabolites tetraniliprole-N-methylquinazoline, tetraniliprole-amide, tetraniliprole-carboxylic acid, tetraniliprole-desmethyl-amide-carboxylic acid, tetraniliprole-N-methyl-quinazolinone-carboxylic acid and tetraniliprole-quinazolinone-carboxylic acid in soil and sediment.

Residues in 20 g soil samples are extracted for 15 minutes in a microwave extractor with a mixture (40 mL) of acetonitrile/water/acetic acid (400/100/3). The extracts are centrifuged to remove fine particles of the soil. Acetic acid (0.9 mL at 0.1 percent) is added to 0.1mL of sample. After further centrifugation to remove fine particle, the sample solution is ready for injection. Identification and quantitation of the active substance is done by high performance liquid chromatography using MS/MS detection in the Multiple Reaction Monitoring mode. The method was validated using three different soils "Höfchen", "Laacher Hof", "Dollendorf" and sediment. The recovery rates were determined for all analytes at fortification levels of 0.002 mg/kg (LOQ) and 0.02 mg/kg (10×LOQ).

Analytical method 01373 was successfully validated by an independent laboratory (ILV), analysing soil from Florida and Washington (Netzband&Jenks, 2016, M-554130-01-1, Report RAFVP017). The results are summarized in Table 58. The mean recovery values for each matrix were generally within the range of 70–120 percent, and RSD were generally less than 20 percent for all analytes at the LOQ and 10× LOQ fortification levels. The validation results for the determination of each of the analytes in soil are summarized in Table 58.

Table 58 Recoveries for the validation of method 01373 for determination of tetraniliprole and metabolites in soil with reported LOQ of 0.002 mg/kg (n=5)

Matrix	Spike level mg/kg	% Recovery mean	% Recovery range	RSD (%)	% Recovery mean	% Recovery range	RSD (%)	Control samples mg/kg	Calibration in solvent	Report reference
Tetraniliprole		Primary transition (m/z 545-356)			Confirmatory transition (m/z 545-376)					
Soil (Höfchen)	0.002	89	78–95	7.7	78	60–95	17	<0.3LOQ (n=4)	0.03–50 µg/L linear r^2>0.99	MR-13/100/ RAFVP019
	0.02	99	94–109	6.4	99	88–109	9.3			
Soil (Laacher)	0.002	104	100–108	3.4	96	67–99 [a]	17			
	0.02	97	89–102	5.1	95	86–100	6.2			
Soil (Dollendorf)	0.002	89	81–108	13	88	81–98	7.6			
	0.02	96	88–101	5.6	98	92–104	4.5			
Sediment	0.002	82	73–94	16	87	60–102	20			
	0.02	87	82–92	4.6	88	82–95	5.5			
Soil (Florida)	0.002	97	92–110	8	98	84–105	9	<0.3LOQ (n=2)	0.03–50 µg/L linear r^2>0.99	RAFVP017 ILV
	0.02	96	89–107	7	97	90–108	9			
Soil (Washington)	0.002	104	94–114	9	101	95–111	7			
	0.02	96	90–101	5	95	90–99	3			
Tetraniliprole-N-methylquinazolinone		Primary transition (m/z 527-389.1)			Confirmatory transition (m/z 527–374.1)					
Soil (Höfchen)	0.002	86	73–96	12	45	0–109 [b]	103	<0.3LOQ (n=4)	0.03–50 µg/L linear r^2>0.99	MR-13/100/ RAFVP019
	0.02	86	81–91	5.5	89	76–112	15			
Soil (Laacher)	0.002	86	76–95	10	80	61–98	20			
	0.02	92	76–98	10	88	79–95	6.9			
Soil (Dollendorf)	0.002	77	64–89	13	57	0–100 [b]	76			
	0.02	85	80–87	3.3	85	78–83	8.9			
Sediment	0.002	68	65–75	5.7	50	0–97 [b]	95			
	0.02	87	78–103	12	84	72–96	11			

Matrix	Spike level mg/kg	% Recovery mean	range	RSD (%)	% Recovery mean	range	RSD (%)	Control samples mg/kg	Calibration in solvent	Report reference
Soil (Florida)	0.002	81	75–88	8	104	88–122	13	<0.3LOQ (n=2)	0.03–50 µg/L r^2>0.99	RAFVP017 ILV
	0.02	102	96–114	7	102	94–110	6			
Soil (Washington)	0.002	90	78–105	11	94	73–111	17			
	0.02	107	100–113	5	106	97–111	6			
Tetraniliprole-amide		Primary transition (m/z 563-356.1)			Confirmatory transition (m/z 563-394.1)					
Soil (Höfchen)	0.002	99	86–111	10	92	72–114	20	<0.3LOQ (n=4)	in solvent 0.03–50 µg/L linear r^2>0.99	MR-13/100/ RAFVP019
	0.02	93	85–101	6.1	95	85–106	8.0			
Soil (Laacher)	0.002	89	76–99	10	83	66–101	16			
	0.02	100	93–109	6.5	96	92–103	4.5			
Soil (Dollendorf)	0.002	101	89–117	11	87	72–99	11			
	0.02	102	100–106	2.2	97	87–108	10			
Sediment	0.002	94	88–100	5.9	96	91–104	5.6			
	0.02	100	93–110	7.0	95	87–103	7.3			
Soil (Florida)	0.002	100	92–112	9	99	89–109	8	<0.3LOQ (n=2)	0.03–50 µg/L linear r^2>0.99	RAFVP017 ILV
	0.02	102	94–112	7	101	95–110	7			
Soil (Washington)	0.002	98	91–105	6	112	91–123	11			
	0.02	100	91–104	5	98	92–105	6			
Tetraniliprole-carboxylic acid		Primary transition (m/z 564-356)			Confirmatory transition (m/z 564-395)					
Soil (Höfchen)	0.002	98	83–106	8.9	107	92–116	8.9	<0.3LOQ (n=4)	0.03–50 µg/L linear r^2>0.99	MR-13/100/ RAFVP019
	0.02	88	83–95	6.0	90	84–98	5.8			
Soil (Laacher)	0.002	100	84–110	11	109	93–124	12			
	0.02	89	84–99	6.7	91	82–100	5.9			
Soil (Dollendorf)	0.002	99	87–118	12	102	93–112	7.2			
	0.02	92	82–101	9.6	95	85–102	7.1			
Sediment	0.002	92	78–101	13	91	81–102	9.8			
	0.02	87	79–95	6.6	87	82–94	5.6			
Soil (Florida)	0.002	88	73–95	12	94	74–111	15	<0.3LOQ (n=2)	0.03–50 µg/L linear r^2>0.99	RAFVP017 ILV
	0.02	96	89–101	6	96	92–99	3			
Soil (Washington)	0.002	98	79–110	12	101	88–111	9			
	0.02	99	93–101	6	99	90–108	7			
Tetraniliprole-desmethyl-amide-carboxylic acid		Primary transition (m/z 550-395.1)			Confirmatory transition (m/z 550-356)					
Soil (Höfchen)	0.002	103	92–109	6.9	96	72–109	15	<0.3LOQ (n=4)	0.03–50 µg/L linear r^2>0.99	MR-13/100/ RAFVP019
	0.02	90	75–99	11	88	75–96	9.2			
Soil (Laacher)	0.002	92	77–100	10	91	82–101	11			
	0.02	82	71–91	11	83	75–94	9.1			
Soil (Dollendorf)	0.002	101	87–115	10	95	72–112	17			
	0.02	88	84–97	6.0	90	84–94	4.4			
Sediment	0.002	83	71–103	17	95	68–114	19			
	0.02	79	75–83	3.7	78	75–80	3.3			
Soil (Florida)	0.002	83	70–102	18	72	59–849	14	<0.3LOQ (n=2)	0.03–50 µg/L linear r^2>0.99	RAFVP017 ILV
	0.02	99	91–105	6	95	92–97	2			
Soil (Washington)	0.002	91	73–114	19	94	81–114	16			
	0.02	112	104–117	5	110	105–117	4			
Tetraniliprole-N-methyl-quinazolinone-carboxylic acid		Primary transition (m/z 546-408.1)			Confirmatory transition (m/z 546-267.1)					
Soil (Höfchen)	0.002	89	72-105	15	[c]		[c]	<0.3LOQ (n=4)	standards in solvent 0.03 -50 µg/L 1/x r^2>0.99	MR-13/100/ RAFVP019
	0.02	89	81-95	5.8	106	82-131	19			
Soil (Laacher)	0.002	86	73-102	14	[c]		[c]			
	0.02	91	80-102	11	96	86-105	9.5			

Matrix	Spike level mg/kg	% Recovery mean	range	RSD (%)	% Recovery mean	range	RSD (%)	Control samples mg/kg	Calibration in solvent	Report reference
Soil (Dollendorf)	0.002	87	77-108	14	[c]		[c]			
	0.02	94	86-101	6.0	95	71-115	20			
Sediment	0.002	83	60-94	16	[c]		[c]			
	0.02	96	89-103	6.3	107	102-113	4.9			
Soil (Florida)	0.002	84	66-95	13	87	70–155 [d]	16	<0.3LOQ (n=2)	0.03 -50 µg/L linear r^2>0.99	RAFVP017 ILV
	0.02	110	105-120	6	113	106-119	4			
Soil (Washington)	0.002	96	85-108	9	88	73-105	14			
	0.02	111	107-121	5	106	95-119	8			
Tetraniliprole-quinalolinone-carboxilic acid		Primary transition (m/z 532-394.1)			Confirmatory transition (m/z 532-366.1)					
Soil (Höfchen)	0.002	105	95-117	9.2	[c]		[c]	<0.3LOQ (n=4)	standards in solvent 0.03 -50 µg/L 1/x r^2>0.99	MR-13/100/ RAFVP019
	0.02	82	69-99	14	80	63-97	19			
Soil (Laacher)	0.002	97	91-108	7.7	[c]		[c]			
	0.02	91	77-110	17	88	81-96	7.3			
Soil (Dollendorf)	0.002	102	92-118	10	[c]		[c]			
	0.02	85	71-99	12	95	76-113	19			
Sediment	0.002	97	75-119	17	[c]		[c]			
	0.02	86	75-103	13	87	71-112	18			
Soil (Florida)	0.002	76	65-91	14	91	69-121	26	<0.3LOQ (n=2)	0.03 -50 µg/L linear r^2>0.99	RAFVP017 ILV
	0.02	92	88-98	4	106	99-112	5			
Soil (Washington)	0.002	99	91-107	7	88	78-96	8			
	0.02	110	105-116	4	109	101-143[e]	9			

Notes:
ILV = Independent Laboratory Validation.
[a] n=4.
[b] Due to a not given sensitivity this fortification level could not be used.
[c] Due to poor sensitivity at this fortification level no acceptable data was generated.
[d] Value of 155% is not included in calculations.
[e] Value of 143% is not included in calculations.

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

Storage stability of spiked residues in plant commodities

Storage stability was investigated by spiking tomato, dry bean (seed), wheat (grain), rape (seed) and grape (bunch of grapes) with 0.20 mg/kg of parent and metabolite tetraniliprole-N-methylquinazolinone [Uceda, 2016, M-565221-01-1, Report 14-10]. The samples stored in High Density Poly Ethylene (HDPE) Nalgene containers at an average temperature of -18 °C or below, were analysed at the nominal storage intervals of 0, 3, 6, 9, 12, 18 and 24 months using analytical method 01414 (see section on analytical methods). The LOQ of the method was 0.01 mg/kg for each compound. Average concurrent fresh recoveries were within the range of 70–110 percent, with RDS <20 percent, except for one recovery mean of 111 percent at the storage interval of 196 days for parent tetraniliprole in dry bean (seed). Control samples had residues below the 0.3LOQ.

Storage stability results (not corrected for concurrent recovery) and concurrent recoveries for the tetraniliprole and tetraniliprole-N-methylquinazolinone are shown in Table 59 and Table 60, respectively.

Table 59 Storage stability at ≤ -18 °C in commodities spiked with 0.2 mg/kg of tetraniliprole and tetraniliprole-N-methylquinazolinone

Commodity	Storage period (days)	% remaining	Mean (%)	% remaining, normalized at day 0	Mean % concurrent recoveries
Tomato	0	110, 107, 106	108	100	109
	126	98, 99	99	91	104
	197	104 (2)	104	97	105
	288	93, 95	94	87	105
	372	96, 97	97	90	101
	565	97, 96	97	90	102
	735	102, 97, 105	103	96	106
Dry bean (seed)	0	103, 101, 94	99	100	101
	125	97, 103	100	101	100
	196	103, 106	105	105	111
	286	100, 97	99	99	97
	371	96, 96	96	97	99
	564	102, 98	100	101	99
	734	100, 98, 102	100	101	99
Wheat (grain)	0	110, 100, 102	104	100	108
	127	100, 98	99	95	100
	199	105, 106	106	101	104
	290	95, 96	96	92	100
	374	98, 98	98	94	99
	567	101, 99	100	96	101
	738	100, 105, 100	102	98	104
Oilseed rape (seed)	0	99 (3)	99	100	96
	126	101, 99	100	101	101
	198	106, 107	107	108	107
	290	92, 96	94	95	97
	373	98, 98	98	99	97
	567	101, 99	100	101	105
	737-741	100, 104, 96, 97, 98	99	100	104
Grape	0	109, 105, 103	106	100	107
	125	96, 95	96	90	102
	196	105, 102	104	98	109
	287	90, 93	92	87	100
	371	94, 101	98	92	97
	564	98, 98	98	93	101
	734	100, 102, 104	102	97	107

Table 60 Storage stability at ≤ -18 °C in commodities spiked with 0.2 mg/kg of tetraniliprole-N-methylquinazolinone

Commodity	Storage period (days)	% remaining	Mean (%)	% remaining, normalized at day 0	Mean % concurrent recoveries
Tomato	0	91, 87, 91	90	100	91
	126	103, 103	103	115	108
	197	87, 88	88	98	92
	288	102, 101	102	113	110
	372	98, 102	100	112	101
	565	103, 102	103	114	105
	735	109, 103,106	108	120	106
Dry bean (seed)	0	89, 84, 88	87	100	86
	125	100, 98	99	114	98

Commodity	Storage period (days)	% remaining	Mean (%)	% remaining, normalized at day 0	Mean % concurrent recoveries
	196	89, 87	88	101	93
	286	96, 96	96	110	98
	371	98, 95	97	111	100
	564	96, 99	98	112	102
	734	98, 99, 98	98	113	98
Wheat (grain)	0	97 (3)	97	100	101
	127	102, 103	103	106	102
	199	97, 96	97	99	99
	290	97, 99	98	101	104
	374	101, 101	101	104	101
	567	99, 101	100	103	103
	738	103, 103,104	103	107	105
Oilseed rape (seed)	0	98, 99, 96	98	100	94
	126	97, 97	97	99	100
	198	94, 96	95	97	99
	290	96, 98	97	99	100
	373	104, 103	104	106	104
	567	101, 105	103	105	106
	737	102, 102, 104	103	105	104
Grape	0	92, 91, 89	91	100	94
	125	101, 102	102	112	105
	196	89, 90	90	99	94
	287	98, 100	99	109	105
	371	100, 102	101	111	101
	564	102, 99	101	111	105
	734	105 (3)	105	116	107

Residues of tetraniliprole and its metabolite tetraniliprole-N-methylquinazolinone are stable for at least 24 months in crop commodities representative of high water (tomato), high oil (oilseed rape), high protein (dry bean), high starch (wheat grain) and high acid (grape) commodity groups when stored under frozen at or below -18 °C.

Storage stability of residues in animal commodities

All samples obtained from livestock feeding studies (see section on farm animal feeding) were extracted and analysed within 30 days of their frozen storage and therefore it was not necessary to conduct freezer storage stability studies for animal products. Additionally, in the animal metabolism studies, samples were extracted and analysed within 5 months.

USE PATTERNS

Tetraniliprole is used as an insecticide on various crops grown under field conditions, applied either by foliar spray, soil applications, in-furrow, or drip chemigation. Tetraniliprole is toxic to bees and should therefore never be applied during bloom.

The Canadian and United States labels for foliar and seed treatments indicate a replanting interval for rotational crops of 30 days for root vegetables, leaves of root and tuber vegetables, bulb vegetables, legume vegetables (except soybeans); foliage of legume vegetables (except soybeans); cucurbits; cereal grains (except corn); forage, fodder, and straw of cereal grains (except corn); rapeseeds; sunflowers, and alfalfa. For all other rotational crops an interval of 120 days is advised. No restrictions or recommendations for the use of adjuvants are given in either the Canadian or United States labels. The registered use patterns that are supported with data are summarized in Table 61.

Table 61 Registered uses of tetraniliprole in the field

Crop	Country	Form	Application method and field or glass house					PHI, days
			Method	Rate g ai/ha (timing)	Spray conc, g ai/hL	Number (maximum seasonal rate, g ai/ha)	Interval, days	
Citrus fruit [a]	United States	200 SC	foliar	45-60 [b]	48-65	3 (180)	5	1
Citrus fruit [a]	United States	200 SC	soil	100-120 [c]	27-32	1 (120)	-	1
Citrus fruit [a]	United States	200 SC	soil + foliar	soil: 100-120 foliar: 45-60 [b] [c]	soil: 27-32 foliar: 48-65	1 + 1 (180; soil + foliar)	20	1
Citrus fruit [a]	United States	200 SC	drip chemigation	120 [c]	n.a.	1 (120)	-	1
Citrus fruit [a]	United States	200 SC	drip chemigation + foliar	drip: 100-120 foliar: 45-60 [b] [c]	drip: n.a. foliar: 27-32	1 + 1 (180; 120 drip + 60 foliar)	20	1
Citrus	Korea	200 SC	foliar	100	4 (2500 L/ha)	2 (200)	7-10	14
Oranges	Cambodia	200 SC	foliar	30	6 (500 L/ha)	1	n.a.	7
Pome fruit [d]	United States	200 SC	foliar	40-60 [e]	2.2-13 (ground)	3 (180)	7	7
Pome fruit [d]	Canada	200 SC	foliar	30-60 [n]	6.7-13.3 (450 L/ha, ground)	3 (180)	7	7
Apple	Korea	200 SC	foliar	100	4 (2500 L/ha)	2 (200)	7-10	14
Pear	Korea	200 SC	foliar	100	4 (2500 L/ha)	2	7-10	14
Sweet persimmon	Korea	200 SC	foliar	100	4 (2500 L/ha)	2	10	14
Stone fruit [f]	United States	200 SC	foliar	40-60 [e]	2.2-13 (ground)	3 (180)	7	5
Stone fruit [f]	Canada	200 SC	foliar	30-60 [n]	230 L/ha (ground)	3 (180)	7	5
Peach	Korea	200 SC	foliar	100	4 (2500 L/ha)	2	7-10	14
Plum	Korea	200 SC	foliar	100	4 (2500 L/ha)	2	7	14
Small fruit vine climbing [g]	United States	200 SC	foliar	30-45 [e]	32-48 (ground)	4 (180)	7	14
Small fruit vine climbing [g]	Canada	200 SC	foliar	45 [n]	10 450 L/ha (ground)	4 (180)	7-10	14
Small fruit vine	United	200	drip	30-45 [e]	n.a.	4	7	14

Crop	Country	Form	Application method and field or glass house					PHI, days
			Method	Rate g ai/ha (timing)	Spray conc, g ai/hL	Number (maximum seasonal rate, g ai/ha)	Interval, days	
climbing [g]	States	SC	chemigation			(180)		
Small fruit vine climbing [g]	United States	200 SC	drip chemigation + foliar	30-45 [e]	drip: n.a. foliar: 32-48 (ground)	1 + 3 (180; drip + foliar)	7	14
Grape	Korea	200 SC	foliar	6	4 (1500 L/ha)	2	10	14
Brassica Head and Stem [i]	United States	200 SC	foliar	30-45	32-48 (ground) 160-240 (air)	4 (180)	5	1
Brassica Head and Stem [i]	Canada	200 SC	foliar	30-45	30-45 (100 L/ha, ground)	4 (180)	5	1
Brassica Head and Stem [i]	United States	200 SC	drip chemigation	30-45	n.a.	1	-	1
Brassica Head and Stem [i]	United States	200 SC	drip chemigation + foliar	30-45	drip: n.a. foliar: 32-48 (ground) 160-240 (air)	1 + 3 (180; drip + foliar)	5	1
Kimchi cabbage	Korea	200 SC	foliar	48	40 (1200 L/ha)	2	10	14
Fruiting vegetables, other than cucurbits [j]	United States	200 SC	foliar	30-45 [k]	32-48 (ground) 160-240 (air)	4 (180)	5	1
Fruiting vegetables, other than cucurbits [j]	Canada	200 SC	foliar	30 [n]	20 (150 L/ha, ground)	4 (120)	5	1
Fruiting vegetables, other than cucurbits [j]	United States	200 SC	drip chemigation	61-200 [n]	n.a.	1 (200)	-	1
Fruiting vegetables, other than cucurbits [j]	United States	200 SC	drip chemigation + foliar	drip: 30-200 [k,n] foliar: 45	drip: n.a. foliar: 32-48 (ground) 160-240 (air)	1 + 3 (200 g; drip + foliar)	5	1
Peppers	Cambodia	200 SC	foliar	30	6-7.5 (400-500 L/ha)	1	n.a.	7
Peppers	Korea	200 SC	foliar	60	4 (1500 L/ha)	2	10	3
Tomato	Korea	200 SC	foliar	60	3.3 g ai/hL (1800	2	10	3

Crop	Country	Form	Application method and field or glass house					PHI, days
			Method	Rate g ai/ha (timing)	Spray conc, g ai/hL	Number (maximum seasonal rate, g ai/ha)	Interval, days	
					L/ha)			
Leafy vegetables [l]	United States	200 SC	foliar	30-45 [h]	32-48 (ground) 160-240 (air)	4 (180)	5	1
Leafy vegetables [l]	Canada	200 SC	foliar	30-45 [h]	30-45 (100 L/ha, ground)	4 (180)	5	1
Leafy vegetables [l]	United States	200 SC	drip chemigation	61-200 [h]	n.a.	1 (200)	n.a.	1
Leafy vegetables [l]	United States	200 SC	drip chemigation + foliar	drip: 30-200 [h] foliar: 30-45	drip: n.a. foliar: 32-48 (ground) 160-240 (air)	1 + 3 (200)	5	1
Soya bean [do not feed or graze soya bean hay or forage for livestock feed]	United States	200 SC	foliar	30-50 [n]	32-54 (ground) 160-270 (air)	4 (200)	3	14
	Canada	200 SC	foliar	30 [n]	30 g ai/hL (100 L/ha, ground)	2 (60)	5	14
	United States	200 SC	in-furrow soil + foliar	in-furrow: 30-200 foliar: 30-45	in-furrow: n.a. foliar: 32-54 (ground) 160-270 (air)	1 + 4 (200; in-furrow + foliar)	n.a. (3)	14
Soya bean	United States	200 SC	in-furrow soil	30-200	n.a.	1	n.a.	n.a.
Soya bean	United States	480 FS	seed treatment	0.045-0.0675 mg ai/kernel	n.a.	1 n.a.	n.a.	n.a.
Soya bean	Canada	480 FS	seed treatment	0.045-0.0675 mg ai/kernel [p]	13-20 mL product/ 140 000 seeds	1 n.a.	n.a.	n.a.
Soya bean	India	200 SC	foliar	50-60	in 500 L water	2	10-15	35
Soya bean	Cambodia	200 SC	foliar	30	6 (500 L/ha)	1	n.a.	7
Tuberous and corm vegetables [m]	United States	200 SC	foliar	30 [n]	162 (ground) 32 (air)	4 (120)	5	14
Tuberous and corm vegetables [m]	Canada	200 SC	foliar	30 [n]	30-60 (100 L/ha by ground and 50 L/ha	2 (60)	10	14

Crop	Country	Form	Application method and field or glass house					PHI, days
			Method	Rate g ai/ha (timing)	Spray conc, g ai/hL	Number (maximum seasonal rate, g ai/ha)	Interval, days	
					for aerial (potato only)			
Tuburous and corm vegetables [m]	United States	200 SC	in-furrow soil at planting	100-200	n.a.	1	n.a.	n.a.
Tuburous and corm vegetables [m]	United States	200 SC	in-furrow soil + foliar	in-furrow: 100-200 foliar: 30 [n]	n.a. foliar: 162 (ground) 32 (air)	1 + 4 (200)	n.a. (5)	14
Tuburous and corm vegetables [m]	Canada	200 SC	in-furrow soil	150	300 (50 L/ha)	1 (150)	n.a.	n.a.
Tuburous and corm vegetables [m]	United States	200 SC	drip chemigation	100-200 [n]	n.a.	1 (200)	n.a.	14
Tuburous and corm vegetables [m]	United States	200 SC	drip chemigation + foliar	drip: 100-200 foliar: 30 [n]	drip: n.a. foliar: 162 (ground) 32 (air)	1 + 4 (200)	n.a. (5)	14
Potato	Cambodia	200 SC	foliar	30	6 (500 L/ha)	1	n.a.	7
Rice	India	200 SC	foliar	50-60	10-12 (500 L/ha)	2 (120)	11-15	43
Rice	Japan	480 FS	seed treatment [q]	264 g ai/ha	11 mL product/kg seed	1	n.a.	n.a.
Rice	Cambodia	200 SC	foliar	30	6 (500 L/ha)	1	n.a.	7
Rice	Korea	200 SC	foliar	30	2 g ai/hL (1500 L/ha)	2	7	14
Maize/Corn (field corn, pop corn and corn grown for seed) [do not feed forage or stover to livestock within PHI 14 days]	United States	200 SC	foliar	30-50 [r]	32-54 (ground) 160-270 (air)	4 (200)	7	14
	Canada	200 SC	foliar	30 [n]	30 (100 L/ha, ground)	4 (120)	7	14
	United States	200 SC	in-furrow soil at planting	30-200	n.a.	1 (200 g ai/ha)	n.a.	n.a.
	United States	200 SC	in-furrow soil + foliar	in-furrow: 30-200 foliar: 30-50 [r]	in-furrow: n.a. foliar: 32-54 (ground) 160-270 (air)	1+3 (200)	n.a. (7)	14
	United States	480 FS	seed treatment	0.25 mg ai/kernel	n.a.	1 n.a.	n.a.	n.a.
	Canada	480 FS	seed treatment	0.25 mg ai/kernel [p]	n.a.	1 n.a.	n.a.	n.a.

Crop	Country	Form	Application method and field or glass house					PHI, days
			Method	Rate g ai/ha (timing)	Spray conc, g ai/hL	Number (maximum seasonal rate, g ai/ha)	Interval, days	
Sweet corn [do not feed forage or stover to livestock within PHI 14 days]	United States	200 SC	foliar	30-50 [r]	32-54 (ground) 160-270 (air)	4 (200)	7	1
	Canada	200 SC	foliar	30 [n]	30 (100 L/ha, ground)	4 (120)	7	1
	United States	200 SC	in-furrow soil at planting	30-200	n.a.	1 (200)	n.a.	n.a.
	United States	200 SC	in-furrow soil + foliar	in-furrow: 30-200 foliar: 30-50 [r]	in-furrow: n.a. foliar: 32-54 (ground) 160-270 (air)	1+3 (200)	n.a. (7)	1
	United States	480 FS	seed treatment	0.25 mg ai/kernel	n.a.	1 n.a.	n.a.	n.a.
Corn	Cambodia	200 SC	foliar	30	6 (500 L/ha)	1	n.a.	7
Tree nuts [o]	United States	200 SC	foliar	30-45 [e]	1.6-9.6 (ground)	4 (180)	7	10
Tree nuts [o]	Canada	200 SC	foliar	30-45 [n]	6.7-13.3 (450 L/ha, ground)	4 (180)	7	10

Notes:

n.a. = Not applicable.

[a] Including: Australian desert lime, Australian finger lime, Australian round lime, Brown River finger lime, calamondin, citron, citrus hybrids, grapefruit, Japanese summer grapefruit, kumquat, lemon, lime, Mediterranean mandarin, Mount White lime, New Guinea wild lime, orange (sour and sweet), pummel, Russell River lime, Satsuma mandarin, sweet lime, tachibana orange, Tahiti lime, tangelo, tangerine (mandarin), tangor, trifoliate orange, uniq fruit and cultivars, varieties, and/or hybrids of these commodities.

[b] Apply post bloom only. Do not make foliar application less than20 days following a soil or drip application.

[c] Apply >21 days prior to bloom or post blooming.

[d] Including: apple, azarole, crabapple, loquat*, mayhaw, medlar, pear, Asian pear, quince, Chinese quince, Japanese quince, tejocote and cultivars, varieties, and/or hybrids of these commodities. * Not in Canadian label.

[e] Apply post-bloom only.

[f] Stone fruit, including apricot, Japanese apricot, capulin*, cherry (black, sweet, tart, and Nanking), Chinese jujube*, nectarine, peach, plum, American plum, beach plum, Canada plum, plum, cherry plum, Chickasaw plum, Damson plum, Japanese plum, Klamath plum, prune, plumcot, sloe and cultivars, varieties, and/or hybrids of these commodities. Canadian label also includes chokecherry. * Not in Canadian label.

[g] Apply at infestation.

[h] Small fruit vine climbing, including amur river grape*, gooseberry, grape, hardy kiwifruit, maypop, schisandra berry, and cultivars, varieties, and/or hybrids of these commodities. Canadian label describes grape as: Grape (American bunch, Muscadine and Vinifera). * Not in Canadian label.

[i] Including: broccoli, Brussels sprouts, cabbage, Chinese cabbage (napa), cauliflower and cultivars, varieties, and/or hybrids of these commodities.

[j] Fruiting vegetables, including African eggplant, bush tomato, cocona, currant tomato, eggplant, garden huckleberry, goji berry, ground cherry, martynia, naranjilla, okra, pea eggplant, pepino, pepper (bell & nonbell), roselle, scarlet eggplant, sunberry, tomatillo, tomato; tree tomato, cultivars, varieties and hybrids of these commodities.

[k] Do not make applications less than 12 days prior to bloom or while plants are blooming.

[l] Leafy vegetables, including amaranth (leafy and Chinese), arugula, Indian aster, blackjack, broccoli raab, Chinese broccoli, abyssinian cabbage, bok choy, seakale cabbage, cat's whiskers, cham-chwi, cham-na-mul, chervil (fresh leaves), chipilin, chrysanthemum (garland), cilantro (fresh leaves), collards, corn salad, cosmos, garden cress, upland cress, dandelion leaves, dang-gwi leaves, dillweed, dock, dol-nam-mul, ebolo, endive, escarole, fameflower, feather cockscomb, Good King Henry, Hanover salad, huauzontle, jute leaves, kale, lettuce (bitter, head and leaf (Romaine)), maca leaves, mizuna, mustard greens, orach, parseley (fresh leaves), plantain (buckhorn), English primrose, purslane (garden and winter), radicchio, radish leaves, rape greens, wild rocket, shepherd's purse, spinach, Malabar spinach, New Zealand spinach, Swiss chard, tanier spinach, turnip greens, Chinese violet leaves, watercress, and cultivars, varieties and/or hybrids of these commodities. Note that for applications made to watercress, production fields must be drained of water at least 24 hours prior to application and water must not be reapplied to the field for a minimum of 24 hours following application. Canadian label also includes Italian corn salad, lamb's lettuce and tree spinach. It is noted that, according to the Codex classification, some of the leafy vegetables mentioned here belong to the fresh herbs.
Restriction for watercress: "For applications made to watercress, production fields must be drained of water at least 24 hours prior to application and water must not be reapplied to the field for a minimum of 24 hours following the application.
[m] Tuberous and corm vegetables, including arracacha, arrowroot, edible canna, cassava (bitter and sweet)*, chayote root*, Chinese artichoke, chufa, dasheen (taro), ginger, Jerusalem artichoke, leren, potato, sweet potato, tanier, true yam, turmeric, yam bean*. * Not in Canadian label. It is noted that, according to the Codex classification, ginger and turmeric belong to the spices.
[n] Apply pre- or post-blooming.
[o] Tree nuts, including: African nut-tree, almond, beechnut, Brazil nut*, Brazilian pine*, bunya*, bur oak nut, butternut, cajou nut*, candle nut*, cashew*, chestnut, chinquapin, coconut*, coquito nut*, dika nut*, ginkgo nut, Guiana chestnut, hazelnut (filbert), heartnut, hickory nut, Japanese horse-chestnut, macadamia nut*, mongongo nut*, monkey-pot*, monkey puzzle nut, Okari nut*, Pachira nut*, peach palm nut*, pecan, pequi*, Pili nut*, pine nut, pistachio*, Sapucaia nut*, tropical almond*, walnut (black & English), yellowhorn and cultivars, varieties, and/or hybrids of these commodities. * Not in Canadian label.
[p] Do not make a subsequent foliar application for a minimum of 60 days after planting the treated seed.
[q] Before soaking and before seeding (seed treatment dressing with coating agent) or after soaking and before seeding (seed treatment dressing during or after seed coating).
[r] For all application methods; only apply up to the V15 (when 15th leaf collar is visible), or after pollen shed (around 1 week after tassel is fully emerged).

RESIDUE RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised residue trials for citrus fruit (orange, mandarin, lemon, and grapefruit), pome fruit (apple and pear), stone fruit (cherry, peach plum), grapes, flowerhead brassicas (broccoli and cauliflower) and head brassicas (head cabbage), fruiting vegetables (tomato, peppers), leafy vegetables (lettuce head, lettuce leaf, spinach, mustard greens), pulses (dry soya beans), tuberous and corm vegetables (potato),cereal grains (rice, maize, sweet corn), tree nuts (almonds and pecans). Table 62 summarizes the trials submitted to the Meeting

Table 62 Overview of the crops considered within the framework of this submission.

Crop subgroup	Code No.	Commodity	Treatment	Table
Lemons and Limes	FC 0002	Lemon	Foliar or a combination of soil + foliar	63
Mandarins	FC 0003	Mandarin	Foliar or a combination of soil + foliar	64
Oranges, Sweet, Sour	FC 0004	Orange	Foliar or a combination of soil + foliar	65
Pummelo and Grapefruit	FC 0005	Grapefruit	Foliar or a combination of soil + foliar	66
Pome fruit	FP 0226 FP 0230	Apple Pear	Foliar	67–69 70–72
Cherries	FS 0013	Cherry	Foliar	73
Plums	FS 0014	Plum	Foliar	74

Crop subgroup	Code No.	Commodity	Treatment	Table
Peaches (including nectarine and apricot)	FS 2001	Peach	Foliar	75
Small fruit vine climbing	FB 0269	Grapes	Foliar	76
Flowerhead Brassicas	VB 0440	Broccoli	Foliar	77
	VB 0404	Cauliflower	Foliar	78
Head Brassicas	VB 0041	Head cabbage	Foliar	79
Tomatoes	VO 0448	Tomato	Foliar	80
Tomatoes	VO 0448	Tomato	Soil application	81
Peppers	VO 0445	Peppers	Foliar	82
Leafy greens	VL 0482	Lettuce head	Foliar	83
	VL 0483	Lettuce leaf		84
	VL 0502	Spinach		85
Leafy greens	VL 0502	Spinach	Soil application	86
Leaves of Brassicaceae	VL 0485	Mustard greens	Foliar	87
Dry beans	VD 0541	Soya bean (dry)	Foliar	88
Dry beans	VD 0541	Soya bean (dry)	In furrow	89
Tuberous and corm vegetables	VR 0589	Potato	Foliar application	90
Tuberous and corm vegetables	VR 0589	Potato	In furrow application	91
Rice Cereals	GC 2088	Rice grain–paddy rice	Foliar Seed	92
Rice Cereals	GC 2088	Rice grain–paddy rice	Seed treatment or a combination of seed treatment and foliar treatment	93
Rice Cereals	GC 0649	Rice, husked, brown rice	Foliar	94
Rice Cereals	GC 0649	Rice, husked, brown rice	Seed treatment, in furrow treatment or combination	95
Maize Cereals	GC 0645	Field corn	Foliar	96
Maize Cereals	GC 0645	Field corn	Seed treatment, in furrow treatment or combination	97
Sweet Corns	GC 2090	Sweet corn	Foliar	98
Sweet Corns	GC 2090	Sweet corn	In furrow application	199
Tree nuts	TN 0660	Almonds	Foliar	100
Tree nuts	TN 0672	Pecans	Foliar	101
Animal feed	AL 1265	Soya bean forage	In-furrow	102
	AL 0541	Soya bean hay	In-furrow	102
	AS 0649	Paddy rice straw	Foliar	103
	AS 0649	Paddy rice straw	Seed treatment or combination of seed and foliar treatment	104
	AS 3545	Paddy rice whole plant	Foliar	105
	AS 3545	Paddy rice whole plant	Seed treatment or combination of seed and foliar treatment	106
	AS 0645	Field corn forage	Foliar	107
	AS 0645	Field corn forage	Seed or in-furrow treatment	108
	AF 3548	Sweet corn forage	Foliar	109
	AF 3548	Sweet corn forage	Seed or in-furrow treatment	110

Crop subgroup	Code No.	Commodity	Treatment	Table
	AF 3557	Field corn stover	Foliar	111
	AF 3557	Field corn stover	Seed treatment, in-furrow or combination of seed &foliar	112
	AF 3557	Sweet corn stover	Foliar	113
		Sweet corn stover	Seed treatment, in-furrow or combination of seed &foliar	114
	AL 1021	Almond hulls	Foliar	115

Application rates, spray concentrations and total residues have been rounded to two figures. Residue data are recorded unadjusted for percentage recoveries or for residue values in control samples unless otherwise stated. Unquantifed residues are shown as below the LOQ of 0.01 mg/kg.

Where multiple analyses were conducted on a single sample, the average value is reported. Where multiple samples were taken from a single plot, the individual and average values are reported. Results are therefore sometimes presented as single values or as duplicate/triplicate values with the (mean) value between brackets. Where results from separate plots with distinguishing characteristics such as different formulations, crop varieties or treatment schedules were reported, results are listed separately for each plot.

Residues from the trials conducted according to the critical GAP, which have been used for the estimation of maximum residue levels, STMR and HR values are underlined.

The residues presented in the tables are given as tetraniliprole and tetraniliprole-N-methylquinazolinone. Tetraniliprole-N-methylquinazolinone was either not found in matrices for human consumption or in quantities <1 percent of the parent (leafy vegetables and incidentally in rice grain). In some animal feed commodities significant levels of the metabolite were observed. In those tables a column with total residues was included. The total column reflects the sum of parent and tetraniliprole-N-methyl-quinazolinone, expressed as parent. The total residues, expressed as parent equivalents are not corrected with a molecular weight conversion factor, since molecular weight deviates only 3 percent.

Citrus fruit

Twenty-three field trials were conducted in the United States to measure the magnitude of tetraniliprole residues in/on grapefruit (6), lemon (5), mandarin (4), and orange (8) following treatment with a tetraniliprole 200 SC formulation (Gould&Dallstream, 2017, M-563131-03-1, Report RAFVP089-02). At each site one plot received a single soil (directed chemigation) plus a single foliar application and two replicate plots received three foliar applications with either concentrated or a dilute spray application, both with equal total rates. Different soil types were included (sand, loam, and clay). Trials were carried out in 2014/2015. Applications were made at actual rates of 115–121 g ai/ha (soil application) or 58–62 g ai/ha each (foliar application), with an application interval of 19–21 days between the soil and foliar applications and an application interval of 4–6 days between the three foliar applications. No adjuvants were used.

Duplicate samples of mature fruit (BBCH 83–89) were harvested from the soil + foliar treatment plots at 5–7 days following soil application, being 12–13 days prior to the foliar application (-12 to -13 DALA), shortly before foliar applications (DALA -0) and at DALA 1 after the foliar application. Single samples of mature fruit (BBCH 83–89) were harvested at the foliar application plots on DALA 0 prior to the final application and at DALA 1 after the third foliar application. Additional decline data were collected from 10 sites, where samples were taken nominally -14, -0, 0 (immediately after the final foliar

application), 1, 7, 14 and 21 days following the final application. Samples were collected from the centre of four trees, from all areas of the plot. Each sample consisted of at least 24 fruits, weighing at least 2 kg, except for one sample of trial FV143-14DA, which consisted of 17 fruits (sample weight was not reported). Which of the samples was smaller was not reported. Since the location consisted of replicate plots using different spraying volumes, but showing similar residue levels and decline rate, the highest value at PHI 1 days was considered suitable for MRL setting.

Samples were stored frozen for a maximum of 489 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using the LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. Totals were only calculated where residues of parent tetraniliprole and tetraniliprole-N-methylquinazolinone are above LOQ of 0.01 mg/kg. In the trials summarized below, levels of the metabolite were always below the LOQ. Therefore, no totals were added to the tables for citrus fruits.

The results of the trials are summarised in Table 63 to Table 66.

Lemons

Table 63 Residues of tetraniliprole in lemons (whole fruits) after a single soil application followed by a single foliar application [a] or three foliar applications using a 200 SC formulation (no adjuvants used) in the United States of America (Study RAFVP089-02)

LEMONS Year, Location (variety)	Date					Residues (mean) (mg/kg) [b]		Trial No. (soil)
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage	DALA [c]	Tetraniliprole	Tetraniliprole-N-methylquinazolinone	
2014, Avalon, Florida (Bearss)	2 [a] (20)	119 61	1.1 5.2	19 Nov., BBCH 85; 09 Dec., BBCH 89	7/-13 -0 1	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.026, 0.021 (0.024)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV150-14HA-TRTD1 (sand)
2014, Avalon, Florida (Bearss)	3 (4)	61 61 61	5.2 5.2 5.2	09 Dec., BBCH 89	-0 1	0.026 <u>0.062</u>	<0.01 <0.01	FV150-14HA-TRTD2
2014, Avalon, Florida (Bearss)	3 (4)	60 60 58	180 180 180	09 Dec., BBCH 89	-0 1	0.012 <0.01	<0.01 <0.01	FV150-14HA-TRTD3
2015, Sanger, California (Lisbon)	2 [a] (18)	120 60	2.2 2.1	13 Nov., BBCH 79; 01 Dec., BBCH 83	5/-13 -0 1	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.050, 0.046 (0.048)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV151-14HB-TRTD1 (sandy loam)
2015, Sanger, California (Lisbon)	3 (6,5)	62 61 60	1.7 2.0 2.0	01 Dec., BBCH 83	-0 1	0.073 <u>0.13</u>	<0.01 <0.01	FV151-14HB-TRTD2
2015, Sanger, California (Lisbon)	3 (6,5)	62 60 61	210 200 200	01 Dec., BBCH 83	-0 1	<0.01 <0.01	<0.01 <0.01	FV151-14HB-TRTD3
2014,	2 [a]	115	1.28	24 Nov.,	7/-14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV152-

LEMONS Year, Location (variety)	Date					Residues (mean) (mg/kg) [b]		Trial No. (soil)
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage	DALA [c]	Tetraniliprole	Tetraniliprole-N-methylquinazolinone	
Navelencia California (Lisbon)	(21)	60	1.3	BBCH 85; 15 Dec., BBCH 85	-0 1	<0.01, <0.01 (<0.01) 0.041, 0.044 (0.043)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	14HA-TRTD1 (loamy sand)
2014, Navelencia California (Lisbon)	3 (5)	59 58 60	1.3 1.3 1.3	15 Dec., BBCH 85	-0 1	0.030 0.058	<0.01 <0.01	FV152-14HA-TRTD2
2014, Navelencia California (Lisbon)	3 (5)	60 60 59	130 180 180	15 Dec., BBCH 85	-0 1	0.060 0.19	<0.01 <0.01	FV152-14HA-TRTD3
2014, Porterville, California (Lisbon)	2 [a] (20)	120 60	0.518 2.1	04 Nov., BBCH 79; 24 Nov., BBCH 85	7/-13 -0 0 1 7 15 22	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.060, 0.064 (0.062) 0.032, 0.050 (0.041) 0.048, 0.043 (0.045) 0.037, 0.037 (0.037) 0.026, 0.017 (0.021)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV153-14DA-TRTD1 (clay loam)
2014, Porterville, California (Lisbon)	3 (6,5)	60 60 60	2.1 2.1 2.1	24 Nov., BBCH 85	-0 0 1 7 15 22	0.094 0.16 0.099 0.14 0.14 0.094	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV153-14DA-TRTD2
2014, Porterville, California (Lisbon)	3 (6,5)	60 60 60	170 160 160	24 Nov., BBCH 83	-0 0 1 7 15 22	0.49 0.83 0.77 0.58 0.49 0.67	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV153-14DA-TRTD3
2015, Orange Cove, California (Lisbon)	2 [a] (20)	120 60	2.5 2.6	28 Jan., BBCH 85; 17 Feb., BBCH 89	7/-13 -0 0 1 7 14 21	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.062, 0.048 (0.055) 0.036, 0.036 (0.036) 0.050, 0.038 (0.044) 0.029, 0.034 (0.032) 0.043, 0.037 (0.040)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV154-14DA-TRTD1 (loam)
2015, Orange Cove, California (Lisbon)	3 (5)	60 60 60	2.6 2.6 2.6	17 Feb., BBCH 89	-0 0 1 7 14 21	0.14 0.26 0.20 0.16 0.11 0.090	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV154-14DA-TRTD2
2015, Orange Cove, California (Lisbon)	3 (5)	60 59 59	160 160 160	17 Feb., BBCH 89	-0 0 1 7 14 21	0.098 0.27 0.14 0.17 0.12 0.068	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV154-14DA-TRTD3

Notes:

RTI = Retreatment Interval; DALA = Days after last application.

Mean residue values presented in parenthesis.

[a] Chemigation application followed by foliar application.
[b] Residues expressed in parent equivalents.
[c] PHI notation of 'X/-X/' represents sampling X days after the chemigation application and -X represents the corresponding number of days before the final foliar; '-0' represents sampling just prior to the final application; '0' represents sampling immediately after the final application.

Mandarins

Table 64 Residues of tetraniliprole in mandarins (whole fruits) after a single soil application followed by a single foliar application or three foliar applications using a 200 SC formulation in the United States of America (Study RAFVP089-02)

MANDARINS Year, Location (variety)	Application					Residues (mean) (mg/kg) [b]		Trial No. (soil)
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [c]	DALA	Tetraniliprole	Tetraniliprole-N-methylquinazolinone	
2014, Clermont, Georgia (Clementine)	2 [a] (20)	120 60	0.86 5.6	09 Oct., BBCH 81; 29 Oct., BBCH 85	6/-14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV146-14HA-TRTD1 (sand)
					-0	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
					1	0.057, 0.053 (0.055)	<0.01, <0.01 (<0.01)	
2014, Clermont, Georgia (Clementine)	3 (4)	60 58 61	5.1 5.4 5.5	29 Oct., BBCH 85	-0	0.090	<0.01	FV146-14HA-TRTD2
					1	0.12	<0.01	
2014, Clermont, Georgia (Clementine)	3 (4)	59 60 61	130 140 130	29 Oct., BBCH 85	-0	0.20	<0.01	FV146-14HA-TRTD3
					1	<u>0.19</u>	<0.01	
2015, Fresno California (Mandarin)	2 [a] (20)	120 59	1.3 3.2	20 Nov., BBCH 83; 10 Dec., BBCH 89	7/-13	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV147-14HB-TRTD1 (sandy loam)
					-0	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
					1	0.032, 0.024 (0.028)	<0.01, <0.01 (<0.01)	
2015, Fresno California (Mandarin)	3 (5)	59 59 59	3.2 3.2 3.2	10 Dec., BBCH 89	-0	0.15	<0.01	FV147-14HB-TRTD2
					1	0.16	<0.01	
2015, Fresno California (Mandarin)	3 (5)	60 59 60	160 160 160	10 Dec., BBCH 89	-0	0.21	<0.01	FV147-14HB-TRTD3
					1	<u>0.17</u>	<0.01	
2014, - Mims, Florida (Minneola)	2 [a] (20)	120 60	2.5 4.2	14 Oct., BBCH 81; 3 Nov., BBCH 83	7/-13	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV148-14DA-TRTD1 (sand)
					-0	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
					0	0.066, 0.064 (0.065)	<0.01, <0.01 (<0.01)	
					1	0.052, 0.054 (0.053)	<0.01, <0.01 (<0.01)	
					7	0.034, 0.034 (0.034)	<0.01, <0.01 (<0.01)	
					14	0.014, 0.014 (0.014)	<0.01, <0.01 (<0.01)	
					21	0.012, 0.011 (0.011)	<0.01, <0.01 (<0.01)	
2014, - Mims, Florida (Minneola)	3 (5)	60 61 60	4.3 4.3 4.3	03 Nov., BBCH 83	-0	0.11	<0.01	FV148-14DA-TRTD2
					0	0.25	<0.01	
					1	<u>0.18</u>	<0.01	
					7	0.13	<0.01	
					14	0.074	<0.01	
					21	0.049	<0.01	

MANDARINS Year, Location (variety)	Application					Residues (mean) (mg/kg) [b]		Trial No. (soil)
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [c]	DALA	Tetraniliprole	Tetraniliprole-N-methylquinazolinone	
2014, - Mims, Florida (Minneola)	3 (5)	59 60 59	130 130 130	03 Nov., BBCH 83	-0 0 1 7 14 21	0.054 0.056 0.062 0.070 0.038 0.012	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV148-14DA-TRTD3
2014, Oviedo, Florida (Minneola)	2 [a] (20)	120 61	2.52 4.3	14 Oct., BBCH 81; 03 Nov., BBCH 81	7/-13	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV149-14DA-TRTD1 (sand)
					-0 0 1 7 14 21	<0.01, <0.01 (<0.01) 0.27, 0.31 (0.29) 0.22, 0.20 (0.21) 0.19, 0.15 (0.17) 0.14, 0.14 (0.14) 0.077, 0.088 (0.083)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
2014, Oviedo, Florida (Minneola)	3 (5)	60 60 60	4.3 4.2 4.2	03 Nov., BBCH 83	-0 0 1 7 14 21	0.50 0.62 0.50 <u>0.54</u> 0.39 0.28	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV149-14DA-TRTD2
2014, Oviedo, Florida (Minneola)	3 (5)	60 59 59	130 130 130	03 Nov., BBCH 83	-0 0 1 7 14 21	0.090 0.11 0.22 0.12 0.10 0.046	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV149-14DA-TRTD3

Notes:
RTI = Retreatment Interval; DALA = Days after last application;
[a] Chemigation application followed by foliar application.
[b] Residues expressed as parent equivalents.
[c] at the last application

Oranges

Table 65 Residues of tetraniliprole in oranges (whole fruits) after a single soil application followed by a single foliar application or three foliar applications using a 200 SC formulation in the United States of America in 2014 (Study RAFVP089-02)

ORANGES Location (variety)	Application					Residues (mean) (mg/kg) [b]		Trial
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [c]	DALA	Tetraniliprole	Tetraniliprole-N-methylquinazolinone	
Winter Garden, Florida (Hamlin)	2 [a] (20)	120 61	1.28 5.1	14 Nov., BBCH 81; 04 Dec., BBCH 85	-15 -0 1	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.066, 0.074 (0.070)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV138-14HA-TRTD1
Winter Garden, Florida (Hamlin)	3 (5)	60 59 60	5.2 5.1 5.1	04 Dec., BBCH 85	-0 1	0.084 <u>0.15</u>	<0.01 <0.01	FV138-14HA-TRTD2

ORANGES Location (variety)	Application					Residues (mean) (mg/kg) [b]		Trial
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [c]	DALA	Tetraniliprole	Tetraniliprole-N-methylquinazolinone	
Winter Garden, Florida Hamlin)	3 (5)	62 60 61	190 190 180	04 Dec., BBCH 85	-0 1	0.028 0.031	<0.01 <0.01	FV138-14HA-TRTD3
Umatilla, Florida (Washington)	2 [a] (20)	120 61	1.06 5.2	19 Nov., BBCH 81; 09 Dec., BBCH 89	-13 -0 1	<0.01, <0.01 (<0.01) <0.01, 0.015 (0.012) 0.035, 0.030 (0.032)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV139-14HA-TRTD1
Umatilla, Florida (Washington)	3 (4)	60 60 60	5.1 5.1 5.1	09 Dec., BBCH 89	-0 1	0.057 0.11	<0.01 <0.01	FV139-14HA-TRTD2
Umatilla, Florida (Washington)	3 (4)	61 59 59	180 180 180	09 Dec., BBCH 89	-0 1	0.058 <u>0.13</u>	<0.01 <0.01	FV139-14HA-TRTD3
Raymondville Texas (Marrs)	2 [a] (19)	121 61	0.371 2.5	26 Nov., BBCH 81; 15 Dec., BBCH 83	-12 -0 1	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.015, 0.015 (0.015)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV140-14HA-TRTD1
Raymondville, Texas (Marrs)	3 (5,4)	61 61 63	2.5 2.5 2.6	15 Dec., BBCH 83	-0 1	0.046 0.041	<0.01 <0.01	FV140-14HA-TRTD2
Raymondville Texas (Marrs)	3 (5,4)	62 62 62	250 250 250	15 Dec., BBCH 83	-0 1	0.028 <u>0.044</u>	<0.01 <0.01	FV140-14HA-TRTD3
Sanger, California (Washington)	2 [a] (19)	120 61	2.0 2.4	12 Nov., BBCH 79; 1 Dec., BBCH 83	-13 -0 1	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.021, 0.020 (0.020)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV141-14HA-TRTD1
Sanger, California (Washington)	3 (5)	61 60 61	2.5 2.4 2.3	01 Dec, BBCH 83	-0 1	0.061 0.10	<0.01 <0.01	FV141-14HA-TRTD2
Sanger, California (Washington)	3 (5)	60 61 62	140 140 140	01 Dec., BBCH 83	-0 1	0.32 <u>0.14</u>	<0.01 <0.01	FV141-14HA-TRTD3
Oviedo, Florida (Navel)	2 [a] (20)	120 61	2.5 4.3	14 Oct., BBCH 81; 03 Nov., BBCH 83	-13 -0 0 1 7 14 21	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.050, 0.047 (0.049) 0.046, 0.030 (0.038) 0.027, 0.031 (0.029) 0.023, 0.023 (0.023) <0.01, 0.010 (0.010)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV142-14DA-TRTD1
Oviedo, Florida (Navel)	3 (5)	61 60 61	4.3 4.2 4.3	03 Nov., BBCH 83	-0 0 1 7 14 21	0.12 0.16 <u>0.14</u> 0.13 0.082 0.024	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV142-14DA-TRTD2

ORANGES Location (variety)	Application					Residues (mean) (mg/kg) [b]		Trial
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [c]	DALA	Tetraniliprole	Tetraniliprole-N-methylquinazolinone	
Oviedo, Florida (Navel)	3 (5)	60 59 59	130 130 130	03 Nov., BBCH 83	-0 0 1 7 14 21	0.044 0.080 0.083 0.047 0.036 0.041	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV142-14DA-TRTD3
Clermont, Florida (Mid sweet)	2 [a] (19)	121 60	1.3 4.7	05 Nov., BBCH 81; 24 Nov., BBCH 89	-12 -0 0 1 7 14 21	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.068, 0.078 (0.073) 0.075, 0.067 (0.071) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01	FV143-14DA-TRTD1 [d]
Clermont, Florida (Mid sweet)	3 (5)	60 59 60	4.1 4.5 4.6	24 Nov., BBCH 89	-0 0 1 7 14 21	0.080 0.14 0.13 0.014 0.018 0.014	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV143-14DA-TRTD2 [d]
Clermont, Florida (Mid sweet)	3 (5)	59 59 60	140 140 140	24 Nov., BBCH 89	-0 0 1 7 14 21	0.11 0.14 <u>0.16</u> 0.042 0.033 0.040	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV143-14DA-TRTD3 [d]
Mims, Florida (Navel)	2 [a] (20)	120 60	2.5 4.2	14 Oct., BBCH 81; 03 Nov., BBCH 83	-13 -0 0 1 7 14 21	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.034, 0.026 (0.030) 0.024, 0.026 (0.025) 0.027, 0.021 (0.024) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV144-14DA-TRTD1
Mims, Florida (Navel)	3 (5)	61 60 61	4.3 4.3 4.3	03 Nov., BBCH 83	-0 0 1 7 14 21	0.085 0.11 0.065 <u>0.066</u> 0.032 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV144-14DA-TRTD2
Mims, Florida (Navel)	3 (5)	60 60 59	130 130 130	03 Nov., BBCH 83	-0 0 1 7 14 21	0.010 0.030 0.017 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV144-14DA-TRTD3
Porterville, California (Atwood)	2 [a] (20)	120 60	1.7 4.3	18 Nov., BBCH 89; 08 Dec., BBCH 89	-13 -0 0 1 7 14 21	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.049, 0.042 (0.045) 0.032, 0.034 (0.033) 0.022, 0.022 (0.022) 0.017, 0.013 (0.015) 0.015, 0.018 (0.017)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV145-14DA-TRTD1

ORANGES Location (variety)	Application					Residues (mean) (mg/kg) [b]		Trial
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [c]	DALA	Tetraniliprole	Tetraniliprole-N-methylquinazolinone	
Porterville, California (Atwood)	3 (5)	59 59 60	4.2 4.2 4.3	08 Dec., BBCH 89	-0 0 1 7 14 21	0.048 0.12 0.062 0.034 0.047 0.031	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV145-14DA-TRTD2
Porterville, California (Atwood)	3 (5)	59 59 61	180 180 190	08 Dec., BBCH 89	-0 0 1 7 14 21	0.27 0.47 0.17 0.26 <u>0.29</u> 0.24	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV145-14DA-TRTD3

Notes:

RTI = Retreatment Interval.

DALA = Days After Last Application.

[a] Chemigation application followed by foliar application.

[b] Residues expressed as parent equivalents.

[c] at the last application

[d] The report mentioned that one sample of trial FV143-14DA consisted of 17 fruits (sample weight was not reported). Which of the samples was smaller was not reported. The location consisted of replicate plots for triple foliar treatment with the same concentration of active substance, but two using different spraying volumes. Since both show similar residue levels and decline rates, the highest value at PHI 1 days was considered suitable for MRL setting.

Grapefruits

Table 66 Residues of tetraniliprole in grapefruits (whole fruits) after a single soil application followed by a single foliar application or three foliar applications using a 200 SC formulation in the United States in 2014 (Study RAFVP089-02)

GRAPE-FRUITS Location (variety)	Application					Residues (mean) (mg/kg) [b]		Trial (soil)
	No. (RTI)	g ai /ha	g ai/ hL	Date, growth stage [c]	DALA	Tetraniliprole	Tetraniliprole-N-methyl-quinazolinone	
Avalon, Florida (Flaming Red)	2 [a] (19)	121 60	1.3 4.6	05 Nov., BBCH 81; 24 Nov., BBCH 85	7/-12 -0 1	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.049, 0.042 (0.046)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV155-14HA-TRTD1 (sand)
Avalon, Florida (Flaming Red)	3 (5)	60 59 61	4.1 4.5 4.7	24 Nov., BBCH 85	-0 1	0.067 0.083	<0.01 <0.01	FV155-14HA-TRTD2
Avalon, Florida (Flaming Red)	3 (5)	61 60 60	140 140 140	24 Nov., BBCH 85	-0 1	0.096 <u>0.19</u>	<0.01 <0.01	FV155-14HA-TRTD3
Umatilla, Florida (Ray Ruby)	2 [a] (20)	120 61	1.1 5.2	19 Nov., BBCH 81; 09 Dec., BBCH 89	7/-13 -0 1	<0.01, <0.01 (<0.01) 0.011, 0.027 (0.019) 0.044, 0.040 (0.042)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV156-14HA-TRTD1 (sand)
Umatilla, Florida (Ray Ruby)	3 (4)	60 60 61	5.1 5.1 5.2	09 Dec., BBCH 89	-0 1	0.012 0.061	<0.01 <0.01	FV156-14HA-TRTD2

GRAPE-FRUITS Location (variety)	Application					Residues (mean) (mg/kg) [b]		Trial (soil)
	No. (RTI)	g ai /ha	g ai/ hL	Date, growth stage [c]	DALA	Tetraniliprole	Tetraniliprole-N-methyl-quinazolinone	
Umatilla, Florida (Ray Ruby)	3 (4)	60 59 59	180 180 180	09 Dec., BBCH 89	-0 1	0.021 <u>0.071</u>	<0.01 <0.01	FV156-14HA-TRTD2
Raymondville Texas (Rio Red)	2 [a] (19)	121 61	0.37 2.5	26 Nov., BBCH 81; 15 Dec., BBCH 83	7/-12	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV157-14HA-TRTD1 (clay)
					-0 1	<0.01, <0.01 (<0.01) 0.018, 0.012 (0.015)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
Raymondville Texas (Rio Red)	3 (5,4)	61 62 61	2.5 2.5 2.5	15 Dec., BBCH 83	-0 1	0.022 0.038	<0.01 <0.01	FV157-14HA-TRTD2
Raymondville Texas (Rio Red)	3 (5,4)	62 62 62	250 250 250	15 Dec., BBCH 83	-0 1	0.032 <u>0.039</u>	<0.01 <0.01	FV157-14HA-TRTD3
Sanger, California (White)	2 [a] (19)	120 61	2.0 2.4	12 Nov., BBCH 79; 01 Dec., BBCH 83	6/-13	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV158-14HA-TRTD1
					-0 1	<0.01, <0.01 (<0.01) 0.020, 0.019 (0.019)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
Sanger, California (White)	3 (5)	61 59 60	2.5 2.4 2.4	01 Dec., BBCH 83	-0 1	0.034 0.057	<0.01 <0.01	FV158-14HA-TRTD2
Sanger, California (White)	3 (5)	62 60 62	130 140 140	01 Dec., BBCH 83	-0 1	0.10 <u>0.49</u>	<0.01 <0.01	FV158-14HA-TRTD3
Mims, Florida (Red Ray)	2 [a] (20)	120 60	2.5 4.3	14 Oct., BBCH 81; 03 Nov., BBCH 83	7/-13	<0.01, <0.01 (<0.01	<0.01, <0.01 (<0.01)	FV159-14DA-TRTD2 (sand)
					-0 0 1 7 14 21	<0.01, <0.01 (<0.01) 0.040, 0.050 (0.045) 0.030, 0.030 (0.030) 0.015, 0.013 (0.014) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
Mims, Florida (Red Ray)	3 (5)	60 60 59	4.3 4.3 4.2	03 Nov., BBCH 83	-0 0 1 7 14 21	0.081 0.12 <u>0.081</u> 0.075 0.012 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV159-14DA-TRTD2
Mims, Florida (Red Ray)	3 (5)	59 59 59	130 130 130	03 Nov., BBCH 83	-0 0 1 7 14 21	0.025 0.031 0.023 0.021 0.016 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV159-14DA-TRTD3
Porterville California (Mellow-gold)	2 [a] (20)	120 59	1.56 4.3	18 Nov., BBCH 89; 08 Dec., BBCH 89	7/-13	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV160-14DA-TRTD1 (loam)
					-0 0 1 7 14 21	<0.01, <0.01 (<0.01) 0.012, 0.033 (0.022) 0.011, 0.010 (0.011) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	

GRAPE-FRUITS Location (variety)	Application					Residues (mean) (mg/kg) [b]		Trial (soil)
	No. (RTI)	g ai /ha	g ai/ hL	Date, growth stage [c]	DALA	Tetraniliprole	Tetraniliprole-N-methyl-quinazolinone	
Porterville California (Mellow-gold)	3 (5)	60 61 60	4.2 4.3 4.3	08 Dec., BBCH 89	-0 0 1 7 14 21	0.064 0.096 <u>0.11</u> 0.037 0.044 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV160-14DA-TRTD2
Porterville California (Mellow-gold)	3 (5)	60 60 60	190 190 190	08 Dec., BBCH 89	-0 0 1 7 14 21	0.061 0.027 0.030 0.018 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	FV160-14DA-TRTD3

Notes:
RTI = Retreatment Interval.
DALA = Days After Last Application.
[a] Chemigation application followed by foliar application.
[b] Residues expressed as parent equivalents.
[c] At the last application.

Pome fruit–Canada and the United States

Twenty-five field trials were conducted in Canada and the United States (Fischer & Roberts, 2016, M-561106-02-1, Report RAFVP104-01) to measure the magnitude of tetraniliprole residues in/on apples (15 trials) and pears (10 trials) following three foliar air-blast applications of tetraniliprole 200 SC. Trials were carried out in 2014/2015. Applications were made at an actual rate of 57–62 g ai/ha, with application intervals of 5–8 days. Applications were made between growth stages BBCH 72–89. There were no adjuvants added to the spray mixture, except in trial FV190-14DA in which a silicone de-foamer was added to the three TRTDC applications (0.005 percent) and one of the TRTDD applications (0.003 percent).

Across all trials, apple fruit and pear fruit were harvested when the RAC was at BBCH 81 to 89. In the harvest trials, samples were collected from plots with a concentrated spray solution (TRTDC) and a diluted spray solution (TRTDD) a day before (-1) or immediately prior to the third treatment (-0), and after the 3rd application at a nominal 7-day pre-harvest interval. Additional decline data was collected from 8 sites, where samples were taken nominally -0, 0, 3, 7, 10 and 14 days following the final application. Each composite pome fruit sample contained a minimum of 24 whole fruit (2 kg), collected from all four quadrants of at least four trees per plot.

Samples were stored frozen for a maximum of 314 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

Pome fruit–Australia

Ten field trials were conducted in Australia (Massault, 2017, M-598808-01-1, Report BCS-0531 and Massault, 2018, M-631139-01-1, Report BCS-0532.01) to measure the magnitude of tetraniliprole residues in/on apples (6 trials) and pears (4 trials) following various foliar treatments with tetraniliprole 200 SC. Trials were carried out in 2017/2018. At each test site, multiple treatment plots were established that

received applications of tetraniliprole at various rates and timings. A non-ionic organic surfactant (Agral) was used at all trial sites.

Across all trials, apple and pear fruit were collected at commercial maturity, with additional decline samples also collected from some treatment plots. With exception of trial 532-2, where only 6 fruits were collected (indicated with [SS]), at least 12 fruits (1–2 kg) were sampled from several places on each tree within the centre of the plot.

Samples were stored frozen for a maximum of 85 days (*ca* 3 months) prior to residue analysis. Samples were analysed using the LC-MS/MS method ATM-0071 and ATM-0071B (see section on analytical methods). The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. The specific recovery data are summarised in the analytical section.

Totals were only calculated where residues of parent tetraniliprole and tetraniliprole-N-methylquinazolinone are above LOQ of 0.01 mg/kg. In the trials summarized below, levels of the metabolite were always below the LOQ. Therefore, no totals were added to the tables for pome fruits.

The results of the trials in apples and pears are summarised in Tables 67 to 72.

Apples

Table 67 Residues of tetraniliprole in apples after three pre-harvest foliar treatments using a 200 SC formulation in field trials in the Australia, Canada and the United States

APPLES Country, Year, Location (variety)	Application					Residues (mg/kg) [b]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]	DALA	Tetraniliprole	T-N-methyl- quinazolinone	
Australia, 2017, Coomboona, Victoria (Pink lady)	3 (10,7)	16 16 16	1.9 2.0 2.0	31 March, BBCH 87 +NIS	0 3 7 13	0.04 0.02 0.04 0.04	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-1 T8
Australia, 2017, Coomboona, Victoria (Pink lady)	3 (10,11)	31 32 33	3.9 4.0 4.0	31 March, BBCH 87 +NIS	0 3 7 13	0.06 0.08 0.11 0.10	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-1 T9
Australia, 2017, Karragullen, Western Australia (Pink lady)	3 (10, 9)	20 23 24	2.0 2.0 2.0	11 April, BBCH 81 +NIS	0 3 7 13	0.22 0.22 0.19 0.22	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-2 T8
Australia, 2017, Karragullen, Western Australia (Pink lady)	3 (10,9)	41 46 48	4.1 4.0 4.1	11 April, BBCH 81 +NIS	0 3 7 13	0.34 0.35 0.37 0.38	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-2 T9
Australia, 2017, Orange, New South Wales (Pink lady)	3 (11,10)	38 40 40	2.0 2.0 2.0	02 March, BBCH 85/87 +NIS	0 3 7 14	0.07 0.07 0.06 0.05	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-3 T8
Australia, 2017, Orange, New South Wales (Pink lady)	3 (11,10)	82 80 80	4.1 4.0 4.0	02 March, BBCH 85/87 +NIS	0 3 7 14	0.28 0.27 0.14 0.08	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-3 T9
Australia, 2018, Nashdale, New South Wales (Crimson Snow)	3 (10,7)	53 52 53	2.6 2.6 2.6	13 April, BBCH 85 +NIS	0 3 7 14	0.05 0.09 0.05 0.05	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-1 T8

APPLES Country, Year, Location (variety)	Application				DALA	Residues (mg/kg) [b]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl- quinazolinone	
Australia, 2018, Nashdale, New South Wales (Crimson Snow)	3 (10,7)	53 52 53	2.6 2.6 2.6	13 April, BBCH 85 +NIS	0 3 7 14	0.13 0.09 0.09 0.03	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-1 T6
Australia, 2018, Nashdale, New South Wales (Crimson Snow)	3 (10,7)	108 104 105	5.2 5.2 5.2	13 April, BBCH 85 +NIS	0 3 7 14	0.24 0.31 0.12 0.16	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-1 T7
Australia, 2018, Nashdale, New South Wales (Crimson Snow)	3 (6,7)	53 53 53	2.6 2.6 2.6	20 April, BBCH 85- 87 +NIS	0 3 7	0.09 0.06 0.05	<0.01 <0.01 <0.01	BCS-0532 532-1 T9
Australia, 2018, Nashdale, New South Wales (Crimson Snow)	3 (6,7)	105 105 105	5.2 5.2 5.2	20 April, BBCH 85- 87 +NIS	0 3 7	0.24 0.22 0.32	<0.01 <0.01 <0.01	BCS-0532 532-1 T10
Australia, 2017/2018, Echunga, South Australia (Granny Smith)	3 (7,8)	26 26 27	2.6 2.6 2.6	12 Jan., BBCH 79- 81 +NIS	0 3 7 13	0.13 0.12 0.15 0.09	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-2 T8 [SS]
Australia, 2017/2018, Echunga, South Australia (Granny Smith)	3 (7,8)	27 27 27	2.6 2.6 2.6	12 Jan., BBCH 79- 81 +NIS	0 3 7 13	0.10 0.10 0.08 0.07	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-2 T6 [SS]
Australia, 2017/2018, Echunga, South Australia (Granny Smith)	3 (7,8)	55 55 55	5.2 5.2 5.2	12 Jan., BBCH 79- 81 +NIS	0 3 7 13	0.20 0.14 0.15 0.15	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-2 T7 [SS]
Australia, 2017/2018, Echunga, South Australia (Granny Smith)	3 (8,10)	26 27 27	2.6 2.6 2.6	22 Jan., BBCH 81- 85 +NIS	0 3 7	0.10 0.06 0.07	<0.01 <0.01 <0.01	BCS-0532 532-2 T9 [SS]
Australia, 2017/2018, Echunga, South Australia (Granny Smith)	3 (8,10)	54 52 55	5.2 5.2 5.2	22 Jan., BBCH 81- 85 +NIS	0 3 7	0.16 0.17 0.16 *(0.18)*	<0.01 <0.01 <0.01	BCS-0532 532-2 T10 [SS]
Australia, 2018, Coomboona, Victoria (Pink Lady)	3 (8,11)	23 23 20	2.6 2.6 2.6	09 April, BBCH 85 +NIS	0 3 7 14	0.15 0.15 0.11 0.12	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-3 T8
Australia, 2018, Coomboona, Victoria (Pink Lady)	3 (8,11)	22 23 20	2.6 2.6 2.6	09 April, BBCH 85 +NIS	0 3 7 14	0.15 0.08 0.11 0.11	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-3 T6
Australia, 2018, Coomboona, Victoria (Pink Lady)	3 (8,11)	44 47 40	5.2 5.2 5.2	09 April, BBCH 85 +NIS	0 3 7 14	0.30 0.31 0.20 0.17	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-3 T7

APPLES Country, Year, Location (variety)	Application				DALA	Residues (mg/kg) [b]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl- quinazolinone	
Australia, 2018, Coomboona, Victoria (Pink Lady)	3 (6,7)	18 20 20	2.6 2.6 2.6	16 April, BBCH 87 +NIS	0 3 7	0.12 0.13 0.15	<0.01 <0.01 <0.01	BCS-0532 532-3 T9
Australia, 2018, Coomboona, Victoria (Pink Lady)	3 (6,7)	37 41 40	5.2 5.2 5.2	16 April, BBCH 87 +NIS	0 3 7	0.44 0.35 0.29	<0.01 <0.01 <0.01	BCS-0532 532-3 T10
Canada, 2015, St. George, Ontario (Paula Red)	3^ (7,7)	61 61 64	5.0 5.0 5.0	11 Aug., BBCH 85	-0 7	0.069 0.13	<0.01 <0.01	RAFVP104- 01FV193-14HA- TRTDD
Canada, 2015, St. George, Ontario (Paula Red)	3^ (7,7)	60 57 62	39 39 39	11 Aug., BBCH 85	-0 7	0.074 <u>0.13</u>	<0.01 <0.01	RAFVP104- 01FV193-14HA- TRTDC
Canada, 2015, St. George, Ontario (Spartan)	3^ (7,7)	61 59 60	5.0 5.0 5.0	08 Sept., BBCH 85	-0 7	0.094 0.097	<0.01 <0.01	RAFVP104- 01FV194-14HA- TRTDD
Canada, 2015, St. George, Ontario (Spartan)	3^ (7,7)	63 58 61	39 38 38	08 Sept., BBCH 85	-0 7	0.099 0.095	<0.01 <0.01	RAFVP104-01, FV194-14HA- TRTDC
United States, 2014, North Rose, New York (Greening)	3^ (7,6)	61 61 61	5.2 5.2 5.2	17 Sept., BBCH 85	-0 7	0.021 0.10	<0.01 <0.01	RAFVP104-01 FV188-14HA- TRTDD
United States, 2014, North Rose, New York (Greening)	3^ (7,6)	61 61 61	14 14 14	17 Sept., BBCH 85	-0 7	0.044 <u>0.11</u>	<0.01 <0.01	RAFVP104-01 FV188-14HA- TRTDC
United States, 2014, Lyons, New York (Twenty Ounce)	3^ (7,7)	59 59 60	4.2 4.2 4.3	07 Oct., BBCH 89	-0 7	0.064 <u>0.10</u>	<0.01 <0.01	RAFVP104-01 FV189-14HA- TRTDD
United States, 2014, Lyons, New York (Twenty Ounce)	3^ (7,7)	60 60 61	21 22 22	07 Oct., BBCH 89	-0 7	0.041 0.089	<0.01 <0.01	RAFVP104-01 FV189-14HA- TRTDC
United States, 2014, Hereford, Pennsylvania (Starkrinson Red Delicious)	3 (6,7)	62 62 61	5.0 5.0 5.0	17 Sept., BBCH 87 +ADJ	-0 0 3 6 9 14	0.12 0.18 0.19 0.20 0.19 0.14	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01 FV190-14DA- TRTDD
United States, 2014, Hereford, Pennsylvania (Starkrimson Red Delicious)	3 (6,7)	61 61 61	16 16 16	17 Sept., BBCH 87 +ADJ	-0 0 3 6 9 14	0.13 0.21 0.22 0.18 0.20 0.15	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01 FV190-14DA- TRTDC
United States, 2014, Cana, Virginia (Rome)	3^ (8,7)	60 60 60	4.4 3.1 3.1	05 Sept., BBCH 87	6	0.21, 0.13 (<u>0.17</u>)	<0.01, <0.01 (<0.01)	RAFVP104-01 FV191-14HA- TRTDD
United States, 2014, Cana, Virginia (Rome)	3^ (8,7)	60 61 61	21 20 20	05 Sept., BBCH 87	6	0.12, 0.087 (0.10)	<0.01, <0.01 (<0.01)	RAFVP104-01 FV191-14HA- TRTDC

APPLES Country, Year, Location (variety)	Application				DALA	Residues (mg/kg) [b]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl- quinazolinone	
United States, 2014, Trilla, Illinois (IL) Red Delicious	3^ (6,7)	60 60 61	5.8 5.9 5.4	04 Sept., BBCH 87	-0 7	0.13 0.12	<0.01 <0.01	RAFVP104-01 FV192-14HA- TRTDD
United States, 2014, Trilla, Illinois (Red Delicious)	3^ (6,7)	60 60 61	21 18 18	04 Sept., BBCH 87	-0 7	0.10 <u>0.20</u>	<0.01 <0.01	RAFVP104-01 FV192-14HA- TRTDC
United States, 2014, Trilla, Illinois (Cortland)	3^ (6,7)	60 60 60	5.8 6.0 5.5	04 Sept., BBCH 87	-0 0 3 7 8 14	0.10 0.13 0.14 0.075 0.11 0.057	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01, FV196-14DA- TRTDD
United States, 2014, Trilla, Illinois (Cortland)	3^ (6,7)	60 62 61	21 18 18	04 Sept., BBCH 87	-0 0 3 7 8 14	0.086 0.17 0.16 0.13 0.074 0.13	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01, FV196-14DA- TRTDC
United States, 2014, Centralia, Illinois (Jonathan Apple)	3^ (7,7)	60 60 60	2.9 3.5 3.3	09 Sept., BBCH 81	-0 0 3 7 10 14	0.069 0.14 0.12 0.088 0.10 0.053	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01, FV195-14DA- TRTDD
United States, 2014, Centralia, Illinois (Jonathan Apple)	3^ (7,7)	60 59 60	13 16 15	09 Sept., BBCH 81	-0 0 3 7 10 14	0.12 0.17 0.19 <u>0.15</u> 0.14 0.11	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01, FV195-14DA- TRTDC
United States, 2015, Perry, Utah (Scarlet gala)	3^ (7,7)	60 61 59	3.2 3.2 3.3	19 Aug., BBCH 85	-0 8	0.084 0.11	<0.01 <0.01	RAFVP104-01, FV197-14HA- TRTDD
United States, 2015, Perry, Utah (Scarlet gala)	3^ (7,7)	61 60 60	19 19 19	19 Aug., BBCH 85	-0 8	0.10 <u>0.15</u>	<0.01 <0.01	RAFVP104-01 FV197-14HA- TRTDC
United States, 2014, Madera, California (Anna)	3^ (7,7)	59 61 61	5.1 5.1 5.1	06 June, BBCH 79	7	0.085	<0.01 <0.01	RAFVP104-01, FV198-14HA- TRTDD
United States, 2014, Madera, California (Anna)	3^ (7,7)	62 61 61	13 13 13	06 June, BBCH 79	7	<u>0.092</u>	<0.01 <0.01	RAFVP104-01, FV198-14HA- TRTDC
United States, 2014, Ephrata, Washington (Ida Red)	3^ (7,7)	60 60 60	4.2 4.3 4.3	11 Sept., BBCH 87	-0 7	0.10 <u>0.16</u>	<0.01 <0.01	RAFVP104-01 FV199-14HA- TRTDD
United States, 2014, Ephrata, Washington (Ida Red)	3^ (7,7)	59 60 59	13 13 13	11 Sept., BBCH 87	-0 7	0.067 0.12	<0.01 <0.01	RAFVP104-01 FV199-14HA- TRTDC

APPLES Country, Year, Location (variety)	Application				DALA	Residues (mg/kg) [b]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl- quinazolinone	
United States, 2014, Ephrata, Washington (Aztec Fuji)	3^ (7,7)	59 60 60	4.2 4.3 4.3	03 Oct., BBCH 87	-0 7	0.077 <u>0.11</u>	<0.01 <0.01	RAFVP104-01 FV201-14HA- TRTDD
United States, 2014, Ephrata, Washington (Aztec Fuji)	3^ (7,7)	59 59 59	13 13 13	03 Oct., BBCH 87	-0 7	0.087 0.094	<0.01 <0.01	RAFVP104-01 FV201-14HA- TRTDC
United States, 2014, Ephrata, Washington (Brookfield Gala)	3^ (7,7)	60 60 60	4.3 4.3 4.3	20 Aug., BBCH 87	-0 0 3 7 9 14	0.096 0.19 0.20 0.17 <u>0.17</u> 0.16	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01, FV202-14DA- TRTDD
United States, 2014, Ephrata, Washington (Brookfield Gala)	3^ (7,7)	59 60 60	13 13 13	20 Aug., BBCH 87	-0 0 3 7 9 14	0.12 0.22 0.18 0.13 0.16 0.16	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01 FV202-14DA- TRTDC
United States, 2014, Hood River, Oregon (Jonagold)	3^ (7,7)	60 61 59	3.6 3.5 3.4	17 Sept., BBCH 85	-1 6	0.053 0.051	<0.01 <0.01	RAFVP104-01 FV200-14HA- TRTDD
United States, 2014, Hood River, Oregon (Jonagold)	3^ (7,7)	60 59 60	15 14 14	17 Sept., BBCH 85	-1 6	0.031 <u>0.064</u>	<0.01 <0.01	RAFVP104-01 FV200-14HA- TRTDC

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.

ADJ = adjuvant used (silicone de-foamer (FoamBuster, Helena Chemical Co.)), trial was not considered for MRL estimation; NIS= Non-ionic organic surfactant Agral.

[SS] = Sample size too small, consisting of 6 instead of 12 fruits; but trials were considered for MRL estimation, since total sample weight was >1 kg.

[a] At last treatment.

[b] Residues expressed as parent equivalents.

Table 68 Residues of tetraniliprole in apples after two pre-harvest foliar treatments using a 200 SC formulation in field trials in Australia

APPLES Year, Location (variety)	Application				DALA	Residues (mg/kg) [b]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl- quinazolinone	
2017, Coomboona, Victoria (Pink lady)	2 (10)	15 16	2.0	20 March, BBCH 86 +NIS	24	0.01	<0.01	BCS-0531 531-1 T4
2017, Coomboona, Victoria (Pink lady)	2 (10)	31 33	4.0 4.0	20 March, BBCH 86 +NIS	24	0.05	<0.01	BCS-0531 531-1 T5

APPLES Year, Location (variety)	Application				DALA	Residues (mg/kg) [b]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl-quinazolinone	
2017, Karragullen, Western (Pink lady)	2 (10)	21 23	2.1 2.0	02 April, BBCH 79 +NIS	22	0.07	<0.01	BCS-0531 531-2 T4
2017, Karragullen, Western Australia (Pink lady)	2 (10)	39 46	4.0 4.0	02 April, BBCH 79 +NIS	22	0.15	<0.01	BCS-0531 531-2 T5
2017, Orange, New South Wales (Pink lady)	2 (11)	42 40	2.0 2.0	20 Feb., BBCH 85 +NIS	24	0.03	<0.01	BCS-0531 531-3 T4
2017, Orange, New South Wales (Pink lady)	2 (11)	80 83	4.0 4.1	20 Feb., BBCH 85 +NIS	24	0.05	<0.01	BCS-0531 531-3 T5
2018, Nashdale, New South Wales (Crimson Snow)	2 (7)	104 105	5.2 5.2	13 April, BBCH 85 +NIS	0 3 7 14	0.13 0.17 0.12 0.24	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-1 T3
2018, Nashdale, New South Wales (Crimson Snow)	2 (7)	52 53	2.6 2.6	13 April, BBCH 85 +NIS	0 3 7 14	0.02 0.03 0.02 <0.01	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-1 T2
2017/2018, Echunga, South Australia (Granny Smith)	2 (8)	26 27	2.6 2.6	12 Jan., BBCH 79-81 +NIS	0 3 7 13	0.06 0.08 0.04 0.04	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-2 T2 [SS]
2017/2018, Echunga, South Australia (Granny Smith)	2 (8)	53 52	5.2 5.2	12 Jan., BBCH 79-81 +NIS	0 3 7 13	0.19 0.12 0.11 0.13	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-2 T3 [SS]
2018, Coomboona, Victoria (Pink Lady)	2 (11)	23 20	2.6 2.6	09 April, BBCH 85 +NIS	0 3 7 14	0.11 0.09 0.08 0.06	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-3 T2
2018, Coomboona, Victoria (Pink Lady)	2 (11)	45 39	5.2 5.2	09 April, BBCH 85 +NIS	0 3 7 14	0.13 0.13 0.19 0.10	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-3 T3

Notes:

RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.

NIS= Non-ionic organic surfactant Agral; [SS] = sample size too small, consisting of 6 instead of 12 fruits, but considered for MRL estimation, since total weight was >1 kg.

[a] Growth stage at last treatment.

[b] Residues are expressed as parent equivalents.

Table 69 Residues of tetraniliprole in apples after a single pre-harvest foliar treatment using a 200 SC formulation in field trials in Australia

APPLES Year, Location (variety)	Application			DALA	Residues (mg/kg) [a]		Study, Trial No.
	g ai/ha	g ai/hL	Date, growth stage		Tetraniliprole	T-N-methyl-quinazolinone	
2017, Coomboona, Victoria (Pink lady)	15	1.9	10 March, BBCH 84 +NIS	34	<0.01	<0.01	BCS-0531 531-1 T2
2017, Coomboona, Victoria (Pink lady)	31	3.9	10 March, BBCH 84 +NIS	34	0.02	<0.01	BCS-0531 531-1 T3
2017, Coomboona, Victoria (Pink lady)	16	2.0	31 March, BBCH 87 +NIS	0 3 7 13	0.04 0.02 0.01 0.03	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-1 T6
2017, Coomboona, Victoria (Pink lady)	32	3.9	31 March, BBCH 87 +NIS	0 3 7 13	0.07 0.07 0.07 0.04	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-1 T7
2017, Karragullen, Western Australia (Pink lady)	20	2.0	23 March, BBCH 75 +NIS	32	0.07	<0.01	BCS-0531 531-2 T2
2017, Karragullen, Western Australia (Pink lady)	40	4.1	23 March, BBCH 75 +NIS	32	0.07	<0.01	BCS-0531 531-2 T3
2017, Karragullen, Western Australia (Pink lady)	24	2.0	11 April, BBCH 81 +NIS	0 3 7 13	0.06 0.07 0.06 0.05	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-2 T6
2017, Karragullen, Western Australia (Pink lady)	49	4.1	11 April, BBCH 81 +NIS	0 3 7 13	0.13 0.10 0.10 0.13	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-2 T7
2017, Orange, New South Wales (Pink lady)	40	2.0	09 Feb., BBCH 81 +NIS	35	0.01	<0.01	BCS-0531 531-3 T2
2017, Orange, New South Wales (Pink lady)	80	4.0	09 Feb., BBCH 81 +NIS	35	0.03	<0.01	BCS-0531 531-3 T3
2017, Orange, New South Wales (Pink lady)	38	2.0	02 March, BBCH 85/87 +NIS	0 3 7 14	0.02 0.02 0.01 0.01	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-3 T6
2017, Orange, New South Wales (Pink lady)	80	4.0	02 March, BBCH 85/87 +NIS	0 3 7 14	0.15 0.05 0.05 0.04	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-3 T7
2018, Nashdale, New South Wales (Crimson Snow)	53	2.6	13 April, BBCH 85 +NIS	0 3 7 14	0.05 0.05 0.02 0.02	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-1 T4
2018, Nashdale, New South Wales (Crimson Snow)	105	5.2	13 April, BBCH 85 +NIS	0 3 7 14	0.10 0.08 0.05 0.05	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-1 T5

APPLES Year, Location (variety)	Application			DALA	Residues (mg/kg) [a]		Study, Trial No.
	g ai/ha	g ai/hL	Date, growth stage		Tetraniliprole	T-N-methyl-quinazolinone	
2017/2018, Echunga, South Australia (Granny Smith)	27	2.6	12 Jan., BBCH 79-81 +NIS	0 3 7 13	0.04 0.04 0.03 0.03	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-2 T4 [SS]
2017/2018, Echunga, South Australia (Granny Smith)	52	5.2	12 Jan., BBCH 79-81 +NIS	0 3 7 13	0.09 0.05 0.07 0.05	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-2 T5 [SS]
2018, Coomboona, Victoria (Pink Lady)	20	2.6	09 April, BBCH 85 +NIS	0 3 7 14	0.03 0.05 0.03 0.06	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-3 T4
2018, Coomboona, Victoria (Pink Lady)	40	5.2	09 April, BBCH 85 +NIS	0 3 7 14	0.06 0.05 0.06 0.05	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-3 T5

Notes:
DALA = Days After Last Application; T = tetraniliprole.
NIS= Non-ionic organic surfactant Agral; [SS] = sample size too small, consisting of 6 instead of 12 fruits.
[a] Residues are expressed as parent equivalents.

Pears

Table 70 Residues of tetraniliprole in pears after three pre-harvest foliar treatments using a 200 SC formulation in field trials in the Australia, Canada and United States.

PEARS Country, Year, Location (variety)	Application				DALA	Residues (mg/kg) [a]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Timing, growth stage[b]		Tetraniliprole	T-N-methyl-quinazolinone	
Australia, 2017/2018, Ashborne, South Australia (Corella)	3 (10,7)	22 22 22	2.6 2.6 2.6	BBCH 79-81 +NIS	0 3 7 15	0.09 0.08 0.15 0.06	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-4 T8
Australia, 2017/2018, Ashborne, South Australia (Corella)	3 (10,7)	21 22 22	2.6 2.6 2.6	04 Jan., BBCH 79-81 +NIS	0 3 7 15	0.09 0.10 0.04 0.08	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-4 T6
Australia, 2017/2018, Ashborne, South Australia (Corella)	3 (10,7)	44 43 43	5.2 5.2 5.2	04 Jan., BBCH 79-81 +NIS	0 3 7 15	0.17 0.18 0.14 0.12	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-4 T7
Australia, 017/2018, Ashborne, South Australia (Corella)	3 (7)	22 22 23	2.6 2.6 2.6	11 Jan., BBCH 81-85 +NIS	0 3 8	0.11 0.13 0.12	<0.01 <0.01 <0.01	BCS-0532 532-4 T9
Australia, 2017/2018, Ashborne, South Australia (Corella)	3 (7)	44 44 43	5.2 5.2 5.2	11 Jan., BBCH 81-85 +NIS	0 3 8	0.15 0.18 0.16	<0.01 <0.01 <0.01	BCS-0532 532-4 T10

PEARS Country, Year, Location (variety)	Application				DALA	Residues (mg/kg) [a]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Timing, growth stage[b]		Tetraniliprole	T-N-methyl-quinazolinone	
Australia, 2018, Shepparton, Victoria (Packham)	3 (9,12)	24 24 26	2.6 2.6 2.6	13 Feb., BBCH 85 +NIS	0 3 6 13	0.05 0.04 0.03 0.02	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-5 T8
Australia, 2018, Shepparton, Victoria (Packham)	3 (9,12)	25 24 26	2.6 2.6 2.6	13 Feb., BBCH 85 +NIS	0 3 6 13	0.05 0.07 0.06 0.05	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-5 T6
Australia, 2018, Shepparton, Victoria (Packham)	3 (9,12)	50 48 47	5.2 5.2 5.2	13 Feb., BBCH 85 +NIS	0 3 6 13	0.09 0.07 0.10 0.07	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-5 T7
Australia, 2018, Shepparton, Victoria (Packham)	3 (8,6)	25 26 25	2.6 2.6 2.6	19 Feb., BBCH 86 +NIS	0 3 7	0.01 0.06 0.05	<0.01 <0.01 <0.01	BCS-0532 532-5 T9
Australia, 2018, Shepparton, Victoria (Packham)	3 (8,6)	50 47 51	5.2 5.2 5.2	19 Feb., BBCH 86 +NIS	0 3 7	0.03 0.10 0.07	<0.01 <0.01 <0.01	BCS-0532 532-5 T10
Australia, 2017, Shepparton East, Victoria (Packham)	3 (11,9)	31 30 27	2.0 2.0 1.9	01 Feb., BBCH 87 +NIS	0 3 6 14	0.08 0.06 0.04 0.02	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-4 T8
Australia, 2017, Shepparton East, Victoria (Packham)	3 (11,9)	60 61 64	4.0 4.0 4.1	01 Feb., BBCH 87 +NIS	0 3 6 14	0.13 0.06 0.04 0.06	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-4 T9
Australia, 2017, Pickering Brook, Western Australia (Packham)	3 (10, 11)	48 48 48	2.0 2.0 2.0	10 March, BBCH 81 +NIS	0 3 7 13	0.13 0.08 0.08 0.11	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-5 T8
Australia, 2017, Pickering Brook, Western Australia (Packham)	3 (10, 11)	95 95 93	4.0 4.0 4.0	10 March, BBCH 81 +NIS	0 3 7 13	0.21 0.30 0.28 0.35	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-5 T9
Canada, 2014 Simcoe, Ontario (Bartlett)	3^ (6,7)	62 62 65	4.9 5.0 5.0	05 Sept., BBCH 85	7	0.044	<0.01	RAFVP104-01 FV204-14HA-TRTDD
Canada, 2014, Simcoe, Ontario (Bartlett)	3^ (6,7)	61 59 62	18 17 18	05 Sept., BBCH 87	7	<u>0.048</u>	<0.01	RAFVP104-01 FV204-14HA-TRTDC
Canada, 2015, Branchton, Ontario (Flemish Beauty)	3^ (7,7)	60 62 60	5.0 39 5.1	16 Sept., BBCH 85	-0 7	0.064 <u>0.081</u>	<0.01 <0.01	RAFVP104-01 FV205-14HB-TRTDD
Canada, 2015, Branchton, Ontario (Flemish Beauty)	3^ (7,7)	60 60 60	39 40 39	16 Sept., BBCH 85	-0 7	0.062 0.062	<0.01 <0.01	RAFVP104-01 FV205-14HB-TRTDC

PEARS Country, Year, Location (variety)	Application				DALA	Residues (mg/kg) [a]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Timing, growth stage[b]		Tetraniliprole	T-N-methyl-quinazolinone	
Canada, 2015, St. Catherines, Ontario (Bartlett)	3^ (5,7)	63 60 60	5.0 5.1 5.1	09 Sept., BBCH 85	-0 0 3 7 9 14	0.21 0.31 0.20 0.13 0.12 0.13	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01 FV206-14DA-TRTDD
Canada, 2015, St. Catherines, Ontario (Bartlett)	3^ (5,7)	61 61 63	39 31 39	09 Sept., BBCH 85	-0 0 3 7 9 14	0.30 0.40 0.35 <u>0.24</u> 0.18 0.18	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01 FV206-14DA-TRTDC
Canada, 2015, St. Catherines, Ontario (Bosc)	3^ (7,7)	59 59 59	5.0 5.0 5.0	09 Sept., BBCH 85	-0 0 3 7 9 14	0.12 0.24 0.15 0.090 0.10 0.089	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01 FV207-14DA-TRTDD
Canada, 2015, St. Catherines, Ontario (Bosc)	3^ (7,7)	61 61 62	39 39 39	09 Sept., BBCH 85	-0 0 3 7 9 14	0.26 0.55 0.34 0.22 0.17 0.21	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01 FV207-14DA-TRTDC
United States, 2014, Williamson, New York (Bartlett)	3^ (5,7)	60 61 61	5.1 5.1 5.2	13 Aug., BBCH 85	-0 7	0.068 0.12	<0.01 <0.01	RAFVP104-01 FV203-14HA-TRTDD
United States, 2014, Williamson, New York (Bartlett)	3^ (5,7)	63 63 63	14 14 14	13 Aug., BBCH 87	-0 7	0.072 <u>0.14</u>	<0.01 <0.01	RAFVP104-01 FV203-14HA-TRTDC
United States, 2015, Wheatland, California (Bartlett)	3^ (6,5)	62 61 60	4.1 4.1 4.1	11 July, BBCH 85	-0 5	0.10 <u>0.13</u>	<0.01 <0.01	RAFVP104-01 FV208-14HB-TRTDD
United States,2015, Wheatland, California (Bartlett)	3^ (6,5)	61 59 61	15 15 15	11 July, BBCH 85	-0 5	0.043 0.084	<0.01 <0.01	RAFVP104-01 FV208-14HB-TRTDC
United States, 2015, Parlier, California (Shinko)	3^ (7,8)	60 60 60	2.6 2.6 2.6	11 Sept., BBCH 87	-0 0 3 7 10 14	0.032 0.047 0.049 0.040 <u>0.044</u> 0.034	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01, FV209-14DA-TRTDD
United States, 2015 Parlier, California (Shinko)	3^ (7,8)	61 59 59	16 16 16	11 Sept., BBCH 87	-0 0 3 7 10 14	0.038 0.056 0.058 0.022 0.026 0.028	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01, FV209-14DA-TRTDC
United States, 2014, Ephrata, Washington (D' Anjou)	3^ (7,7)	59 60 61	4.2 4.2 4.3	09 Sept., BBCH 85	-0 6	0.13 0.14	<0.01 <0.01	RAFVP104-01 FV210-14HA-TRTDD

PEARS Country, Year, Location (variety)	Application				DALA	Residues (mg/kg) [a]		Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Timing, growth stage[b]		Tetraniliprole	T-N-methyl-quinazolinone	
United States, 2014, Ephrata, Washington (D' Anjou)	3^ (7,7)	59 60 60	13 13 13	09 Sept., BBCH 85	-0 6	0.096 <u>0.16</u>	<0.01 <0.01	RAFVP104-01 FV210-14HA-TRTDC
United States, 2014, Hood River, Oregon (Columbia Red Anjou)	3^ (6,7)	62 59 61	3.5 3.5 3.5	10 Sept., BBCH 85	-0 7	0.070 <u>0.080</u>	<0.01 <0.01	RAFVP104-01 FV211-14HA-TRTDD
United States, 2014, Hood River, Oregon (Columbia Red Anjou)	3^ (6,7)	61 60 61	15 15 14	10 Sept., BBCH 85	-0 7	0.063 0.064	<0.01 <0.01	RAFVP104-01 FV211-14HA-TRTDC
United States, 2014, Hood River, Oregon (Starkrimson)	3^ (7,7)	60 59 60	4.8 4.6 5.0	11 Aug., BBCH 87	-0 0 3 7 10 14	0.052 0.091 0.092 0.065 0.067 0.050	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01 FV212-14DA-TRTDD
United States, 2014, Hood River, Oregon (Starkrimson)	3^ (7,7)	61 60 60	16 15 15	11 Aug., BBCH 87	-0 0 3 7 10 14	0.056 0.076 0.078 0.067 0.060 0.049	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RAFVP104-01 FV212-14DA-TRTDC

Notes:
RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.
NIS= Non-ionic organic surfactant Agral.
[a] Residues expressed as parent equivalents.
[b] Growth stage at last treatment.

Table 71 Residues of tetraniliprole in pears after two pre-harvest foliar treatments using a 200 SC formulation in field trials in Australia

PEARS Year, Location (variety)	Application				DALA	Residues (mg/kg) [a]		Study, Reference, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl-quinazolinone	
2017/2018, Ashborne, South Australia (Corella)	2 (7)	22 22	2.6 2.6	04 Jan., BBCH 79-81 +NIS	0 3 7 15	0.08 0.10 0.04 0.05 *(0.23)*	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-4 T2
2017/2018, Ashborne, South Australia (Corella)	2 (7)	44 43	5.2 5.2	04 Jan., BBCH 79-81 +NIS	0 3 7 15	0.19 0.21 0.14 0.10	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-4 T3
2018, Shepparton, Victoria (Packham)	2 (12)	24 25	2.6 2.6	13 Feb., BBCH 85 +NIS	0 3 6 13	0.04 0.05 0.02 0.04	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-5 T2
2018, Shepparton, Victoria (Packham)	2 (12)	49 49	5.2 5.2	13 Feb., BBCH 85 +NIS	0 3 6 13	0.06 0.09 0.09 0.05	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-5 T3

PEARS Year, Location (variety)	Application				DALA	Residues (mg/kg) [a]		Study, Reference, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl-quinazolinone	
2017, Shepparton East, Victoria (Packham)	2 (11)	32 30	2.1 2.0	23 Jan., BBCH 82 +NIS	23	<0.01	<0.01	BCS-0531 531-4 T4
2017, Shepparton East, Victoria (Packham)	2 (11)	60 60	4.0 4.0	23 Jan., BBCH 82 +NIS	23	0.03	<0.01	BCS-0531 531-4 T5
2017, Pickering Brook, Western Australia (Packham)	2 (10)	48 48	2.0 2.0	27 Feb., BBCH 76 +NIS	24	0.05	<0.01	BCS-0531 531-5 T4
2017, Pickering Brook, Western Australia (Packham)	2 (10)	95 95	4.0 4.0	27 Feb., BBCH 76 +NIS	24	0.22	<0.01	BCS-0531 531-5 T5

Notes:
RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.
NIS= Non-ionic organic surfactant Agral;
[a] Residues expressed as parent equivalents.
[b] at the last application

Table 72 Residues of tetraniliprole in pears after a single pre-harvest foliar treatment using a 200 SC formulation in field trials in Australia

Year, Location PEARS (variety)	Application			DALA	Residues (mg/kg) [a]		Study, Reference, Trial No.
	g ai/ha	g ai/hL	Date, growth stage		Tetraniliprole	T-N-methyl-quinazolinone	
2017/2018, Ashborne, South Australia (Corella)	22	2.6	04 Jan., BBCH 79-81 +NIS	0 3 7 15	<0.01 0.08 0.10 0.05	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-4 T4
2017/2018, Ashborne, South Australia (Corella)	43	5.2	04 Jan., BBCH 79-81 +NIS	0 3 7 15	0.07 0.07 0.05 0.05	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-4 T5
2018, Shepparton, Victoria (Packham)	26	2.6	13 Feb., BBCH 85 +NIS	0 3 6 13	0.02 0.02 0.02 0.02	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-5 T4
2018, Shepparton, Victoria (Packham)	52	5.2	13 Feb., BBCH 85 +NIS	0 3 6 13	0.04 0.05 0.04 0.03	<0.01 <0.01 <0.01 <0.01	BCS-0532 532-5 T5
2017, Shepparton East, Victoria (Packham)	32	2.1	12 Jan., BBCH 78 +NIS	34	<0.01	<0.01	BCS-0531 531-4 T2
2017, Shepparton East, Victoria (Packham)	59	4.0	12 Jan., BBCH 78 +NIS	34	0.01	<0.01	BCS-0531 531-4 T3
2017, Shepparton East, Victoria (Packham)	26	1.9	01 Feb., BBCH 87 +NIS	0 3 6 14	0.02 0.05 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-4 T6

Year, Location PEARS (variety)	Application			DALA	Residues (mg/kg) [a]		Study, Reference, Trial No.
	g ai/ha	g ai/hL	Date, growth stage		Tetraniliprole	T-N-methyl-quinazolinone	
2017, Shepparton East, Victoria (Packham)	55	3.8	01 Feb., BBCH 87 +NIS	0 3 6 14	0.05 0.02 0.01 0.01	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-4 T7
2017, Pickering Brook, Western Australia (Packham)	47	2.0	17 Feb., BBCH 74-75 +NIS	34	0.03	<0.01	BCS-0531 531-5 T22017
2017, Pickering Brook, Western Australia (Packham)	95	4.0	17 Feb., BBCH 74-75 +NIS	34	0.07	<0.01	BCS-0531 531-5 T3
2017, Pickering Brook, Western Australia (Packham)	48	2.0	10 March, BBCH 81 +NIS	0 3 7 13	0.05 0.04 0.03 0.03	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-5 T6
2017, Pickering Brook, Western Australia (Packham)	93	4.0	10 March, BBCH 81 +NIS	0 3 7 13	0.09 0.09 0.10 0.07	<0.01 <0.01 <0.01 <0.01	BCS-0531 531-5 T7

Notes:
DALA = Days After Last Application; NIS= Non-ionic organic surfactant Agral; T = tetraniliprole.
[a] Residues expressed as parent equivalents.

Stone fruit

Thirty-eight field trials were conducted to measure the magnitude of tetraniliprole residues in/on cherry (12 trials; Greenland, 2016a, M-570651-01-1, Report SARS-15-15), peach (16 trials; Greenland, 2016b, M-572119-01-1, Report SARS-15-16) and plum (10 trials; Greenland, 2016c, Document M-572124-01-1, Report SARS-14-01) following three foliar air-blast applications of tetraniliprole 200 SC. Trials were carried out in 2014/2015. Applications were made at an actual rate of 58-62 g ai/ha, with application intervals of 5–9 days. Applications were made between growth stage BBCH 74–89. In some trials an adjuvant (non-ionic surfactant (NIS) or crop oil concentrate (COC)) was added. At two of the plum test sites, additional treatment plots were established that were treated at an exaggerated rate to provide samples for processing.

Fruit were harvested when the RAC was at BBCH 81 to 89 (normal commercial harvest), 5 days after the last application (DALA)). Additional decline data was collected from five sites, where samples were taken nominally 0, 3, 5, 7 and 10 days following the final application. For each cherry sample a composite sample, weighing at least 1 kg, from several places on at least four trees within each plot was collected. The cherries were pitted and destemmed and the remaining fruit processed to a fine consistency in the presence of dry ice using a food processor prior to extraction. Samples of peach fruit or plums (a minimum of 24 fruit per sample) weighing at least 2 kg were taken from several places from at least four trees across the plot. Peach and plum stems and stones (pits) were removed prior to sample homogenization

Samples were stored frozen for a maximum of 344 days (*ca* 11 months) prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414 (LOQ of 0.01 mg/kg). The concurrent recoveries were within the acceptable range of 70–120 percent.

Totals were only calculated where residues of tetraniliprole and tetraniliprole-N-methylquinazolinone are above LOQ of 0.01 mg/kg. Except for one trial in plums, which was not considered for MRL estimation, levels of the metabolite were always < LOQ. Therefore, no totals were added to the tables for stone fruits. The results of the trials are summarised in Table 73 to Table 75.

Cherries

Table 73 Residues of tetraniliprole in cherries (fruits, pit removed) after pre-harvest foliar treatments using a 200 SC formulation in field trials in Canada and the United States in 2015 (Study SARS-15-15)

CHERRIES Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole (mg/kg)	T-N-methyl-quinazolinone	
Canada, British Columbia (sweet–Lapin)	3 (7)	60 62 62	15 15 15	22 June BBCH 87 + NIS	5	0.089, 0.080 (0.085)	<0.01, <0.01 (<0.01)	SARS-15-15-BC
United States, Wayne, New York (sour-Montmorency)	3^ (7)	60 60 60	4.9 4.9 4.9	10 July, BBCH 87	5	0.27, 0.28 (0.28)	<0.01, <0.01 (<0.01)	SARS-15-15-NY
United States, Oceana, Michigan sour-Montmorency)	3 (7)	60 60 61	13 16 16	09 July, BBCH 85 +COC	5	0.66, 0.66 (0.66)	<0.01, <0.01 (<0.01)	SARS-15-15-MI2
United States, Oceana, Michigan (sweet – Skeena)	3^ (7)	61 61 60	5.5 5.2 5.2	09 July, BBCH 85	5	0.24, 0.24 (0.24)	<0.01, <0.01 (<0.01)	SARS-15-15-MI1
United States, Ottawa, Michigan (sweet – Sam)	3 (6,7)	60 60 60	1.5 1.5 1.5	28 June, BBCH 87 + NIS	0	0.45, 0.38 (0.42)	<0.01, <0.01 (<0.01)	SARS-15-15-MI3
					3	0.18, 0.24 (0.21)	<0.01, <0.01 (<0.01)	
					5	0.24, 0.24 (0.24)	<0.01, <0.01 (<0.01)	
					7	0.23, 0.26 (0.24)	<0.01, <0.01 (<0.01)	
					10	0.20, 0.19 (0.20)	<0.01, <0.01 (<0.01)	
United States, Berks, Pennsylvania (sour-Montmorency)	3 (6,7)	60 59 60	14 14 14	29 June, BBCH 87 + NIS	0	0.34, 0.36 (0.35)	<0.01, <0.01 (<0.01)	SARS-15-15-PA
					3	0.25, 0.25 (0.25)	<0.01, <0.01 (<0.01)	
					5	0.27, 0.25 (0.26)	<0.01, <0.01 (<0.01)	
					7	0.28, 0.27 (0.27)	<0.01, <0.01 (<0.01)	
					10	0.19, 0.18 (0.18)	<0.01, <0.01 (<0.01)	
United States, Cache, Utah (sour–Montmorency)	3^ (7)	61 61 61	5.2 5.1 5.2	25 July, 95% ripe	5	0.43, 0.45 (0.44)	<0.01, <0.01 (<0.01)	SARS-15-15-UT1
United States, Box Elder, Utah (sour-Montmorency)	3 (6,8)	61 59 60	14 14 14	09 July, BBCH 87 +COC	5	0.64, 0.48 (0.56)	<0.01, <0.01 (<0.01)	SARS-15-15-UT2
United States, Door, Wisconsin (sour-Montmorency)	3 (7)	58 60 60	5.2 5.1 5.2	23 July, All fruit red + NIS	5	0.50, 0.48 (0.49)	<0.01, <0.01 (<0.01)	SARS-15-15-WI
United States, Yuba, California (sweet–Bing)	3 (7)	61 60 60	4.6 4.6 5.8	07 May, BBCH 87 + NIS	5	0.25, 0.33 (0.29)	<0.01, <0.01 (<0.01)	SARS-15-15-CA1
United States, Fresno, California (sweet – Coral Champagne)	3^ (6,7)	59 60 60	13 13 13	1 May, BBCH 87	5	0.13, 0.12 (0.12)	<0.01, <0.01 (<0.01)	SARS-15-15-CA2
United States, Washington, Idaho (sweet – Bing)	3 (7)	60 60 60	5.2 5.2 5.2	17 June, BBCH 86 + NIS	5	0.38, 0.39 (0.38)	<0.01, <0.01 (<0.01)	SARS-15-15-ID

Notes:
+COC = adjuvant added (Crop Oil Concentrate); +NIS= adjuvant added (non-ionic surfactant); ^no adjuvant added; GS = Growth stage at last treatment; RTI = Retreatment Interval; DALA = Days After Last Application; Mean residue values presented in parenthesis; T = tetraniliprole.
[a] Residues expressed as parent equivalents.
[b] At the last application.

Plums

Table 74 Residues of tetraniliprole in plums (pits removed) after pre-harvest foliar treatments using a 200 SC formulation in field trials in Canada and the United States (Study SARS-14-01)

PLUMS Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
Canada, 2015, Fraser Valley, British Columbia (PRM1 grafter on Moyer)	3 (9,5)	300 299 308	30 30 30	22 Aug., BBCH 83 +NIS	5	0.37, 0.45 (0.41)	<0.01, <0.01 (<0.01)	SARS-14-01-BC-3 [p]
Canada, 2015, Waterloo Region Ontario (German)	3^ (7)	58 61 62	16 16 16	10 Sept., BBCH 87	5	0.14, 0.11 (0.13)	<0.01, <0.01 (<0.01)	SARS-14-01-ON-2
United States, 2015, Clackamas, Oregon (Moyer)	3^ (7)	59 59 59	18 18 18	24 Aug., BBCH 87	5	0.014, <0.01 (0.012)	<0.01, <0.01 (<0.01)	SARS-14-01-OR-2
United States, 2014, Wright, Minnesota (Black 16)	3^ (7)	61 61 61	4.8 4.8 4.8	26 Aug., BBCH 89	5	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-14-01-MN-2
United States, 2015, Fresno, California (Honey Punch)	3 (7)	61 61 61	5.1 5.1 5.1	20 July, BBCH 87 + NIS	5	0.10, 0.058 (0.081)	<0.01, <0.01 (<0.01)	SARS-14-01-CA2-2
United States, 2015, Fresno, California (Honey Punch)	3 (7)	60 60 59	16 16 16	20 July, BBCH 85 + NIS	5	0.071, 0.086 (0.079)	<0.01, <0.01 (<0.01)	01SARS-14-01-CA4-2
United States, 2014, Fresno, California (Fortune)	3 (7)	60 59 59	13 13 13	09 July, 100% coloured fruit +COC	0	0.039, 0.043 (0.041)	<0.01, <0.01 (<0.01)	01SARS-14-01-CA1-2
					3	0.033, 0.029 (0.031)	<0.01, <0.01 (<0.01)	
					5	0.034, 0.044 (0.039)	<0.01, <0.01 (<0.01)	
					7	0.041, 0.038 (0.039)	<0.01, <0.01 (<0.01)	
					10	0.034, 0.027 (0.031)	<0.01, <0.01 (<0.01)	
United States, 2015, Madera, California (Fortune)	3 (7)	64 60 60	5.3 5.1 5.1	25 June, Fully coloured +COC	5	0.039, 0.013 (0.026)	<0.01, <0.01 (<0.01)	SARS-14-01-CA3-2
United States, 2015, Grant, Washington (Italian Plum)	3^ (7)	59 60 61	13 13 13	21 Aug., BBCH 83	5	0.013, 0.019 (0.016)	<0.01, <0.01 (<0.01)	SARS-14-01-WA1-2
United States, 2014, Grant, Washington (Italian Plum)	3^ (7)	297 297 300	64 64 64	21 Aug., BBCH 83	5	0.073, 0.13 (0.10)	<0.01, 0.021 (0.015) Total: 0.073, 0.15 (0.12)	SARS-14-01-WA1-3[p]

Notes:
+COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; GS = Growth stage at last treatment; RTI = Retreatment Interval; DALA = Days After Last Application; Mean residue values presented in parenthesis; T = tetraniliprole.
[p] = See processing section for data on prunes.
[a] Residues expressed as parent equivalents.
[b] At the last application.

Peaches

Table 75 Residues of tetraniliprole in peaches pits (removed) after pre-harvest foliar treatments using a 200 SC formulation in field trials in the United States in 2015 (Study SARS-15-16)

PEACHES Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
Wayne, New York (Virgil)	3^ (7)	62 61 61	5.2 5.2 5.2	10 Aug., BBCH 87	5	0.10, 0.070 (0.086)	<0.01, <0.01 (<0.01)	SARS-15-16-NY
Berks, Pennsylvania (Red skin)	3 (7,8)	60 60 60	14 14 14	12 Aug., BBCH 81 + COC	5	0.10, 0.077 (0.091)	<0.01, <0.01 (<0.01)	SARS-15-16-PA
Bingham, Idaho (Later Elberta)	3 (7)	59 61 61	4.9 5.0 5.1	17 Sept., BBCH 89 + NIS	5	0.46, 0.42 (0.44)	<0.01, <0.01 (<0.01)	SARS-15-16-ID1
Canyon, Idaho (Red Golbe)	3^ (7,6)	60 58 57	16 16 16	04 Aug., BBCH 87	0	0.065, 0.062 (0.063)	<0.01, <0.01 (<0.01)	SARS-15-16-ID2
					3	0.072, 0.076 (0.074)	<0.01, <0.01 (<0.01)	
					5	0.057, 0.047 (0.052)	<0.01, <0.01 (<0.01)	
					7	0.046, 0.046 (0.046)	<0.01, <0.01 (<0.01)	
					10	0.11, 0.051 (0.080)	<0.01, <0.01 (<0.01)	
Clarke, Georgia (Contender)	3^ (7)	60 60 59	16 16 16	10 July, BBCH 87	5	0.068, 0.072 (0.070)	<0.01, <0.01 (<0.01)	SARS-15-16-GA2
Sumter, Georgia (Redskin Elberta)	3 (7)	60 60 60	5.3 5.1 5.3	05 Aug., BBCH 85 + COC	5	0.017, 0.044 (0.030)	<0.01, <0.01 (<0.01)	SARS-15-16-GA3
Tift, Georgia (June Prince)	3 (7)	61 61 61	4.6 4.7 4.7	26 May, BBCH 85 + NIS	0	0.20, 0.15 (0.18)	<0.01, <0.01 (<0.01)	SARS-15-16 SARS-15-16-GA1
					3	0.15, 0.13 (0.14)	<0.01, <0.01 (<0.01)	
					5	0.11, 0.076 (0.095)	<0.01, <0.01 (<0.01)	
					7	0.085 0.062 (0.073)	<0.01, <0.01 (<0.01)	
					10	0.043, 0.050 (0.047)	<0.01, <0.01 (<0.01)	
Major, Oklahoma	3^ (7)	60 62 61	17 19 19	01 July, BBCH 83	5	0.073, 0.054 (0.064)	<0.01, <0.01 (<0.01)	SARS-15-16-OK
Madera, California (Springcrest)	3 (7)	61 61 60	14 14 14	27 May, BBCH 89 + NIS	5	0.085, 0.094 (0.089)	<0.01, <0.01 (<0.01)	SARS-15-16-CA1

PEACHES Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
Fresno, California (Kaweah)	3^ (7)	61 60 60	5.4 5.3 5.4	27 July, BBCH 89	5	0.17, 0.14 (0.15)	<0.01, <0.01 (<0.01)	SARS-15-16-CA2
Yolo, California (Babcock – White Peach)	3 (7)	60 60 59	18 18 17	19 June, BBCH 86 +COC	5	0.048, 0.034 (0.041)	<0.01, <0.01 (<0.01)	SARS-15-16-CA3
Kent, Michigan (Vinegold)	3 (7)	60 60 60	14 15 14	03 Aug., BBCH 87 +COC	5	0.17, 0.12 (0.15)	<0.01, <0.01 (<0.01)	SARS-15-16-MI3
Oceana, Michigan (Coralstar)	3 (7)	60 61 60	16 16 16	19 Aug., BBCH 79 + NIS	5	0.38, 0.38 (0.38)	<0.01, <0.01 (<0.01)	SARS-15-16-MI1
Oceana, Michigan (Babygold)	3^ (7)	62 60 60	5.1 5.1 5.1	19 Aug., BBCH 79	5	0.18, 0.090 (0.13)	<0.01, <0.01 (<0.01)	SARS-15-16-MI2
Muskegon, Michigan (Babygold)	3 (8,6)	60 59 60	5.1 4.9 4.9	27 Aug., BBCH 87 +NIS	5	0.22, 0.20 (0.21)	<0.01, <0.01 (<0.01)	SARS-15-16-MI4
Medina, Texas (La Feliciana)	3 (7)	61 60 61	5.4 4.7 4.9	17 June, BBCH 85 +COC	5	0.065, 0.046 (0.056)	<0.01, <0.01 (<0.01)	SARS-15-16-TX

Notes:
+COC = adjuvant added (Crop Oil Concentrate); +NIS= adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.
[a] Residues expressed as parent equivalents.
[b] At the last application.

Berries and other small fruits

Grapes

Fifteen field trials were conducted in Canada and the United States to measure the magnitude of tetraniliprole residues in/on grapes (Greenland, 2016d, M-572121-01-1, Report SARS-14-7) following four foliar applications of a tetraniliprole 200 SC formulation. Trials were carried out in 2014/2015. Applications were made at an actual rate of 44–54 g ai/ha, with application intervals 6– days. Some trials were carried out without the use of adjuvants and in other trials an adjuvant was used (COC or NIS).

At two trial sites (trials SARS-14-07-CA8-3 and -NY-3) additional treatment plots were established that received an exaggerated application (5×) to provide samples for processing.

Samples of grape (a minimum of 12 bunches or parts of 12 bunches) weighing at least 1 kg were taken from random areas across the plots at normal commercial harvest, nominally 14 days after the last application. Additional decline data was collected from 1 site, where samples were taken 0, 7, 14, 21 and 28 days following the final application.

Samples were stored frozen for a maximum of 212 days (*ca* 7 months) prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes. The concurrent recoveries were within the acceptable range of 70–120 percent. The results of the trials are summarised in Table 77.

Table 76 Residues of tetraniliprole in grapes (bunches of grapes) after pre-harvest foliar treatments using an 200 SC formulation in field trials in Canada and the United States in 2014 (Study SARS-14-07)

GRAPES Country, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
Canada, Waterloo Region, Ontario (Sabrevois)	4 (7)	44 44 44 54	12 12 12 12	12 Sept., BBCH 85 +ADJ	14	0.28, 0.25 (0.27)	<0.01, <0.01 (<0.01)	SARS-14-07-ON-2
Canada, Okanagan Valley, British Columbia (Cabernet Sauvignon)	4 (8,7)	44 45 44 44	4.0 4.0 4.0 4.0	03 Oct., BBCH 85 +ADJ	14	1.0, 0.62 (0.82)	<0.01, <0.01 (<0.01)	SARS-14-07-BC-2
United States, Dane, Wisconsin (Concorde)	4 (7)	45 45 45 45	4.3 4.2 4.1 4.3	17 Sept., BBCH 89 +ADJ	14	0.21, 0.18 (0.19)	<0.01, <0.01 (<0.01)	SARS-14-07-WI1-2
United States, Dane, Wisconsin (Concorde)	4^ (6-7)	45 45 45 45	4.0 4.0 3.3 4.4	16 Sept., BBCH 84	14	0.35, 0.233 (0.29)	<0.01, <0.01 (<0.01)	SARS-14-07-WI2-2
United States, Monterey, California (Syrah)	4 (7)	44 47 45 47	3.5 3.4 3.3 3.7	12 Oct., BBCH 89 +ADJ	14	0.073, 0.50 (0.28)	<0.01, <0.01 (<0.01)	SARS-14-07-CA7-2
United States, Fresno, California (Thompson Seedless)	4^ (7-8)	229 226 471 228	54 54 112 54	01 Aug., BBCH 85	14	1.0, 0.56 (0.78)	<0.01, <0.01 (<0.01)	SARS-14-07-CA8-3 [p]
United States, Fresno, California (Thompson Seedless)	4^ (7-8)	46 45 45 46	11 11 11 11	01 Aug., BBCH 85	14	0.061, 0.052 (0.057)	<0.01, <0.01 (<0.01)	SARS-14-07-CA8-2
United States, Fresno, California (Thompson Seedless)	4 (7)	46 46 46 46	12 12 12 12	24 Aug., BBCH 87 +ADJ	14	0.32, 0.21 (0.27)	<0.01, <0.01 (<0.01)	SARS-14-07-CA4-2
United States, Madera, California (Thompson Seedless)	4^ (7)	45 45 45 45	10 10 10 10	07 Aug., BBCH 87	14	0.10, 0.087 (0.094)	<0.01, <0.01 (<0.01)	SARS-14-07-CA2-2
United States, Madera, California (Thompson Seedless)	4 (7)	46 46 46 46	3.5 3.5 3.5 3.5	31 July, BBCH 87 +ADJ	14	0.38, 0.40 (0.39)	<0.01, <0.01 (<0.01)	SARS-14-07-CA1-2
United States, Madera, California (Merlot)	4 (7)	46 46 45 46	3.5 3.5 3.5 3.5	08 Aug., BBCH 88 +ADJ	14	0.23, 0.30 (0.27)	<0.01, <0.01 (<0.01)	SARS-14-07-CA3-2
United States, San Luis Obispo, California (Cabernet)	4 (7)	46 45 44 45	10 10 10 10	05 Sept., BBCH 85 +ADJ	14	0.26, 0.25 (0.25)	<0.01, <0.01 (<0.01)	SARS-14-07-CA6-2

GRAPES Country, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
United States, San Luis Obispo, California (Syrah Noir)	4^ (7)	45 44 44 46	4.2 4.1 4.0 4.1	05 Sept., BBCH 85	14	0.15, 0.13 (0.14)	<0.01, <0.01 (<0.01)	SARS-14-07-CA5-2
United States, Yates, New York (DeChaunac)	4^ (7)	45 45 45 44	3.8 3.8 3.8 3.8	09 Sept., BBCH 85	14	0.23, 0.17 (0.20)	<0.01, <0.01 (<0.01)	SARS-14-07-NY-2
United States, Yates, New York (DeChaunac)	4^ (7)	225 226 226 226	19 19 19 19	09 Sept., BBCH 85	14	2.6, 1.4 (2.0)	<0.01, <0.01 (<0.01)	SARS-14-07-NY-3[p]
United States, Lehigh, Pennsylvania (Noiret)	4 (7)	45 45 45 45	10 10 10 10	19 Sept., BBCH 87 +ADJ	0 7 14 21 28	0.30, 0.30 (0.30) 0.27, 0.17 (0.22) 0.28, 0.25 (0.26) 0.22, 0.30 (0.26) 0.10, 0.15 (0.12)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	SARS-14-07-PA-2
United States, 2014 Grant, Washington (Chardonnay)	4 (7)	45 45 45 45	12 12 12 12	17 Sept., BBCH 85 +ADJ	14	0.90, 0.95 (0.92)	<0.01, <0.01 (<0.01)	SARS-14-07-WA-2

Notes:

ADJ = adjuvant added, either COC or NIS.
^no adjuvant added.
RTI = Retreatment Interval.
DALA = Days After Last Application.
T = tetraniliprole.
[p] See section on processing for data on raisins and juice.
[a] Residues are expressed as parent equivalents.
[b] At the last application.

Brassica vegetables, except brassica leafy vegetables

Twenty field trials were conducted to measure the magnitude of tetraniliprole residues in/on broccoli, cabbage, and cauliflower following four applications of a tetraniliprole 200 SC formulation (Netzband & Roberts, 2016, M-565724-02-1, Report RAFVP096-01). Trials were carried out in 2014. Applications were made at actual rates of 43–47 g ai/ha, with application intervals of 4–5 days without addition of an adjuvant.

Samples of florets (cauliflower), curd with stalks (broccoli) and heads with or without wrapper leaves (head cabbage) were collected at maturity, 1-day after the final application. Additional decline data was collected from eight sites, where samples were generally taken 0, 1, 5, 10 and 14 days following the final application. Each sample of broccoli, cauliflower, and head cabbage consisted of a composite from at least 12 individual plants and, for broccoli and cauliflower, weighed a minimum of 1 kg (with the exception of the residue reduction samples and trials FV262-14DA and FV267-14DA, indicated with [SS]).

For broccoli and cauliflower samples, the flower head and stem were collected by hand. For cabbage, the head was collected by hand, and if applicable, wrapper leaves were manually removed.

Samples were stored frozen for a maximum of 633 days (broccoli), 617 days (cauliflower), and 699 days (*ca* 23 months, cabbage) prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. In the trials summarized below, levels of the metabolite were always below the LOQ. Therefore, no totals were added to the tables for flowerhead brassicas.

The results of the trials are summarised in Table 77 to Table 79.

Broccoli

Table 77 Residues of tetraniliprole in broccoli (curd with stalk) after pre-harvest foliar treatments using a 200 SC formulation in field trials in the United States in 2014 (Study RAFVP096-01)

BROCCOLI Year, Location (variety)	Application					Residues (mean) (mg/kg) [a]		, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
Richland, Iowa (Walthan 29)	4^ (4,5,4)	45 45 45 46	20 16 20 20	06 Aug., BBCH 49	0 1 4 8 12	0.080, 0.14 (0.11) 0.14, 0.16 (<u>0.15</u>) 0.11, 0.069 (0.089) 0.029, 0.028 (0.028) <0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV262-14DA [SS]
Lime springs, Iowa (Packman)	4^ (4,4,5)	45 46 43 45	19 19 19 19	28 July, BBCH 49	1	0.17, 0.20 (<u>0.18</u>)	<0.01, <0.01 (<0.01)	FV263-14HA
Uvalde, Texas (Marathon)	4^ (5)	46 45 45 46	19 19 19 19	13 Jan., BBCH 45	1	0.12, 0.096 (<u>0.11</u>)	<0.01, <0.01 (<0.01)	FV264-14HB
Madera, California (Heritage)	4^ (4,5,5)	44 47 47 47	16 16 16 16	24 Nov., BBCH 47	0 1 4 10 14	0.23, 0.21 (0.22) 0.25, 0.23 (<u>0.24</u>) 0.12, 0.13 (0.12) 0.035, 0.062 (0.049) 0.029, 0.022 (0.025)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV265-14DB
Santa Maria, California (Heritage)	4^ (5,5,5)	45 46 45 46	16 16 16 16	14 Dec., BBCH 46	1	0.17, 0.11 (<u>0.14</u>)	<0.01, <0.01 (<0.01)	FV266-14HA[p]

Notes:

^No adjuvant added; RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.

[p] See section on processing for data on cooked and washed curd.

[SS] Samples size <1 kg.

[a] Residues are expressed as parent equivalents.

[b] At the last application.

Cauliflower

Table 78 Residues of tetraniliprole in cauliflower (curd) after pre-harvest foliar treatments using an 200 SC formulation in field trials in the United States in 2014 (Study RAFVP096-01)

CAULIFLOWER Location (variety)	Application					Residues (mean) (mg/kg) [a]		Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
Richland, Iowa (Early snowball)	4^ (4)	45 46 45 45	20 20 20 20	14 Aug., BBCH 46	0 1 4 8 12	0.29, 0.23 (0.26) 0.17, 0.15 (<u>0.16</u>) 0.071, 0.078 (0.075) 0.016, 0.024 (0.020) <0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV267-14HA [SS]
Bagley, Iowa (Early snowball)	4^ (5,4,5)	43 44 46 45	20 23 23 21	27 Aug., BBCH 49	1	0.19, 0.19 (<u>0.19</u>)	<0.01, <0.01 (<0.01)	FV268-14HA
Yuba City, California (Snow Ball)	4^ (5)	45 45 46 45	24 24 24 24	10 June, BBCH 49	0 1 5 10 12	0.079, 0.088 (0.083) 0.066, 0.066 (<u>0.066</u>) 0.065, 0.035 (0.050) 0.012, 0.016 (0.014) <0.01, 0.015 (0.13)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV269-14HA
King City, California (Snowball)	4^ (4,5,5)	44 47 46 46	9.7 9.8 9.7 9.8	05 Jan., BBCH 49	1	0.14, 0.075 (<u>0.11</u>)	<0.01, <0.01 (<0.01)	FV270-14HA
Hillsboro, Oregon (Symphony)	4^ (5)	46 46 46 46	25 24 24 25	14 Aug., BBCH 49	1	0.028, 0.044 (<u>0.036</u>)	<0.01, <0.01 (<0.01)	FV271-14HA

Notes:
^No adjuvant added; DALA = Days After Last Application; RTI = Retreatment Interval; T = tetraniliprole.
[SS] Samples size <1 kg.
[a] Residues are expressed as parent equivalents.
[b] At the last application.

Cabbage

Table 79 Residues of tetraniliprole in cabbage head with wrapper leaves and without whapper leaves after pre-harvest foliar treatments using a 200 SC formulation in field trials in the United States in 2014 (Study RAFVP096-01)

CABBAGE Location (variety)	Application						Residues (mean) (mg/kg) [a]		Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Commodity	Tetraniliprole	T-N-methyl-quinazolinone	
Alton, New York (Fario)	4^ (5,4,5)	45 45 45 45	24 24 24 24	14 Aug., BBCH 46	0 1 5 9 14	with wrapper leaves	0.25, 0.24 (0.24) 0.12, 0.075 (<u>0.096</u>) 0.080, 0.089 (0.085) 0.062, 0.085 (0.074) 0.039, 0.025 (0.032)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV272-14DA

CABBAGE Location (variety)	Application					Commodity	Residues (mean) (mg/kg) [a]		Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA		Tetraniliprole	T-N-methyl-quinazolinone	
					0	without whapper leaves	0.022, 0.014 (0.018)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
					1		<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
					5		<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
					9		<0.01, <0.01 (<0.01)		
					14		<0.01, <0.01 (<0.01)		
Chula, Georgia (Cheer)	4^ (4,5,4)	45 44 46 46	22 21 22 21	16 Nov., BBCH 49	1	with wrapper leaves	0.045, 0.046 (0.046)	<0.01, <0.01 (<0.01)	FV273-14HA
					1	without whapper leaves	<0.01, 0.010 (0.010)	<0.01, <0.01 (<0.01)	
Center Hill, Florida (Benelli)	4^ (3,4,4,)	46 47 46 46	16 16 16 16	14 April, BBCH 46	1	with wrapper leaves	0.97, 1.2 (1.1)	<0.01, <0.01 (<0.01)	FV274-14HA
					1	without whapper leaves	0.017, 0.023 (0.020)	<0.01, <0.01 (<0.01)	
Carlyle, Illinois (Stone head)	4^ (5,4,5)	46 46 46 46	21 20 22 21	02 July, BBCH 48	0	with wrapper leaves	0.040, 0.032 (0.036)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV275-14DA
					1		0.036, 0.068 (0.052)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
					5		0.088, 0.058 (0.073)	<0.01, <0.01 (<0.01)	
					9		0.018, 0.020 (0.019)		
					14		<0.01, <0.01 (<0.01)		
					0	without whapper leaves	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
					1		0.011, <0.01 (0.011)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
					5		<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
					9		<0.01, <0.01 (<0.01)		
					14		<0.01, <0.01 (<0.01)		
Northwood, North Dakota (Stone head)	4^ (5,4,5)	44 45 46 45	24 24 24 24	05 Aug., BBCH 49	0	with wrapper leaves	0.33, 0.33 (0.33)	<0.01, <0.01 (<0.01)	FV276-14DA
					1		0.31, 0.40 (0.35)	<0.01, <0.01 (<0.01)	
					5		0.29, 0.36 (0.33)	<0.01, <0.01 (<0.01)	
					9		0.21, 0.22 (0.21)	<0.01, <0.01 (<0.01)	
					14		0.17, 0.22 (0.20)	<0.01, <0.01 (<0.01)	

CABBAGE Location (variety)	Application				DALA	Commodity	Residues (mean) (mg/kg) [a]		Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]			Tetraniliprole	T-N-methyl-quinazolinone	
					0 1 5 9 14	without whapper leaves	0.021, 0.016 (0.019) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
Stafford, Kansas (Stonehead cabbage)	4^ (5,4,5)	46 45 45 45	37 37 37 37	25 June, BBCH 47	0 1 5 8 14	with wrapper leaves	0.61, 0.685 (0.65) 0.25, 0.36 (0.31) 0.24, 0.19 (0.21) 0.14, 0.13 (0.13) 0.10, 0.091 (0.096)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV277-14DA
					0 1 5 8 14	without whapper leaves	0.032, 0.026 (0.029) 0.015, 0.025 (0.020) 0.012, <0.01 (0.11) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
Springfield, Nebraska (Early Dutch)	4^ (4,5,4)	45 45 44 45	34 35 34 34	15 June, BBCH 49	1	with wrapper leaves	0.079, 0.095 (0.087)	<0.01, <0.01 (<0.01)	FV278-14HA
						without whapper leaves	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
Delavan, Wisconsin (Vantage Point)	4^ (4,5,5)	44 43 44 44	18 18 17 18	20 Oct., BBCH 49	1		0.11, 0.13 (0.12)	<0.01, <0.01 (<0.01)	FV279-14HA
					1	without whapper leaves	0.016, <0.01 (0.013)	<0.01, <0.01 (<0.01)	
Uvalde, Texas (Pennant)	4^ (5,4,4)	45 45 46 45	24 26 27 26	17 June, BBCH 49	1	with whapper leaves	0.19, 0.11 (0.15)	<0.01, <0.01 (<0.01)	FV280-14HA
						without whapper leaves	0.033, 0.018 (0.026)	<0.01, <0.01 (<0.01)	
Madera, California (Golden Cross)	4^ (4,5,5)	46 46 46 46	19 19 19 19	26 May, BBCH 49	1	with whapper leaves	0.38, 0.58 (0.48)	<0.01, <0.01 (<0.01)	FV281-14HA

CABBAGE Location (variety)	Application						Residues (mean) (mg/kg) [a]		Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Commodity	Tetraniliprole	T-N-methyl-quinazolinone	
						without whapper leaves	0.037, <0.01 (0.024)	<0.01, <0.01 (<0.01)	

Notes:

^No adjuvant added; DALA = Days After Last Application; RTI = Retreatment Interval; T = tetraniliprole.

[a] Residues are expressed as parent equivalents.

[b] At the last application.

Fruiting vegetables, other than cucurbits

Tomato

Nineteen field trials were conducted in the United States in 2014/2015 to measure the magnitude of tetraniliprole residues in/on tomato following four foliar applications of a tetraniliprole 200 SC formulation (Greenland, 2016e, M-572627-01-1, Report SARS-14-19). Applications were made at an actual rate of 43–47 g ai/ha, with application intervals of 4–6 days. Three trials included an additional plot which was treated with one soil drench application of tetraniliprole 200 SC at the maximum label rate (200 g ai/ha). Data on the soil types were not reported. At two of the trial sites (NY-3 and GA-3) additional treatment plots were established that received an exaggerated application rate (4 times 222–231 g ai/ha), to provide samples for processing.

Samples of fruit (a minimum of 24 medium (or 12 large) fruits weighing at least 2 kg, taken from random areas across the plots) were collected at maturity, nominally 1-day after the last foliar application. Additional information provided by the applicant showed that the typical fruits size varied from small sized varieties 28–81 g, medium sized varieties (170–280 g), to large varieties (up to 900 g). For the plots treated with a soil application, samples were harvested at maturity (14 days after treatment). Additional decline data was collected from 2 sites, where samples were taken 0, 1, 3, 5 and 10 days following the final foliar application, and 3, 7, 14, 21 and 28 days following the soil application.

Samples were stored frozen for a maximum of 299 days (*ca* 10 months) prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. In the trials summarized below, levels of the metabolite were always below the LOQ. Therefore, no totals were added to the tables for tomatoes.

The results of the trials are summarised in Table 80 and Table 81.

Table 80 Residues of tetraniliprole in tomato after pre-harvest foliar treatments using a 200 SC formulation in field trials in the United States performed (Study SARS-140-19)

TOMATO, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole (mg/kg)	T-N-methyl-quinazolinone	
2014, Tift, Georgia (BHN 602- 280-340 g)	4 (5)	46 46 46 45	6.6 6.5 20 21	21 Oct., BBCH 76 +COC	1	0.082, 0.068 (0.075)	<0.01, <0.01 (<0.01)	SARS-14-19-GA-2

TOMATO, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole (mg/kg)	T-N-methyl-quinazolinone	
2014, Tift, Georgia (BHN 602 – 280-340 g)	4 (5)	222 231 228 229	33 33 102 104	21 Oct., BBCH 76 +COC	1	0.39, 0.35 (0.37)	<0.01, <0.01 (<0.01)	SARS-14-19-GA-3 [p]
2014, Stearns, Minnesota (Roma – Plum Roma – 57 g)	4^ (5)	43 43 43 43	23 23 23 23	03 Sept., BBCH 89	1	0.026, 0.033 (0.030)	<0.01, <0.01 (<0.01)	SARS-14-19-MN-2
2014, Shelby, Missouri (Celebrity F1–200-230 g)	4 (5)	46 45 46 46	22 23 23 22	02 Sept, Green and ripe fruit, +COC	1	0.060, 0.047 (0.053)	<0.01, <0.01 (<0.01)	SARS-14-19-MO1-2
2014, Fresno, California (Ouali +23 – 255-280 g)	4 (5)	46 45 46 46	24 24 24 24	07 Aug., BBCH 82 +NIS	1	0.074, 0.050 (0.062)	<0.01, <0.01 (<0.01)	SARS-14-19-CA2-2
2014, Fresno, California (Roma–Plum Roma–57 g)	4 (5)	46 45 46 45	24 24 24 24	14 Aug., BBCH 73 +COC	0 1 3 5 10	0.075, 0.12 (0.095) 0.093, 0.13 (0.11) 0.32, 0.15 (0.23) 0.050, 0.16 (0.10) 0.14, 0.084 (0.11)	<0.01, <0.01 (<0.01)	SARS-14-19-CA1-2
2014, Wayne, New York (Mountain fresh)	4^ (5)	45 46 46 46	24 24 24 24	04 Sept., Green and red fruit	1	0.046, 0.038 (0.042)	<0.01, <0.01 (<0.01)	SARS-14-19-NY-2
2014, Wayne, New York (Mountain fresh)	4^ (5)	225 226 225 231	120 120 120 120	04 Sept., Green and red fruit	1	0.31, 0.46 (0.39)	<0.01, <0.01 (<0.01)	SARS-14-19-NY-3 [p]
2015, Cass, North Dakota (Early Girl)	4 (5)	45 47 45 46	24 24 24 24	28 Aug., BBCH 81 +NIS	1	0.057, 0.050 (0.053)	<0.01, <0.01 (<0.01)	SARS-14-19-ND-2
2015, Freeborn, Minnesota (Celebrity F1)	4 (5)	45 46 45 45	20 21 21 21	10 Sept., BBCH 86 +COC	1	0.047, 0.036 (0.042)	<0.01, <0.01 (<0.01)	SARS-14-19-MN2-2
2015, Shelby, Missouri (Celebrity F1)	4 (5)	45 45 45 45	18 17 17 16	08 Oct., Flowering to baring red fruit +NIS	1	0.059, 0.086 (0.072)	<0.01, <0.01 (<0.01)	SARS-14-19-MO2-2
2015, Shelby, Missouri (Juliet – mini Roma)	4^ (5)	45 45 45 45	16 17 17 16	21 Aug., Flower to ripe fruit	1	0.19, 0.25 (0.22)	<0.01, <0.01 (<0.01)	SARS-14-19-MO3-2
2015, Sutter California (Heinz 144107 – medium oval fruit)	4^ (5)	46 46 45 45	16 16 16 16	06 Aug., BBCH 86	1	0.088, 0.070 (0.079)	<0.01, <0.01 (<0.01)	SARS-14-19-CA4-2

TOMATO, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole (mg/kg)	T-N-methyl-quinazolinone	
2015 San Luis Obispo, California (Sungold – Cherry)	4 (5)	44 44 44 48	10 9.4 9.4 9.3	16 Sept., BBCH 88 +COC	1	0.35, 0.30 (0.32)	<0.01, <0.01 (<0.01)	SARS-14-19-CA5-2
2015, Fresno, California (Q27 – large to very large)	4^ (5)	46 45 46 46	24 24 24 24	31 July, BBCH 89	1	0.028, 0.052 (0.040)	<0.01, <0.01 (<0.01)	SARS-14-19-CA7-2
2015, Fresno California (DRI 319)	4 (5)	45 44 45 45	16 16 16 16	25 Aug., BBCH 87 + NIS	1	0.074, 0.059 (0.066)	<0.01, <0.01 (<0.01)	SARS-14-19-CA3-2
2015, Kings, California (5608)	4 (6,5,5)	43 46 45 45	9.3 9.7 9.6 9.7	11 July, BBCH 89 +NIS	1	0.062, 0.066 (0.064)	<0.01, <0.01 (<0.01)	SARS-14-19-CA6-2
2015, Seminole, Florida (Better boy)	4 (5)	47 46 45 46	16 16 16 16	BBCH 81 +NIS [c]	1	0.053, 0.061 (0.057)	<0.01, <0.01 (<0.01)	SARS-14-19-FL1-2
2015, Madison, Florida (Red bounty)	4 (5)	45 43 44 46	11 11 11 11	21 Oct., 75% fruit typical size +COC	1	0.12, 0.11 (0.12)	<0.01, <0.01 (<0.01)	SARS-14-19-FL2-2
2015, Jefferson Iowa (Delicious)	4^ (5)	45 45 45 45	21 21 21 21	16 Aug., BBCH 75	1	0.075, 0.086 (0.080)	<0.01, <0.01 (<0.01)	SARS-14-19-IA-2
2015, York, Nebraska (Beef eater)	4 (5,5,4)	46 45 44 45	22 21 21 21	09 Sept., BBCH 81 + NIS	1	0.036, 0.033 (0.034)	<0.01, <0.01 (<0.01)	SARS-14-19-NE-2

Notes:
DALA = Days After Last Application; +COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval; T = tetraniliprole.
[a] Residue are expressed as parent equivalents.
[b] At the last application.
[c] NIS applied only once with first application.
[p] See section on processing for data on paste and puree.

Table 81 Residues of tetraniliprole in tomato after a soil drench application using a 200 SC formulation in field trials in the United States performed in 2015 (SARS-14-19)

TOMATO Location (variety)	Application			Residues (mg/kg) [a]			Trial No.
	g ai/ha	g ai/hL	Date, growth stage	DALA	Tetraniliprole (mg/kg)	T-N-methyl-quinazolinone	
Shelby, Missouri (Celebrity F1)	197	137	25 Sept., Flowering to green fruit +NIS	14	<0.01, 0.021 (mean: 0.016)	<0.01 (2)	SARS-14-19-MO2-4

TOMATO Location (variety)	Application			Residues (mg/kg) [a]			Trial No.
	g ai/ha	g ai/hL	Date, growth stage	DALA	Tetraniliprole (mg/kg)	T-N-methyl-quinazolinone	
Sutter, California (Heinz 144107)	200	31	24 July, BBCH 69	14	<0.01 (2)	<0.01 (2)	SARS-14-19-CA4-4
Jefferson, Iowa (Delicious)	200	31	01 Aug., BBCH 69	3 7 14 21 28	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.01 (2)	SARS-14-19-IA-4

Notes:
DALA = Days After Last Application; +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; T = tetraniliprole.
[a] Residues are expressed a parent equivalents.

Peppers

Thirteen field trials were conducted in the United States to measure the magnitude of tetraniliprole residues in/on pepper (sweet and chili) following four foliar applications of a tetraniliprole 200 SC formulation (Greenland, 2016f, M-570122-01-1, Report SARS-14-20). Applications were made at an actual rate of 43–47 g ai/ha with application intervals of 5–6 days. Trials were carried out in 2014/2015. The field trials were carried out with (+COC or +NIS) or without (indicated by ^) use of adjuvants.

Samples of fruit (a minimum of 24 medium (or 12 large) fruits weighing at least 2.4 kg, taken from random areas across the plots) were collected at maturity, nominally 1-day after the last foliar application. Additional decline data was collected from 1 site, where samples were taken 0, 1, 3, 5 and 10 days following the final application.

Samples were stored frozen for a maximum of 285 days (*ca* 9 months) prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. The specific recovery data are summarised in the analytical section.

Totals were only calculated where residues of parent tetraniliprole and tetraniliprole-N-methylquinazolinone are above LOQ of 0.01 mg/kg. In the trials summarized below, levels of the metabolite were always below the LOQ. Therefore, no totals were added to the tables for peppers.

The results of the trials in sweet peppers and chili peppers are summarised in Table 82.

Table 82 Residues of tetraniliprole in sweet peppers and chili-pepper (CP) after four pre-harvest foliar treatments using a 200 SC formulation in field trials in the United States (Study SARS-14-20)

PEPPER Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai /ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
2014, Fresno, California (Encore)	4^ (5)	46 46 46 45	24 24 24 24	28 Oct., BBCH 89	1	0.072, 0.086 (0.079)	<0.01, <0.01 (<0.01)	SARS-14-20-CA1
2014, Armstrong, Texas (Big Jim Numex)	4 (5)	46 46 44 46	22 22 21 22	04 Sept., BBCH 89 +NIS	1	0.011, <0.01 (0.011)	<0.01, <0.01 (<0.01)	SARS-14-20-TX2

PEPPER Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai /ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
2014, Stearns, Minnesota (King Arthur)	4 (5)	43 43 43 43	23 23 23 23	13 Sept., BBCH 89 +NIS	1	0.053, 0.044 (0.048)	<0.01, <0.01 (<0.01)	SARS-14-20-MN1
2014, Stearns, Minnesota (Wisconsin Lakes)	4^ (5)	45 45 46 45	24 24 24 24	14 Sept., BBCH 89	1	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-14-20-MN2
2014, Wharton, Texas (X3R Camelot)	4 (5)	45 45 45 45	27 25 25 25	23 Oct., BBCH 73 +COC	1	0.11, 0.19 (0.15)	<0.01, <0.01 (<0.01)	SARS-14-20-TX1
2014, Seminole, Florida (California Wonder)	4 (5)	47 46 45 45	16 16 16 16	30 Nov., BBCH 72 +NIS	1	0.080, 0.071 (0.075)	<0.01, <0.01 (<0.01)	SARS-14-20-FL
2014, Tift, Georgia (Aristole)	4^ (5)	44 45 46 45	11 11 20 20	21 Oct., BBCH 86	0 1 3 5 10	0.025, 0.023 (0.024) 0.022, 0.020 (0.021) 0.025, 0.022 (0.024) 0.016, 0.021 (0.019) 0.015, 0.015 (0.015)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	SARS-14-20-GA
2015, Shelby, Missouri (King Arthur)	4 (5)	46 45 46 46	24 24 24 24	21 Aug., BBCH 85 +COC	1	0.20, 0.19 (0.20)	<0.01, <0.01 (<0.01)	SARS-14-20-MO
2015, San Luis Obispo, California (Crusader)	4^ (5)	45 45 45 45	9.3 9.3 9.3 9.4	23 Oct., BBCH 89	1	0.092, 0.095 (0.093)	<0.01, <0.01 (<0.01)	SARS-14-20-CA3
2015, Walworth, Wisconsin (Garfield)	4^ (5)	46 46 45 45	28 27 25 26	11 Aug., BBCH 85	1	0.046, 0.036 (0.041)	<0.01, <0.01 (<0.01)	SARS-14-20-WI
2015, Cass, North Dakota (Red Knight)	4 (5)	45 45 44 45	24 24 24 24	28 Aug., BBCH 80 +COC	1	0.071, 0.083 (0.077)	<0.01, <0.01 (<0.01)	SARS-14-20-ND
2014, Fresno, California (Compadre) [CP]	4 (5)	46 45 47 46	24 24 24 24	28 Oct., BBCH 89 +COC	1	0.083, 0.092 (0.087)	<0.01, <0.01 (<0.01)	SARS-14-20-CA2
2015, Cache, Utah (Early Jalapeno) [CP]	4 (5)	47 44 45 43	31 32 30 31	27 Aug., BBCH 89 +NIS	1	0.10, 0.11 (0.11)	<0.01, <0.01 (<0.01)	SARS-14-20-UT

Notes:

+COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); [CP] = Chili pepper; GS = Growth stage at last treatment; RTI = Retreatment Interval; DALA = Days After Last Application; [a] Residues are expressed as parent equivalents.

[b] At the last application

Leafy vegetables (including Brassica leafy vegetables)

Leafy greens

Twenty-six field trials were conducted in the United States to measure the magnitude of tetraniliprole residues in/on leaf lettuce (11 trials) (Greenland, 2016g, M572118-01-1, Report SARS-14-11), head lettuce (six trials) (Greenland, 2016h, M570646-01-1, Report SARS-15-12) and spinach (nine trials) (Greenland, 2016i, M5720124-01-1, Report SARS-14-14), following four foliar applications of a tetraniliprole SC formulation. Applications were made at an actual rate of 43–50 g ai/ha with application intervals of 2–4 days. Trials were carried out in 2014/2015. The field trials were carried out with (+COC or +NIS) or without (indicated by ^) use of adjuvants. At three of the spinach trial sites, an additional treatment plot was established that received a single soil application at the base of the spinach plants at a rate of 201–202 g ai/ha, 14 days prior to harvest.

Samples of lettuce leaves, lettuce heads (with and without wrapper leaves) and spinach leaves were collected at maturity, nominally 1 day following the final foliar application at BBCH 47-49 and were taken from random areas across the plots. Samples of leaf lettuce consisted of above ground portions of a minimum of 12 plants, weighing at least 1 kg. Samples of lettuce heads consisted of a minimum of 12 heads (or sections of 12 heads) with wrapper leaves or twelve heads (or sections of 12 heads) without wrapper leaves) weighing at least 1 kg. Samples of spinach leaves, weighing at least 1 kg, were taken from at least 12 plants. Following the soil application, spinach leaves were collected after 14 days (at maturity).

Additional decline data was collected from 3 foliar treated plot sites, where samples were taken 0, 1, 3, 5 and 10 days following the final application. Decline samples were also collected from the soil treated plot at 3, 7-, 14-, 21-, and 28-days following application.

Samples were stored frozen for a maximum of 268 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. The results of the trials are summarised in Tables 83 to Table 86.

Head lettuce

Table 83 Residues of tetraniliprole in head lettuce (heads with (WWL) or without (WOWL) wrapper leaves) after pre-harvest foliar treatments using a 200 SC formulation in field trials in the United States in 2015 (Study SARS-15-12)

HEAD LETTUCE Location (varietly)	Application					Residues (mean) (mg/kg) [a]		Trial No. sample
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methylquinazolinone	
Wayne, New York (NI244-4402)	4^ (3)	46 45 46 46	18 18 18 18	31 Aug., Vegetative	0 1 3 5 10	1.5, 1.5 (1.5) 2.1, 1.9 (2.0) 0.98, 1.0 (1.0) 0.85, 0.80 (0.83) 0.72, 0.38 (0.55)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	SARS-15-12-NY, *WWL*
					0 1 3 5 10	0.24, 0.16 (0.20) 0.13, 0.13 (0.13) 0.21, 0.28 (0.24) 0.26, 0.25 (0.25) 0.014, 0.012 (0.013)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	*WOWL*

HEAD LETTUCE Location (varietly)	Application					Residues (mean) (mg/kg) [a]		Trial No. sample
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methylquinazolinone	
Yolo, California (Great Lakes 659)	4 (3)	45 45 45 45	16 16 16 16	17 Dec., BBCH 49 +COC	1	2.7, 2.8 (2.7)	<0.01, <0.01 (<0.01)	SARS-15-12-CA1, *WWL*
					1	0.98, 0.76 (0.87)	<0.01, <0.01 (<0.01)	*WOWL*
2015, Fresno, California (Great Lakes)	4 (3)	45 45 46 45	24 24 24 24	27 Nov., BBCH 49 +NIS	1	0.43, 0.58 (0.51)	<0.01, <0.01 (<0.01)	SARS-15-12-CA4, *WWL*
					1	0.011, 0.029 (0.020)	<0.01, <0.01 (<0.01)	*WOWL*
Fresno, California (Crispino MTO 06)	4^ (4,2,2)	45 45 45 45	16 16 16 16	14 Nov., BBCH 49	1	1.4, 1.1 (1.3)	<0.01, <0.01 (<0.01)	SARS-15-12-CA2, *WWL*
					1	0.014, 0.020 (0.017)	<0.01, <0.01 (<0.01)	*WOWL*
Palm Beach, Florida (Iceberg)	4 (3)	45 45 45 45	30 31 30 30	22 Dec., BBCH 49 +COC	1	0.41, 0.47 (0.43)	<0.01, <0.01 (<0.01)	SARS-15-12-FL, *WWL*
					1	0.023, 0.022 (0.023)	<0.01, <0.01 (<0.01)	*WOWL*
San Luis Obispo, California (Regency)	4 (3)	46 45 46 44	16 16 16 16	30 June, BBCH 49 +COC	1	1.3, 1.2 (1.2)	<0.01, <0.01 (<0.01)	SARS-15-12-CA3,*WWL*
					1	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	*WOWL*

Notes:
+COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; DALA = Days After Last Application;; RTI = Retreatment Interval; T = tetraniliprole.
[a] Residues are expressed as parent equivalents.
[b] At the last application

Leaf lettuce

Table 84 Residues of tetraniliprole in leaf lettuce (leaves) after pre-harvest foliar treatments using a 200 SC formulation in field trials in the United States (Study SARS-14-11)

Year, Location LEAF LETTUCE (variety)	Application					Residues (mean) (mg/kg) [a]		Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methylquinazolinone	
2014, Fresno, California (Green Leaf)	4 (3)	46 46 46 46	24 24 24 24	24 Oct., BBCH 49 +NIS	0 1 3 5 10	2.2, 2.0 (2.1) 3.6, 3.6 (3.6) 3.3, 3.6 (3.4) 0.85, 0.54 (0.70) 0.86, 1.4 (1.1)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	SARS-14-11-CA1
2014, Stearns, Minnesota (Ruby Sky)	4^ (3)	45 45 45 45	24 24 24 24	25 Sept., BBCH 49	1	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-14-11-MN1
2015, Cass, North Dakota (Grand Rapids)	4 (3)	47 45 44 46	24 24 24 24	30 July, BBCH 47 +NIS	1	1.8, 1.6 (1.7)	0.011, <0.01 (0.011) (total: 1.8, 1.6 (1.7))	SARS-14-11-ND
2015, Fresno, California (Green Star)	4 (3)	45 45 45 45	16 16 16 16	28 Oct., BBCH 49 +NIS	1	3.3, 2.8 (3.1)	<0.01, <0.01 (<0.01)	SARS-14-11-CA4

Year, Location LEAF LETTUCE (variety)	Application					Residues (mean) (mg/kg) [a]		Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methylquinazolinone	
2015, Monterey, California (Bergram's)	4^ (3,3,2)	47 45 45 45	16 16 16 16	29 Oct., BBCH 49	1	2.0, 2.4 (2.2)	<0.01, <0.01 (<0.01)	SARS-14-11-CA2
2015, Yolo, California (Salad Bowl)	4 (3)	45 45 45 45	16 16 16 16	04 June, BBCH 49 +COC	1	3.0, 3.5 (3.2)	0.015, 0.017 (0.016) (total: 3.0, 3.5 (3.2))	SARS-14-11-CA3
2015, Clarke, Georgia (Salad Bowl)	4^ (3,4,2)	45 45 45 45	12 13 13 13	12 Nov., BBCH 48-49	1	3.5, 3.0 (3.2)	0.012, <0.01 (0.011) (total: 3.5, 3.0 (3.2))	SARS-14-11-GA
2015, Dane, Wisconsin (Loose leaf)	4^ (3,3,2)	45 50 43 45	21 21 20 20	15 July, BBCH 48-49	1	2.2, 2.1 (2.2)	<0.01, <0.01 (<0.01)	SARS-14-11-WI
2015, Jackson, Florida (Heirloom)	4 (3)	46 44 46 45	25 25 24 24	28 Nov., BBCH 48 +COC	1	8.0, 8.0 (8.0)	0.022, 0.022 (0.020) (total: 8.0, 8.0 (8.0))	SARS-14-11-FL
2015, Jefferson, Iowa (Kodiak)	4 (3)	45 45 46 46	33 34 33 21	18 June, BBCH 48-49 +NIS	1	2.7, 3.2 (2.9)	<0.01, <0.01 (<0.01)	SARS-14-11-IA
2015, Freeborn, Minnesota (Salad Bowl – Green)	4 (3)	45 45 45 45	21 21 21 21	30 July, Vegetative +COC	1	2.3, 2.3 (2.3)	<0.01, <0.01 (<0.01)	SARS-14-11-MN2

Notes:
+COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.
[a] Residues are expressed as parent equivalents.
[b] At the last application

Spinach

Table 85 Residues of tetraniliprole in spinach (leaves) after pre-harvest foliar treatments using a 200 SC formulation in field trials in Canada and the United States (Study SARS-14-14)

SPINACH Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methylquinazolinone	
United States, 2014, Fresno, California (Corvette)	4 (3)	46 45 46 46	24 24 24 24	26 Oct., BBCH 49 +NIS	0 1 3 5 10	7.1, 14 (10) 8.7, 8.6 (8.7) 5.8, 6.2 (6.0) 5.3, 4.1 (4.7) 1.6, 1.7 (1.7)	0.017, <0.01 (0.013) (total: 7.1, 14 (10)) 0.020, 0.022 (0.021) 0.017, 0.017 (0.017) 0.013, 0.013 (0.013) 0.010, <0.01 (0.010)	SARS-14-14-CA1-2

SPINACH Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methylquinazolinone	
United States, 2014, Wright, Minnesota (SV2157VB)	4 (3)	43 44 43 43	23 23 22 22	29 Sept., BBCH 47-49 +NIS	1	0.17, 0.32 (0.25)	<0.01, <0.01 (<0.01)	SARS-14-14-MN2-2
United States, 2014, Madera, California (Renegade)	4^ (3)	45 45 45 45	16 16 16 16	22 Nov., Mature	1	6.9, 7.0 (7.0)	<0.01, <0.01 (<0.01)	SARS-14-14-CA2-2 GL
United States, 2014, Stearns, Minnesota (Corvair)	4 (3)	43 44 43 43	23 23 23 23	20 Sept., BBCH 48-49 +COC	1	0.32, 0.67 (0.50)	<0.01, <0.01 (<0.01)	SARS-14-14-MN1-2
United States, 2014, Webster, Georgia, (Sakota)	4^ (3)	44 44 45 45	21 21 21 22	21 Dec., BBCH 48	1	2.9, 3.6 (3.3)	<0.01, <0.01 (<0.01)	SARS-14-14-GA
United States, 2015, Jerome, Idaho (Unipack 151)	4 (3)	45 46 46 46	22 22 23 21	12 Oct., BBCH 49 +NIS	1	4.2, 4.8 (4.5)	<0.01, <0.01 (<0.01)	SARS-14-14-UT-2
United States, 2015, Wharton, Texas (Bloomsdale Longstanding)	4 (3)	46 45 47 46	18 17 17 18	30 Jan., BBCH 45 +COC	1	5.0, 6.1 (5.6)	<0.01, 0.014 (0.012) (total: 5.0, 6.1 (5.6))	SARS-14-14-TX-2
United States, 2015, Wayne, New York (Longstanding Bloomsdale)	4^ (3)	45 45 45 45	19 19 19 19	10 Aug., > 12 Leaf	1	8.0, 7.6 (7.8)	0.039, 0.032 (0.035) (total: 8.0, 7.6 (7.8))	SARS-14-14-NY-2
Canada, 2015, Grey, Manitoba (Vancouver)	4^ (3)	46 45 46 45	45 45 45 45	02 July, Bolted	1	7.6, 5.6 (6.6)	0.025, 0.026 (0.026) (total: 7.6, 5.6 (6.6))	SARS-14-14-MB-2

Notes:
+COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.
[a] Parents are expressed as parent equivalents.
[b] At the last application.

Table 86 Residues of tetraniliprole in spinach (leaves) after single soil drench application using a 200 SC formulation in field trials in the United States in 2015 (Study SARS-14-14)

Location SPINACH (variety)	Application			Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	g ai/hL	Date, growth stage	DALA	Tetraniliprole	T-N-methylquinazolinone	
Jerome, Idaho (Unipack 151)	201	23	29 Sept., BBCH 33	3 7 14 21 28	7.0, 7.6 (7.3) 5.0, 6.1 (5.5) 3.0, 4.2 (3.6) 1.2, 1.2 (1.2) 1.2, 1.5 (1.4)	<0.01, <0.01 (<0.01)	14-UT-3

Location SPINACH (variety)	Application			Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	g ai/hL	Date, growth stage	DALA	Tetraniliprole	T-N-methylquinazolinone	
Wayne, New York (Longstanding Bloomsdale)	201	12	28 July, > 12 Leaf	14	0.68, 0.76 (0.72)	<0.01, <0.01 (<0.01)	SARS-14-14-NY-3
Madera, California (Renegade)	203	61	09 Nov., 10+ leaves	14	0.79, 1.2 (1.0)	<0.01, <0.01 (<0.01)	SARS-14-14-CA2-3

Notes:
DALA = Days After Last Application; T = tetraniliprole.
[a] Residues are expressed as parent equivalents.

Brassica leafy vegetables

Mustard greens

Five field trials were conducted to measure the magnitude of tetraniliprole residues in/on mustard greens following four applications of a tetraniliprole 200 SC formulation (Miller & Jerkins, 2016, M-557177-01-1, Report RAFVN036). Applications were made at a rate of 45–49 g ai/ha, with application intervals of 4–5 days. Trials were carried out in 2014/2015. No adjuvants were used.

Samples of leaves weighing at least 1 kg were collected at maturity from at least 12 separate areas of the plot, nominally 1-day after the final application. One bulk sample of 4 kg was collected from plot FV306-14HA for processing purposes. Additional decline data were collected from 2 sites, where samples were taken nominally 0, 1, 5, 10 and 14 days following the final application.

All samples were stored frozen for a maximum of 427 days (*ca* 14 months) prior to residue analysis. Samples were analysed for tetraniliprole and tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. Totals were only calculated where residues of parent tetraniliprole and tetraniliprole-N-methylquinazolinone are above LOQ of 0.01 mg/kg. In the trials summarized below, levels of the metabolite were always below the LOQ.

The results of the trials are summarised in Table 87.

Table 87 Residues of tetraniliprole in mustard greens (leaves) after pre-harvest foliar treatments using a 200 SC formulation in field trials in the United States (Study RAVN036)

MUSTARD GREENS Year, Location (variety)	Application					Residues (mean) (mg/kg) [a]		Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
2015, Raymondville, Texas (Florida Broadleaf)	4^ (5,4,5)	46 45 46 46	24 24 24 24	02 April, BBCH 16	0 1 5 10 14	5.8, 5.8 (5.8) 4.6, 3.9 (4.2) 1.6, 1.6 (1.6) 0.31, 0.22 (0.27) 0.056, 0.053 (0.054)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV305-14DA-TRTD2015
2014, Elko, South Carolina (Florida Broadleaf)	4^ (5,5,4)	46 45 45 45	21 21 21 21	12 Nov., BBCH 16	0 1 4 9 14	4.0, 3.9 (3.9) 4.0, 4.1 (4.0) 3.6, 3.4 (3.5) 2.5, 2.5 (2.5) 0.37, 0.24 (0.31)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	FV302-14DA-TRTD

MUSTARD GREENS Year, Location (variety)	Application					Residues (mean) (mg/kg) [a]		Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
2014, Cheneyville, Louisiana (Florida Broadleaf)	4^ (5,5,4)	47 48 49 46	16 16 16 16	14 Nov., BBCH 19	1	7.3, 7.2 (7.3)	<0.01, <0.01 (<0.01)	FV303-14HA-TRTD
2014, Richland, Iowa (Southern Giant Curled)	4^ (4,5,5)	45 45 45 45	28 28 27 25	16 June, BBCH 19	1	3.4, 3.9 (3.6)	<0.01, <0.01 (<0.01)	FV304-14HA-TRTD
2014, Madera, California (Florida Broadleaf)	4^ (4,5,5)	45 46 46 46	16 16 16 16	23 June, BBCH 49	1	3.2, 3.2, (3.2)	<0.01, <0.01 (<0.01)	FV306-14HA-TRTD

Notes:

^No adjuvant added; RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.

[a] Residues are expressed a parent equivalents.

[b] At the last application.

Pulses

Soya bean (dry)

Twenty-one field trials were conducted in Canada and the United States to measure the magnitude of tetraniliprole residues in/on soya bean raw agricultural commodities following four foliar applications of a tetraniliprole 200 SC formulation (Greenland, 2016j, M-574330-02-1, Report SARS-15-03). Applications were made at an actual rate of 48–52 g ai/ha, with application intervals of 2–4 days. Three trials also included a separate plot where soya bean was treated with one in-furrow soil application of tetraniliprole 200 SC at an actual rate of 203–240 g ai/ha. Trials were carried out in 2014/2015.

An additional treatment plot was established at two sites that was treated at an exaggerated rate (5×) to provide samples for processing. Samples of seed were collected at normal maturity (BBCH not reported), nominally 14 days after the last foliar application. Additional decline data was collected from 2 sites, where samples were taken 0, 7, 14, 21 and 28 days following the final foliar application.

Seed samples, weighing at least 1 kg, were collected from a mechanical harvester in at least twelve areas within the plot as the harvester proceeded through the plot, or plants were harvested from at least twelve areas within the plot by hand and then threshed.

Soybean seed samples for processing and aspirated grain fractions (AGF) were collected at the normal commercial harvest time, fourteen (14) days after the last test substance foliar application. Samples were stored frozen for a maximum of 227 days (*ca* 7 months) prior to residue analysis. Samples were analysed for tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

The results of the trials are summarised in Table 88 (Foliar applications) and Table 89 (In-furrow treatments). In the trials summarized below, levels of the metabolite were always below the LOQ. Therefore, no totals were added to the tables for soya bean seeds.

Table 88 Residues of tetraniliprole in soya bean (dry seeds) after pre-harvest foliar treatments using a 200 SC formulation in field trials in Canada and the United States in 2015 (Study SARS-15-03)

SOYA BEAN Country, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole (mg/kg)	T-N-methyl-quinazolinone	
Canada, Brant, Ontario (Absolute RR)	4 (3)	52 50 51 48	25 25 25 25	24 Sept., BBCH 97 +NIS	14	<0.01, 0.014 (0.012)	<0.01, <0.01 (<0.01)	SARS-15-03-ON-2
United States, Clarke, Georgia (AG-4933)	4^ (3)	50 50 50 50	14 14 14 14	01 Oct., BBCH 81-83	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-03-GA1-2
United States, Sherburne, Minnesota (PB0879NRR2)	4 (3,3,2))	51 51 51 51	27 27 27 27	26 Sept., BBCH 87 +COC	14	0.16, 0.11 (0.14)	<0.01, <0.01 (<0.01)	SARS-15-03-MN1-2
United States, York, Nebraska (S51112199)	4 (3)	50 50 51 50	25 26 26 25	08 Oct., BBCH 89 +NIS	13	0.030, 0.035 (0.033)	<0.01, <0.01 (<0.01)	SARS-15-03-NE1-2
United States, Jefferson, Iowa (P28T33R)	4 (3)	50 50 50 49	23 23 23 22	01 Oct., BBCH 81-83 +NIS	14	0.016, 0.020 (0.018)	<0.01, <0.01 (<0.01)	SARS-15-03-IA-2
United States, Freeborn, Minnesota (Pioneer P15T83R)	4 (3)	50 50 50 50	29 28 30 30	18 Sept., BBCH 85-89 +COC	0 7 14 21 28	0.022, 0.019 (0.021) 0.012, 0.015 (0.014) 0.010, 0.011 (0.011) <0.010, 0.014 (0.012) <0.010, <0.010 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	SARS-15-03-MN4-2
United States, Walworth, Wisconsin (AG2031/A124341)	4^ (3)	49 49 49 50	26 27 28 26	01 Oct., BBCH 81-83	14	0.024, 0.028 (0.026)	<0.01, <0.01 (<0.01)	SARS-15-03-WI1-2
United States, Stearns, Minnesota (AG013RRY2)	4 (3)	50 50 50 50	31 31 31 31	25 Sept., BBCH 87-88 +COC	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-03-MN3-2
United States, Stearns, Minnesota (AG013RRY2)	4^ (3)	50 49 50 50	31 31 31 31	25 Sept., BBCH 87-88	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-03-MN2-2
United States, Stearns, Minnesota (AG013RRY2)	4^ (3)	250 250 250 250	158 158 158 159	25 Sept., BBCH 87-88	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-03-MN2-4
United States, Washington, Mississippi (Armour 4744)	4^ (3)	50 50 50 49	35 35 35 35	08 Oct., BBCH 84	14	0.043, 0.033 (0.038)	<0.01, <0.01 (<0.01)	SARS-15-03-MS-2

SOYA BEAN Country, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole (mg/kg)	T-N-methyl-quinazolinone	
United States, Dunklin, Missouri (AG-4632)	4 (3)	50 50 50 50	27 27 27 27	12 Oct., BBCH 86 +COC	14	0.017, 0.015 (0.016)	<0.01, <0.01 (<0.01)	SARS-15-03-MO1-2
United States, Cass, North Dakota (A 1025962)	4 (3)	51 52 50 52	27 27 27 27	19 Sept., BBCH 86 +NIS	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-03-ND-2
United States, Shelby, Missouri (P3ST58R)	4^ (3)	49 50 50 50	27 27 28 27	25 Sept., BBCH 81-83	14	0.049, 0.025 (0.037)	<0.01, <0.01 (<0.01)	SARS-15-03-MO3-2
United States, Garfield, Oklahoma (S44-K7)	4^ (2,4,3)	52 51 52 51	17 17 17 17	29 Oct., BBCH 97	15	0.027, 0.032 (0.030)	<0.01, <0.01 (<0.01)	SARS-15-03-OK-2
United States, Crittenden, Arkansas (HBK4953LL)	4 (3,4,3)	51 50 50 50	54 53 53 53	02 Oct., BBCH 88 +NIS	14	0.050, 0.045 (0.048)	<0.01, <0.01 (<0.01)	SARS-15-03-AR-2
United States, Crittenden, Arkansas (HBK4953LL)	4 (3,4,3)	251 251 251 250	270 270 270 270	02 Oct., BBCH 88 +NIS	14	0.45, 0.45 (0.45)	<0.01, <0.01 (<0.01)	SARS-15-03-AR-4
United States, Butler, Missouri (48E3RR)	4 (3)	50 49 49 50	27 27 27 27	01 Oct., BBCH 87 +NIS	14	0.052, 0.054 (0.053)	<0.01, <0.01 (<0.01)	SARS-15-03-MO2-2
United States, Dane, Wisconsin (S17-G8)	4 (3)	51 51 51 51	22 23 23 23	01 Oct., BBCH 81-83 +COC	14	0.020, 0.016 (0.018)	<0.01, <0.01 (<0.01)	SARS-15-03-WI2-2
United States, York, Nebraska (AG2733)	4 (3)	50 50 50 50	22 22 22 22	26 Sept., BBCH 85 +NIS	0 7 14 21 28	0.10, 0.064 (0.084) 0.037, 0.040 (0.039) 0.043, 0.025 (0.034) 0.046, 0.11 (0.079) 0.089, 0.094 (0.092)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	SARS-15-03-NE2-2
United States, Webster, Georgia (95470)	4 (3)	50 50 49 50	25 25 25 25	15 Oct., BBCH 87 +COC	14	<0.01, 0.040 (0.025)	<0.01, <0.01 (<0.01)	SARS-15-03-GA2-2
United States, Shelby, Missouri (Missouri Pride)	4 (3)	51 49 49 49	28 28 27 26	01 Oct., BBCH 81-83 +COC	14	0.12, 0.14 (0.13)	<0.01, <0.01 (<0.01)	SARS-15-03-MO4-2
United States, Stafford, Kansas (P31T11R-SA2P)	4^ (3)	49 50 51 50	30 30 30 30	24 Sept., BBCH 85-87	14	0.024, 0.027 (0.026)	<0.01, <0.01 (<0.01)	SARS-15-03-KS-2

Notes:
+COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; GS = Growth stage at last treatment; RTI = Retreatment Interval; DALA = Days After Last Application; Mean residue values presented in parenthesis.
[a] Residues are expressed as parent equivalents.
[b] At the last application.

Table 89 Residues of tetraniliprole in soya bean (dry seeds) after in-furrow treatment at planting using an 200 SC formulation in field trials in the United States in 2015 (Study SARS-15-03)

SOYA BEAN Location (variety)	Application			Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	g ai/hL	Date	DALA	Tetraniliprole	T-N-methyl-quinazolinone	
Clarke, Georgia (AG-4933)	203	107	08 June,	129	<0.01, 0.017 (0.013)	<0.01, <0.01 (<0.01)	SARS-15-03-GA1-3
York, Nebraska (S51112199)	199	220	09 June, +Buffer	134	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-03-NE1-3
Dane, Wisconsin (S17-G8)	200	240	02 June, +acidifier	135	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-03-WI2-3

Notes:
DALA = Days After Last Application.
[a] Residues are expressed as parent equivalents.

Root and tuber vegetables

Potato

Twenty-six field trials were conducted in Canada and the United States to measure the magnitude of tetraniliprole residues in/on potato following one in furrow or four foliar applications of a tetraniliprole 200 SC formulation (Dallstream & Jerkins, 2016b, M-557979-01-1, Report RAFVP074). Foliar applications were made at an actual rate of 25–32 g ai/ha, with application intervals of 3–7 days. In-furrow treatments were made at a nominal rate of 200 g ai/ha. Trials were carried out in 2014/2015. No adjuvants were used.

Samples of tubers were collected at maturity, generally 14 days after the final foliar application. Samples were also collected at maturity following the in-furrow applications. Additional decline data was collected from 4 sites, where samples were taken nominally 3, 7, 14, 21 and 28 days following the final foliar application.

Samples were stored frozen a maximum of 455 days (*ca* 15 months) prior to residue analysis. Samples were analysed for tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

The results of the trials are summarised in Table 90 (Foliar applications) and Table 91 (In-furrow treatments). In the trials summarized below, levels were always below the LOQ.

Table 92 Residues of tetraniliprole in potato tubers after 4 pre-harvest foliar treatments using a 200 SC formulation in field trials in Canada and the United States (Study RAVFP074).

POTATO Country, Year, Location (variety)	Application				Residues (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
Canada, 2014, Taber, Alberta (Blazer Russet)	4^ (6,4,4)	31 30 31 31	30 30 30 30	02 Sept., BBCH 49	14	<0.01 (2)	<0.01 (2)	FV101-14HA-TRTF
Canada, 2014, Taber, Alberta (Norland, Red skin potato)	4^ (5,4,4)	31 30 31 30	30 30 30 30	19 Aug., BBCH 49	14	<0.01 (2)	<0.01 (2)	FV102-14HA-TRTF
Canada, 2014, Josephburg Alberta (Russet Burbank)	4^ (5,3,5)	31 31 31 31	31 31 31 31	17 Sept., BBCH 46	14	<0.01 (2)	<0.01 (2)	FV111-14HA-TRTF
Canada, 2015, Coalhurst, Alberta Sangre, red skinned)	4^ (5)	30 31 31 30	30 30 30 30	19 Aug., BBCH 46-48	14	<0.01 (2)	<0.01 (2)	FV112-14HB-TRTF
United States, 2014, Bagley Iowa (Yukon God)	4^ (5,4,5)	30 30 31 31	25 26 28 28	18 June, BBCH 47	14	<0.01 (2)	<0.01 (2)	FV096-14HA-TRTF
United States, 2014, Richland, Iowa (Yukon Gold; Seed Potato)	4^ (5,5,4)	30 30 31 30	25 25 25 23	16 June, BBCH 47	14	<0.01 (2)	<0.01 (2)	FV099-14HA-TRTF
United States, 2014, Kerman, California (Yukon Gold)	4^ (5)	30 30 30 30	27 27 27 27	13 June, BBCH 46	14	<0.01 (2)	<0.01 (2)	FV104-14HA-TRTF
United States, 2014, High Springs, Florida (Red Pontiac)	4^ (5,5,4)	32 30 30 30	26 24 25 24	14 May, BBCH 46	14	<0.01 (2)	<0.01 (2)	FV095-14HA-TRTF
United States, 2014, Payette, Idaho (Russet Norkotab)	4^ (5)	31 31 31 31	24 24 24 24	08 Sept., BBCH 48	13	<0.01 (2)	<0.01 (2)	FV106-14HA-TRTF
United States, 2014, Rupert Idaho (Russet Norkotah)	4^ (5)	30 30 31 30	26 26 27 25	14 Aug., BBCH 48	14	<0.01 (2)	<0.01 (2)	FV105-14HA-TRTF
United States, 2014, Rupert, Idaho (Russet Burbank)	4^ (5)	30 30 30 30	27 27 25 26	19 Aug., BBCH 47	3 7 14 21 28	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	FV110-14DA-TRTF
United States, 2014, Jerome, Idaho (Dark Red Norland)	4^ (7,7,5)	30 30 30 30	25 25 24 25	20 Aug., BBCH 48	14	<0.01 (2)	<0.01 (2)	FV103-14HA-TRTF

POTATO Country, Year, Location (variety)	Application				Residues (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
United States, 2014, Jerome, Idaho (Ranger)	4^ (5,6,4)	30 30 30 30	23 23 24 23	11 Sept., BBCH 48	14	<0.01 (2)	<0.01 (2)	FV107- 14HA- TRTF
United States, 2014, Seven Springs North Carolina (Red Pontiac)	4^ (5,5,4)	30 30 30 31	25 25 25 24	06 June, BBCH 45	45	<0.01 (2)	<0.01 (2)	FV094- 14HA- TRTF
United States, 2014, Lenexa, Kansas (Kennebec)	4^ (5)	31 30 31 30	25 23 24 24	16 June, BBCH 47	14	<0.01 (2)	<0.01 (2)	FV098- 14HA- TRTF
United States, 2014, Brooklyn, Wisconsin (Superior)	4^ (5,5,4)	30 30 31 30	27 25 25 25	19 Aug., BBCH 47	3 7 13 20 27	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	FV100- 14DA- TRTF
United States, 2014, Phelps, New York (Sifra)	4^ (5)	30 30 30 30	25 25 25 25	02 Sept., BBCH 48	14	<0.01 (2)	<0.01 (2)	FV091- 14HA- TRTF [c]
United States, 2014, Sodus, New York (Reba)	4^ (4,5,5)	30 30 30 30	23 23 23 23	01 Sept., BBCH 46	14	<0.01 (2)	<0.01 (2)	FV087- 14HA- TRTF [c]
United States, 2014, Lyons, New York (Yukon Gold)	4^ (4,5,5)	30 30 30 30	23 23 23 23	02 Sept., BBCH 47	14	<0.01 (2)	<0.01 (2)	FV090- 14HA- TRTF [c]
United States, 2014, Lyons, New York (Gold Rush)	4^	30 30 30 30	23 23 23 23	02 Sept., BBCH 47	3 7 14 21 27	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	FV089- 14DA- TRTF [c]
United States, 2014, Lyons, New York (Red Norland)	4^ (4,5,5)	30 30 30 30	23 23 23 23	01 Sept., BBCH 46	14	<0.01 (2)	<0.01 (2)	FV092- 14HA- TRTF [c]
United States, 2014, LeRoy, New York (Sifra)	4^ (5)	30 30 30 30	25 25 25 25	02 Sept., BBCH 48	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV093- 14HA- TRTF [c]
United States, 2014, North Rose, New York (Modoc)	4^ (5)	31 31 31 31	25 25 25 25	20 Aug., BBCH 48	3 7 14 21 28	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	FV088- 14DA- TRTF
United States, 2014, Northwood North Dakota (Atlantic)	4^ (5)	31 30 30 31	32 32 32 32	10 Sept., BBCH 48	14	<0.01 (2)	<0.01 (2)	FV097- 14HA- TRTF

POTATO Country, Year, Location (variety)	Application				Residues (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
United States, 2014, Ephrata, Washington (Norland Dark Red)	4^ (5)	30 30 30 30	32 32 32 32	12 Aug., BBCH 48	14	<0.01 (2)	<0.01 (2)	FV109-14HA-TRTF
United States, 2014, Ephrata, Washington (Russet Burbank)	4^ (5)	30 30 31 31	32 32 32 32	03 Sept., BBCH 48	14	<0.01 (2)	<0.01 (2)	FV108-14HA-TRTF

Notes:
ADJ = adjuvant, either COC or NIS; ^no adjuvant added; RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.
[a] Residues are expressed as parent equivalents.
[b] At the last application.
[c] Five of the trials were carried out at the same time in Phelps (1) and Sodus (1), and Lyons (3); The cities are geographically <30 km apart. When considering the application date, the trials in Phelps and Sodus were not considered independent, nor were the three trials in Lyons.

Table 91 Residues of tetraniliprole in potato tubers after one in furrow application at planting using a 200 SC formulation in field trials in Canada and the United States (Study RAFP074)

POTATO Country, Year, Location (variety)	Application			Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	g ai/hL	Date	DALA	Tetraniliprole	T-N-methyl quinazolinone	
Canada, 2014, Taber, Alberta (Blazer Russet)	192	200	22 May,	117	0.017, 0.013 (0.015)	<0.01, <0.01 (<0.01)	FV101-14HA-TRTI
Canada, 2014, Taber, Alberta (Norland, Red skin)	200	167	22 May,	103	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV102-14HA-TRTI
Canada, 2014, Josephburg, Alberta (Russet Burbank)	206	186	03 June,	120	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV111-14HA-TRTI
Canada, 2015, Coalhurst, Alberta (Sangre, red skinned)	200	200	14 May,	111	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV112-14HB-TRTI
United States, 2014, Bagley, Iowa (Yukon Gold)	206	190	17 April,	104	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV096-14HA-TRTI
United States, 2014, Richland, Iowa (Yukon Gold; Seed Potato)	200	190	07 April,	114	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV099-14HA-TRTI2014
United States, 2014, Kerman, California (Yukon Gold)	200	210	14 March,	105	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV104-14HA-TRTI
United States, 2014, High Springs, Florida (Red Pontiac)	204	180	14 March,	75	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV095-14HA-TRTI
United States, 2014, Payette, Idaho (Russet Norkotab)	198	160	17 May,	127	0.013, 0.011 (0.012)	<0.01, <0.01 (<0.01)	FV106-14HA-TRTI

POTATO Country, Year, Location (variety)	Application			Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	g ai/hL	Date	DALA	Tetraniliprole	T-N-methyl quinazolinone	
United States, 2014, Rupert, Idaho (Russet Norkotah)	200	170	15 May,	105	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV105-14HA-TRTI
United States, 2014, Rupert, Idaho (Russet Burbank)	198	150	24 April,	131	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV110-14DA-TRTI
United States, 2014, Jerome, Idaho (Dark Red Norland)	200	180	05 May,	121	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV103-14HA-TRTI
United States, 2014, Jerome, Idaho (Rangert)	203	360	02 May,	146	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV107-14HA-TRTI
United States, 2014, Seven Springs, North Carolina (Red Pontiac)	198	240	14 March,	97	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV094-14HA-TRTI
United States, 2014, Lenexa, Kansas (Kennebec)	202	190	01 April,	120	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV098-14HA-TRTI
United States, 2014, Brooklyn, Wisconsin (Superior)	200	310	16 June,	87	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV100-14DA-TRTI
United States, 2014, LeRoy, New York (Sifra)	202	140	12 June,	96	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV093-14HA-TRTI
United States, 2014, Phelps, New York (Sifra)	202	140	11 June,	97	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV091-14HA-TRTI [b]
United States, 2014, Sodus, New York (Reba)	200	140	24 June,	83	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV087-14HA-TRTI [b]
United States, 2014, Lyons, New York (Yukon Gold)	200	140	26 June,	82	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV090-14HA-TRTI [b]
United States, 2014, Lyons, New York (Gold Rush)	197	140	26 June,	82	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV089-14DA-TRTI [b]
United States, 2014, Lyons, New York (Red Norland)	200	140	26 June,	81	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV092-14HA-TRTI [b]
United States, 2014, North Rose, New York (Modoc)	203	140	07 June,	88	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV088-14DA-TRTI
United States, 2014, Northwood, North Dakota (Atlantic)	201	210	03 June,	113	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV097-14HA-TRTI
United States, 2014, Ephrata, Washington (Norland Dark Red)	204	210	24 April,	124	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV109-14HA-TRTI
United States, 2014, Ephrata, Washington (Russet Burbank)	198	210	24 April,	146	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	FV108-14HA-TRTI

Notes:

DALA = Days After Last Application; T = tetraniliprole.

[a] Residues are expressed as parent equivalents.

[b] Five of the trials were carried out at the same time in Phelps (1) and Sodus (1), and Lyons (3); The cities are geographically <30 km apart. When considering the application dates, the trials in Phelps and Sodus were not considered independent, nor were the three trials in Lyons.

Cereal grains

Rice–Thailand, India and Vietnam

Twelve field trials were conducted throughout Thailand, India and Vietnam to measure the magnitude of tetraniliprole and tetraniliprole-N-methylquinazolinone residues in/on paddy rice following 2 foliar applications of a thiacloprid 480 SC formulation containing 120 g tetraniliprole/L (Woodard, 2019a, Report RAFV0085, Document M-669757-01-1). Applications were made at actual rates of 38–42 g ai/ha, with application intervals of 7–17 days. In addition, twelve field trials were conducted in the same countries to measure the magnitude of tetraniliprole and tetraniliprole-N-methylquinazoline residues in/on paddy rice following either 1 seed treatment (TRTD1), 1 seed treatment followed by on foliar treatment (TRTD2) or 3 foliar treatments (TRTD3) with a tetraniliprole 480 FS formulation or a tetraniliprole 200 SC formulation (Woodard, 2019b, Report RAFV0014, Document M-667200-01-1). Seed treatments were made at application rates ranging from 240 to 244 g ai/ha, foliar treatments were made at actual rates of 58–68 g ai/ha. All the trials were carried out in 2018/2019. No adjuvants were used in either study.

In both studies samples of rice grain and brown rice were collected from each site at normal commercial harvest (*ca* 43 days after the final application). Several sites in both studies included additional samples of grain, collected approximately 38, 43, 50 and 53 days following the final application to provide residue decline data.

For the whole grain (paddy rice) samples, collected at 40 (actual 38–40 days), 45 (actual 43 – 45 days), 50 (actual 48 – 50 days), 55 days (actual 53 days), and normal commercial harvest (NHC), sample fractions were generated according to local practice. At crop maturity (approximately BBCH 89), sufficient paddy rice plants were harvested from the control plot first and then from the treated plot to obtain minimum sample sizes for all commodities (1 kg grain). In a clean area away from the test plots, the whole grain rice was separated from the rice straw and allowed to dry by sun drying up to 5 days if needed, according to local practice. The rice grain was then dehulled and milled, using a dehulling machine, by hand or other method. The dehulling process produced unpolished, brown rice and rice hulls. Brown rice samples were collected and the rice hulls were discarded.

Samples were stored frozen for a maximum of 261 days and 136 days. In both studies samples were analysed for residues of tetraniliprole and metabolite tetraniliprole_N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

The results of the trials are summarised in Table 92 and Table 95 (Whole grain/paddy rice) and Table 96 and Table 97 (Brown rice).

Rice–Brazil

Six field trials were conducted in Brazil to measure the magnitude of tetraniliprole and tetraniliprole-N-methylquinazolinone residues in/on paddy rice following 3 foliar applications with a tetraniliprole 200 SC formulation (Carvalho, 2016, M-567467-02-1, Report I14-046). Applications were made at actual rates of 40–43 g ai/ha, with application intervals of 14 days. All the trials were carried out in 2015.

Samples of rice (grain) were collected from each site at normal commercial harvest (*ca* 14 days after the final application). Additional samples were collected from three sites to provide residue decline data (PHI 7, 14 and 21 days). Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole_N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. The period of the storage of the frozen samples to tetraniliprole and its metabolite tetraniliprole-N-methylquinazolinone was at maximum 95 days. Totals were only calculated where residues of parent tetraniliprole and tetraniliprole-N-methylquinazolinone are above LOQ of 0.01 mg/kg.

The results of the trials on rice are summarised in Tables 92 to 95..

Table 92 Residues of tetraniliprole paddy rice (grain) after foliar treatment using SC formulation in trials performed in Brazil, India, Thailand and Vietnam

PADDDY RICE GRAIN Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]				Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	Total	
Brazil, 2015 Rio Prado, (Puita Inta-CL)	3 (14)	41 41 41	27 27 27	01 April, BBCH 79	14	0.11	<0.01	0.11	I14-046 I14-046-01
Brazil, 2015, Novo Cabrais, (Puita Inta-CL)	3 (14)	40 41 41	27 27 27	02 April, BBCH 79	14	0.057	<0.01	0.057	I14-046 I14-046-02
Brazil, 2015, Vera Cruz, (Irga 108)	3 (14)	43 43 43	29 29 29	23 March, BBCH 85	14	0.10	<0.01	0.10	I14-046 I14-046-03
Brazil, 2015, Candelaria, (Puita Inta CL)	3 (14)	43 42 43	29 28 29	16 March, BBCH 79	7 14 21	0.64 0.65 0.19	0.013 0.013 0.010	0.65 0.66 0.20	I14-046 I14-046-04
Brazil, 2015, Santa Cruz do Sul, (Irga 108)	3 (14)	41 42 43	27 28 29	18 March, BBCH 81	7 14 21	0.51 0.12 0.075	<0.01 <0.01 <0.01	0.51 0.12 0.075	I14-046 I14-046-05
Brazil, 2015, Passo do Sobrado, (Puita Inta CL)	3 (14)	43 42 42	29 28 29	09 April, BBCH 79	7 14 21	0.24 0.18 0.26	<0.01 <0.01 <0.01	0.24 0.18 0.26	I14-046 I14-046-07
India, 2018, Ippili, Srikakulam District, Andhra Pradesh (MTU 1010)	2 (7)	41 40	10 10	15 Oct., BBCH 55	38	0.095, 0.099 (0.097)	<0.01, <0.01 (<0.01)	0.095, 0.099 (0.097)	RAFV0085-G-DA-TRTD
					43	0.12, 0.14 (0.13)	<0.01, <0.01 (<0.01)	0.12, 0.14 (0.13)	
					48	0.16, 0.085 (0.12)	<0.01, <0.01 (<0.01)	0.16, 0.085 (0.12)	
					53	0.084, 0.13 (0.11)	<0.01, <0.01 (<0.01)	0.084, 0.13 (0.11)	
India, 2018, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	2 (7)	39 39	10 11	24 Oct., BBCH 56	43	0.28, 0.28 (0.28)	<0.01, <0.01 (<0.01)	0.28, 0.28 (0.28)	RAFV0085-H-HA-TRTD

PADDDY RICE GRAIN Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]				Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	Total	
India, 2018, Seedhi, Srikakulam District, Andhra Pradesh (MTU1121)	2 (16)	40 42	10 10	01 Nov., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085-I-HA-TRTD
India, 2018, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	2 (15)	41 40	10 11	03 Nov., BBCH 51	43	0.018, 0.021 (0.020)	<0.01, <0.01 (<0.01)	0.018, 0.021 (0.020)	RAFV0085-J-HA-TRTD
India, 2018, Ippili Village, Srikakulam District, Andhra Pradesh (MTU1010)	3 (5,2)	62 60 59	-	15 Oct., BBCH 57	38	0.37, 0.29 (0.33)	<0.01, <0.01 (<0.01)	0.37, 0.29 (0.33)	RAFV0014-G-DA-TRTD3
					43	0.32, 0.26 (0.29)	<0.01, <0.01 (<0.01)	0.32, 0.26 (0.29)	
					48	0.29, 0.29 (0.29)	<0.01, <0.01 (<0.01)	0.29, 0.29 (0.29)	
					53	0.22, 0.22 (0.22)	<0.01, <0.01 (<0.01)	0.22, 0.22 (0.22)	
India, 2018, Purli Village, Srikakulam District, Andhra Pradesh (RNR 15048)	3 (7,7)	59 59 59	-	24 Oct., BBCH 56	43	0.69, 0.67 (0.68)	0.018, 0.018 (0.018)	0.71, 0.69 (0.70)	RAFV0014-H-HA-TRTD3
India, 2018, Seedhi Village, Srikakulam District, Andhra Pradesh (MTU 1121)	3 (7,16)	58 59 61	-	01 Nov., BBCH 51	43	0.023, 0.027 (0.025)	<0.01, <0.01 (<0.01)	0.023, 0.027 (0.025)	RAFV0014-I-HA-TRTD3
India, 2018, Sancham Village, Srikakulam District, Andhra Pradesh (MTU 1121)	3 (7,15)	60 59 59	-	03 Nov., BBCH 51	43	0.050, 0.048 (0.049)	<0.01, <0.01 (<0.01)	0.050, 0.048 (0.049)	RAFV0014 RAFV0014-J-HA-TRTD3
Thailand, 2018, Kamphaeng Saen, Nakhon Pathom (RD41)	2 (11)	40 40	10 10	10 Dec., BBCH 58	38	1.9, 1.7 (1.8)	0.020, 0.017 (0.019)	1.9, 1.7 (1.8)	RAFV0085 RAFV0085-A-DA-TRTD
					43	1.7, 1.5 (1.6)	0.017, 0.014 (0.016)	1.7, 1.5 (1.6)	
					48	1.1, 0.99 (1.0)	0.011, 0.013 (0.012)	1.1, 0.99 (1.0)	
					53	1.0, 1.3 (1.2)	0.013, 0.019 (0.016)	1.0, 1.3 (1.2)	
Thailand, 2018, Tamung Kanchanaburi (RD56)	2 (13)	40 40	10 10	12 Dec., BBCH 56	38	0.087, 0.076 (0.082)	<0.01, <0.01 (<0.01)	0.087, 0.076 (0.082)	RAFV0085-B-DA-TRTD
					43	0.057, 0.069 (0.063)	<0.01, <0.01 (<0.01)	0.057, 0.069 (0.063)	
					48	0.036, 0.038 (0.037)	<0.01, <0.01 (<0.01)	0.036, 0.038 (0.037)	

PADDDY RICE GRAIN Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]				Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	Total	
					53	0.056, 0.053 (0.055)	<0.01, <0.01 (<0.01)	0.056, 0.053 (0.055)	
Thailand, 2018, Yanyao A Samchuk, Suphan Buri (RD41)	2 (8)	40 40	10 10	09 Dec., BBCH 58	43	0.33, 0.37 (0.35)	<0.01, <0.01 (<0.01)	0.33, 0.37 (0.35)	RAFV0085-C-HA-TRTD
Thailand, 2019, Banma, Bangsai, Ayutthaya (Pitsanloke)	2 (11)	40 39	10 10	20 Jan., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085-D-HA-TRTD
Thailand, 2019, Chorakaerong, Chaiyo, Ang Thong (RD47)	2 (11)	40 40	10 10	18 Jan., BBCH 51	43	0.021, 0.016 (0.019)	<0.01, <0.01 (<0.01)	0.021, 0.016 (0.019)	RAFV0085-E-HA-TRTD
Thailand, 2019, Namfarn Inburi Sing Buri Province (RD49)	2 (17)	39 40	10 10	25 Jan., BBCH 51	45	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085-F-HA-TRTD
Vietnam, 2018, Hau My Trinh Village, Cai Be District, Tien Giang (OM5451)	2 (11)	40 39	11 10	03 Sept., BBCH 51	40	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085-K-DA-TRTD
					43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
					50	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
					53	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
Thailand, 2018, Bangkokyae T. Sraseemum, A. Kamphaeng Saen, Nakhon Pathom (RD41)	3 (7,11)	64 61 61	-	10 Dec., BBCH 58	38	1.7, 1.5 (1.6)	0.011, 0.010 (0.011)	1.7, 1.5 (1.6)	RAFV0014 RAFV0014-A-DA-TRTD3
					43	1.4, 1.2 (1.3)	<0.01, 0.010 (0.010)	1.4, 1.2 (1.3)	
					48	1.1, 1.1 (1.1)	<0.01, <0.01 (<0.01)	1.1, 1.1 (1.1)	
					53	1.2, 1.0 (1.1)	<0.01, <0.01 (<0.01)	1.2, 1.0 (1.1)	
Thailand, 2018, Muangchum A. Tamung Kanchanaburi (RD56)	3 (7,13)	68 60 61	-	12 Dec., BBCH 56	38	0.14, 0.15 (0.15)	<0.01, <0.01 (<0.01)	0.14, 0.15 (0.15)	RAFV0014 RAFV0014-B-DA-TRTD3
					43	0.085, 0.088 (0.087)	<0.01, <0.01 (<0.01)	0.085, 0.088 (0.087)	
					48	0.097, 0.088 (0.093)	<0.01, <0.01 (<0.01)	0.097, 0.088 (0.093)	
					53	0.088, 0.094 (0.091)	<0.01, <0.01 (<0.01)	0.088, 0.094 (0.091)	

PADDDY RICE GRAIN Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]				Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	Total	
Thailand, 2018, Yanyao A Samchuk, Suphan Buri 72130 (RD41)	3 (7,8)	61 61 61	-	09 Dec., BBCH 58	43	0.66, 0.69 (0.68)	<0.01, <0.01 (<0.01)	0.66, 0.69 (0.68)	RAFV0014-C-HA TRTD3
Thailand, 2018, 39 M2 Banma, Bangsai, Ayutthaya Province (Pitsanloke)	3 (12,11)	66 60 60	-	20 Jan., BBCH 51	43	0.021, 0.024 (0.023)	<0.01, <0.01 (<0.01)	0.021, 0.024 (0.023)	RAFV0014-D-HA-TRTD3
Thailand, 2018, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	3 (10,11)	61 61 61	-	18 Jan., BBCH 51	43	0.030, 0.031 (0.031)	<0.01, <0.01 (<0.01)	0.030, 0.031 (0.031)	RAFV0014-E-HA-TRTD3
Thailand, 2018, Namfarn Inburi Sing Buri (RD49)	3 (10,17)	62 60 60	-	25 Jan., BBCH 51	45	0.016, 0.012 (0.014)	<0.01, <0.01 (<0.01)	0.016, 0.012 (0.014)	RAFV0014-F-HA-TRTD3
Vietnam, 2018, Hau My Trinh, Cai Be District, Tien Giang (OM5451)	3 (7,11)	64 60 60	-	03 Sept., BBCH 51	40	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-K-DA-TRTD3
					43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
					50	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
					53	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
Vietnam, 2018, Hamlet, Phong My, Giong Trom District, Ben Tre (Nang hoa 9)	3 (7,12)	60 59 60	-	19 Sept., BBCH 51	43	0.012, 0.011 (0.012)	<0.01, <0.01 (<0.01)	0.012, 0.011 (0.012)	RAFV0014-L-HB-TRTD3

Notes:
DALA = Days After Last Application; RTI = Retreatment Interval; T = tetraniliprole;
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Table 93 Residues of tetraniliprole paddy rice (rice grain) after seed treatment (480 SC) or a combination of seed treatment and foliar treatment (200 SC) in trials performed in India,Thailand or Vietnam (Study RAFV0014).

PADDDY RICE Country, Year, Location (variety)	Application			Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	Date, growth stage [a]	DALA	Tetraniliprole	T-N-methyl quinazolinone	Total	

PADDDY RICE Country, Year, Location (variety)	Application			Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	Date, growth stage [a]	DALA	Tetraniliprole	T-N-methyl quinazolinone	Total	
India, 2018, Ippili, Srikakulam District, Andhra Pradesh (MTU1010)	240 [ST]	08 Aug., NA	NCH	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<u><0.01</u>)	RAFV0014-G-DA-TRTD1
India, 2018, Ippili, Srikakulam District, Andhra Pradesh (MTU1010)	240 [ST]59	15 Oct., BBCH 57	38	0.25, 0.15 (0.20)	<0.01, <0.01 (<0.01)	0.25, 0.15 (0.20)	RAFV0014-G-DA-TRTD2
			43	0.17, 0.27 (0.22)	<0.01, <0.01 (<0.01)	0.17, 0.27 (0.22)	
			48	0.12, 0.14 (0.13)	<0.01, <0.01 (<0.01)	0.12, 0.14 (0.13)	
			53	0.14, 0.13 (0.14)	<0.01, <0.01 (<0.01)	0.14, 0.13 (0.14)	
India, 2018, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	240 [ST]	06 Aug., NA	NCH	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<u><0.01</u>)	RAFV0014-H-HA-TRTD1
India, 2018, Purli Village, Srikakulam District, Andhra Pradesh (RNR 15048)	240 [ST] 58	24 Oct., BBCH 56	43	0.60, 0.51 (0.56)	0.013, <0.01 (0.012)	0.61, 0.51 (0.56)	RAFV0014-H-HA-TRTD2
India, 2018, Seedhi Village, Srikakulam District, Andhra Pradesh (MTU 1121)	240 [ST]	04 Aug., NA	NCH	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<u><0.01</u>)	RAFV0014-I-HA-TRTD1
India Seedhi, Srikakulam District, Andhra Pradesh	240 [ST] 60	01 Nov., BBCH 51	43	0.025, 0.016 (0.021)	<0.01, <0.01 (<0.01)	0.025, 0.016 (0.021)	RAFV0014-I-HA-TRTD2
India, 2018, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	240 [ST]	10 Aug., NA	NCH	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<u><0.01</u>)	RAFV0014-J-HA-TRTD1
India, 2018, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	240 [ST] 59	03 Nov., BBCH 51	43	0.013, 0.011 (0.012)	<0.01, <0.01 (<0.01)	0.013, 0.011 (0.012)	RAFV0014-J-HA-TRTD2
Thailand, 2018, Bangkokyae T. Sraseemum, A. Kamphaeng Saen, Nakhon Pathom (RD41)	244 [ST]	27 Sept. NA	NCH	0.012, 0.014 (<u>0.013</u>)	<0.01, <0.01 (<0.01)	0.012, 0.014 (<u>0.013</u>)	RAFV0014-A-DA-TRTD1
Thailand, 2018, Bangkokyae T. Sraseemum, A. Kamphaeng Saen, Nakhon Pathom (RD41)	244 [ST] 63	10 Dec., BBCH 58	38	1.9, 1.9 (1.9)	0.015, 0.013 (0.014)	1.9, 1.9 (1.9)	RAFV0014-A-DA-TRTD2
			43	1.5, 1.4 (1.5)	<0.01, <0.01 (<0.01)	1.5, 1.4 (1.5)	
			48	1.0, 1.2 (1.1)	<0.01, <0.01 (<0.01)	1.0, 1.2 (1.1)	
			53	0.79, 0.84 (0.82)	<0.01, <0.01 (<0.01)	0.79, 0.84 (0.82)	

PADDDY RICE Country, Year, Location (variety)	Application			Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	Date, growth stage [a]	DALA	Tetraniliprole	T-N-methyl quinazolinone	Total	
Thailand, 2018, Muangchum A. Tamung Kanchanaburi (RD56)	244 [ST]	25 Sept., NA	NCH	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<u>≤0.01</u>)	RAFV001 4-B-DA-TRTD1
Thailand, 2018, Muangchum A. Tamung Kanchanaburi (RD56)	244 [ST] 62	12 Dec., BBCH 56	38	0.062, 0.057 (0.060)	<0.01, <0.01 (<0.01)	0.062, 0.057 (0.060)	RAFV001 4-B-DA-TRTD2
			43	0.031, 0.029 (0.030)	<0.01, <0.01 (<0.01)	0.031, 0.029 (0.030)	
			48	0.026, 0.022 (0.024)	<0.01, <0.01 (<0.01)	0.026, 0.022 (0.024)	
			53	0.023, 0.024 (0.024)	<0.01, <0.01 (<0.01)	0.023, 0.024 (0.024)	
Thailand, 2018, Yanyao A, Samchuk, Suphan Buri (RD41)	244 [ST]	26 Sept., NA	NCH	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<u>≤0.01</u>)	RAFV001 4-C-HA TRTD1
Thailand, 2018, Yanyao A Samchuk, Suphan Buri 72130 (RD41)	244 [ST] 61	09 Dec., BBCH 58	43	0.58, 0.54 (0.56)	<0.01, <0.01 (<0.01)	0.58, 0.54 (0.56)	RAFV001 4-C-HA-TRTD2
Thailand, 2018, Banma, Bangsai, Ayutthaya Province (Pitsanloke)	244 [ST]	08 Nov., NA	NCH	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<u>≤0.01</u>)	RAFV001 4-D-HA-TRTD1
Thailand, 2018, Banma, Bangsai, Ayutthaya Province (Pitsanloke)	244 [ST] 61	20 Jan., BBCH 51	43	0.017, 0.017 (0.017)	<0.01, <0.01 (<0.01)	0.017, 0.017 (0.017)	RAFV001 4-D-HA-TRTD2
Thailand, 2018, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	244 [ST]	09 Nov., NA	NCH	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<u>≤0.01</u>)	RAFV001 4-E-HA-TRTD1
Thailand, 2018, 28/2 M2 Chorakaerong, Chaiyo, Ang Thong Province (RD47)	244 [ST] 62	18 Jan., BBCH 51	43	0.022, 0.020 (0.021)	<0.01, <0.01 (<0.01)	0.022, 0.020 (0.021)	RAFV001 4-E-HA-TRTD2
Thailand, 2018, 13 M5 Namfarn Inburi Sing Buri Province (RD49)	244 [ST]	10 Nov., NA	NCH	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<u>≤0.01</u>)	RAFV001 4-F-HA-TRTD1
Thailand, 2018, 13 M5 Namfarn Inburi Sing Buri Province (RD49)	244 [ST] 61	25 Jan., BBCH 51	45	0.012, 0.011 (0.012)	<0.01, <0.01 (<0.01)	0.012, 0.011 (0.012)	RAFV001 4-F-HA-TRTD2
Vietnam, 2018, Hau My Trinh, Cai Be District, Tien Giang Province (OM5451)	240 [ST]	03 July, NA	NCH	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<u>≤0.01</u>)	RAFV001 4-K-DA-TRTD1
Vietnam, 2018, Hau My Trinh, Cai Be District, Tien Giang Province (OM5451)	240 [ST] 59	03 Sept., BBCH 51	40	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV001 4-K-DA-TRTD2
			43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
			50	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	

PADDDY RICE Country, Year, Location (variety)	Application		DALA	Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
			53	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	
Vietnam, 2018, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	240 [ST]	18 July, NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-L-HB-TRTD1
Vietnam, 2018, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	240 [ST] 58	19 Sept., BBCH 51	43	0.014, 0.011 (0.013)	<0.01, <0.01 (<0.01)	0.014, 0.011 (0.013)	RAFV0014-L-HB-TRTD2

Notes:
DALA = Days After Last Application; [ST]=seed treatment.
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Table 94 Residues of tetraniliprole brown rice (husked rice) after foliar treatment using a 120 or 200 SC formulation in trials performed in India, Thailand or Vietnam.

HUSKED RICE Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
India, 2018, Ippili, Srikakulam District, Andhra Pradesh (MTU 1010)	2 (7)	41 40	10 10	15 Oct., BBCH 55	43	0.014, 0.010 (0.012)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-G-DA-TRTD
India, 2018, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	2 (7)	39 39	10 11	24 Oct., BBCH 56	43	0.023, 0.019 (0.021)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-H-HA-TRTD
India, 2018, Seedhi, Srikakulam District, Andhra Pradesh State (MTU1121)	2 (16)	40 42	10 10	01 Nov., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-I-HA-TRTD
India, 2018, Sancham, Srikakulam District, Andhra Pradesh State (MTU 1121)	2 (15)	41 40	10 11	03 Nov., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-J-HA-TRTD
India, 2018, Ippili, Srikakulam District, Andhra Pradesh (MTU1010)	3 (5,2)	62 60 59	-	15 Oct., BBCH 57	43	0.025, 0.036 (0.031)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-G-DA TRTD3
India, 2018, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	3 (7,7)	59 59 59	-	24 Oct., BBCH 56	43	0.033, 0.033(0.033)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-H-HA-TRTD3
India, 2018, Seedhi, Srikakulam District, Andhra Pradesh (MTU 1121)	3 (7,16)	58 59 61	-	01 Nov., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-I-HA-TRTD3

HUSKED RICE Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
India, 2018, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	3 (7,15)	60 59 59	-	03 Nov., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-J-HA-TRTD3
Thailand, 2018, Kamphaeng Saen, Nakhon Pathom (RD41)	2 (11)	40 40	10 10	10 Dec., BBCH 58	43	0.043, 0.047 (0.045)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-A-DA-TRTD
Thailand, 2018, Tamung Kanchanaburi (RD56)	2 (13)	40 40	10 10	12 Dec., BBCH 56	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RFV0085 RAFV0085-B-DA-TRTD
Thailand, 2018, A Samchuk, Suphan Buri (RD41)	2 (8)	40 40	10 10	09 Dec., BBCH 58	43	0.019, 0.019 (0.019)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-C-HA-TRTD
Thailand, 2019, Banma, Bangsai, Ayutthaya (Pitsanloke)	2 (11)	40 39	10 10	20 jan., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-D-HA-TRTD
Thailand, 2019, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	2 (11)	40 40	10 10	18 Jan., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-E-HA-TRTD
Thailand, 2019, Namfarn Inburi Sing Buri (RD49)	2 (17)	39 40	10 10	25 jan., BBCH 51	45	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-F-HA-TRTD
Vietnam, 2018, Hau My Trinh Village, Cai Be District, Tien Giang Province (OM5451)	2 (11)	40 39	11 10	03 Sept., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-K-DA-TRTD
Vietnam, 2018, Phong My Village, Giong Trom District, Ben Tre Province (Nang hoa 9)	2 (12)	39 38	10 10	19 Sept., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085 RAFV0085-L-HA-TRTD
Thailand, 2018, Sraseemum, A. Kamphaeng Saen, Nakhon Pathom (RD41)	3 (7,11)	64 61 61	-	10 Dec., BBCH 58	43	0.052, 0.046 (0.049)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-A-DA-TRTD3
Thailand, 2018, Muangchum A. Tamung Kanchanaburi (RD56)	3 (7,13)	68 60 61	-	12 Dec., BBCH 56	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-B-DA TRTD3
Thailand, 2018, Yanyao A, Samchuk, Suphan Buri 72130 (RD41)	3 (7,8)	61 61 61	-	09 Dec., BBCH 58	43	0.021, 0.020 (0.021)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-C-HA-TRTD3
Thailand, 2018, Banma, Bangsai, Ayutthaya (Pitsanloke)	3 (12,11)	66 60 60	-	20 Jan., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-D-HA-TRTD3
Thailand, 2018, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	3 (10,11)	61 61 61	-	18 Jan., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-E-HA-TRTD3
Thailand, 2018, Namfarn Inburi, Sing Buri Province (RD49)	3 (10,17)	62 60 60	-	25 Jan., BBCH 51	45	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-F-HA-TRTD3

HUSKED RICE Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Study, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
Vietnam, 2018, Hau My Trinh Village, Cai Be District, Tien Giang Province (OM5451)	3 (7,11)	64 60 60	-	03 Sept., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-K-DA-TRTD3
Vietnam, 2018, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	3 (7,12)	60 59 60	-	19 Sept., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-L-HB-TRTD3

Notes:
DALA = Days After Last Application; RTI = Retreatment Interval; T = tetraniliprole.
[a] Residues are expressed as parent equivalents.
[b] At the last application.

Table 95 Residues of tetraniliprole brown rice (husked rice) after seed treatment [ST] or a combination of seed treatment and a foliar treatment using SC formulation for foliar treatment or a 480 SC formulation for seed treatment in trials performed in India, Thailand or Vietnam in 2018 (Study RAFV0014)

HUSKED RICE Country, Year, Location (variety)	Application		Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
India, Ippili, Srikakulam District, Andhra Pradesh (MTU1010)	240 [ST]	08 Aug., NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-G-DA-TRTD1
India, Ippili, Srikakulam District, Andhra Pradesh (MTU1010)	240 [ST] 59	15 Oct., BBCH 57	43	0.012, 0.019 (0.016)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-G-DA-TRTD2
India, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	240 [ST]	06 Aug., NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-H-HA-TRTD1
India, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	240 [ST] 58	24 Oct., BBCH 56	43	0.037, 0.039 (0.038)	<0.01, <0.01 (<0.01)	RAFV0014-H-HA-TRTD2
India, Seedhi, Srikakulam District, Andhra Pradesh (MTU 1121)	240 [ST]	04 Aug., NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-I-HA-TRTD1
India, Seedhi, Srikakulam District, Andhra Pradesh	240 [ST] 60	01 Nov., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-I-HA-TRTD2
India, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	240 [ST]	10 Aug., NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-J-HA-TRTD1
India, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	240 [ST] 59	03 Nov., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-J-HA-TRTD2
Thailand, Sraseemum, A. Kamphaeng Saen, Nakhon Pathom 73140 (RD41)	244 [ST]	27 Sept., NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-A-DA-TRTD1

HUSKED RICE Country, Year, Location (variety)	Application		Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
Thailand, Sraseemum, A. Kamphaeng Saen, Nakhon Pathom 73140 (RD41)	244 [ST] 63	10 Dec., BBCH 58	43	0.054, 0.061 (0.058)	<0.01, <0.01 (<0.01)	RAFV0014-A-DA-TRTD2
Thailand, Muangchum A. Tamung Kanchanaburi (RD56)	244 [ST]	25 Sept. NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-B-DA-TRTD1
Thailand, Muangchum A. Tamung Kanchanaburi (RD56)	244 [ST] 62	12 Dec., BBCH 56	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-B-DA-TRTD2
Thailand, Samchuk, Suphan Buri 72130 (RD41)	244 [ST]	26 Sept., NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-C-HA-TRTD1
Thailand, Samchuk, Suphan Buri 72130 (RD41)	244 [ST] 61	09 Dec., BBCH 58	43	0.021, 0.018 (0.020)	<0.01, <0.01 (<0.01)	RAFV0014-C-HA-TRTD2
Thailand, Bangsai, Ayutthaya (Pitsanloke)	244 [ST]	08 Nov., NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-D-HA-TRTD1
Thailand, Bangsai, Ayutthaya (Pitsanloke)	244 [ST] 61	20 Jan., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-D-HA-TRTD2
Thailand, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	244 [ST]	09 Nov., NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-E-HA-TRTD1
Thailand, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	244 [ST] 62	18 Jan., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-E-HA-TRTD2
Thailand, 13 M5 Namfarn Inburi, Sing Buri Province (RD49)	244 [ST]	10 Nov., NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-F-HA-TRTD1
Thailand, 13 M5 Namfarn Inburi Sing Buri Province (RD49)	244 [ST] 61	25 Jan., BBCH 51	45	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-F-HA-TRTD2
Vietnam, Hau My Trinh, Cai Be District, Tien Giang Province (OM5451)	240 [ST]	03 July NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-K-DA-TRTD1
Vietnam, Hau My Trinh, Cai Be District, Tien Giang Province (OM5451)	240 [ST] 59	03 Sept., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-K-DA-TRTD2
Vietnam, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	240 [ST]	18 July, NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-L-HB-TRTD1
Vietnam, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	240 [ST] 58	19 Sept., BBCH 51	43	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-L-HB-TRTD2

Notes:

DALA = Days After Last Application; [ST] = Seed Treatment;

[a] Residues are expressed in parent equivalents.

[b] At the last application.

Maize/field corn

Twenty one field trials were conducted in Canada and the United States to measure the magnitude of tetraniliprole residues in/on field corn raw agricultural commodities following four foliar applications of a tetraniliprole 200 SC formulation (Stewart & Greenland, 2016, M-574645-01-2, Report SARS-15-06).

Foliar applications were generally made at applications rates ranging between 49 and 52 g ai/ha, with application intervals of 6–8 days, except for one trial that had 13 days between the 2nd and 3rd applications to plot 2. Three trials also included a separate plot where field corn was treated with one in-furrow soil application of a tetraniliprole 200 SC formulation, at application rates ranging from 198 to 202 g ai/ha. Three trials also included two additional plots that were each planted with field corn seed that was treated with a tetraniliprole 480 FS formulation, corresponding with 30–48 g ai/ha. One of these seed treated plots was also treated with three foliar applications of a tetraniliprole 200 SC formulation at 50–51 g ai/ha. In some trials adjuvants (NIS or COC) were used.

At two of the sites, an additional treatment plot was established that received four foliar applications at an exaggerated rate (5×) of nominally 250 g ai/ha, to provide samples for processing. All trials were carried out in 2015.

Samples were collected for analysis at the normal commercial harvest time, fourteen (14) days after the last test substance application. Foliar applications in plots designated for grain sampling (plots 3 and 6) were scheduled so that the last application occurred fourteen (14) days before crop maturity–normal grain harvest. Additional decline samples were collected for analysis at 0, 7, 14 (normal harvest), 21, and 28 days after the last application (DALA).

Grain samples, weighing at least 1 kg, were collected from a mechanical harvester in at least twelve areas within the plot as the harvester proceeded through the plot, or ears were removed from the plants by hand and then shelled. Treated bulk grain samples weighing >200 kg for processing were mechanically harvested.

Samples were stored frozen for a maximum of 158 days (*ca* 5 months) prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone (BCS-CQ63359) using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. The results on grain are summarised in Table 37.

Totals were only calculated where residues of parent tetraniliprole and tetraniliprole-N-methylquinazolinone are above LOQ of 0.01 mg/kg. In the trials summarized below, levels of the metabolite were always below the LOQ therefore, no totals were added to the table.

Table 96 Residues of tetraniliprole in maize grains after foliar treatment using a 200 SC formulation in trials performed in Canada and the United States in 2015 (Study SARS-15-06)

MAIZE Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
Canada, Valley, British Columbia (N09VGT)	4 (7,7,8)	50 50 51 51	17 17 17 17	02 Sept., BBCH 85 +NIS	14	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-BC-3

MAIZE Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
Canada, Wentworth, Ontario (Pioneer 35F38)	4^ (7)	49 49 49 52	25 25 25 25	07 Oct., BBCH 85	0 7 14 23 28	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<u><0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	SARS-15-06-ON-3
United States, Dane, Wisconsin (NP2643GT)	4 (8,6,8)	50 50 51 50	24 24 24 24	15 Oct., BBCH 87 +COC	14	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-WI2-3
United States, Walworth, Wisconsin (DKC49.94R.B.)	4^ (7)	49 49 49 50	26 26 26 27	07 Oct., R6	14	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-WI1-3
United States, York, Nebraska (NP2643GT)	4 (7)	50 50 50 50	26 27 26 26	25 Sept., BBCH 87 +NIS	0 7 14 21 28	<0.010, <0.010 (<0.01) 0.014, <0.010 (0.012) <0.010, <0.010 (<0.01) 0.010, <0.010 (0.010) 0.010, 0.012 (<u>0.011</u>)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	SARS-15-06-NE1-3
United States, York, Nebraska (DKC 60-67 RIB)	4^ (7)	50 51 50 51	23 23 23 23	02 Oct., BBCH 87	15	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-NE2-3
United States, Sherburne Minnesota (NP2643GT)	4 (7)	50 51 51 51	27 27 27 27	18 Sept. BBCH 87 +COC	14	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-MN3-3
United States, Sherburne, Minnesota (DeKalb 41-32)	4^ (7)	51 51 51 50	27 27 27 27	18 Sept., BBCH 87	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-MN1-3
United States, Stearns, Minnesota (DeKalb)	4 (7)	50 50 50 50	32 32 32 31	10 Oct., BBCH 89 +COC	14	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-MN2-3
United States, Stearns, Minnesota (DeKalb)	4 (7)	251 254 261 251	159 160 165 158	10 Oct., BBCH 89 +COC	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-MN2-7
United States, Freeborn, Minnesota (Pioneer 9256)	4 (6,8,7)	50 50 50 50	23 23 23 23	07 Oct., R6 +NIS	14	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-MN4-3

MAIZE Country, Year, Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
United States, Wayne, New York (X19318WP.O)	4^ (7)	52 51 53 52	20 20 20 20	01 Oct., BBCH 87	14	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-NY-3
United States, Wayne, New York (X19318WP.O)	4^ (7)	254 252 262 255	100 99 100 100	01 Oct., BBCH 87	14	0.015, 0.017 (0.016)	<0.01, <0.01 (<0.01)	SARS-15-06-NY-7
United States, Wayne, North Carolina (DKC68-03)	4 (7)	49 50 51 51	19 25 25 23	17 Aug., BBCH 87 +COC	14	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-NC-3
United States, Miami, Ohio (A6408VT3PRIB)	4 (7,7,8)	52 51 52 52	37 37 37 37	02 Oct., R6 +NIS	14	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-OH-3
United States, Shelby, Missouri (G11U58-GT)	4 (7)	50 49 50 50	27 27 27 27	18 Sept., R6 +COC	14	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-MO3-3
United States, Shelby, Missouri (G11U58GT)	4^ (7)	49 49 50 49	27 26 28 27	21 Oct., BBCH 89	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-MO2-3
United States, Macon, Missouri (R1313NT2P)	4 (7)	51 50 49 51	27 26 27 27	10 Sept., Dent/Black Layer +NIS	14	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-MO4-3
United States, Butler, Missouri (Mycogen 2C797)	4 (7)	50 50 49 51	27 27 27 27	01 Sept., BBCH 87 +COC	14	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-MO1-3
United States, Jefferson, Iowa (P1023AM)	4^ (7)	94 50 50 50	23 24 22 23	15 Sept., Early R6	14	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-IA-3
United States, Wharton, Texas (Pioneer P1234AM)	4 (7)	50 50 50 50	40 40 39 40	27 July, BBCH 86 +COC	14	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-TX-3
United States, Cass, North Dakota (01053928)	4 (7)	52 49 51 67	35 27 27 35	01 Oct., BBCH 87 +NIS	14	<0.01, <0.01 (<u>≤0.01</u>)	<0.01, <0.01 (<0.01)	SARS-15-06-ND-3

Notes:

+COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; GS = Growth stage at last treatment; RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.

[a] Residues are expressed in parent equivalents.

[b] At the last application.

Table 97 Residues of tetraniliprole in maize grains after seed treatment (480 SC), in-furrow treatment or a combination of both seed treatment and foliar treatment (200 SC) in trials performed in the United States in 2015 (Study SARS-15-06)

MAIZE Location (variety)	Application				Residues (mg/kg) [a]			Trial No. (soil type)
	No (RTI)	g ai/ha	g ai/hL	Dae, growth stage [b]	DALA	Tetraniliprole	T-N-methy lquinazolinone	
Dane, Wisconsin (NP2643GT)	1^	30	19	01 June, Seed treatment	150	<0.01 (2)	<0.01 (2)	SARS-15-06-WI2-5 (silt loam)
Dane, Wisconsin (NP2643GT)	4 (122,6 ,8)	30 [c] 50 50 50	19 24 24 24	15 Oct., BBCH 87 +COC	14	<0.01 (2)	<0.01 (2)	SARS-15-06-WI2-6 (silt loam)
York, Nebraska (NP2643GT)	1^	32	19	13 May, Seed treatment	149	<0.01 (2)	<0.01 (2)	SARS-15-06-NE1-5 (clay loam)
York, Nebraska (NP2643GT)	4 (121,7 ,7)	32 [c] 50 50 50	19 27 26 26	25 Sept., BBCH 87	14	<0.01 (2)	<0.01 (2)	SARS-15-06-NE1-6 (clay loam)
York, Nebraska (DKC 60-67 RIB)	1^	202	95	09 June, In-furrow at planting	130	<0.01 (2)	<0.01 (2)	SARS-15-06-NE2-4 (clay loam)
Sherburne Minnesota (NP2643GT)	1^	48	20	27 May, Seed treatment	128	<0.01 (2)	<0.01 (2)	SARS-15-06-MN3-5 (loamy sand)
Sherburne Minnesota (NP2643GT)	4 (100,7 ,7)	48 [c] 51 51 51	20 27 27 27	18 Sept. BBCH 87 +COC	14	<0.01 (2)	<0.01 (2)	SARS-15-06-MN3-6 (loamy sand)
Shelby, Missouri (G11U58GT)	1^	198	420	30 June, In-furrow at planting	127	<0.01 (2)	<0.01 (2)	SARS-15-06-MO2-4 (silt loam)
Jefferson, Iowa (P1023AM)	1 [c]	200	250	19 May, In-furrow at planting	133	<0.01 (2)	<0.01 (2)	SARS-15-06-IA-4 (silty clay loam)

Notes:
DALA = Days After Last Application; +COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; GS = Growth stage at last treatment; RTI = Retreatment Interval; Mean residue values presented in parenthesis; T = tetraniliprole.
[a] Residues are expressed in parent equivalents.
[b] At the last application.
[c] Seed treatment.

Maize/sweet corn

Fifteen field trials were conducted in the United States and Canada to measure the magnitude of tetraniliprole residues in/on sweet corn raw agricultural commodities following four foliar applications of a tetraniliprole 200 SC formulation at an actual rate of 48–54 g ai/ha, with application intervals of 6–10 days (Greenland&Stewart, 2016, M-574351-01-2, Report SARA-15-05). Three trials also included a separate plot where sweet corn was treated with one in-furrow soil application of tetraniliprole 200 SC at an application rate of 199–202 g ai/ha. Furthermore, three trials also included two separate plots that were planted with sweet corn seed that was treated with a tetraniliprole 480 FS formulation at a rate of

49–63 g ai/ha. One of these seed treated plots was also treated with three foliar applications of tetraniliprole 200 SC at a rate of 48–52 g ai/ha. Trials were carried out in 2015. In some trials adjuvants (NIS or COC) were used.

All sample collection dates were collected on normal harvest of sweet corn for fresh market, the milk stage. Forage and kernel plus cob with husk removed (K+CWHR) samples were collected at normal harvest, one (1) day after the last foliar application (DALA). Stover samples were collected one month later (1 DALA + one month), corresponding to about 80 to 85 percent dry matter, except at the NE site stover samples were not collected due to technician error of destroying plants before stover sampling date. Forage and K+CWHR samples from the seed treatment only plot were collected on the same day as the other plots. Decline samples of forage and K+CWHR were collected at 0, 1 (normal harvest), 3, 7, and 14 DALA. Stover decline samples were collected at 0 DALA + one month, 1 (normal harvest) DALA + 1 month, 3 DALA + 1 month, 7 DALA + 1 month, and 14 DALA + 1 month. Forage and stover samples were composites of 12 plants. Each stem, with leaves attached, was divided into three (3) equal lengths. Top, middle and bottom portions were randomly selected from each of the three groups of four stems to ensure that parts of all 12 stems were included in the sample. The exception was at the ND site where the entire plant was harvested instead of dividing it into three parts (see Deviation 1). K+CWHR samples consisted of at least 12 ears from different plants in each plot and weighed at least 2 kg.

Samples were stored frozen for a maximum of 316 days (*ca* 10 months) prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. In the trials summarized below, levels of tetraniliprole-N-methylquinazolinone were always below the LOQ. The results on kernels (cobs with husks removed) are summarised in Table 98 and Table 99.

Table 98 Residues of tetraniliprole in sweet corn kernel with cobs with husks removed (K+CWHR) after foliar treatments with a 200 SC formulation in trials performed in Canada and the United States in 2015 (Study SARS-15-05)

SWEET CORN Country, Location (variety)	Application				Residues (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
Canada, , Waterloo, Ontario (Pioneer Ambrosia)	4 (7)	49 52 50 51	25 25 25 25	18 Aug., BBCH 73 +COC	0 1 3 7 14	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	SARS-15-05- ON-2
Canada, , Portage La Prairie, Manitoba (Earlivee)	4^ (7,7,6)	49 50 49 50	50 50 50 50	02 Sept., BBCH 79	1	<0.01 (2)	<0.01 (2)	SARS-15-05- MB-2
Canada, , Fraser Valley, British Columbia (Honey and Cream)	4 (7)	52 51 51 49	13 13 13 13	04 Sept., BBCH 75+NIS	1	<0.01 (2)	<0.01 (2)	SARS-15-05- BC-2
United States, Dane, Wisconsin (Silver King F1)	4 (7,6,8)	51 49 50 50	24 25 25 24	25 Aug., Milk +NIS	1	<0.01 (2)	<0.01 (2)	SARS-15-05- WI-2

SWEET CORN Country, Location (variety)	Application				Residues (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
United States, Lehigh, Pennsylvania (Spring Treat F1)	4 (7,7,6)	49 48 49 48	17 17 17 17	05 Aug., R3 +COC	1	<0.01 (2)	<0.01 (2)	SARS-15-05-PA-2
United States, , Madera. California (Cuppa Joe)	4^ (7)	51 52 51 51	18 18 18 18	03 Aug., late milk	0 1 3 7 14	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	SARS-15-05-CA-2
United States, , Clarke, Georgia (Silver queen)	4 (7)	51 50 50 49	21 19 19 19	06 Aug., BBCH 71-74 +NIS	1	<0.01 (2)	<0.01 (2)	SARS-15-05-GA-2
United States, , Cass, North Dakota (Golden)	4 (8,7,7)	54 50 50 49	36 36 35 36	30 Aug., BBCH 81+COC	1	<0.01 (2)	<0.01 (2)	SARS-15-05-ND-2
United States, Wayne, New York (Supersweet Jubilee plus)	4^ (7)	51 50 51 50	20 20 20 20	27 Aug., Milk stage	1	<0.01 (2)	<0.01 (2)	SARS-15-05-NY-2
United States, , Stearns, Minnesota (Ambrosia)	4^ (7)	50 50 50 49	32 32 32 32	05 Sept., BBCH 75	1	<0.01 (2)	<0.01 (2)	SARS-15-05-MN-2
United States, Shelby, Missouri (Jackpot)	4 (7)	51 50 51 51	27 27 27 27	27 Aug., Milk +COC	1	<0.01 (2)	<0.01 (2)	SARS-15-05-MO1-2
United States, Shelby, Missouri (Incredible)	4 (7)	51 50 53 53	29 27 26 27	30 July, Late milk +NIS	1	<0.01 (2)	<0.01 (2)	SARS-15-05-MO2-2
United States, Hall, Nebraska (Obsession II)	4 (7)	50 50 50 50	26 26 26 28	10 Aug., BBCH 79 +NIS	1	<0.01 (2)	<0.01 (2)	SARS-15-05-NE-2
United States, Seminole, Florida (Primus)	4^ (7,6,7)	51 49 49 49	18 18 18 18	02 Dec., BBCH 79	1	<0.01 (2)	<0.01 (2)	SARS-15-05-FL-2
United States, Bingham, Idaho (Ambrosia)	4 (7)	52 50 49 48	44 36 36 35	04 Sept., R3, Milk +COC	1	<0.01 (2)	<0.01 (2)	SARS-15-05-ID-2

Notes:

+COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.

[a] Residues are expressed in parent equivalents.

[b] At the last application.

Table 99 Residues of tetraniliprole in sweet corn kernel with cobs with husks removed (K+CWHR) after a single in- furrow application using a 200 SC formulation, a seed treatment with a 480 SC formulation or a combination of seed treatment and foliar treatments in trials performed in the United States in 2015 (Study SARS-15-05)

SWEET CORN Location (variety)	Application				Residues (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
Dane, Wisconsin (Silver King F1)	1^	63	-	02 June, Seed treatment	85	<0.01 (2)	<0.01 (2)	SARS-15-05-WI-4
Dane, Wisconsin (Silver King F1)	4 (70,6,8)	63 50 50 50	- 25 25 24	25 Aug., Milk +NIS	1	<0.01 (2)	<0.01 (2)	SARS-15-05-WI-5
Lehigh, Pennsylvania (Spring Treat F1)	1^	60	-	25 May, Seed treatment	73	<0.01 (2)	<0.01 (2)	SARS-15-05 SARS-15-05-PA-4
Lehigh, Pennsylvania (Spring Treat F1)	4 (59,7,6)	60 [a] 48 49 50	- 17 17 17	05 Aug., R3 +COC	1	<0.01 (2)	<0.01 (2)	SARS-15-05 SARS-15-05-PA-5
Madera. California (Cuppa Joe)	1^	49	-	12 May, Seed treatment	84	<0.01 (2)	<0.01 (2)	SARS-15-05-CA-4
Madera. California (Cuppa Joe)	4 (59,7,7)	50 [a] 52 51 51	- 18 18 18	03 Aug., late milk	1	<0.01 (2)	<0.01 (2)	SARS-15-05-CA-5
Clarke, Georgia (Silver queen)	1^	202	110	20 May, In-furrow at planting	79	<0.01 (2)	<0.01 (2)	SARS-15-05-GA-3
Wayne, New York (Supersweet Jubilee plus)	1^	199	240	05 June, In furrow at planting	84	<0.01 (2)	<0.01 (2)	SARS-15-05-NY-3
Shelby, Missouri (Jackpot)	1^	201	410	11 June, In-furrow at planting	78	<0.01 (2)	<0.01 (2)	SARS-15-05-MO1-3

Notes:
+COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Tree nuts

Almonds

Five field trials were conducted in the United States to measure the magnitude of tetraniliprole residues in/on almond, following four foliar applications of a tetraniliprole 200 SC formulation (Greenland, 2016k, M-572123-01-1, Report SARS-15-17). Applications were made at an actual rate of 44–46 g ai/ha with application intervals of 7 days. Trials were carried out in 2015. In some trials an adjuvant (NIS or COC) was used.

Samples of nutmeat were collected at maturity, 10 days after the final application. Additional decline data was collected from 1 site, where samples were taken 0, 5, 10, 15 and 20 days following the final application. Almonds were taken by hand, taking care to avoid the plot boundaries, from several places from upper, middle and lower portions of the trees across the plot. The nutmeats were separated from the hulls. Samples of almond nutmeats (weighing at least 1 kg) were kept cool until they were placed in freezers

Samples were stored frozen for a maximum of 176 days (*ca* 6 months) prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone (BCS-CQ63359) using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. The results of the trials are summarised in Table 100.

Totals were only calculated where residues of parent tetraniliprole and tetraniliprole-N-methylquinazolinone are above LOQ of 0.01 mg/kg. In the trials summarized below, levels of the metabolite were always below the LOQ therefore, no totals were added to the table.

Table 100 Residues of tetraniliprole in almond (nutmeat) after foliar treatment using a 200 SC formulation in trials performed in the United States in 2015 (Study SARS-15-17)

ALMOND Location (variety)	Application				Residues (mean) (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
Fresno, California (Nonpareil)	4^ (7)	45 45 45 45	12 12 12 12	03 Aug., BBCH 85	10	0.01, <0.01 (0.01)	<0.01, <0.01 (<0.01)	SARS-15-17-CA1
Fresno, California (Monterey)	4^ (7)	45 45 45 45	38 38 38 38	20 Aug., BBCH 85	10	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-17-CA4
Fresno, California (Butte)	4 (7)	46 45 45 45	12 12 12 12	31 Aug., BBCH 89 +COC	0 5 10 15 20	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.010, 0.010 (0.010) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	SARS-15-17-CA5
Glenn, California (Nonpareil)	4 (7)	44 44 44 45	38 38 38 38	04 Aug., BBCH 85 +COC	10	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-17-CA2
Yolo, California (Butte)	4 (7)	46 45 45 46	13 13 13 13	07 Aug., BBCH 87 +NIS	10	0.014, 0.017 (0.015)	<0.01, <0.01 (<0.01)	SARS-15-17-CA3

Notes:

+COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval; DALA = Days After Last Application; T = tetraniliprole.

[a] Residues are expressed in parent equivalents.

[b] At the last application.

Pecan

Eight field trials were conducted in the United States to measure the magnitude of tetraniliprole residues in/on pecan, following four foliar applications of a tetraniliprole 200 SC formulation [Greenland, 2016l, M-570119-01-1, Report SARS-14-02]. Applications were made at a rate of 44–47 g ai/ha with application intervals of 7–8 days. The trials were carried out in 2014/2015.

Samples of nutmeat were collected at maturity, generally 10 days after the final application. Additional decline data was collected from 1 site, where samples were taken 0, 5, 10, 15 and 20 days following the final application. Samples of pecan nutmeat weighing at least 1 kg were taken from several places from at least four trees across the plot, except the treated samples from the SARS-14-02-TX3 site were only 0.79 and 0.90 kg.

Samples were stored frozen for a maximum of 245 days (*ca* 8 months) prior to residue analysis. Samples were analysed for residues of tetraniliprole and tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

In the trials summarized below, levels of tetraniliprole and tetraniliprole-N-methylquinazolinone were always below the LOQ. The results of the trials are summarised in Table 101.

Table 101 Residues of tetraniliprole in pecan (nutmeat) after foliar treatment using a 200 SC formulation in trials performed in the United States (Study SARS-14-02)

PECAN Year, Location (variety)	Application				Residues (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
2015 Frio, Texas (Caddo)	4 (7)	45 45 45 46	10 12 12 12	09 Oct., Shuck split + NIS	10	<0.01 (2)	<0.01 (2)	SARS-14-02-TX3 [SS]
2015, Frio, Texas (Cheyenne)	4 (7)	46 44 45 45	3.5 3.5 3.7 3.6	10 Oct., Husks cracking and opening over 50% +COC	10	<0.01 (2)	<0.01 (2)	SARS-14-02-TX1
2014, Briscoe Texas (Cherokee)	4^ (7)	45 46 46 44	14 14 14 13	27 Nov., Full Nut Closed Shuck	0 5 10 15 20	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	SARS-14-02-TX2
2015 Hardeman, Texas (Choctaw)	4 (7)	45 45 45 45	4.7 4.7 4.6 4.6	05 Nov., Late shuck split +COC	10	<0.01 (2)	<0.01 (2)	SARS-14-02-TX4
2014, Mississippi, Missouri (Pawnee)	4 (7)	45 45 45 45	16 16 16 16	07 Nov., BBCH 89 +NIS	10	<0.01 (2)	<0.01 (2)	SARS-14-02-MO1
2015, Mississippi, Missouri (Pawnee)	4^ (7)	45 45 45 45	12 3.9 3.9 3.9	09 Nov., BBCH 85	10	<0.01 (2)	<0.01 (2)	SARS-14-02-MO2
2014, Crisp, Georgia (Desirable)	4^ (7)	47 45 45 44	15 14 15 14	27 Oct., 100% shuck split, fruit ripening	10	<0.01 (2)	<0.01 (2)	SARS-14-02-GA1

PECAN Year, Location (variety)	Application				Residues (mg/kg) [a]			Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	
2014, Irwin, Georgia (Summer)	4 (7)	45 45 45 45	3.4 3.7 3.4 3.3	07 Nov., 70% of pecans ripe for harvest +COC	10	<0.01 (2)	<0.01 (2)	SARS-14-02-GA2

Notes:
DALA = Days After Last Application; +COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant);^no adjuvant added; RTI = Retreatment Interval; T = tetraniliprole.
[SS] = sample size < 1 kg, actual size 0.79 and 0.90 kg.
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Animal feed items

Soya bean forage and soya bean hay

A total of three field trials on soya bean were conducted in the United States in the 2014/2015 growing season [Greenland, 2016j, M-574330-02-1, Report SARS-15-03] resulting in residue data of tetraniliprole in soya bean forage and soya bean hay. In separate plots soya bean was treated with one in-furrow soil application of tetraniliprole 200 SC at an actual rate of 203–240 g ai/ha. Trials were carried out in 2014/2015. Forage and hay samples were cut at approximately BBCH 70 (mid-to-full bloom to pods 50 percent developed). Forage was collected immediately after cutting and hay was collected after drying to about 10 to 20 percent moisture. Samples of soya bean forage weighing at least 1 kg and soya bean hay weighing at least 0.5 kg were taken from random areas across the plots.

Samples were stored frozen for a maximum of 224 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and tetraniliprole-N-methylquinazolinone (BCS-CQ63359) using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. The results are presented in Table 104 and Table 105.

Table 102 Residues of tetraniliprole in soya bean forage and hay after in-furrow treatment at planting using an 200 SC formulation in field trials in the United States in 2015 (Study SARS-15-03)

SOYA BEAN FORAGE Location (variety)	Application		DALA	Sample	Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	g ai/hL			Tetraniliprole	T-N-methyl quinazolinone	Total [b]	
Clarke, Georgia (AG-4933)	203	107	51	forage	0.025, 0.037 (0.031)	<0.01, <0.01 (<0.01)	0.025, 0.037 (0.031)	SARS-15-03-GA1-3
				Hay	0.090, 0.083 (0.086)	0.025, 0.024 (0.024)	0.12, 0.11 (0.11)	
York, Nebraska (S51112199)	199	220	56	forage	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-03-NE1-3
				Hay	0.014, 0.014 (0.014)	<0.01, <0.01 (<0.01)	0.014, 0.014 (0.014)	
Dane, Wisconsin (S17-G8)	200	240	69	forage	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-03-WI2-3
				hay	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	

Notes:

[a] Residues are expressed in parent equivalents.

Rice straw, and whole plant silage

Twelve field trials were conducted throughout Thailand, India and Vietnam to measure the magnitude of tetraniliprole and tetraniliprole-N-methylquinazolinone residues in/on paddy rice following 2 foliar applications of a thiacloprid 480 SC formulation containing 120 g tetraniliprole/L (Woodard, 2019a, Report RAFV0085, Document M-669757-01-1). Applications were made at actual rates of 38–42 g ai/ha, with application intervals of 7–17 days. In addition, twelve field trials were conducted in the same countries following either 1 seed treatment, 1 seed treatment followed by on foliar treatment or 3 foliar treatments with a tetraniliprole 480 FS formulation or a tetraniliprole 200 SC formulation (Woodard, 2019b, Report RAFV0014, Document M-667200-01-1). Seed treatments were made at application rates ranging from 240 to 244 g ai/ha, foliar treatments were made at actual rates of 58–68 g ai/ha. All the trials were carried out in 2018/2019. No adjuvants were used in either study.

For the whole plant (no roots), panicles removed and panicles samples at 33 (actual 31-33 days) day interval, sufficient paddy rice plants were harvested from the control plot first and then from the treated plot to obtain minimum sample sizes (minimum of 12 plants and 0.5 kg) for each whole plant (no roots, panicles removed) and panicles sample. The rice plant was cut about 2 cm above the surface of the water. Panicles were separated from the rest of the plant to create 2 samples; panicles and whole plant (no roots) panicles removed. BBCH growth stage varied between the samples.

For straw samples at 40 (actual 38–40 days), 45 (actual 43–45 days), 50 (actual 48–50 days), 55 days (actual 53 days), and normal commercial harvest samples (NHC). At crop maturity (approximately BBCH 89), sufficient paddy rice plants were harvested from the control plot first and then from the treated plot to obtain minimum sample sizes for all commodities (0.5 kg straw). Sample fractions were generated according to local practice. In a clean area away from the test plots, the whole grain rice was separated from the rice straw. The separated rice grain and straw from both plots was then sun dried up to 5 days, if necessary.

Samples were stored frozen for a maximum of 261 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and tetraniliprole_N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

The results on paddy rice straw and paddy rice whole plant silage are presented in Table 103 to Table 106.

Table 103 Residues of tetraniliprole paddy rice (straw) after foliar treatment using a 120 or 200 SC formulation in trials performed in India,Thailand and Vietnam

RICE STRAW Country, Year, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Study-Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
India, 2018, Ippili, Srikakulam District, Andhra Pradesh (MTU 1010)	2 (7)	41 40	10 10	15 Oct., BBCH 55	38	0.56, 0.58 (0.57)	0.016, 0.017 (0.017)	0.58, 0.60 (0.070)	RAFV0085-G-DA-TRTD
					43	0.57, 0.46 (0.52)	0.016, 0.018 (0.017)	0.59, 0.48 (0.69)	
					48	0.69, 0.94 (0.82)	0.024, 0.031 (0.028)	0.71, 0.97 (0.85)	
					53	1.1, 1.3 (1.2)	0.037, 0.051 (0.044)	1.1, 1.4 (1.2)	

RICE STRAW Country, Year, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Study-Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
India, 2018, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	2 (7)	39 39	10 11	24 Oct., BBCH 56	43	1.5, 1.3 (1.4)	0.036, 0.032 (0.034)	1.5, 1.3 (1.4)	RAFV0085-H-HA-TRTD
India, 2018, Seedhi, Srikakulam District, Andhra Pradesh (MTU1121)	2 (16)	40 42	10 10	01 Nov., BBCH 51	43	0.38, 0.31 (0.35)	0.038, 0.027 (0.033)	0.42, 0.34 (0.38)	RAFV0085-I-HA-TRTD
India, 2018, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	2 (15)	41 40	10 11	03 Nov., BBCH 51	43	1.2, 1.2 (1.2)	0.025, 0.027 (0.026)	1.2, 1.2 (1.2)	RAFV0085-J-HA-TRTD
India, 2018, Ippili Village, Srikakulam District, Andhra Pradesh (MTU1010)	3 (5,2)	62 60 59	-	15 Oct., BBCH 57	38	2.1, 2.3 (2.2)	0.077, 0.061 (0.069)	2.2, 2.4 (2.3)	RAFV0014-G-DA TRTD3
					43	1.4, 1.2 (1.3)	0.044, 0.034 (0.039)	1.4, 1.2 (1.3)	
					48	1.8, 1.4 (1.6)	0.057, 0.039 (0.048)	1.9, 1.4 (1.6)	
					53	2.2, 2.1 (2.2)	0.089, 0.088 (0.089)	2.3, 2.2 (2.3)	
India, 2018, Purli,Srikakulam District, Andhra Pradesh (RNR 15048)	3 (7,7)	59 59 59	-	24 Oct., BBCH 56	43	4.2, 4.7 (4.5)	0.12, 0.11 (0.12)	4.3, 4.8 (4.6)	RAFV0014-H-HA-TRTD3
India, 2018, Seedhi, Srikakulam District, Andhra Pradesh (MTU 1121)	3 (7,16)	58 59 61	-	01 Nov., BBCH 51	43	1.4, 0.90 (1.2)	0.094, 0.077 (0.086)	1.5, 0.98 (1.3)	RAFV0014-I-HA-TRTD3
India, 2018, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	3 (7,15)	60 59 59	-	03 Nov., BBCH 51	43	2.7, 3.1 (2.9)	0.061, 0.078 (0.070)	2.8, 3.2 (3.0)	RAFV0014-J-HA-TRTD3
Thailand, 2018, Kamphaeng Saen, Nakhon Pathom (RD41)	2 (11)	40 40	10 10	10 Dec., BBCH 58	38	5.6, 4.3 (5.0)	0.19, 0.15 (0.17)	5.8, 4.5 (5.2)	RAFV0085-A-DA-TRTD
					43	4.8, 4.2 (4.5)	0.21, 0.17 (0.19)	5.0, 4.4 (4.7)	
					48	4.8, 4.6 (4.7)	0.23, 0.22 (0.23)	5.0, 4.8 (4.9)	
					53	4.8, 5.1 (5.0)	0.33, 0.39 (0.36)	5.1, 5.5 (5.4)	
Thailand, 2018, Tamung Kanchanaburi (RD56)	2 (13)	40 40	10 10	12 Dec., BBCH 56	38	3.1, 3.1 (3.1)	0.11, 0.095 (0.10)	3.2, 3.2 (3.2)	RAFV0085-B-DA-TRTD
					43	3.7, 3.3 (3.5)	0.057, 0.076 (0.067)	3.8, 3.4 (3.6)	
					48	3.5, 2.9 (3.2)	0.057, 0.081 (0.069)	3.6, 3.0 (3.3)	

RICE STRAW Country, Year, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Study-Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
					53	2.8, 2.3 (2.6)	0.084, 0.076 (0.080)	2.9, 2.4 (2.7)	
Thailand, 2018, Yanyao A Samchuk, Suphan Buri (RD41)	2 (8)	40 40	10 10	09 Dec., BBCH 58	43	3.8, 3.7 (3.8)	0.12, 0.12 (0.12)	3.9, 3.8 (3.9)	RAFV0085-C-HA-TRTD
Thailand, 2019, Banma, Bangsai, Ayutthaya (Pitsanloke)	2 (11)	40 39	10 10	20 Jan., BBCH 51	43	1.5, 1.7 (1.6)	0.063, 0.063 (0.063)	1.6, 1.8 (1.7)	RAFV0085-D-HA-TRTD
Thailand, 2019, Chorakaerong, Chaiyo, Ang Thong (RD47)	2 (11)	40 40	10 10	18 Jan., BBCH 51	43	2.1, 1.6 (1.9)	0.070, 0.054 (0.062)	2.2, 1.7 (2.0)	RAFV0085-E-HA-TRTD
Thailand, 2019, Namfarn Inburi Sing Buri Province (RD49)	2 (17)	39 40	10 10	25 Jan., BBCH 51	45	1.8, 1.6 (1.7)	0.068, 0.069 (0.069)	1.9, 1.7 (1.8)	RAFV0085-F-HA-TRTD
					45	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085-F-HA-TRTD
Vietnam, 2018, Hau My Trinh, Cai Be District, Tien Giang Province (OM5451)	2 (11)	40 39	11 10	03 Sept., BBCH 51	40	0.26, 0.17 (0.22)	0.020, 0.018 (0.019)	0.28, 0.19 (0.24)	RAFV0085-K-DA-TRTD
					43	0.23, 0.24 (0.24)	0.016, 0.016 (0.016)	0.25, 026 (0.26)	
					50	0.17, 0.19 (0.18)	0.018, 0.014 (0.016)	0.19, 0.20 (0.20)	
					53	0.14, 0.18 (0.16)	0.013, 0.019 (0.016)	0.15, 0.20 (0.18)	
Vietnam, 2018, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	2 (12)	39 38	10 10	19 Sept., BBCH 51	43	0.24, 0.28 (0.26)	0.029, 0.030 (0.030)	0.27, 0.31 (0.29)	RAFV0085-L-HA-TRTD
Thailand, 2018, Bangkokyae T. Sraseemum, A. Kamphaeng Saen, Nakhon Pathom 73140 (RD41)	3 (7,11)	64 61 61	-	10 Dec., BBCH 58	38	4.4, 3.7 (4.1)	0.17, 0.15 (0.16)	4.6, 3.9 (4.3)	RAFV0014-A-DA-TRTD3
					43	4.8, 5.5 (5.2)	0.19, 0.21 (0.20)	5.0, 5.7 (5.4)	
					48	5.6, 6.2 (5.9)	0.21, 0.21 (0.21)	5.8, 6.4 (6.1)	
					53	6.6, 6.5 (6.6)	0.29, 0.31 (0.30)	6.9, 6.8 (6.9)	
Thailand, 2018, 19/8 M1 T. Muangchum A. Tamung Kanchanaburi (RD56)	3 (7,13)	68 60 61	-	12 Dec., BBCH 56	38	5.1, 6.9 (6.0)	0.15, 0.13 (0.14)	5.4, 7.0 (6.1)	RAFV0014-B-DA-TRTD3
					43	6.8, 6.0 (6.4)	0.11, 0.10 (0.11)	6.9, 6.1 (6.5)	
					48	5.5, 6.2 (5.9)	0.16, 0.21 (0.19)	5.7, 6.4 (6.1)	
					53	6.3, 5.6 (6.0)	0.14, 0.14 (0.14)	6.4, 5.7 (6.1)	
Thailand, 2018, Yanyao A Samchuk, Suphan Buri (RD41)	3 (7,8)	61 61 61	-	09 Dec., BBCH 58	43	5.3, 4.8 (5.1)	0.17, 0.16 (0.17)	5.5, 5.0 (5.3)	RAFV0014-C-HA-TRTD3

RICE STRAW Country, Year, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Study-Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Thailand, 2018, Banma, Bangsai, Ayutthaya Province (Pitsanloke)	3 (12,11)	66 60 60	-	20 Jan., BBCH 51	43	3.1, 1.8 (2.5)	0.11, 0.064 (0.087)	3.2, 1.9 (2.6)	RAFV0014-D-HA-TRTD3
Thailand, 2018, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	3 (10,11)	61 61 61	-	18 Jan., BBCH 51	43	4.6, 5.1 (4.9)	0.14, 0.16 (0.15)	4.7, 5.3 (5.0)	RAFV0014-E-HA-TRTD3
Thailand, 2018, 13 M5 Namfarn Inburi, Sing Buri Province (RD49)	3 (11,17)	62 60 60	-	25 Jan., BBCH 51	45	2.4, 1.7 (2.1)	0.11, 0.078 (0.094)	2.5, 1.8 (2.2)	RAFV0014-F-HA-TRTD3
Vietnam, 2018, Hau My Trinh, Cai Be District, Tien Giang Province (OM5451)	3 (7,11)	64 60 60	-	03 Sept., BBCH 51	40	0.52, 0.37 (0.45)	0.038, 0.028 (0.033)	0.56, 0.40 (0.48)	RAFV0014-K-DA-TRTD3
					43	0.41, 0.40 (0.41)	0.023, 0.025 (0.024)	0.43, 0.43 (0.43)	
					50	0.35, 0.30 (0.33)	0.027, 0.015 (0.021)	0.38, 0.32 (0.35)	
					53	0.26, 0.22 (0.24)	0.029, 0.027 (0.028)	0.29, 0.25 (0.27)	
Vietnam, 2018, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	3 (7,12)	60 59 60	-	19 Sept., BBCH 51	43	0.46, 0.45 (0.46)	0.049, 0.048 (0.049)	0.51, 0.50 (0.51)	RAFV0014-L-HB-TRTD3

Notes:
DALA = Days After Last Application; RTI = Retreatment Interval;
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Table 104 Residues of tetraniliprole paddy rice (straw) after seed treatment or a combination of seed treatment [ST] and one foliar treatment [ST, FT] using a 200 SC formulation for foliar treatment or a 480 SC formulation for seed treatment in trials performed in India, Thailand or Vietnam in 2018 (Study RAFV0014)

RICE STRAW Country, Location (variety)	Application		Date, growth stage [b]	DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No.	g ai/ha			Tetraniliprole	T-N-methyl quinazolinone	Total	
India, Ippili, Srikakulam District, Andhra Pradesh (MTU1010)	1 [ST]	240	08 Aug., NA	NCH	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014-G-DA-TRTD1
India, Ippili, Srikakulam District, Andhra Pradesh (MTU1010)	2 [ST, FT]	240 59	15 Oct., BBCH 57	38	0.88, 1.1 (0.99)	0.038, 0.061 (0.050)	0.92, 1.2 (1.0)	RAFV0014-G-DA-TRTD2
				43	0.87, 0.67 (0.77)	0.029, 0.029 (0.029)	0.90, 0.70 (0.80)	
				48	0.76, 0.78 (0.77)	0.040, 0.042 (0.041)	0.80, 0.82 (0.81)	

RICE STRAW Country, Location (variety)	Application No.	Application g ai/ha	Date, growth stage [b]	DALA	Residues (mean) (mg/kg) [a] Tetraniliprole	T-N-methyl quinazolinone	Total	Trial No.
				53	0.98, 0.93 (0.96)	0.039, 0.051 (0.045)	1.0, 0.98 (1.0)	
India, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	1 [ST]	240	06 Aug., NA	NCH	0.016, 0.018 (0.017)	<0.01, <0.01 (<0.01)	0.016, 0.018 (0.017)	RAFV0014-H-HA-TRTD1
India, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	2 [ST, FT]	240 58	24 Oct., BBCH 56	43	2.4, 2.4 (2.4)	0.079, 0.058 (0.069)	2.5, 2.5 (2.5)	RAFV0014-H-HA-TRTD2
India, Seedhi Village, Srikakulam District, Andhra Pradesh (MTU 1121)	1 [ST]	240	04 Aug., NA	NCH	0.013, 0.015 (0.014)	<0.01, <0.01 (<0.01)	0.013, 0.015 (0.014)	RAFV0014-I-HA-TRTD1
India Seedhi, Srikakulam District, Andhra Pradesh	2 [ST, FT]	240 60	01 Nov., BBCH 51	43	0.90, 1.1 (1.0)	0.10, 0.088 (0.094)	1.0, 1.2 (1.1)	RAFV0014-I-HA-TRTD2
India, Sancham Srikakulam District, Andhra Pradesh (MTU 1121)	1 [ST]	240	10 Aug., NA	NCH	0.015, 0.024 (0.020)	0.017, 0.018 (0.018)	0.032, 0.042 (0.038)	RAFV0014-J-HA-TRTD1
India, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	2 [ST, FT]	240 59	03 Nov., BBCH 51	43	1.6, 1.7 (1.7)	0.033, 0.042 (0.038)	1.6, 1.7 (1.7)	RAFV0014-J-HA-TRTD2
Thailand, Sraseemum, A. Kamphaeng Saen, Nakhon Pathom 73140 (RD41)	1 [ST]	244	27 Sept. NA	NCH	3.9, 0.11 (2.0)	0.17, 0.018 (0.094)	4.1, 0.13 (2.1)	RAFV0014-A-DA-TRTD1
Thailand, Sraseemum, A. Kamphaeng Saen, Nakhon Pathom 73140 (RD41)	2 [ST, FT]	244 63	10 Dec., BBCH 58	38	4.4, 5.2 (4.8)	0.17, 0.17 (0.17)	4.6, 5.4 (5.0)	RAFV0014-A-DA-TRTD2
				43	4.1, 5.3 (4.7)	0.13, 0.19 (0.16)	4.2, 5.5 (4.9)	
				48	5.5, 4.9 (5.2)	0.22, 0.20 (0.21)	5.7, 5.1 (5.4)	
				53	4.8, 5.1 (5.0)	0.25, 0.25 (0.25)	5.1, 5.4 (5.3)	
Thailand, Muangchum A. Tamung Kanchanaburi (RD56)	1 [ST]	244	25 Sept., NA	NCH	0.011, 0.010 (0.011)	<0.01, <0.01 (<0.01)	0.011, 0.010 (0.011)	RAFV0014-B-DA-TRTD1
Thailand, Muangchum A. Tamung Kanchanaburi (RD56)	2 [ST, FT]	244 62	12 Dec., BBCH 56	38	4.5, 3.4 (4.0)	0.077, 0.078 (0.078)	4.6, 3.5 (4.1)	RAFV0014 RAFV0014-B-DA-TRTD2
				43	4.5, 4.3 (4.4)	0.061, 0.085 (0.073)	4.6, 4.4 (4.5)	
				48	3.8, 3.0 (3.4)	0.082, 0.086 (0.084)	3.9, 3.1 (3.5)	
				53	4.0, 3.5 (3.8)	0.083, 0.061 (0.072)	4.1, 3.6 (3.9)	

RICE STRAW Country, Location (variety)	Application		Date, growth stage [b]	DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No.	g ai/ha			Tetraniliprole	T-N-methyl quinazolinone	Total	
Thailand, Yanyao A Samchuk, Suphan Buri 72130 (RD41)	1 [ST]	244	26 Sept., NA	NCH	0.031, 0.036 (0.034)	0.015, 0.013 (0.014)	0.046, 0.049 (0.048)	RAFV0014-C-HA-TRTD1
Thailand, Yanyao A Samchuk, Suphan Buri 72130 (RD41)	2 [ST, FT]	244 61	09 Dec., BBCH 58	43	3.9, 3.9 (3.9)	0.16, 0.18 (0.17)	4.1, 4.1 (4.1)	RAFV0014-C-HA-TRTD2
Thailand, Bangsai, Ayutthaya Province (Pitsanloke)	1 [ST]	244	08 Nov., NA	NCH	0.024, 0.020 (0.022)	<0.01, <0.01 (<0.01)	0.024, 0.020 (0.022)	RAFV0014-D-HA-TRTD1
Thailand, Bangsai, Ayutthaya Province (Pitsanloke)	2 [ST, FT]	244 61	20 Jan., BBCH 51	43	2.0, 1.6 (1.8)	0.065, 0.049 (0.057)	2.1, 1.6 (1.9)	RAFV0014-D-HA-TRTD2
Thailand, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	1 [ST]	244	09 Nov., NA	NCH	0.020, 0.025 (0.023)	0.015, 0.015 (0.015)	0.035, 0.040 (0.038)	RAFV0014-E-HA-TRTD1
Thailand, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	2 [ST, FT]	244 62	18 Jan., BBCH 51	43	3.0, 2.7 (2.9)	0.094, 0.080 (0.087)	3.1, 2.8 (3.0)	RAFV0014-E-HA-TRTD2
Thailand, Inburi Sing Buri Province (RD49)	1 [ST]	244	10 Nov., NA	NCH	0.034, 0.033 (0.034)	<0.01, <0.01 (<0.01)	0.034, 0.033 (0.034)	RAFV0014-F-HA-TRTD1
Thailand, Inburi Sing Buri Province (RD49)	2 [ST, FT]	244 61	25 Jan., BBCH 51	45	1.4, 1.8 (1.6)	0.048, 0.062 (0.055)	1.4. 1.9 (1.7)	RAFV0014-F-HA-TRTD2
Vietnam, Hau My Trinh, Cai Be District, Tien Giang Province (OM5451)	1 [ST]	240	03 July, NA	NCH	0.020, 0.017 (0.019)	<0.01, <0.01 (<0.01)	0.020, 0.017 (0.019)	RAFV0014-K-DA-TRTD1
Vietnam, Hau My Trinh, Cai Be District, Tien Giang Province (OM5451)	2 [ST, FT]	240 59	03 Sept., BBCH 51	40	0.32, 0.17 (0.25)	0.018, 0.025 (0.022)	0.34, 0.20 (0.27)	RAFV0014-K-DA-TRTD2
				43	0.27, 0.21 (0.24)	0.016, 0.017 (0.017)	0.29, 0.23 (0.26)	
				50	0.20, 0.21 (0.21)	0.018, 0.015 (0.017)	0.22, 0.22 (0.22)	
				53	0.23, 0.25 (0.24)	0.023, 0.022 (0.023)	0.25, 0.27 (0.26)	
Vietnam, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	1 [ST]	240	18 July, NA	NCH	0.046, 0.056 (0.051)	0.013, 0.017 (0.015)	0.059, 0.073 (0.066)	RAFV0014-L-HB-TRTD1

RICE STRAW Country, Location (variety)	Application		Date, growth stage [b]	DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No.	g ai/ha			Tetraniliprole	T-N-methyl quinazolinone	Total	
Vietnam, Phong My,Giong Trom District, Ben Tre Province (Nang hoa 9)	2 [ST, FT]	240 58	19 Sept., BBCH 51	43	0.38, 0.38 (0.38)	0.038, 0.039 (0.039)	0.42, 0.42 (0.42)	RAFV0014-L-HB-TRTD2

Notes:
DALA = Days After Last Application; [FT] = Foliar Treatment; [ST] = Seed Treatment;
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Table 105 Residues of tetraniliprole paddy rice whole plants w/o roots, panicles, rest of plants after foliar treatment, using a 120 or 200 SC formulation in trials performed in India, Thailand or Vietnam.

RICE SILAGE Country, Year, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Study-Trial No. Sample
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
India, 2018, Ippili, Srikakulam District, Andhra Pradesh (MTU 1010)	2 (7)	41 40	10 10	BBCH 55	0	0.88, 0.95 (0.92)	<0.01, <0.01 (<0.01)	0.88, 0.95 (0.92)	RAFV0085-G-DA-TRTD Whole plant without roots
					33	0.43, 0.29 (0.36)	0.01, <0.01 (0.01)	0.44, 0.29 (0.37)	RAFV0085-G-DA-TRTD Rest of Plant
					33	0.13, 0.08 (0.11)	<0.01, <0.01 (<0.01)	0.13, 0.08 (0.11)	RAFV0085-G-DA-TRTD Pinacles
India, 2018, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	2 (7)	39 39	10 11	BBCH 56	0	2.8, 3.2 (3.0)	<0.01, 0.013 (0.012)	2.8, 3.2 (3.0)	RAFV0085-H-HA-TRTD Whole plant without roots
India, 2018, Seedhi, Srikakulam District, Andhra Pradesh (MTU1121)	2 (16)	40 42	10 10	BBCH 51	0	1.0, 1.2 (1.1)	<0.01, <0.01 (<0.01)	1.0, 1.2 (1.1)	RAFV0085-I-HA-TRTD Whole plant without roots
India, 2018, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	2 (15)	41 40	10 11	BBCH 51	0	0.89, 0.75 (0.82)	<0.01, <0.01 (<0.01)	0.89, 0.75 (0.82)	RAFV0085-J-HA-TRTD Whole plant without roots
India, 2018, Ippili, Srikakulam District, Andhra Pradesh (MTU1010)	3 (5,2)	62 60 59	-	15 Oct., BBCH 57	0	1.5, 1.7 (1.6)	<0.01, <0.01 (<0.01)	1.5, 1.7 (1.6)	RAFV0014-G-DA-TRTD3 Whole plant without roots
					33	1.0, 1.2 (1.1)	0.022, 0.019 (0.021)	1.0, 1.2 (1.1)	RAFV0014-G-DA-TRTD3 Rest of Plant

RICE SILAGE Country, Year, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Study-Trial No. Sample
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
					33	0.27, 0.33 (0.30)	<0.01, <0.01 (<0.01)	0.27, 0.33 (0.30)	RAFV0014-G-DA-TRTD3 Pinacles
India, 2018, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	3 (7,7)	59 59 59	-	24 Oct., BBCH 56	0	2.7, 3.2 (3.0)	<0.01, <0.01 (<0.01)	2.7, 3.2 (3.0)	RAFV0014-H-HA-TRTD3 Whole plant without roots
India, 2018, Seedhi Village, Srikakulam District, Andhra Pradesh (MTU 1121)	3 (7,16)	58 59 61	-	01 Nov., BBCH 51	0	2.5, 2.4 (2.5)	0.015, 0.020 (0.018)	2.5, 2.4 (2.5)	RAFV0014-I-HA-TRTD3 Whole plant without roots
India, 2018, Sancham Village, Srikakulam District, Andhra Pradesh (MTU 1121)	3 (7,15)	60 59 59	-	03 Nov., BBCH 51	0	2.1, 1.8 (2.0)	<0.01, <0.01 (<0.01)	2.1, 1.8 (2.0)	RAFV0014-J-HA-TRTD3 Whole plant without roots
Thailand, 2018, Kamphaeng Saen, Nakhon Pathom (RD41)	2 (11)	40 40	10 10	BBCH 58	0	3.5, 2.7 (3.1)	<0.01, <0.01 (<0.01)	3.5, 2.7 (3.1)	RAFV0085-A-DA-TRTD Whole plant without roots
					32	1.2, 0.94 (1.1)	0.043, 0.037 (0.040)	1.2, 0.98 (1.1)	RAFV0085-A DA-TRTD Rest of Plant
					32	1.8, 2.4 (2.1)	0.017, 0.016 (0.017)	1.8, 2.4 (2.1)	RAFV0085-A-DA-TRTD Pinacles
Thailand, 2018, Tamung Kanchanaburi (RD56)	2 (13)	40 40	10 10	BBCH 56	0	1.9, 1.9 (1.9)	<0.01, <0.01 (<0.01)	1.9, 1.9 (1.9)	RAFV0085-B-DA-TRTD Whole plant without roots
					31	0.57, 0.50 (0.54)	0.01, <0.01 (0.01)	0.58, 0.50 (0.55)	RAFV0085-B-DA-TRTD Rest of Plant
					31	0.12, 0.20 (0.16)	<0.01, <0.01 (<0.01)	0.12, 0.20 (0.16)	RAFV0085-B-DA-TRTD Pinacles
Thailand, 2018, Yanyao A Samchuk, Suphan Buri (RD41)	2 (8)	40 40	10 10	BBCH 58	0	1.3, 2.3 (1.8)	<0.01, <0.01 (<0.01)	1.3, 2.3 (1.8)	RAFV0085-C-HA-TRTD Whole plant without roots
Thailand, 2019, Banma, Bangsai, Ayutthaya Province (Pitsanloke)	2 (11)	40 39	10 10	BBCH 51	0	1.6, 1.8 (1.7)	<0.01, <0.01 (<0.01)	1.6, 1.8 (1.7)	RAFV0085-D-HA-TRTD Whole plant without roots

RICE SILAGE Country, Year, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Study-Trial No. Sample
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Thailand, 2019, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	2 (11)	40 40	10 10	BBCH 51	0	1.8, 1.8 (1.8)	<0.01, <0.01 (<0.01)	1.8, 1.8 (1.8)	RAFV0085-E-HA-TRTD Whole plant without roots
Thailand, 2019, Namfarn Inburi Sing Buri Province (RD49)	2 (17)	39 40	10 10	BBCH 51	0	1.0, 1.7 (1.4)	<0.01, <0.01 (<0.01)	1.0, 1.7 (1.4)	RAFV0085-F-HA-TRTD Whole plant without roots
Vietnam, 2018, Hau My Trinh Village, Cai Be District, Tien Giang Province (OM5451)	2 (11)	40 39	11 10	BBCH 51	0	1.2, 1.1 (1.2)	<0.01, <0.01 (<0.01)	1.2, 1.1 (1.2)	RAFV0085-K-DA-TRTD Whole plant without roots
					33	0.10, 0.071 (0.086)	<0.01, <0.01 (<0.01)	0.10, 0.071 (0.086)	RAFV0085-K-DA-TRTD Rest of Plant
					33	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0085-K-DA-TRTD Pinacles
Vietnam, 2018, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	2 (15)	39 38	10 10	BBCH 51	45	1.6, 1.4 (1.5)	<0.01, <0.01 (<0.01)	1.6, 1.4 (1.5)	RAFV0085-L-HA-TRTD Whole plant without roots
Thailand, 2018, Sraseemum, A. Kamphaeng Saen, Nakhon Pathom 73140 (RD41)	3 (7,11)	64 61 61	-	10 Dec., BBCH 58	0	3.0, 3.6 (3.3)	0.011, 0.014 (0.013)	3.0, 3.6 (3.3)	RAFV0014-A-DA-TRTD3 Whole plant without roots
					32	1.1, 1.2 (1.2)	0.049, 0.062 (0.056)	1.1, 1.3 (1.3)	RAFV0014-A-DA-TRTD3 Rest of Plant
					32	2.3, 3.1 (2.7)	0.014, 0.020 (0.017)	2.3, 3.1 (2.7)	RAFV0014-A-DA-TRTD3 Pinacles
Thailand, 2018, Muangchum A. Tamung Kanchanaburi (RD56)	3 (7,13)	68 60 61	-	12 Dec., BBCH 56	0	2.5, 2.5 (2.5)	<0.01, <0.01 (<0.01)	2.5, 2.5 (2.5)	RAFV0014-B-DA-TRTD3 Whole plant without roots
					31	1.0, 1.5 (1.3)	0.018, 0.034 (0.026)	1.0, 1.5 (1.3)	RAFV0014-B-DA-TRTD3 Rest of Plant
					31	0.11, 0.20 (0.16)	<0.01, <0.01 (<0.01)	0.11, 0.20 (0.17)	RAFV0014-B-DA-TRTD3 Pinacles
Thailand, 2018, Yanyao A Samchuk, Suphan Buri 72130 (RD41)	3 (7,8)	61 61 61	-	09 Dec., BBCH 58	0	3.0, 3.0 (3.0)	0.010, 0.010 (0.010)	3.0, 3.0 (3.0)	RAFV0014-C-HA-TRTD3 Whole plant without roots

RICE SILAGE Country, Year, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Study-Trial No. Sample
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Thailand, 2018, Banma, Bangsai, Ayutthaya Province (Pitsanloke)	3 (12,11)	66 60 60	-	20 Jan., BBCH 51	0	2.5, 1.9 (2.2)	<0.01, <0.01 (<0.01)	2.5, 1.9 (2.2)	RAFV0014-D-HA-TRTD3 Whole plant without roots
Thailand, 2018, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	3 (10,11)	61 61 61	-	18 Jan., BBCH 51	0	2.1, 2.6 (2.4)	<0.01, <0.01 (<0.01)	2.1, 2.6 (2.4)	RAFV0014-E-HA-TRTD3 Whole plant without roots
Thailand, 2018, Namfarn Inburi Sing Buri Province (RD49)	3 (11,17)	62 60 60	-	19 Jan., BBCH 51	0	2.2, 2.3 (2.3)	<0.01, <0.01 (<0.01)	2.2, 2.3 (2.3)	RAFV0014-F-HA-TRTD3 Whole plant without roots
Vietnam, 2018, Hau My Trinh, Cai Be District, Tien Giang Province (OM5451)	3 (7,11)	64 60 60	-	03 Sept., BBCH 51	0	1.3, 2.1 (1.7)	<0.01, <0.01 (<0.01)	1.3, 2.1 (1.7)	RAFV0014-K-DA-TRTD3 Whole plant without roots
					33	0.098, 0.10 (0.099)	<0.01, <0.01 (<0.01)	0.11, 0.11 (0.11)	RAFV0014-K-DA-TRTD3 Rest of Plant
					33	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	RAFV0014-K-DA-TRTD3 Pinacles
Vietnam, 2018, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	3 (7,12)	60 59 60	-	19 Sept., BBCH 51	0	2.1, 2.1 (2.1)	0.012, <0.01 (0.011)	2.1, 2.1 (2.1)	RAFV0014-L-HB-TRTD3 Whole plant without roots

Notes:
DALA = Days After Last Application; RTI = Retreatment Interval;
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Table 106 Residues of tetraniliprole paddy rice whole plants w/o roots, panicles, rest of plants after seed treatment or a combination of seed treatment with a 480 SC formulation and a foliar treatment using a 200 SC formulation in trials performed in India, Thailand or Vietnam in 2018 (Study RAFV0014)

Country, Location RICE SILAGE (variety)	Application			DALA	Residues (mean) (mg/kg) [a]			Trial No. Sample
	No	g ai/ha	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Thailand, 2018, Sraseemum, A. Kamphaeng Saen, Nakhon	2 [ST& FT]	244 63	10 Dec., BBCH 58	0	4.8, 4.6 (4.7)	0.012, 0.010 (0.011)	4.8, 4.6 (4.7)	RAFV0014-A-DA-TRTD2 Whole plant without roots

Country, Location RICE SILAGE (variety)	Application			DALA	Residues (mean) (mg/kg) [a]			Trial No. Sample
	No	g ai/ha	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Pathom (RD41)				32	0.80, 1.2 (1.0)	0.039, 0.044 (0.042)	0.84, 1.2 (1.0)	RAFV0014-A-DA-TRTD2 Rest of Plant
				32	3.1, 2.7 (2.9)	0.019, 0.019 (0.019)	3.1, 2.7 (2.9)	RAFV0014-A-DA-TRTD2 Pinacles
Thailand, 2018, Muangchum A. Tamung Kanchanaburi (RD56)	2 [ST& FT]	244 62	12 Dec., BBCH 56	0	1.5, 1.6 (1.6)	<0.01, <0.01 (<0.01)	1.5, 1.6 (1.6)	RAFV0014-B-DA-TRTD2 Whole plant without roots
				31	0.80, 0.48 (0.64)	0.013, 0.020 (0.017)	0.81, 0.50 (0.66)	RAFV0014-B-DA-TRTD2 Rest of Plant
				31	0.17, 0.11 (0.14)	<0.01, <0.01 (<0.01)	0.17, 0.11 (0.14)	RAFV0014-B-DA-TRTD2 Pinacles
Thailand, Yanyao A Samchuk, Suphan Buri 72130 (RD41)	2 [ST& FT]	244 61	09 Dec., BBCH 58	0	2.5, 3.3 (2.9)	<0.01, <0.01 (<0.01)	2.5, 3.3 (2.9)	RAFV0014-C-HA-TRTD2 Whole plant without roots
Thailand, Banma, Bangsai, Ayutthaya Province (Pitsanloke)	2 [ST& FT]	244 61	20 Jan., BBCH 51	0	2.5, 2.0 (2.3)	<0.01, <0.01 (<0.01)	2.5, 2.0 (2.3)	RAFV0014-D-HA-TRTD2 Whole plant without roots
Thailand, Chorakaerong, Chaiyo, Ang Thong Province (RD47)	2 [ST& FT]	244 62	18 Jan., BBCH 51	0	1.6, 1.9 (1.8)	<0.01, <0.01 (<0.01)	1.6, 1.9 (1.8)	RAFV0014-E-HA-TRTD2 Whole plant without roots
Thailand, 2018, Namfarn Inburi Sing Buri Province (RD49)	2 [ST& FT]	244 61	19 Jan., BBCH 51	0	2.0, 1.8 (1.9)	<0.01, <0.01 (<0.01)	2.0, 1.8 (1.9)	RAFV0014-F-HA-TRTD2 Whole plant without roots
India, Ippili, Srikakulam District, Andhra Pradesh (MTU1010)	2 [ST& FT]	240 59	15 Oct., BBCH 57	0	1.2, 1.2 (1.2)	<0.01, <0.01 (<0.01)	1.2, 1.2 (1.2)	RAFV0014-G-DA-TRTD2 Whole plant without roots
				33	0.49, 0.24 (0.37)	0.012, <0.01 (0.011)	0.50, 0.24 (0.38)	RAFV0014-G-DA-TRTD2 Rest of Plant
				33	0.13, 0.17 (0.15)	<0.01, <0.01 (<0.01)	0.13, 0.17 (0.15)	RAFV0014-G-DA-TRTD2 Pinacles
India, Purli, Srikakulam District, Andhra Pradesh (RNR 15048)	2 [ST& FT]	240 58	24 Oct., BBCH 56	0	2.1, 1.8 (2.0)	<0.01, <0.01 (<0.01)	2.1, 1.8 (2.0)	RAFV0014-H-HA-TRTD2 Whole plant without roots

Country, Location RICE SILAGE (variety)	Application			DALA	Residues (mean) (mg/kg) [a]			Trial No. Sample
	No	g ai/ha	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
India, Seedhi, Srikakulam District, Andhra Pradesh (MTU 1121)	2 [ST, FT]	240 60	01 Nov., BBCH 51	0	1.9, 2.0 (2.0)	<0.01, <0.01 (<0.01)	1.9, 2.0 (2.0)	RAFV0014-I-HA-TRTD2 Whole plant without roots
India, Sancham, Srikakulam District, Andhra Pradesh (MTU 1121)	2 [ST& FT]	240 59	03 Nov., BBCH 51	0	1.2, 1.3 (1.3)	<0.01, <0.01 (<0.01)	1.2, 1.3 (1.3)	RAFV0014-J-HA-TRTD2 Whole plant without roots
Vietnam, Hau My Trinh, Cai Be District, Tien Giang Province (OM5451)	2 [ST& FT]	240 59	03 Sept., BBCH 51	0	0.99, 0.78 (0.89)	<0.01, <0.01 (<0.01)	0.99, 0.78 (0.89)	RAFV0014-K-DA-TRTD2 Whole plant without roots
				33	0.090, 0.082 (0.086)	<0.01, <0.01 (<0.01)	0.090, 0.082 (0.086)	RAFV0014-K-DA-TRTD2 Rest of Plant
				33	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	RAFV0014 RAFV0014-K-DA-TRTD2 Pinacles
Vietnam, Phong My, Giong Trom District, Ben Tre Province (Nang hoa 9)	2 [ST& FT]	240 58	19 Sept., BBCH 51	0	1.2, 1.4 (1.3)	<0.01, <0.01 (<0.01)	1.2, 1.4 (1.3)	RAFV0014-L-HB-TRTD2 Whole plant without roots

Notes:
DALA = Days After Last Application; [FT] = Foliar Treatment; [ST] = Seed Treatment
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Maize forage and stover

Supervised field residue trials on maize/field corn were conducted in Canada and the United States in the 2018/2019 growing season (Stewart & Greenland, 2016, M-574645-01-2, Report SARS-15-06).

At all locations plots received four foliar applications of a tetraniliprole 200 SC formulation. Foliar applications were generally made at applications rates ranging between 49 and 52 g ai/ha, with application intervals of 6–8 days, except for one trial that had 13 days between the 2nd and 3rd applications to plot 2. Three trials also included a separate plot where field corn was treated with one in-furrow soil application of a tetraniliprole 200 SC formulation, at application rates ranging from 198 to 202 g ai/ha. Three trials also included two additional plots that were each planted with field corn seed that was treated with a tetraniliprole 480 FS formulation, corresponding with 30–48 g ai/ha. One of these seed treated plots was also treated with three foliar applications of a tetraniliprole 200 SC formulation at 50–51 g ai/ha. In some trials adjuvants (NIS or COC) were used.

Samples were collected for analysis at the normal commercial harvest time, fourteen (14) days after the last test substance application. Foliar applications in plots designated for forage sampling (plot 2) were scheduled so that the last application occurred fourteen (14) days before the late dough/early

dent stage–normal forage harvest. Foliar applications in plots designated for stover sampling (plots 3 and 6) were scheduled so that the last application occurred fourteen (14) days before crop maturity–normal stover harvest. Forage and stover samples from in-furrow treatment (plot 4) and seed only treatment (plot 5) were harvested at the same time as the forage and stover were harvested in plots 2 and 3. Additional decline data was collected from 2 sites, where samples were taken nominally 0, 7, 14, 21 and 28 days following the final foliar application. Decline samples were collected for analysis at 0, 7, 14 (normal harvest), 21, and 28 days after the last application (DALA).

Forage and stover samples were composites of twelve plants. Treated bulk grain samples for generating aspirated grain fractions were mechanically harvested.

Samples were stored frozen for a maximum of 158 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

In a second study, fifteen field trials were conducted in Canada and the United States to measure the magnitude of tetraniliprole residues in/on sweet corn raw agricultural commodities following four foliar applications of a tetraniliprole 200 SC formulation at an actual rate of 48–54 g ai/ha, with application intervals of 6–10 days [Greenland&Stewart, 2016, M-574351-01-2, Report SARA-15-05]. Three trials also included a separate plot where sweet corn was treated with one in-furrow soil application of tetraniliprole 200 SC at an application rate of 199–202 g ai/ha. Furthermore, three trials also included two separate plots that were planted with sweet corn seed that was treated with a tetraniliprole 480 FS formulation at a rate of 49–63 g ai/ha. One of these seed treated plots was also treated with three foliar applications of tetraniliprole 200 SC at a rate of 48–52 g ai/ha. Trials were carried out in 2015. In some trials adjuvants (NIS or COC) were used.

All sample collection dates were keyed on normal harvest of sweet corn for fresh market, the milk stage. Forage samples were collected at this stage, one (1) day after the last foliar application (DALA). Stover samples were collected one month later (1 DALA + one month), corresponding to about 80 to 85 percent dry matter, except at the NE site stover samples were not collected due to technician error of destroying plants before stover sampling date. Forage samples from the seed treatment only plot were collected on the same day as the other plots. Decline samples of forage were collected at 0, 1 (normal harvest), 3, 7, and 14 DALA. Stover decline samples were collected at 0 DALA + one month, 1 (normal harvest) DALA + 1 month, 3 DALA + 1 month, 7 DALA + 1 month, and 14 DALA + one month. Forage and stover samples were composites of 12 plants. Each stem, with leaves attached, was divided into three (3) equal lengths. Top, middle and bottom portions were randomly selected from each of the three groups of four stems to ensure that parts of all 12 stems were included in the sample. The exception was at the ND site where the entire plant was harvested instead of dividing it into three parts.

Samples were stored frozen for a maximum of 316 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

The results on maize forage and stover are presented in Tables 107 to 113.

Table 107 Residues of tetraniliprole in field maize orage after foliar treatment using a 200 SC formulation in trials performed in Canada and the United States in 2015 (Study SARS-15-06)

MAIZE FORAGE, Country, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Canada, Valley, British Columbia (N09VGT)	4 (7)	50 50 52 50	17 17 17 17	04 Aug., BBCH 75 +NIS	14	2.2, 2.1 (2.2)	<0.01, <0.01 (<0.01)	2.2, 2.1 (2.2)	SARS-15-06-BC-2
Canada, Wentworth, Ontario (Pioneer 35F38)	4^ (7)	51 51 52 54	25 25 25 25	25 Aug., BBCH 75	0 7 14 21 29	2.0, 1.9 (2.0) 1.4, 2.2 (1.8) 1.1, 1.1 (1.1) 0.61, 0.76 (0.68) 0.66, 0.80 (0.74)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, 0.013 (0.013) 0.013, 0.016 (0.014) 0.020, 0.025 (0.022)	2.0, 1.9 (2.0) 1.4, 2.2 (1.8) 1.1, 1.1 (1.1) 0.62, 0.78 (0.69) 0.68, 0.82 (0.76)	SARS-15-06-ON-2
United States, Dane, Wisconsin (NP2643GT)	4 (7,6,8)	51 50 50 50	25 24 24 24	26 Aug., Milk +COC	14	0.50, 0.57 (0.53)	<0.01, <0.01 (<0.01)	0.50, 0.57 (0.53)	SARS-15-06-WI2-2
United States, York, Nebraska (NP2643GT)	4 (7)	50 50 50 51	26 28 27 27	11 Aug., BBCH 79 +NIS	0 7 14 21 28	0.46, 0.36 (0.41) 0.37, 0.37 (0.37) 0.24, 0.24 (0.24) 0.25, 0.24 (0.24) 0.13, 0.17 (0.15)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	0.46, 0.36 (0.41) 0.37, 0.37 (0.37) 0.24, 0.24 (0.24) 0.25, 0.24 (0.24) 0.13, 0.17 (0.15)	SARS-15-06-NE1-2
United States, Sherburne Minnesota (NP2643GT)	4 (7)	49 50 50 50	27 27 27 27	14 Aug., Milk-Early dough +COC	14	0.56, 0.79 (0.68)	<0.01, <0.01 (<0.01)	0.56, 0.79 (0.68)	SARS-15-06-MN3-2
United States, Sherburne, Minnesota (DeKalb 41-32)	4^ (7)	50 50 50 50	27 27 27 27	14 Aug., BBCH 75	14	0.17, 0.29 (0.23)	<0.01, <0.01 (<0.01)	0.17, 0.29 (0.23)	SARS-15-06-MN1-2
United States, Stearns, Minnesota (DeKalb)	4 (7)	49 49 50 50	32 31 32 31	28 Aug., BBCH 79-83 +COC	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-MN2-2
United States, Freeborn, Minnesota (Pioneer 9256)	4 (7)	50 50 50 50	23 23 23 23	14 Aug., R3 +NIS	14	0.21, 0.17 (0.19)	<0.01, <0.01 (<0.01)	0.21, 0.17 (0.19)	SARS-15-06-MN4-2

MAIZE FORAGE, Country, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
United States, Miami, Ohio (A6408VT3PRIB)	4 (8,6,7)	53 53 55 52	37 37 37 37	02 Sept., R4 +NIS	14	0.90, 1.0 (0.96)	0.021, 0.026 (0.024)	0.92, 1.0 (0.97)	SARS-15-06-OH-2
United States, Wayne, New York (X19318WP.O)	4^ (7)	50 50 50 50	20 20 20 20	20 Aug., Milk stage	14	0.45, 0.65 (0.55)	<0.01, <0.01 (<0.01)	0.45, 0.65 (0.55)	SARS-15-06-NY-2
United States, Wayne, North Carolina (DKC68-03)	4 (7)	50 50 51 51	17 21 21 19	27 July, BBCH 89 +COC	14	1.2, 1.4 (1.3)	0.025, 0.019 (0.022)	1.2, 1.4 (1.3)	SARS-15-06-NC-2
United States, Stafford Kansas (P1105AM-N502)	4 (7)	54 51 51 52	30 30 30 30	06 Aug., BBCH 65-67 +NIS	14	0.44, 0.45 (0.44)	<0.01, <0.01 (<0.01)	0.44, 0.45 (0.44)	SARS-15-06-KS-2
United States, York, Nebraska (DKC 60-67 RIB)	4^ (7,7,6)	50 51 49 49	23 23 23 23	26 Aug., BBCH 75	14	0.41, 0.58 (0.49)	<0.01, <0.01 (<0.01)	0.41, 0.58 (0.49)	SARS-15-06-NE2-2
United States, York, Nebraska (DKC 60-67 RIB)	4^ (7)	50 51 50 51	23 23 23 23	02 Oct., BBCH 87	15	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-NE2-3
United States, Shelby, Missouri (G11U58-GT)	4 (7)	52 50 52 51	25 27 26 27	14 Aug., Dough +COC	14	1.0, 0.98 (1.0)	0.019, 0.012 (0.016)	1.0, 1.0 (1.0)	SARS-15-06-MO3-2
United States, Shelby, Missouri (G11U58GT)	4^ (7)	50 51 51 50	27 27 27 28	18 Sept., R3	14	3.6, 2.6 (3.1)	<0.01, <0.01 (<0.01)	3.6, 2.6 (3.1)	SARS-15-06-MO2-2
United States, Macon, Missouri (R1313NT2P)	4 (7)	50 50 51 50	29 27 25 27	31 July, Late Milk/Early Dough +NIS	14	0.074, 0.14 (0.11)	<0.01, <0.01 (<0.01)	0.074, 0.14 (0.11)	SARS-15-06-MO4-2
United States, Butler, Missouri (Mycogen 2C797)	4 (7)	50 49 51 51	27 27 27 27	20 July, BBCH 71-73 +COC	14	0.52, 0.45 (0.48)	0.023, 0.016 (0.020)	0.54, 0.47 (0.50)	SARS-15-06-MO1-2
United States, Walworth, Wisconsin (DKC49.94R.B.)	4^ (7,8,6)	49 50 49 50	27 28 27 26	27 Aug., R3 Milk	14	0.036, 0.044 (0.040)	<0.01, <0.01 (<0.01)	0.036, 0.044 (0.040)	SARS-15-06-WI1-2

MAIZE FORAGE, Country, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
United States, Jefferson, Iowa (P1023AM)	4^ (7)	49 51 50 51	33 20 24 24	07 Aug., R3	14	0.54, 0.35 (0.44)	<0.01, <0.01 (<0.01)	0.54, 0.35 (0.44)	SARS-15-06-IA-2
United States, Wharton, Texas (Pioneer P1234AM)	4 (7,13, 7)	50 49 50 50	45 41 41 43	02 July, BBCH 79 +COC	14	1.5, 1.1 (1.3)	0.023, 0.031 (0.027)	1.5, 1.1 (1.3)	SARS-15-06-TX-2
United States, Cass, North Dakota (01053928)	4 (7)	51 51 49 51	35 35 27 27	14 Sept, BBCH 84 +NIS	14	2.1, 0.71 (1.4)	0.014, 0.013 (0.014)	2.1, 0.72 (1.4)	SARS-15-06-ND-2

Notes:
DALA = Days After Last Application; +COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant);^no adjuvant added; RTI = Retreatment Interval.
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Table 108 Residues of tetraniliprole in field corn forage after seed treatment or in-furrow treatment a 480 SC formulation for seed treatment and a 200 SC formulation for in-furrow treatment in trials performed in the United States in 2015 (Study SARS-15-06)

MAIZE FORAGE Location (variety)	Application			DALA	Residues (mean) (mg/kg) [a]			Trial No.
	g ai/ha	g ai/hL	Date treatment		Tetraniliprole	T-N-methyl quinazolinone	Total	
Dane, Wisconsin (NP2643GT)	30	19	01 June, Seed treatment	100	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-WI2-5
York, Nebraska (NP2643GT)	32	19	13 May, Seed treatment	104	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-NE1-5
Sherburne Minnesota (NP2643GT)	48	20	27 May, Seed treatment	93	0.60, 0.75 (0.67)	<0.01, <0.01 (<0.01)	0.60, 0.75 (0.67)	SARS-15-06-MN3-5
York, Nebraska (DKC 60-67 RIB)	202	95	09 June, In-furrow at planting	92	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-NE2-4
Shelby, Missouri (G11U58GT)	198	420	30 June, In-furrow at planting	94	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-MO2-4
Jefferson, Iowa (P1023AM)	200	250	19 May, In-furrow at planting	94	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-IA-4

Notes:
DALA = Days After Last Application.
[a] Residues are expressed in parent equivalents.

Table 109 Residues of tetraniliprole in sweet corn forage after 4 foliar treatments using a 200 SC formulation in trials performed in Canada and the United States in 2015 (Study SARS-15-05)

MAIZE FORAGE Country, Location (variety	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total [b]	
Canada, Waterloo, Ontario (Pioneer Ambrosia)	4 (7)	49 52 50 51	25 25 25 25	18 Aug., BBCH 73 +COC	0 1 3 7 14	2.1, 2.4 (2.3) 2.0, 1.7 (1.8) 1.9, 2.4 (2.2) 2.3, 2.1 (2.2) 1.9, 2.4 (2.2)	<0.01, 0.010 (0.010) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) 0.017, 0.020 (0.018)	2.1, 2.4 (2.3) 2.0, 1.7 (1.8) 1.9, 2.4 (2.2) 2.3, 2.1 (2.2) 1.9, 2.4 (2.2)	SARS-15-05-ON-2
Canada, Portage La Prairie, Manitoba (Earlivee)	4^ (7,7,6)	49 50 49 50	50 50 50 50	02 Sept., BBCH 79	1	1.6, 2.6 (2.1)	0.014, 0.011 (0.013)	1.6, 2.6 (2.1)	SARS-15-05-MB-2
Canada, Fraser Valley, British Columbia (Honey and Cream)	4 (7)	52 51 51 49	13 13 13 13	04 Sept., BBCH 75 +NIS	1	1.1, 1.4 (1.2)	<0.01, <0.01 (<0.01)	1.1, 1.4 (1.2)	SARS-15-05-BC-2
United States, Dane, Wisconsin (Silver King F1)	4 (7,6,8)	51 49 50 50	24 25 25 24	25 Aug., Milk +NIS	1	0.67, 0.78 (0.72)	<0.01, <0.01 (<0.01)	0.67, 0.78 (0.72)	SARS-15-05-WI-2
United States, Lehigh, Pennsylvania (Spring Treat F1)	4 (7,7,6)	49 48 49 48	17 17 17 17	05 Aug., R3 +COC	1	3.7, 3.0 (3.4)	0.017, 0.013 (0.015)	3.7, 3.0 (3.4)	SARS-15-05-PA-2
United States, Madera. California (Cuppa Joe)	4^ (7)	51 52 51 51	18 18 18 18	03 Aug., Mature corn for harvest – late milk	0 1 3 7 14	1.2, 1.7 (1.4) 2.0, 2.6 (2.3) 2.5, 2.6 (2.5) 1.5, 2.0 (1.8) 1.4, 2.7 (2.0)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, 0.013 (0.012) <0.01, <0.01 (<0.01) <0.01, 0.012 (0.012)	1.2, 1.7 (1.4) 2.0, 2.6 (2.3) 2.5, 2.6 (2.5) 1.5, 2.0 (1.8) 1.4, 2.7 (2.0)	SARS-15-05-CA-2
United States, Clarke, Georgia (Silver queen)	4 (7)	51 50 50 49	21 19 19 19	06 Aug., BBCH 71-74 +NIS	1	1.2, 0.94 (1.1)	0.020, 0.019 (0.020)	1.2, 0.96 (1.1)	SARS-15-05-GA-2
United States, Cass, North Dakota (Golden)	4 (8,7,7)	54 50 50 49	36 36 35 36	30 Aug., BBCH 81 +COC	1	1.6, 1.8 (1.7)	<0.01, <0.01 (<0.01)	1.6, 1.8 (1.7)	SARS-15-05-ND-2
United States, Wayne, New York (Supersweet Jubilee plus)	4^ (7)	51 50 51 50	20 20 20 20	27 Aug., Milk stage	1	1.7, 1.8 (1.8)	0.015, 0.017 (0.016)	1.7, 1.8 (1.8)	SARS-15-05-NY-2

MAIZE FORAGE Country, Location (variety	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total [b]	
United States, Stearns, Minnesota (Ambrosia)	4^ (7)	50 50 50 49	32 32 32 32	05 Sept., BBCH 75	1	0.053, 0.076 (0.064)	<0.01, <0.01 (<0.01)	0.053, 0.076 (0.064)	SARS-15-05-MN-2
United States, Shelby, Missouri (Jackpot)	4 (7)	51 50 51 51	27 27 27 27	27 Aug., Milk +COC	1	3.4, 3.4 (3.4)	<0.01, 0.011 (0.011)	3.4, 3.4 (3.4)	SARS-15-05-MO1-2
United States, Shelby, Missouri (Incredible)	4 (7)	51 50 53 53	29 27 26 27	30 July, Late milk +NIS	1	2.9, 2.8 (2.8)	0.015, 0.018 (0.017)	2.9, 2.8 (2.8)	SARS-15-05-MO2-2
United States, Hall, Nebraska (Obsession II)	4 (7)	50 50 50 50	26 26 26 28	10 Aug., BBCH 79 +NIS	1	1.0, 1.4 (1.2)	<0.01, <0.01 (<0.01)	1.0, 1.4 (1.2)	SARS-15-05-NE-2
United States, Seminole, Florida (Primus)	4^ (7,6,7)	51 49 49 49	18 18 18 18	02 Dec., BBCH 79	1	1.1, 1.7 (1.4)	<0.01, <0.01 (<0.01)	1.1, 1.7 (1.4)	SARS-15-05-FL-2
United States, Bingham, Idaho (Ambrosia)	4 (7)	52 50 49 48	44 36 36 35	04 Sept., R3, Milk +COC	1	1.4, 1.5 (1.5)	<0.01, <0.01 (<0.01)	1.4, 1.5 (1.5)	SARS-15-05-ID-2

Notes:
DALA = Days After Last Application; +COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval.
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Table 110 Residues of tetraniliprole in sweet corn forage after seed treatment (480 SC) or an in-furrow application (200 SC) or a combination of seed treatment and 3 foliar applications (200 SC) in trials performed in the United States in 2015 (Study SARS-15-05)

SWEET CORN FORAGE Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Dane, Wisconsin (Silver King F1)	1^	63	-	02 June, Seed treatment	85	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-05-WI-4
Dane, Wisconsin (Silver King F1)	4 (70, 6,8)	63[c] 50 50 50	- 25 25 24	25 Aug., Milk +NIS	1	0.48, 0.53 (0.51)	<0.01, <0.01 (<0.01)	0.48, 0.53 (0.51)	SARS-15-05-WI-5
Lehigh, Pennsylvania (Spring Treat F1)	1^	60	-	25 May, Seed treatment	73	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-05-PA-4
Lehigh, Pennsylvania (Spring Treat F1)	4 (59, 7,6)	60[c] 48 49 50	- 17 17 17	05 Aug., R3 +COC	1	2.3, 2.7 (2.5)	<0.01, <0.01 (<0.01)	2.3, 2.7 (2.5)	SARS-15-05-PA-

SWEET CORN FORAGE Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Madera. California (Cuppa Joe)	1^	49	-	12 May, Seed treatment	84	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-05-CA-4
Madera. California (Cuppa Joe)	4 (59, 7,7)	50[c] 52 51 51	- 18 18 18	03 Aug., late milk	1	2.6, 2.0 (2.3)	<0.01, <0.01 (<0.01)	2.6, 2.0 (2.3)	SARS-15-05-CA-5
Clarke, Georgia (Silver queen)	1^	202	110	20 May, In-furrow at planting	79	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-05-GA-3
Wayne, New York (Supersweet Jubilee plus)	1^	199	240	05 June, In furrow at planting	84	0.014, <0.01 (0.012)	<0.01, <0.01 (<0.01)	0.014, <0.01 (0.012)	SARS-15-05-NY-3
Shelby, Missouri (Jackpot)	1^	201	410	11 June, In-furrow at planting	78	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-05-MO1-3

Notes:

DALA = Days After Last Application; +COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant);^no adjuvant added; GS = Growth stage at last treatment; RTI = Retreatment Interval;

[a] Residues are expressed in parent equivalents.

[b] At the last application.

[c] Seed treatment.

Table 111 Residues of tetraniliprole in field corn stover after foliar treatment using a 200 SC formulation in trials performed in Canada and the United States in 2015 (Study SARS-15-06).

MAIZE STOVER Country, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Canada, Wentworth, Ontario (Pioneer 35F38)	4^ (7)	51 51 52 54	25 25 25 25	25 Aug., BBCH 75	0 7 14 21 29	2.0, 1.9 (2.0) 1.4, 2.2 (1.8) 1.1, 1.1 (1.1) 0.61, 0.76 (0.68) 0.66, 0.80 (0.74)	<0.010, <0.010 (<0.01) <0.010, <0.010 (<0.01) <0.010, 0.013 (0.013) 0.013, 0.016 (0.014) 0.020, 0.025 (0.022)	2.0, 1.9 (2.0) 1.4, 2.2 (1.8) 1.1, 1.1 (1.1) 0.62, 0.78 (0.69) 0.68, 0.82 (0.76)	SARS-15-06-ON-2
Canada, Wentworth, Ontario (Pioneer 35F38)	4^ (7)	49 49 49 52	25 25 25 25	07 Oct., BBCH 85	0 7 14 23 28	1.5, 1.4 (1.5) 0.85, 0.92 (0.88) 0.32, 0.42 (0.37) 0.15, 0.22 (0.19) 0.19, 0.24 (0.22)	0.011, 0.011 (0.011) 0.020, 0.017 (0.019) 0.021, 0.022 (0.021) 0.026, 0.030 (0.028) 0.029, 0.027 (0.028)	1.5, 1.4 (1.5) 0.87, 0.94 (0.90) 0.34, 0.44 (0.39) 0.18, 0.25 (0.22) 0.22, 0.27 (0.25)	SARS-15-06-ON-3
Canada, Valley, British Columbia	4 (7,7,8)	50 50	17 17	02 Sept., BBCH 85	14	2.8, 3.1 (2.9)	<0.01, <0.01 (<0.01)	2.8, 3.1 (2.9)	SARS-15-06-

MAIZE STOVER Country, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
(N09VGT)		51 51	17 17	+NIS					BC-3
United States, Dane, Wisconsin (NP2643GT)	4 (8,6,8)	50 50 51 50	24 24 24 24	15 Oct., BBCH 87 +COC	14	2.1, 2.5 (2.3)	<0.01, <0.01 (<0.01)	2.1, 2.5 (2.3)	SARS-15-06-WI2-3
United States, York, Nebraska (NP2643GT)	4 (7)	50 50 50 50	26 27 26 26	25 Sept., BBCH 87 +NIS	0 7 14 21 28	0.55, 0.60 (0.57) 0.27, 0.26 (0.26) 0.20, 0.16 (0.18) 0.16, 0.24 (0.20) 0.16, 0.067 (0.11)	<0.010, <0.010 (<0.01) <0.010, <0.010 (<0.01) <0.010, <0.010 (<0.01) 0.010, 0.016 (0.013) 0.016, 0.011 (0.014)	0.55, 0.60 (0.57) 0.27, 0.26 (0.26) 0.20, 0.16 (0.18) 0.17, 0.26 (0.21) 0.18, 0.078 (0.12)	SARS-15-06-NE1-3
United States, Sherburne Minnesota (NP2643GT)	4 (7)	50 51 51 51	27 27 27 27	18 Sept. BBCH 87 +COC	14	3.6, 3.5 (3.5)	<0.01, <0.01 (<0.01)	3.6, 3.5 (3.5)	SARS-15-06-MN3-3
United States, Sherburne, Minnesota (DeKalb 41-32)	4^ (7)	51 51 51 50	27 27 27 27	18 Sept., BBCH 87	14	4.0, 4.8 (4.4)	0.019, 0.026 (0.022)	4.0, 4.8 (4.4)	SARS-15-06-MN1-3
United States, Stearns, Minnesota (DeKalb)	4 (7)	50 50 50 50	32 32 32 31	10 Oct., BBCH 89 +COC	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-MN2-3
United States, Freeborn, Minnesota (Pioneer 9256)	4 (6,8,7)	50 50 50 50	23 23 23 23	07 Oct., R6 +NIS	14	2.2, 3.0 (2.6)	0.034, 0.040 (0.037)	2.2, 3.0 (2.6)	SARS-15-06-MN4-3
United States, Wayne, New York (X19318WP.O)	4^ (7)	52 51 53 52	20 20 20 20	01 Oct., BBCH 87	14	0.35, 0.22 (0.28)	<0.01, <0.01 (<0.01)	0.35, 0.22 (0.28)	SARS-15-06-NY-3
United States, Wayne, North Carolina (DKC68-03)	4 (7)	49 50 51 51	19 25 25 23	17 Aug., BBCH 87 +COC	14	2.6, 2.1 (2.4)	0.13, 0.12 (0.13)	2.7, 2.2 (2.5)	SARS-15-06-NC-3
United States, Miami, Ohio (A6408VT3PRIB)	4 (7,7,8)	52 51 52 52	37 37 37 37	02 Oct., R6 +NIS	14	3.4, 2.4 (2.9)	0.089, 0.062 (0.076)	3.5, 2.5 (3.0)	SARS-15-06-OH-3
United States, Shelby, Missouri (G11U58-GT)	4 (7)	50 49 50 50	27 27 27 27	18 Sept., R6 +COC	14	8.6, 7.1 (7.9)	0.060, 0.049 (0.054)	8.7, 7.1 (8.0)	SARS-15-06-MO3-3
United States, Stafford Kansas (P1105AM-N502)	4 (7)	49 48 50 49	30 30 30 30	16 Sept., BBCH 87 +NIS	14	11, 9.1 (10)	0.019, 0.013 (0.016)	11, 9.1 (10)	SARS-15-06-KS-3
United States,	4^	50	23	02 Oct.,	15	2.1, 2.9 (2.5)	0.022, 0.028	2.1, 2.9 (2.5)	SARS-

MAIZE STOVER Country, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
York, Nebraska (DKC 60-67 RIB)	(7)	51 50 51	23 23 23	BBCH 87			(0.025)		15-06-NE2-3
United States, Shelby, Missouri (G11U58GT)	4^ (7)	49 49 50 49	27 26 28 27	21 Oct., BBCH 89	14	3.8, 4.8 (4.3)	<0.01, <0.01 (<0.01)	3.8, 4.8 (4.3)	SARS-15-06-MO2-3
United States, Butler, Missouri (Mycogen 2C797)	4 (7)	50 50 49 51	27 27 27 27	01 Sept., BBCH 87 +COC	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-MO1-3
					14	3.4, 2.8 (3.1)	0.060, 0.041 (0.051)	3.5, 2.8 (3.2)	SARS-15-06-MO1-3
United States, Macon, Missouri (R1313NT2P)	4 (7)	51 50 49 51	27 26 27 27	10 Sept., Dent/Black Layer +NIS	14	6.0, 5.8 (5.9)	0.17, 0.25 (0.21)	6.2, 6.0 (6.1)	SARS-15-06-MO4-3
United States, Walworth, Wisconsin (DKC49.94R.B.)	4^ (7)	49 49 49 50	26 26 26 27	07 Oct., R6	14	0.92, 0.68 (0.80)	<0.01, <0.01 (<0.01)	0.92, 0.68 (0.80)	SARS-15-06-WI1-3
United States, Jefferson, Iowa (P1023AM)	4^ (7)	94 50 50 50	23 24 22 23	15 Sept., Early R6	14	2.8, 3.0 (2.9)	0.092, 0.078 (0.085)	2.9, 3.1 (3.0)	SARS-15-06-IA-3
United States, Wharton, Texas (Pioneer P1234AM)	4 (7)	50 50 50 50	40 40 39 40	27 July, BBCH 86 +COC	14	3.4, 3.1 (3.2)	0.065, 0.080 (0.073)	3.5, 3.2 (3.3)	SARS-15-06-TX-3
United States, Cass, North Dakota (01053928)	4 (7)	52 49 51 67	35 27 27 35	01 Oct., BBCH 87 +NIS	14	11, 6.8 (9.1)	0.011, 0.014 (0.012)	11, 6.8 (9.1)	SARS-15-06-ND-3

Notes:
DALA = Days After Last Application; +COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval;
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Table 112 Residues of tetraniliprole in maize stover after seed treatment, in-furrow treatment at planting or a combination of seed treatment and 3 foliar applications using a 200 SC formulation for foliar treatment and a 480 SC formulation for seed treatment in trials performed in the United States in 2015 (Study SARS-06-15)

MAIZE STOVER Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Dane, Wisconsin (NP2643GT)	1^	30	19	01 June, Seed treatment	150	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-WI2-5
Dane,	4	30[c]	19	15 Oct.,	14	1.4, 1.4	<0.01, <0.01	1.4, 1.4 (1.4)	SARS-

MAIZE STOVER Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [a]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Wisconsin (NP2643GT)	(122, 6,8)	50 50 50	24 24 24	BBCH 87 + COC		(1.4)	(<0.01)		15-06-WI2-6
York, Nebraska (NP2643GT)	1^	32	19	13 May, Seed treatment	149	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-NE1-5
York, Nebraska (NP2643GT)	4 (121, 7,7)	32[c] 50 50 50	19 27 26 26	25 Sept., BBCH 87	14	1.2, 1.1 (1.1)	<0.01, <0.01 (<0.01)	1.2, 1.1 (1.1)	SARS-15-06-NE1-6
Sherburne Minnesota (NP2643GT)	1^ [c]	48	20	27 May, Seed treatment	128	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-MN3-5
Sherburne Minnesota (NP2643GT)	4 (100, 7,7)	48[c] 51 51 51	20 27 27 27	18 Sept. BBCH 87 + COC	14	3.8, 6.7 (5.3)	<0.01, 0.012 (0.011)	3.8, 6.7 (5.3)	SARS-15-06-MN3-6
York, Nebraska (DKC 60-67 RIB)	1^ [d]	202	95	09 June, In-furrow	130	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-NE2-4
Shelby, Missouri (G11U58GT)	1	198	420	30 June, In-furrow	127	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-06-MO2-4
Jefferson, Iowa (P1023AM)	1	200	250	19 May, In-furrow	133	0.012, 0.010 (0.011)	<0.01, <0.01 (<0.01)	0.012, 0.010 (0.011)	SARS-15-06-IA-4

Notes:

DALA = Days After Last Application; +COC = adjuvant added (Crop Oil Concentrate); RTI = Retreatment Interval;

[a] Residues are expressed in parent equivalents.

[b] At the last application.

[c] Seed treatment.

Table 113 Residues of tetraniliprole in sweet corn stover after foliar treatments with a 200 SC formulation in trials performed in Canada and the United States in 2015 (Study SARS-15-05)

SWEET CORN STOVER Country, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Canada, Waterloo, Ontario (Pioneer Ambrosia)	4 (7)	49 52 50 51	25 25 25 25	18 Aug., BBCH 73 +COC	28 29 31 36 45	4.0, 3.3 (3.7) 3.5, 6.1 (4.8) 5.0, 9.3 (7.2) 9.6, 6.2 (7.9) 6.6, 5.9 (6.3)	0.049, 0.034 (0.041) 0.051, 0.039 (0.045) 0.095, 0.064 (0.079) 0.074, 0.062 (0.068) 0.23, 0.19 (0.21)	4.0, 3.3 (3.7) 3.6, 6.1 (4.8) 5.1, 9.4 (7.3) 9.7, 6.3 (8.0) 6.8, 6.1 (6.5)	SARS-15-05-ON-2
Canada, Portage La Prairie,	4^ (7,7,6)	49 50 49	50 50 50	02 Sept., BBCH 79	14	2.4, 2.5 (2.5)	0.058, 0.065 (0.061)	2.5, 2.6 (2.6)	SARS-15-05-MB-2

SWEET CORN STOVER Country, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Manitoba (Earlivee)		50	50						
Canada, Fraser Valley, British Columbia (Honey and Cream)	4 (7)	52 51 51 49	13 13 13 13	04 Sept., BBCH 75 +NIS	31	0.63, 0.44 (0.54)	0.019, 0.014 (0.016)	0.65, 0.45 (0.56)	SARS-15-05-BC-2
United States, Dane, Wisconsin (Silver King F1)	4 (7,6,8)	51 49 50 50	24 25 25 24	25 Aug., Milk +NIS	31	0.093, 0.17 (0.13)	0.031, 0.031 (0.031)	0.12, 0.20 (0.16)	SARS-15-05-WI-2
United States, Lehigh, Pennsylvania (Spring Treat F1)	4 (7,7,6)	49 48 49 48	17 17 17 17	05 Aug., R3 +COC	30	1.6, 2.6 (2.2)	0.052, 0.078 (0.065)	1.7, 2.7 (2.3)	SARS-15-05-PA-2
United States, Madera. California (Cuppa Joe)	4^ (7)	51 52 51 51	18 18 18 18	03 Aug., Mature corn for harvest – late milk	30 31 33 37 44	8.6, 13 (11) 15, 16 (16) 14, 17 (16) 13, 4.5 (8.8) 12, 15 (14)	<0.010, 0.012 (0.011) 0.012, 0.012 (0.012) 0.012, 0.015 (0.014) 0.012 0.011 (0.012) <0.010, 0.0101 (0.010)	8.6, 13 (11) 15, 16 (16) 14, 17 (16) 13, 4.5 (8.8) 12, 15 (14)	SARS-15-05-CA-2
United States, Clarke, Georgia (Silver queen)	4 (7)	51 50 50 49	21 19 19 19	06 Aug., BBCH 71-74 +NIS	20	1.1, 0.37 (0.72)	0.17, 0.13 (0.15)	1.3, 0.50 (0.87)	SARS-15-05-GA-2
United States, Cass, North Dakota (Golden)	4 (8,7,7)	54 50 50 49	36 36 35 36	30 Aug., BBCH 81 +COC	31	0.79, 0.81 (0.80)	0.020, 0.014 (0.017)	0.81, 0.82 (0.82)	SARS-15-05-ND-2
United States, Wayne, New York (Supersweet Jubilee plus)	4^ (7)	51 50 51 50	20 20 20 20	27 Aug., Milk stage	29	<0.01, 0.018 (0.014)	<0.01, <0.01 (<0.01)	<0.01, 0.018 (0.014)	SARS-15-05-NY-2
United States, Stearns, Minnesota (Ambrosia)	4^ (7)	50 50 50 49	32 32 32 32	05 Sept., BBCH 75	31	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-05-MN-2
United States, Shelby, Missouri (Jackpot)	4 (7)	51 50 51 51	27 27 27 27	27 Aug., Milk +COC	29	5.7, 5.2 (5.4)	0.089, 0.066 (0.078)	5.8, 5.3 (5.5)	SARS-15-05-MO1-2
United States, Shelby, Missouri (Incredible)	4 (7)	51 50 53 53	29 27 26 27	30 July, Late milk +NIS	29	1.1, 1.3 (1.2)	0.075, 0.11 (0.091)	1.2, 1.4 (1.3)	SARS-15-05-MO2-2
United States, Seminole, Florida	4^ (7,6,7)	51 49 49	18 18 18	02 Dec., BBCH 79	32	1.2, 1.6 (1.4)	0.056, 0.065 (0.061)	1.3, 1.7 (1.5)	SARS-15-05-FL-2

SWEET CORN STOVER Country, Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
(Primus)		49	18						
United States, Bingham, Idaho (Ambrosia)	4 (7)	52 50 49 48	44 36 36 35	04 Sept., R3, Milk +COC	31	1.3, 1.6 (1.4)	<0.01, <0.01 (<0.01)	1.3, 1.6 (1.4)	SARS-15-05-ID-2

Notes:
DALA = Days After Last Application; +COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval.
[a] Residues are expressed in parent equivalents.
[b] At the last application.

Table 114 Residues of tetraniliprole in sweet corn stover after a seed treatment (480 SC), in furrow application at at planting (200 SC), or a combination of a seed treatment with 3 foliar applications in trials performed in the United States in 2015 (Study SARS-15-05)

SWEET CORN STOVER Location (variety)	Application				Residues (mean) (mg/kg) [a]				Trial No.
	No (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]	DALA	Tetraniliprole	T-N-methyl quinazolinone	Total	
Dane, Wisconsin (Silver King F1)	1^	63	-	02 June, Seed treatment	115	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-05-WI-4
Dane, Wisconsin (Silver King F1)	4 (70,6,8)	63 [c] 50 50 50	- 25 25 24	25 Aug., Milk +NIS	31	0.072, 0.086 (0.079)	0.026, 0.028 (0.027)	0.098, 0.11 (0.10)	SARS-15-05-WI-5
Lehigh, Pennsylvania (Spring Treat F1)	1^	60	-	25 May, Seed treatment	102	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-05-PA-4
Lehigh, Pennsylvania (Spring Treat F1)	4 (59,7,6)	60 [c] 48 49 50	- 17 17 17	05 Aug., R3 +COC	30	1.2, 1.1 (1.1)	0.031, 0.033 (0.032)	1.2, 1.1 (1.1)	SARS-15-05-PA-5
Madera. California (Cuppa Joe)	1^	49	-	12 May, Seed treatment	114	0.013, <0.01 (0.011)	<0.01, <0.01 (<0.01)	0.013, <0.01 (0.011)	SARS-15-05-CA-4
Madera. California (Cuppa Joe)	4 (59,7,7)	50 [c] 52 51 51	- 18 18 18	03 Aug., late milk	31	12, 9.8 (11)	<0.01, <0.01 (<0.01)	12, 9.8 (11)	SARS-15-05-CA-5
Clarke, Georgia (Silver queen)	1^	202	110	20 May, In-furrow	98	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-05-GA-3
Wayne, New York (Supersweet Jubilee plus)	1^	199	240	05 June, In-furrow	112	1.6, 2.0 (1.8)	0.081, 0.079 (0.080)	1.7, 2.1 (1.9)	SARS-15-05-NY-3
Shelby, Missouri (Jackpot)	1^	201	410	11 June, In-furrow	106	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	SARS-15-05-MO1-3

Notes:

DALA = Days After Last Application in days; +COC = adjuvant added (Crop Oil Concentrate); +NIS = adjuvant added (non-ionic surfactant); ^no adjuvant added; RTI = Retreatment Interval in days.
[a] Residues are expressed in parent equivalents.
[b] At the last application.
[c] Seed treatment.

Almond hulls

Five field trials were conducted in the United States to measure the magnitude of tetraniliprole residues in/on almond, following four foliar applications of a tetraniliprole 200 SC formulation [Greenland, 2016k, M-572123-01-1, Report SARS-15-17]. Applications were made at an actual rate of 44–46 g ai/ha with application intervals of 7 days. Trials were carried out in 2015.

Samples of almond hulls were collected at maturity, 10 days after the final application. Additional decline DALA was collected from 1 site, where samples were taken 0, 5, 10, 15 and 20 days following the final application. Almonds were taken by hand, taking care to avoid the plot boundaries, from several places from upper, middle and lower portions of the trees across the plot. The nutmeats were separated from the hulls. Samples of almond hulls (weighing at least 1 kg) were kept cool until they were placed in freezers

Samples were stored frozen for a maximum of 176 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent. The results of the trials are summarised in Table 114.

Table 115 Residues of tetraniliprole in almond hulls after foliar treatment using a 200 SC formulation in trials performed in the United States in 2015 (Study SARS-15-17)

ALMOND HULLS Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Study reference, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Fresno, California (Nonpareil)	4 (7)	45 45 45 45	12 12 12 12	03 Aug., BBCH 85	10	1.6, .2.0 (1.8)	<0.01, <0.01 (<0.01)	1.6, .2.0 (1.8)	SARS-15-17-CA1
Fresno, California (Monterey)	4 (7)	45 45 45 45	38 38 38 38	20 Aug., BBCH 85	10	1.1, 1.0 (1.1)	<0.01, <0.01 (<0.01)	1.1, 1.0 (1.1)	SARS-15-17-CA4
Fresno, California (Butte)	4 (7)	46 45 45 45	12 12 12 12	31 Aug., BBCH 89	0 5 10 15 20	1.0, 0.97 (1.0) 0.78, 0.82 (0.80) 0.66, 0.87 (0.77) 0.30, 0.34 (0.32) 0.26, 0.27 (0.26)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	1.0, 0.97 (1.0) 0.78, 0.82 (0.80) 0.66, 0.87 (0.77) 0.30, 0.34 (0.32) 0.26, 0.27 (0.26)	SARS-15-17-CA5
Glenn, California (Nonpareil)	4 (7)	44 44 44 45	38 38 38 38	04 Aug., BBCH 85	10	0.21, 0.22 (0.22)	<0.01, <0.01 (<0.01)	0.21, 0.22 (0.22)	SARS-15-17-CA2

ALMOND HULLS Location (variety)	Application				DALA	Residues (mean) (mg/kg) [a]			Study reference, Trial No.
	No. (RTI)	g ai/ha	g ai/hL	Date, growth stage [b]		Tetraniliprole	T-N-methyl quinazolinone	Total	
Yolo, California (Butte)	4 (7)	46 45 45 46	13 13 13 13	07 Aug., BBCH 87	10	0.78, 0.82 (0.80)	<0.01, <0.01 (<0.01)	0.78, 0.82 (0.80)	SARS-15-17-CA3

Notes:
DALA = Days After Last Application; RTI = Retreatment Interval;
[a] Residues are expressed in parent equivalents.
[b] At the last application.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data.

Nature of residues during processing

The hydrolytic stability of tetraniliprole was investigated in aqueous buffer solutions at 3 pH values and temperatures to simulate representative processing conditions (Bongartz&Schmeling, 2014c, M-475964-01-1, Report EnSa-13-1093). The study was performed at pH 4, 5 and 6 and temperatures of 90 °C, 100 °C and 120 °C, for 20, 60 and 20 minutes, respectively, representing pasteurization, baking/brewing/boiling and sterilization. Temperatures were maintained constant throughout incubation and no significant variation of the pH values was observed in the buffered solutions.

[Pyrazole-carboxamide-^{14}C]-tetraniliprole and [phenyl-carbamoyl-^{14}C]-tetraniliprole were prepared in buffer solution (pH 4, 5 and 6) at a nominal concentration of 1 mg/L. The radioactivity in the samples at start and end of the study was determined by LSC. Material balances ranged from 94 to 107 percent of the applied radioactivity, and at least 97 percent of the radioactivity in the samples was identified by reverse phase HPLC.

A summary of the results are shown in Table 115.

Table 115 Radioactive residues of [pyrazole-carboxamide-^{14}C]-tetraniliprole (py-^{14}C) and of [phenyl-carbamoyl-^{14}C]-tetraniliprole (phe-^{14}C) and hydrolysis products under representative processing conditions.

	Processing conditions					
	pH 4 / 90 °C / 20 min pasteurisation		pH 5 / 100 °C / 60 min baking/brewing/boiling		pH 6 / 120 °C / 20 min sterilisation	
Tetraniliprole , % RA	89.93	94.93	64.61	67.92	1.14	1.46
T-pyrazole-5-N-methyl-amide, % RA	1.04	NA	3.03	NA	1.98	NA
T-esamino-methyl-carboxylic acid, % RA	0.54	--	2.45	2.48	2.09	1.52
T-N-methyl-quinazolinone, % RA	8.49	3.93	29.91	26.61	93.62	94.30
Total identified, % RA	100	98.86	100	97.01	98.83	97.28
Total characterised, % RA	--	1.13	--	2.99	1.18	2.72
Number of unknown peaks	--	2	--	2	2	3
Largest unknown peak, % RA	NA	0.65	NA	2.41	0.68	1.24
Accountability, % RA	100	100	100	100	100	100

Notes:
RA = Radioactivity; NA = not applicable.

Tetraniliprole was shown to be predominantly stable at pH 4 / 90 °C / 20 min (pasteurisation). However, conversion into degradation compounds increased under conditions with higher pH- and temperature values. Under sterilisation conditions nearly all parent compound was hydrolysed. The main hydrolysis product for both labels was tetraniliprole-N-methyl-quinazolinone. Only low amounts of tetraniliprole-pyrazole-5-N-methyl-amide (specific for the pyrazole-carboxamide label) and tetraniliprole-desamino-methyl-carboxylic acid were detected.

Magnitude of residues during processing

Oranges

Two field trials were conducted, in the United States (Veal&Jerkins, 2016b, M-560734-01-1, Report RAFVN026), to measure the magnitude of tetraniliprole residues in/on orange processed commodities following three foliar applications of tetraniliprole 200 SC at an exaggerated (5×) nominal rate of 300 g ai/ha per application, without the use of an adjuvant. Applications were made to oranges (Valencia) at actual total rates of 900–910 g ai/ha with application intervals of 4–5 days at BBCH 83 to 89. Spray volumes ranged from 465–1414 L/ha. Samples of orange whole fruit were harvested 1-day after the final application at commercial maturity (BBCH 83–89). Single composite samples of orange fruit were harvested from the treated and untreated plots one day after the last application, at BBCH growth stages 83 to 89. Individual sub-samples of control and treated unwashed orange fruit, the raw agricultural commodity (RAC), were removed and frozen for subsequent analysis. Oranges were processed according to simulated commercial procedures into washed whole fruit, peel, washed peel, pulp, juice, raw juice, peel without oil, oil, wet pomace, dry pomace, dried pulp and marmalade. Throughout the study one representative sample of untreated control and two (duplicate) samples of treated processed commodities were taken.

Samples were stored frozen (<-18 °C) for a maximum of 687 days (*ca* 23 months) prior to analysis. All samples were analysed for residues of tetraniliprole and tetraniliprole-N-methyl-quinazolinone using analytical method 01414.

Oranges were weighed after removal from storage or upon receipt. Representative unwashed fruit sample fractions were taken (sample: *raw agricultural commodity*). The remaining fruit was hand inspected for undesirable fruit or field debris, and if observed, it was removed, weighed, and discarded.

Washing and peeling

An aliquot of unwashed fruit (3.9–6.2 kg) was peeled, and the unwashed peel (0.78–1.8 kg) was collected (sample: *unwashed peel)*. The peeled fruit was weighed (2.7–4.4 kg) and discarded. The remaining oranges (195–202 kg) were 'batch tub' washed for 5 minutes. Representative samples of washed fruit fractions were collected (sample: *washed whole fruit)*. An aliquot of washed fruit (4.1–6.1 kg) was peeled, and representative sample (sample: *washed peel)* was taken from the washed peel (1.0/1.8 kg) as well as a sample (sample: *peeled orange (flesh)*) from the peeled fruit (2.9–4.3 kg). The remaining peeled fruit was discarded. The yield factor for peeling unwashed fruit ranged from 0.67 to 0.76. The yield factor for peeling of washed fruit ranged from 0.70 to 0.78.

Marmalade

An aliquot of washed fruit was set aside for producing marmalade (2.0–2.5 kg). The rind of the oranges for marmalade processing was removed with a vegetable peeler/zester (33–117 g). The rind was then chopped in a food processor and cooked for 20 minutes, resulting in 71–172 g (weight gain of -5.3–55 g) and set aside. The remaining rind on the oranges was removed and the seeds discarded (1.1–1.5 kg). The orange flesh was chopped in the Robot Coupe food processor (0.65–1.1 kg) and cooked on the stove for 40 minutes with added water (20 percent of fruit by weight), being 123–210 g. The rind and fruit were then combined with 3 tablespoons of lemon juice per kilogram of fruit, and sugar at 1.5 times the total weight of the fruit mixture. The total mixture was then boiled for 3 minutes, pectin (0.05 kg) was added, and the mixture (1.1–1.8 kg) was boiled for an additional 2 minutes. The finished marmalade (1.6–2.8 kg) was packed into sterilized jars and cooled. Representative jars of marmalade were collected (sample: *marmalade*). Marmalade in excess of needs was discarded. The yield factor from washed fruit to marmalade ranged from 0.71–1.1.

Oil processing

The remaining washed oranges (183–191 kg) were transferred to the modified Hobart Abrasive Peeler for scarifying. Approximately 2.42 kg (average) of oranges per batch were abraded with water for about 90 seconds to scarify the flavedo for oil recovery. The resulting oil–water emulsion was collected. The scarified fruit was weighed (191–213 kg, with weight gain of 8.3–22 kg) and an aliquot retained for juice processing (35–44 kg) and for peeling (4.5–5.1 kg). The remaining scarified fruit was discarded.

The collected oil-water emulsion from the scarification process was transferred to the Sweco Sifter and screened using a 94 TBC screen (-180 jim) to separate any flavedo fragments from the oil-water emulsion. The scarified flavedo (+94 TBC screen) was set aside for later addition to the shredded peel. The oil-water emulsion (-94 TBC screen) was processed through the cream separator and IEC centrifuge to separate the oil and placed in the freezer. The residual emulsion was frozen for a minimum of overnight. After removing the emulsion from the freezer, the sample was thawed, centrifuged, and the oil removed with a volumetric pipette. The entire sample of oil recovered (337–629 gram) was collected (sample: *cold extracted peel oil*). The yield factor from washed fruit to oil ranged from 0.0018–0.0034.

An aliquot of scarified oranges (4.5–5.1 kg) were peeled and representative samples (sample: *peel without oil*) were collected from the recovered peel (1.2–1.6 kg).

Juicing

An aliquot of the scarified oranges (35–44 kg) was transferred to the Hollymatic Juice Extractor to recover juice. The juice (10–15 kg, including a ca. 2 L lost to spillage in one sample) and peel recovered (2.3–3.0 kg) from the juice extraction were weighed and the peel set aside for further processing. The collected juice (10–15 kg) was transferred to the pulper finisher and screened using a -1.19 mm screen to remove vesicular membranes, seeds, segment membranes, and peel fragments from the juice. The recovered, screened juice was weighed and ° Brix taken on the fresh juice (7.5–14 kg (0.49–2.6 kg loss)). The collected rag and seeds (170–350 g) were set aside for later addition to the shredded peel. Representative samples of the fresh juice were taken (sample: *raw juice*). The remaining juice (6.5–13 kg) was pasteurized at 88 – 91 °C for a minimum of 15 seconds, cooled to less than 38 °C, and representative samples (sample: *pasteurized juice*) were taken from the pasteurized juice (6.2–12 kg- loss of 220–530 g). The remaining juice was discarded. The yield of washed fruit to raw juice ranged from 0.28–0.37 and the yield from washed fruit to pasteurized juice 0.27 to 0.35.

Wet pomace

The peel from the Hollymatic Juice Extractor (23–30 kg) was shredded using the Robot Coupe Food Processor. The shredded peel (23–29 kg) was combined with the scarified flavedo (0.17–4.3 kg) from the scarification process and rag and seeds (170–350 g) from the juice finisher process to generate the unpressed wet pomace (24–30 kg).

Wet pomace: A portion (10–14 kg) of the wet pomace was pressed to remove moisture (1.5–4.0 kg) using the Suntech Fruit Press, resulting in 6.6–10 kg pressed wet pomace), and representative samples were taken (sample: *wet pomace*). Calculated weight loss in pressing was 1.0–2.0 kg. The remaining pressed wet pomace (5.6–9.3 kg) was reserved for drying (dry pomace). The yield from RAC (washed oranges) to pressed wet pomace corrected for subsampling ranged from 0.42–0.53.

Limed wet pomace: The remaining unpressed wet pomace (12–16 kg) was used to generate limed wet pomace. Lime (-95 percent CaO) was added to the unpressed wet pomace and mixed on the Hobart mixer for 17 minutes. The limed wet pomace (12–16 kg) was pH tested, pressed using the Suntech Fruit Press resulting in 5.2–8.0 kg limed wet pomace, and the expressed liquid was weighed (6.3–8.6 kg), checked for pH, ° Brix, and discarded. The calculated weight loss during pressing was 0.40–0.77 kg. No sample was taken from the limed wet pomace product.

Dry pomace

Unlimed dry pomace: The unlimed wet pomace (5.6–9.3 kg) was placed on the Laboratory Bin Air Dryer and dried to below 10 percent moisture, resulting in a loss of 4.3–7.3 kg. The remaining dry pomace (1.2–2.0 kg) was milled using the Suntech Fruit Press hammermill resulting in 1.2–2.0 kg, with a loss of 20 g. Representative samples of the dry pomace were taken (sample: *dry pomace*). The remaining dry pomace was discarded. The yield of unlimed wet pomace to unlimed dry pomace ranged from 0.20 to 0.22. The yield from RAC (washed fruit) to unlimed dry pomace ranged from 0.094–0.11.

Limed dry pomace: The pressed, wet, limed pomace (5.2–8.0 kg) was placed on the Laboratory Bin Air Dryer and dried to below 10 percent moisture with a loss of 4.8–6.9 kg. The remaining dried pomace (1.4–2.2 kg) was milled using the Suntech Fruit Press hammermill resulting in 1.3–2.1 kg (loss of 10–30 g). Representative samples of the dry limed pomace were removed (sample: *limed dry pomace*). The remaining limed dry pomace was discarded. The yield of limed wet pomace to limed dry pomace ranged from 0.25 to 027. The yield form RAC (washed fruit) to limed dry pomace ranged from 0.84–0.97.

Figure 7 summarizes the citrus processing procedure.

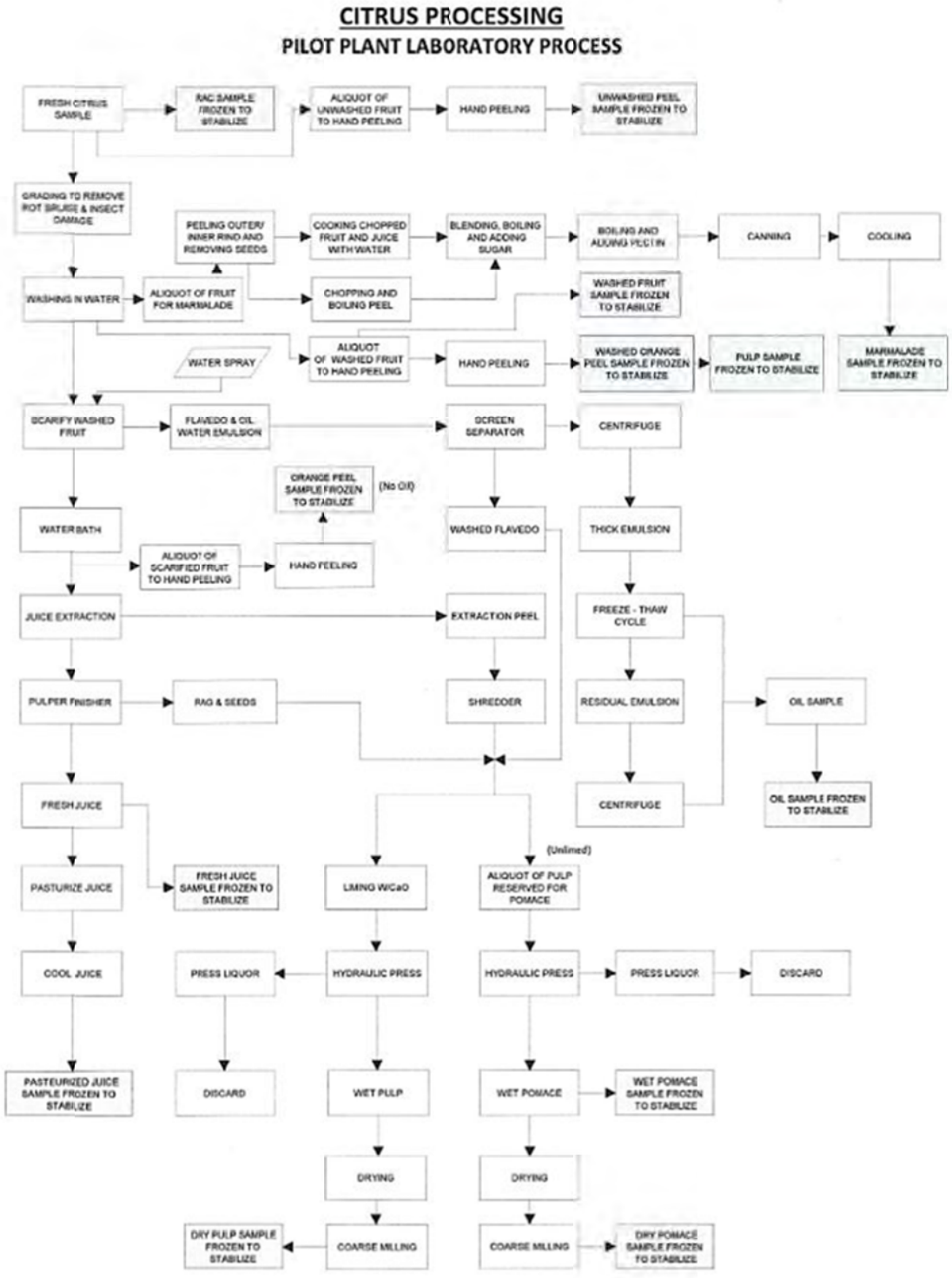


Figure 7 Citrus processing

Processing factors were derived for several orange processed commodities. Trial data, residue levels and processing factors for each type of commodity are summarised Table 116. Note that total residues represent parent + tetraniliprole-N-methyl-quinazolinone, unless levels of the metabolite were <LOQ of 0.01 mg/kg. This generally results in the same PFs, except for cold extracted peel and limed dry pomace in one trial, which is still very similar.

Table 116 Tetraniliprole residues and processing factors in orange whole fruit and processed commodities (Report RAFVN026)

Trial information	Sample	Residues (mg/kg) [a]			PF [b]	
		Tetraniliprole	T- N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
FV298-14PA, Oviedo, Florida United States, 2014 (variety: Valencia) 3 x ca. 300 g ai/ha (no ADJ) at BBCH 83-89, RTI = 4-5 days; DALA=1 day	Orange (RAC)	0.31	<0.01	0.31	-	-
	Washed whole fruit	0.17	<0.01	0.17	0.55	0.55
	Raw juice	<0.01	<0.01	<0.01	<0.03	<0.03
	Pasteurized juice	<0.01	<0.01	<0.01	<0.03	<0.03
	Marmalade	<0.01	<0.01	<0.01	<0.03	<0.03
	Wet peel without oil	0.11	<0.01	0.11	0.35	0.35
	Wet Peel	0.94	<0.01	0.94	3.0	3.0
	Washed peel	0.33	<0.01	0.33	1.1	1.1
	Cold extracted peel oil	3.4	0.56	3.96	11	12
	Flesh (peeled orange)	0.039	<0.01	0.039	0.13	0.13
	Wet pomace	0.075	<0.01	0.075	0.24	0.24
	Dry pomace	0.33	<0.01	0.33	1.1	1.1
	Limed dry pomace	0.38	0.025	0.41	1.2	1.3
FV299-14PC, Navelencia, California, United States, 2016 (variety: Valencia) 3 x ca. 300 g ai/ha (no ADJ) at BBCH 83-89, RTI= 4-5 days; DALA= 1 day	Orange (RAC)	0.47	<0.01	0.47	-	-
	Washed whole fruit	0.29	<0.01	0.29	0.62	0.62
	Raw juice	<0.01	<0.01	<0.01	<0.02	<0.02
	Pasteurized Juice	<0.01	<0.01	<0.01	<0.02	<0.02
	Marmalade	0.01	<0.01	0.01	0.021	0.021
	Washed peel	0.75	<0.01	0.75	1.6	1.6
	Wet Peel	1.20	<0.01	1.20	2.6	2.6
	Wet peel without oil	0.12	<0.01	0.12	0.26	0.26
	Cold pressed peel Oil	2.9	0.03	2.93	6.2	6.2
	Flesh, peeled orange	0.038	<0.01	0.038	0.081	0.081
	Wet pomace	0.073	<0.01	0.073	0.16	0.16
	Dry pomace	0.29	<0.01	0.29	0.62	0.62
	Limed dry pomace	0.45	<0.01	0.45	0.96	0.96

Notes:
DALA = Days After Last Application; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval.
[a] Expressed as parent tetraniliprole.
[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

Apples

Two field trials were conducted, in the United States, to measure the magnitude of tetraniliprole residues in/on apple processed commodities (Dallstream&Jerkins, 2016c, M-560638-01-1, Report RAFVP0640. Three foliar applications of tetraniliprole 200 SC at exaggerated rates (5×) were applied. Applications were made at actual rates of 296–307 g ai/ha, with application intervals of 7 days. Samples of apple whole fruit were collected 7 days after the final application at commercial maturity (BBCH 87–89). Apples were processed according to simulated commercial procedures into wet pomace, juice, washed fruit, peeled fruit, apple sauce, dried fruit, dried pomace, raw juice and peel. Throughout the study one representative sample of untreated control and two (duplicate) samples of treated processed commodities were taken. Figure 8 summarizes the apple processing procedure.

Washing

A batch of 70–93 kg apples was available. An aliquot of unwashed apples (4.9–7.2 kg) was reserved for peeling. Unwashed apples were peeled, resulting in 4.7–6.3 kg peeled apples and 0.84–1.3 kg unwashed peel. Representative samples of unwashed, peeled fruit (sample: *unwashed peeled fruit*) and fruit peel (sample: *unwashed fruit peel*) were taken. The remaining apples (60–83 kg) were washed in a stainless-steel wash cart using a ratio of ~2 kg of cold water to each 1 kg of fruit for 5 minutes. Representative samples were taken (sample: *wash water* and sample: *whole washed fruit*). Aliquots of washed apples were set aside for processing into apple sauce (12–13 kg), dried apple (18 kg), and apple juice (26–43 kg). The apples in excess were discarded.

Juicing

Washed apples for juice processing (26–43 kg) were fed into the Suntech Fruit Press hammermill assembly and reduced to crushed apple pulp (25–42 kg) with a loss to the process of 0.53–0.94 kg. The crushed apple pulp was transferred to the 35 L Swept Surface Steam Jacketed Kettle and heated with low-pressure steam until the temperature of the apple pulp reached 40–50 °C. 1.5 g of pectic enzyme per kg of apple pulp (38–62 g) was then added and mixed for approximately 2 minutes and permitted to react for approximately 2 hours, then pressed using the Suntech Fruit Press.

The collected wet pomace (5.5–10 kg) was analysed for moisture content. A representative wet pomace sample was taken (sample: *wet pomace*). The yield from washed fruit to wet pomace ranged from 0.20 to 0.24. The remaining wet pomace (4.4–9.2 kg) was placed on the Laboratory Bin Air Dryer and dried to below 10 percent moisture, resulting in a weight of dry pomace of 1.0–2.5 kg, with a calculated weight loss of 3.2–6.7 kg. The yield from wet pomace to dry pomace ranged from 0.23 to 0.27. The dry pomace was milled using the Suntech Fruit Press hammermill (1.0–2.4 kg remaining). A representative sample of the dry pomace was taken (sample: *dry pomace*).

The fresh juice recovered from the fruit press (18–29 kg) was filtered over a U.S. #40 screen to remove any coarse solids, resulting in 18–28 kg fresh/raw juice (yield = 0.66–0.70). A representative fresh/raw unpasteurized fresh juice sample taken (sample: *fresh/raw juice*). Part of the fresh raw juice was reserved for pasteurization (11–16 kg). The fresh raw juice in excess was discarded. The calculated loss to the pressing process was 1.6–3.2 kg and to the filtering process 0.04–0.07 kg.

An aliquot of raw fresh juice (11–16 kg) for the pasteurized juice fraction was then heated to approximately 93° C for 15–30 seconds to deactivate the pectic enzymes and then cooled to approximately 30 °C, resulting in a weight of 9.9–15 kg, due to a loss by deactivation process of 0.54–0.74 kg. The unfiltered pasteurized apple juice was placed in refrigerated storage overnight to permit settling of solids. The juice was racked off, resulting in 2.8–9.6 kg juice and settled solids were discarded.

Part of the clear juice (7.54 kg) was discarded (trial FV001 only). Another part of the clear juice (1.3–3.6 kg) was then vacuum filtered using a vacuum pump, vacuum flasks and Büchner vacuum funnels. Diatomaceous earth and filter paper were used as filtering aids. After filtering, the juice (1.1–3.3 kg) was heated again to 93 ± 3 °C for approximately 30 seconds, packed in sterilized cans, and sealed. The cans were inverted for sterilization of the lids and the cans cooled in a cold-water bath. Pasteurized juice weighed 1.0–3.2 kg. The yield of pasteurized juice from washed fruit, corrected for sub-fractionation ranged from 0.18 to 0.41. Samples were taken (sample: *pasteurized juice*). The remaining pasteurized juice was discarded.

Apple sauce

The aliquot of washed apples reserved for apple sauce processing (12–13 kg) were peeled, cored, and sliced. The peel and cores removed were weighed (3.2–5.2 kg) and discarded. The peeled/cored/sliced apples (7.1–9.4 kg) were placed in cold water containing salt to prevent enzymatic browning. The sliced apples were then cooked with water at ~85–95 °C, until completely soft and weighing 8.5–12 kg. The cooked apples were then strained through the pulper finisher and separated into raw apple sauce (6.3–10 kg) and apple fiber (strain rest) weighing 0.58–1.1 kg (loss through the process of 0.70–1.7 kg). The strain rest was weighed and discarded and sugar (0.90–1.6 kg) was added to the apple sauce until a minimum of 18° Brix was achieved. The apple sauce (7.2–12 kg) was then reheated to a minimum of 90 °C and a final Brix taken before pasteurized sauce (6.6–12 kg) is packed in cans and sealed. Loss due to the process of pasteurization and canning is 0.29–0.71 kg. The cans were processed for 10 minutes in boiling water and cooled in cold water. A representative sample of the pasteurized, canned apple sauce was taken (sample: *apple sauce*). The yield of washed apple to pasteurized apple sauce ranged from 0.53 to 0.95.

Drying of apples

The washed aliquot of apples (18 kg) reserved for dried apple processing, were peeled, cored and spiral sliced to ~0.64 cm thickness (11–13 kg). The peel and cores removed were weighed (5.4–7.6 kg) and discarded. A calculated weight loss due to the process of peeling slicing and coring is 0–0.39 kg. The peeled/cored/sliced apples (11–13 kg) were placed in cold water containing salt to prevent enzymatic browning. The sliced apples are then diced using a potato French fry strip culler producing a 0.64 cm square apple piece. The diced apples are then dipped for 5 minutes in a prepared solution of 0.5 percent of potassium meta-bisulfite and 0.2 percent citric acid. The diced apples are evenly divided into three trays and the trays are placed in the tray dryer. The calculated weight loss in dicing and transfer to dryer is 0.0–1.1 kg. The trays are periodically rotated in the dryer and the diced apples stirred and weighed until a target moisture of ~2.5 percent moisture is obtained, resulting 1.0–1.4 kg of dried apple slices, with a calculated weight loss due to drying of 8.6–11.4 kg (corresponding with 88–90 percent). A representative sample of the dried apple was taken (sample: *dried apple*). The yield of washed apple to dried apple slices ranged from 0.055–0.077. Figure 7 shows the processing procedure for apple

Samples were stored frozen (<-18 °C) for a maximum of 259 days prior to analysis. All samples were analysed for residues of tetraniliprole and tetraniliprole-N-methylquinazolinone using analytical method 01414.

Trial data, residue levels and processing factors for each type of commodity are summarised in Table 117. The concentration of residues observed in pomace (wet) and peel suggest that the residue is present on the peel and the PF is due to fractionation. The higher residues in dry pomace correspond with concentration due to the loss on drying.

Note that total residues represent parent + tetraniliprole-N-methyl-quinazolinone, unless levels of the metabolite were <LOQ of 0.01 mg/kg. This generally results in the same PFs, except for the washings in one trial, but still with a similar PF.

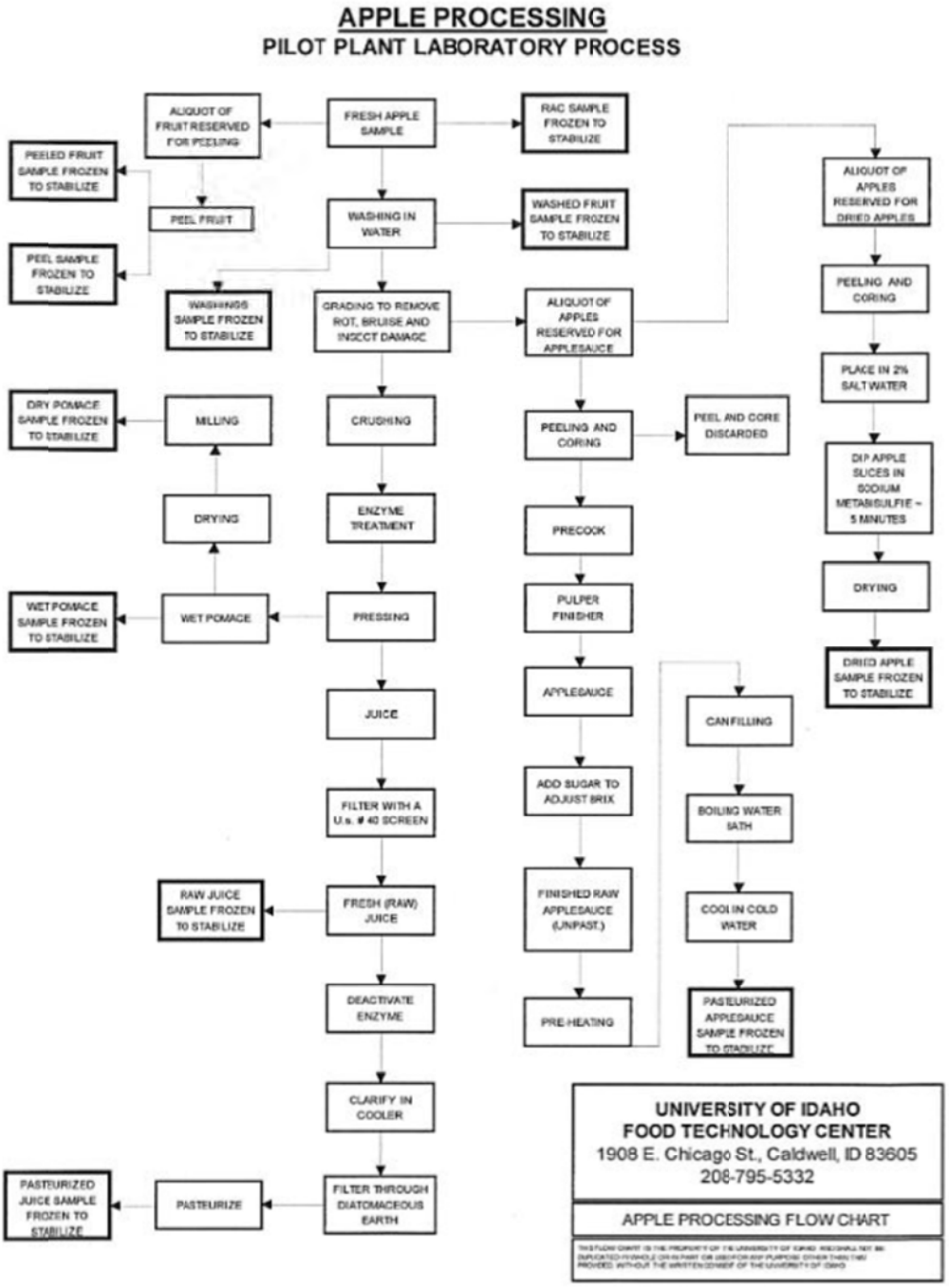


Figure 8 Apple processing

Table 117 Tetraniliprole residues and processing factors in apple whole fruit and processed commodities (Report RAFVP064)

Trial information	Sample	Residues (mean) (mg/kg) [a]			PF [b]	
		Tetraniliprole	T- N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
FV001-15PA, Jefferson, Illinois, United States, 2015	Apple (RAC)	0.59, 0.49, 0.54 (0.54)	3 x <0.01 (<0.01)	0.54	-	-
	Washed whole fruit	0.41, 0.40, 0.52	3 x <0.01 (<0.01)	0.44	0.83	0.83

Trial information	Sample	Residues (mean) (mg/kg) [a]			PF [b]	
		Tetraniliprole	T- N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
(variety: Jonathan Apple) 3 x ca. 300 g ai/ha (no ADJ) at BBCH 77-85 (18 Aug.), RTI = 7 days; DALA=7 days		(0.44)				
	Raw juice	0.37	<0.01	0.37	0.69	0.69
	Pasteurized Juice	0.34	<0.01	0.34	0.62	0.62
	Washings	0.041	<0.01	0.041	0.076	0.076
	Peeled fruit	0.041	<0.01	0.041	0.077	0.077
	Peel	3.9	<0.01	3.9	7.3	7.3
	Apple sauce	<0.01	<0.01	<0.01	<0.019	<0.019
	Dried fruit	0.017	<0.01	0.017	0.031	0.031
	Wet pomace	0.98	<0.01	0.98	1.8	1.8
	Dry pomace	3.7	0.031	3.7	6.8	6.9
FV002-15PA, Payette, Idaho, United States, 2015 (variety: Early Spur Rome) 3 x ca. 300 g ai/ha (no ADJ) at BBCH 81-85 (29 Sept.), RTI= 7 days; DALA= 7 days	Apple (RAC)	0.43, 0.20, 0.36 (0.33)	3 x <0.01 (<0.01)	0.33	-	-
	Washed whole fruit	0.29, 0.18, 0.21 (0.23)	3 x <0.01 (<0.01)	0.23	0.70	0.70
	Raw juice	0.10	<0.01	0.10	0.31	0.31
	Pasteurized Juice	0.082	<0.01	0.082	0.25	0.25
	Washings	0.050	0.024	0.074	0.15	0.22
	Peeled fruit	0.099	<0.01	0.099	0.30	0.30
	Peel	4.8	<0.01	4.8	15	15
	Apple sauce	<0.01	<0.01	<0.01	<0.030	<0.030
	Dried fruit	0.012	<0.01	0.012	0.036	0.036
	Wet pomace	0.53	<0.01	0.53	1.6	1.6
	Dry pomace	2.4	<0.01	2.4	7.3	7.3

Notes:
DALA = Days After Last Application; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval; T-N-MQZ = tetraniliprole-N-methylquinazolinone.
[a] Expressed as parent tetraniliprole.
[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

Plums and prunes

Two trials on plums with exaggerated rates (5×) were conducted (Greenland, 2016c, M-572124-01-1 Report SARS-14-01) to provide samples for processing to measure the magnitude of tetraniliprole residues in/on plum and prunes. Three foliar air-blast applications of a tetraniliprole 200 SC formulation were made between growth stage BBCH 74–89 with retreatment intervals of approximately 7 days and samples collected 5 days after the last application. A minimum of 11.8 kg per sample of plums were collected at normal harvest for processing into prunes. The control and treated samples were processed on the day of harvest and the processing procedures were identical for the untreated and treated samples. The fresh plum samples were weighed and arranged on the drying trays of the dehydrator. Plums were dried for one or two days at 54–57 °C. Actual sample weights to establish material balance were not reported. According to the Standing Operating Procedures in the report, the samples had to be dried until they weighed 1/3 of the original weight.

Samples were stored frozen for a maximum of 267 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

Trial data, residue levels and processing factors for each type of commodity are summarized Table 118. Processing factors were derived for prunes, where a concentration of residues was observed, consistent with a concentration due to weight loss by drying.

Note that total residues represent parent + tetraniliprole-N-methyl-quinazolinone, unless levels of the metabolite were <LOQ of 0.01 mg/kg. This results either in the same or a similar PF.

Table 118 Residues of tetraniliprole and processing factors in plums/prunes [Report SARS-14-01]

Trial information	Sample	Residues (mean) (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	total	PF_{parent}	PF_{total}
SARS-14-01-BC-3, Fraser Valley, British Columbia Canada, 2015 (Variety: PRM1 grafter on Moyer) 3 x foliar treatment; 299-308 g ai/ha, RTI = 9 & 5 days; adjuvant (NIS), DALA = 5 days	Plum (RAC)	0.37, 0.45 (0.41)	<0.01, <0.01 (<0.01)	0.37, 0.45 (0.41)	-	-
	Prune	2.0, 1.7 (1.9)	<0.01, <0.01 (<0.01)	2.0, 1.7 (1.9)	4.6	4.6
SARS-14-01-WA1-3, Grant, Washington, United States, 2014 (Italian Plum) 3 x foliar treatment; 297-300 g ai/ha, RTI=7 days; no adjuvant added; DALA= 5 days	Plum (RAC)	0.073, 0.13 (0.10)	<0.01, 0.021 (0.015)	0.073, 0.15 (0.12)	-	-
	Prune	0.35, 0.36 (0.36)	<0.01, <0.01 (<0.01)	0.35, 0.36 (0.36)	3.6	3.6

Notes:
DALA = Days After Last Application; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval.
[a] Expressed as parent tetraniliprole.
[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

Grapes

Two field trials (one with white grapes and one withe red grapes) were conducted in the United States to provide samples for processing (Greenland, 2016d, M-572121-01-1, Report SARS-14-7). Applications were made at an exaggerated (5×) rate of 225-229 g ai/ha, with one exception of 471 g ai/ha. Applications were made with intervals of 7 days. A minimum of 23 kg per sample of grapes were collected at normal commercial harvest 14 days after the last application for processing into juice or raisins according to simulated commercial procedures. Throughout the study one representative sample of untreated control and two (duplicate) samples of treated processed commodities were taken.

Whole grapes from random bunches were carefully destemmed by hand and packed one layer deep on dehydrator trays which were placed in the dehydrator. Grapes were dehydrated for about 26 hours at 54 to 60 °C to achieve a moisture range between 15–18 percent and samples were collected (sample: *raisin*).

The remaining grapes were passed through a Crusher/Destemmer to remove stems and produce grape mash. The mash was layered into cloth stacks on a hydraulic press and pressed to separate the juice from the wet pomace and samples were collected (sample: *juice, raw*).

According to the protocol for processing small lots of grapes weighing approximately 44 kg are used as starting point, of which 2.3 kg is subtracted before processing. Typically, 6.8–7.7 kg destemmed grapes are used to produce raisins (typically 1.1–1.4 kg), 40-45 percent is raw juice (typically 14–16 kg), 35–40 percent (typically 10–12 kg) is wet pomace, 1–3 percent is grape stem loss and 11–20 percent is

operation loss. Figure 9 shows the processing procedure from grape to juice, wet pomace, raisin and dry pomace.

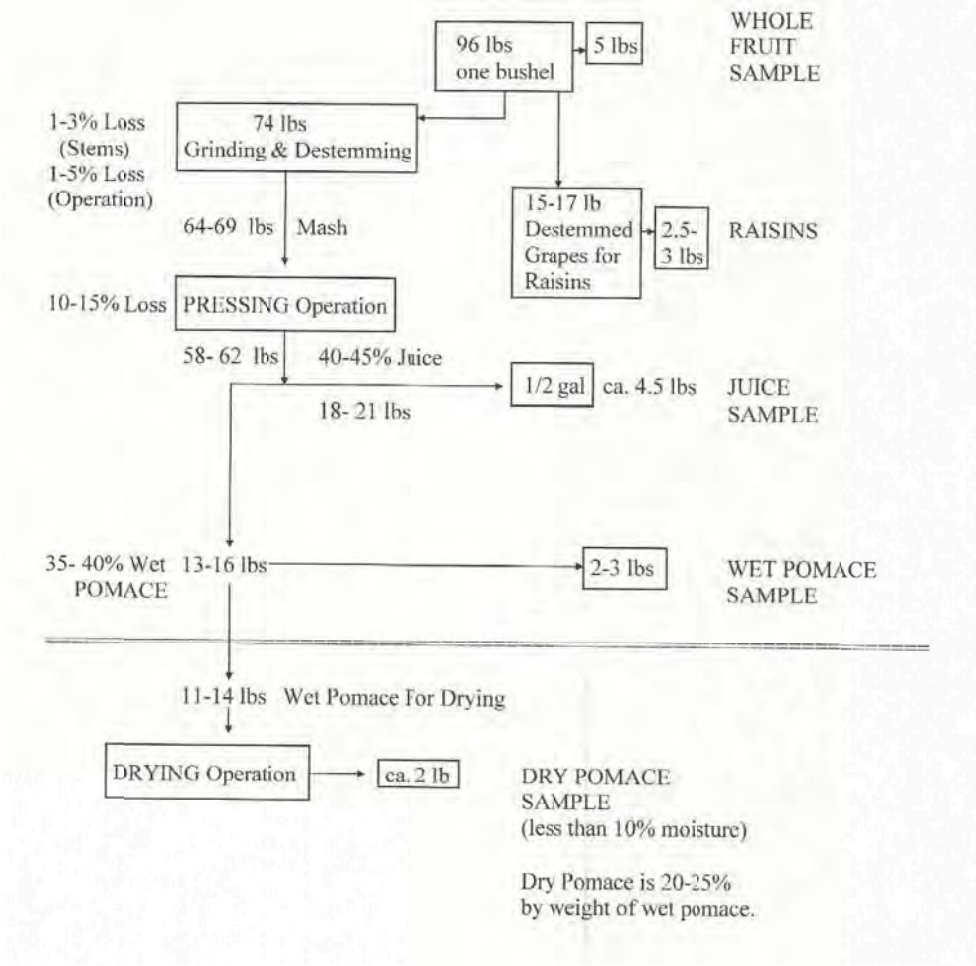


Figure 9 Grape processing flow chart

Samples were stored frozen for a maximum of 212 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414 with a LOQ of 0.01 mg/kg for both analytes. The concurrent recoveries were within the acceptable range of 70–120 percent.

Trial data, residue levels and processing factors for each type of commodity are summarised Table 119.

Table 119 Residues of tetraniliprole and processing factors in grape and processed commodities (Report SARS-14-7)

Trial information	Sample	Residues (mean) (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
SARS-14-07-CA8-3, Fresno, California, United States, 2014 (Thompson eedless)	Grape (RAC)	1.0, 0.56 (0.78)	<0.01, <0.01 (<0.01)	1.0, 0.56 (0.78)	-	-
	Raw Juice	0.25, 0.24 (0.24)	<0.01, <0.01 (<0.01)	0.25, 0.24 (0.24)	0.31	0.31

Trial information	Sample	Residues (mean) (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
4 x foliar treatment; 226-471 g ai/ha; RTI = days; DALA = 14 days	Raisin	0.65, 0.80 (0.72)	0.013, 0.013 (0.013)	0.66, 0.81 (0.73)	0.92	0.94
SARS-14-07-NY-3, Yates, New York, United States, 2014 (DeChaunac) 4 x foliar treatment; 225-226 g ai/ha; RTI = days; DALA = 14 days	Grape (RAC)	2.6, 1.4 (2.0)	<0.01, <0.01 (<0.01)	2.6, 1.4 (2.0)	-	-
	Raw Juice	0.14, 0.39 (0.27)	<0.01, <0.01 (<0.01)	0.14, 0.39 (0.27)	0.14	0.14
	Raisin	2.8, 3.3 (3.1)	0.078, 0.058 (0.068)	2.9, 3.4 (3.2)	1.6	1.6

Notes:
DALA = Days After Last Application; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval; T-N-MQZ = tetraniliprole-N-methylquinazolinone.
[a] Expressed as parent tetraniliprole.
[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value in the processed commodity is <LOQ, a value of 0.01 or 0.02 mg/kg has been used for calculation of a PF Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

In a second study (Freitag & Hoffmeister, 2017, M-577324-01-1, Report 14-03404), four processing trials (two with red grapes and two with white grapes) were conducted throughout Europe to measure the magnitude of tetraniliprole and tetraniliprole-N-methylquinazolinone in grapes and processed grape commodities following one foliar application of tetraniliprole 200 SC. Applications were made at actual rates of 200 g ai/ha at BBCH 85 and samples of grapes were collected approximately 30 days after application (BBCH 89). Grapes were processed according to simulated commercial procedures into juice, pomace and wine.

Juicing of red grapes (trials 14-3404-02 and 14-3404-04)

Deep-frozen bunches of grapes (21–24 kg) were weighed in and destemmed into berries (20–23 kg) and stalks and stems. Unripe, damaged or rot fruits were sorted out, if necessary. The berries were washed (water rate of 2 kg/kg berries), resulting in 20–21 kg and a sample taken (sample: *washed berries*). The remaining sample of washed berries (19–20 kg) was shredded in a cutter into mash. To obtain a good yield of colour in the juice and for enzyming the mash was heated up to approx. 50 °C and then mixed with the pectolytic enzyme product. The residence time took place 2 hours. For trial 02 the enzymed mash was stored overnight at room-temperature and for 04-T the enzyme mash was pressed directly. The enzyme mash (18–19 kg) was pressed in a high-pressure-press into raw juice (15–16 kg) and wet pomace (2.7–3.2 kg) and samples were taken (sample: *raw juice* and sample: *wet pomace*). The yield from RAC to wet pomace corrected for fractionation ranged from 0.12–0.16 and from RAC to raw juice 0.72–0.76. The remaining sample of wet pomace (1.7–2.2 kg) was dried in a convection oven at a temperature of 100 °C for 2.0–3.0 hours until a water content <10 percent (02-T = 6.94 percent; 04-T = 3.15 percent) was reached, resulting in 0.67–0.94 kg dry pomace (yield from wet pomace to dry pomace = 0.40–0.44) and a sample was taken (sample: *dry pomace*). The fining of the remaining raw juice started with the enzymation using a pectolytic-enzyme. First the raw juice (13–15 kg) was heated up to approximately 50 °C and mixed with the enzyme product. The reaction time took place for 1 hour and further left to stand overnight at room temperature for settling down the lees particles. On the next day the enzymed raw juice was decanted, centrifuged and filtered to obtain clear juice and retentate. Clarified juice was pasteurized (77–78 °C) in a plate-heat-exchanger, Brix-values measured, and a sample taken (sample: *juice,*

pasteurized) from the resulting 6.9–10 kg pasteurized juice. After correction for fractionation, the yield from RAC to pasteurized juice ranged from 0.39 to 0.49.

Juicing of white grapes (trials 14-3404-01 and 14-3404-03)

Deep-frozen bunches of grapes were weighed in (20–22 kg) and destemmed into berries (19–22 kg) and stalks and stems. Unripe, damaged or rot fruits were sorted out, if necessary. The berries were washed (water rate of 2 kg/kg berries), resulting in 17–21 kg, and a sample taken (sample: *washed berries*). The remaining sample of the washed berries (16–20 kg) was shredded in a cutter into mash. In contrast to the red grape juice no heating and enzymation took place before pressing. The mash (15–19 kg) was pressed in a high-pressure-press into raw juice (12–15 kg) and wet pomace (2.4–4.1 kg) and samples were taken (sample: *juice, raw* and sample: *wet pomace* (one trial only)). The yield from RAC to wet pomace corrected for fractionation ranged from 0.12–0.19 and from RAC to raw juice 0.63–0.70. The remaining sample of wet pomace (2.2–3.1 kg) was dried in a convection oven at a temperature of 100 °C for 2.5–4.5 hours until a water content <10 percent (01-T = 6.48 percent, 03-T = 3.79 percent) was reached, resulting in 0.83–0.91 kg dry pomace (yield from wet pomace to dry pomace = 0.29-0.38), and a sample was taken (sample: *dry pomace*). The fining of the remaining raw juice started with the enzymation using a pectolytic-enzyme. First the raw juice (11–14 kg) was heated up to approximately 50° and mixed with the enzyme product. The reaction time took place for 1 hour and further left to stand overnight at room temperature for settling down the lees particles. On the next day the enzymed raw juice was decanted, centrifuged and filtered to obtain clear juice and retentate. Clarified juice was pasteurized (~78 °C) in a plate-heat-exchanger, Brix-values measured, resulting in 5.2–8.9 kg pasteurized juice, and a sample was taken (sample: *juice, pasteurized*). After correction for fractionation, the yield from RAC to pasteurized juice ranged from 0.30 to 0.45. The processing procedure is shown in Figure 10.

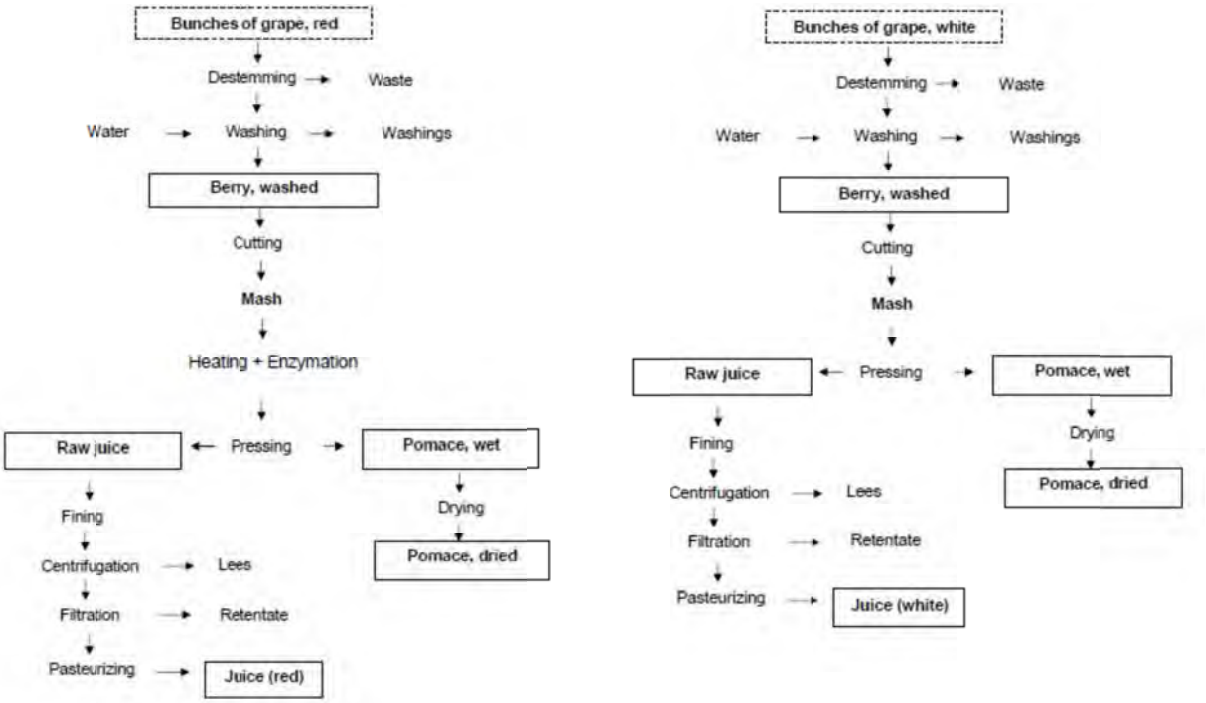


Figure 10 Grape juice production processes

Processing of red grape into must (trials 14-3404-02 and 14-3404-04)

Bunches of frozen grapes were defrosted at ~13 °C for processing into must. The defrosted red grapes (62–63 kg) were destemmed and milled into mash in a screw-extruder. The obtained mash of grapes was filled into the heating machine and heated up to approx. 80 °C for about 2 minutes under constant stirring. Afterwards the mash was cooled down. The mash was pressed in a wine press (vinification plant) to obtain must and pomace (apparatus: Trester). After pressing, it was not necessary to add sugar to the must. To prevent any oxidation processes, the must was treated with potassium disulfide (ratio: 100 mg/L). Furthermore, to prevent any protein diffusion, bentonite was added to the must (ratio: 1 g/L). For the following clarification the must was left to stand for approx. 12–15 hours. After the settling time, the preclarified must was decanted, weighed (43–54 kg), and sampled (sample: *must*).

Processing of grapes into red wine (trials 14-3404-02 and 14-3404-04)

Samples of the preclarified must (22–23 kg) were filled into fermentation vessels and yeast was added (ratio: 20 g/100 L). The following fermentation occurred in absence of air and the disappeared carbon dioxide was measured until the process was finished. For first racking the clear young wine was decanted from the wine lees and was then treated with potassium disulfide (ratio: 30 mg/L). After approximately 2 weeks the second racking followed. The young wine was decanted a second time and treated with potassium disulfide one more time (ratio: 35–40 mg/L) after which the young wine was filtered using a disinfection-filter, weighed (14 kg), and sampled (sample: *first red wine at bottling*). The remaining of the young wine was filled into bottles and stored at approx. 12 °C for approx. 6–7 months. After this maturation time this wine was sampled (sample: red *wine at first taste*). The yield from RAC to young wine is 0.42–0.51. The procedure is shown in Figure 11.

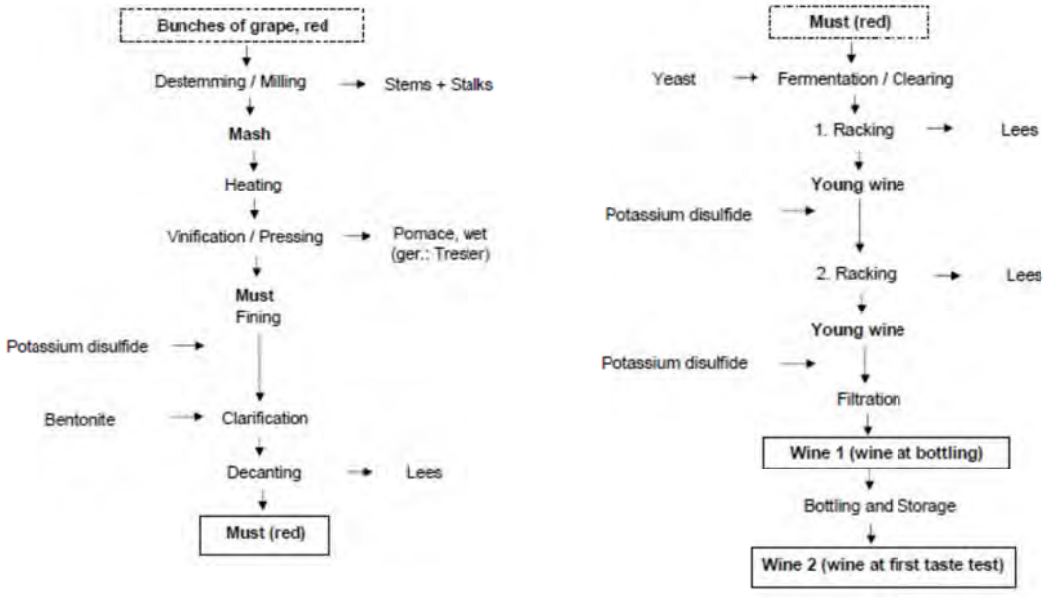


Figure 11 Processing steps in red wine production

Processing of white grape into must (trials 14-3404-01 and 14-3404-03)

Bunches of fresh grapes (14-3404-01) or frozen grapes (14-3404-03) were used for processing into must. The fresh or defrosted white grapes (75.26/72.58 kg) were milled into mash in a screw-extruder without destemming. In contrast to the process with red grapes no heating took place before the vinification. The mash was pressed in a wine press (vinification plant) to obtain must and pomace (apparatus Trester). After pressing the must weight was measured, it was not necessary to add sugar to the must. To prevent any oxidation processes, the must was treated with potassium disulfide (ratio: 100 mg/L). Furthermore, to prevent any protein diffusion, bentonite was added to the must (ratio: 1 g/L). For the following clarification the treated must were left to stand for approx. 12–15 hours. After the settling time, the preclarified must was decanted, weighed (47.94/54.34 kg) and a sample was taken (sample: *must*).

Processing of grapes into white wine (trials 14-3404-01 and 14-3404-03)

An aliquot of the preclarified must (20.0/22.88 kg) was filled into fermentation vessels and yeast was added (ratio: 20 g/100 L). The following fermentation occurred in absence of air and the disappeared carbon dioxide was measured until the process was finished. The fermentation started for 14-3404-01 on 2014-09-25 and was finished on 2014-10-06 (11 days). The fermentation for 14-3404-03 started on 2014-11-28 and was finished on 2014-012-22 (24 days). For first racking the clear young wine was decanted from the wine lees and was then treated with potassium disulfide (ratio: 50 mg/L for 14-3404-01 and 30 mg/L for 14-3404-03). After approx. 2 weeks the second racking followed, though only for trial 14-3404-03. The young wine was decanted a second time and treated with potassium disulfide one more time (ratio: 45 mg/L) after which the young wine was filtered using a disinfection-filter, weighed (15.14/17.40 kg), and sampled (sample: *first white wine at bottling*). The remaining of the young wine was filled into bottles and stored at approx. 12 °C for approx. 6–7 months. After this maturation time this wine was sampled (sample: *white wine at first taste*). The yield from RAC to young red wine is 0.43–0.57.

Samples were stored frozen (< -18 °C) for a maximum of 411 days prior to analysis. This is within the demonstrated storage stability period for tetraniliprole and tetraniliprole-N-methylquinazolinone residues in high acid content commodities (see section on storage stability). All samples were analysed for residues of tetraniliprole and tetraniliprole-N-methylquinazolinone using the validated analytical method 01414.

Processing factors were derived for grape processed commodities. A concentration of residues was observed in pomace (wet and dry), consistent with a concentration due to weight loss by pressing and drying. Residues were diluted in all other commodities. Trial data, residue levels and processing factors for each type of commodity are summarised Table 120.

Note that total residues represent parent + tetraniliprole-N-methyl-quinazolinone, unless levels of the metabolite were <LOQ of 0.01 mg/kg. This generally results in the same or very similar PFs.

Table 120 Residues of tetraniliprole and processing factors in grape and processed commodities (Report SARS-14-7)

Trial information	Sample	Residues (mean) (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
14-3404-01-1, Maikammer, Germany, 2014 (Weißburgunder White) 1 x foliar spray at	Grape, white (RAC)	0.42, 0.36 (0.39)	<0.01, <0.01 (<0.01)	0.42, 0.36 (0.39)	-	-
	Berries washed	0.28	<0.01	0.28	0.72	0.72
	Juice, raw	0.068	<0.01	0.068	0.17	0.17
	Juice, pasteurized	<0.01	<0.01	<0.01	<0.026	<0.026
	Must	0.085	<0.01	0.085	0.22	0.22

Trial information	Sample	Residues (mean) (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
200 g ai/ha, BBCH 85 (25 Aug.), DALA = 30 days	Wine at bottling	0.065	<0.01	0.065	0.17	0.17
	Wine at first taste	0.080	<0.01	0.080	0.21	0.21
	Wet pomace	0.97	<0.01	0.97	2.5	2.5
	Dry pomace	1.9	0.05	1.95	4.9	5.0
14-3404-03-1, Cerveteri (RM), Italy, 2014 (Trebbiano White) 1 x foliar spray at 200 g ai/ha, BBCH 81 (20 Aug.), DALA = 30 days	Grape, white (RAC)	0.066, 0.055 (0.061)	<0.01, <0.01 (<0.01)	0.066, 0.055 (0.061)	-	-
	Berries washed	0.035	<0.01	0.035	0.55	0.55
	Juice, raw	0.016	<0.01	0.016	0.25	0.25
	Juice, pasteurized	<0.01	<0.01	<0.01	<0.16	<0.16
	Must	0.04	<0.01	0.04	0.62	0.62
	Wine at bottling	0.025	<0.01	0.025	0.39	0.39
	Wine at first taste	0.017	<0.01	0.017	0.27	0.27
	Wet pomace	0.14	<0.01	0.14	2.2	2.2
	Dry pomace	0.49	0.014	0.50	7.7	8.2
14-3404-02-1, Saint Nicolas de Bourgueil, France, 2014 (Cabernet Franc Red) 1 x foliar spray at 200 g ai/ha, BBCH 85 (25 Aug.), DALT = 30 days	Grape, red (RAC)	0.33, 0.32 (0.33	<0.01, <0.01 (<0.01)	0.33, 0.32 (0.33)	-	-
	Berries washed	0.16	<0.01	0.16	0.48	0.48
	Juice, raw	0.078	<0.01	0.078	0.24	0.24
	Juice, pasteurized	0.026	<0.01	0.026	0.079	0.079
	Must	0.19	<0.01	0.19	0.58	0.58
	Wine at bottling	0.15	<0.01	0.15	0.45	0.45
	Wine at first taste	0.10	<0.01	0.10	0.30	0.30
	Wet pomace	0.68	<0.01	0.68	2.1	2.1
	Dry pomace	1.0	0.02	1.0	3.0	3.0
14-3404-03-1, Piera, Spain, 2014 (Tempranillo Red) 1 x foliar spray at 200 g ai/ha, BBCH 83 (31 July), DALT = 30 days	Grape, red (RAC)	0.30, 0.26 (0.28)	<0.01, <0.01 (<0.01)	0.30, 0.26 (0.28)	-	-
	Berries washed	0.17	<0.01	0.17	0.61	0.61
	Juice, raw	0.10	<0.01	0.10	0.36	0.36
	Juice, pasteurized	0.021	<0.01	0.021	0.075	0.075
	Must	0.24	<0.01	0.24	0.86	0.86
	Wine at bottling	0.16	<0.01	0.16	0.57	0.57
	Wine at first taste	0.15	<0.01	0.15	0.54	0.54
	Wet pomace	0.77	<0.01	0.77	2.8	2.8
	Dry pomace	2.2	0.031	2.2	7.9	7.9

Notes:
DALA = Days After Last Application; RAC = Raw Agricultural Commodity; RTI = Re-treatment Interval.
[a] Expressed as parent tetraniliprole.
[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value in the processed commodity is <LOQ, a value of 0.01 or 0.02 mg/kg has been used for calculation of a PF. Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

Broccoli

One field trial was conducted to provide samples to determine the effect of washing or washing and cooking on tetraniliprole residues in/on broccoli treated with four applications of a tetraniliprole 200 SC formulation (Netzband&Roberts, 2016, M-565724-02-1, Report RAFVP096-01). Applications were made at actual rates of 45–46 g ai/ha, with application intervals of 4–5 days, without addition of an adjuvant.

Samples of stalks/curd were collected at maturity, nominally 1-day after the final application and consisted of a composite of 12 plants, weighing at least 1 kg. Throughout the study one representative sample of untreated control and two (duplicate) samples of treated processed commodities were taken.

The washing and cooking procedures were performed in a manner similar to normal household practice. For all broccoli samples (RAC, washed, and washed and cooked), the leaves, withered florets, and any woody part of the stem were removed and discarded (sample: *broccoli, head and stem*). The washed subsample was rinsed under lukewarm to cool running tap water for approximately 30 seconds to ensure that all portions of the sample were rinsed. Excessive water was shaken off and the washed subsample was allowed to drain on paper towels for at least 2 minutes (sample: *broccoli, washed*). After draining, the broccoli head with stem was halved lengthwise. The lengthwise cuts were placed in a pan containing a small amount of boiling water and cooked, covered, for 10 minutes. Cooked broccoli was drained and then allowed to cool to room temperature (sample: *broccoli, washed and cooked*). Actual weight data from RAC, washed and cooked broccoli were not reported. Yield factors could not be calculated.

Samples were stored frozen for a maximum of 633 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414 (LOQ of 0.01 mg/kg for both analytes in all commodities). The concurrent recoveries were within the acceptable range of 70–120 percent. Trial data, residue levels and processing factors for each type of commodity are summarised Table 121.

Table 121 Residues of tetraniliprole and processing factors in raw and cooked broccoli (Report RAFVP096-01)

Trial information	Sample	Residues (mean) (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
RAFVP096-01 FV266-14HA, Santa Maria, California, United States, 2014 (Heritage) 4 x foliar treatment; 45-46 g ai/ha, RTI = 5 days, no adjuvant added, DALT = 1 day	Broccoli (RAC)	0.17, 0.11 (0.14)	<0.01, <0.01 (<0.01)	0.17, 0.11 (0.14)	-	-
	Broccoli head and stem (preprocessing)	0.24	<0.01	0.24	-	-
	Broccoli, washed	0.18	<0.01	0.18	1.3	1.3
	Broccoli washed and cooked	0.048	0.020	0.068	0.34	0.49

Notes:
DALA = Days After Last Application; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval.
[a] Expressed as parent tetraniliprole.
[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg).
Where the value in the processed commodity is <LOQ, a value of 0.01 or 0.02 mg/kg has been used for calculation of a PF.
Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF.
Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

Tomato

Two field trials were conducted in the United States to provide samples for processing of tomatoes to paste and puree (Greenland, 2016e, M-572627-01-1, Report SARS-14-19). Applications were made at an exaggerated (5×) rate of 225–229 g ai/ha, with one exception of 471 g ai/ha. Applications were made with intervals of 5 days. A minimum of 23.8 kg per sample of tomatoes were collected at normal commercial harvest for processing into paste or puree. Throughout the study one representative sample of untreated control and two (duplicate) samples of treated processed commodities were taken.

The fresh tomato fruits were washed by placing the fruit in wire baskets and submerging three times in water. The water was changed and this washing process was repeated. The wash water was discarded. The washed tomatoes were quartered and pushed through an electric food strainer/sauce maker to produce raw/fresh puree. One representative sample of untreated control puree and two (duplicate) samples of treated puree were collected, packaged, labelled and frozen (sample: *tomato puree, unpasteurized*). The remaining puree was collected in a kettle and heated between 82 and 93 °C under occasional stirring for 6-10 hours to produce paste. One representative sample of untreated control paste and two (duplicate) samples of treated paste were collected, packaged, labelled and frozen (sample: *tomato paste*). Excess tomato paste was discarded. A bulk of treated tomatoes was received (23.8/24.5 kg). After sampling (2.0 and 2.3/2 × 3.2 kg) a total of 19.5/18.1 kg of treated tomatoes were used to produce 11/7.3 kg of puree and 2.6/1.6 kg of paste. Duplicate samples of puree (1.13/1.02 kg) and of paste (1.24/0.79 kg) were taken. Yield factors from fruit to puree ranged from 0.40–0.56 and from 0.16 to 0.17 for fruit to paste.

Samples were stored frozen for a maximum of 299 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414, with a LOQ of 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

Trial data, residue levels and processing factors for each type of commodity are summarised Table 122. Tetraniliprole residues were found to increase in paste, but less than expected based on weight loss. Overall the data suggest a degradation of the residue due to processing.

Table 122 Residues of tetraniliprole and processing factors in tomato fruit, paste and puree (Report SARS-14-19)

Trial information	Sample	Residues (mean) (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
SARS-14-19-GA-3 Tift, Georgia, United States, 2014 (BHN 602) 4 x foliar treatment; 222-231 g ai/ha, RTI = 5 days; Adjuvant COC added; DALA = 1 day	Tomato (RAC)	0.39, 0.35 (0.37)	<0.01, <0.01 (<0.01)	0.39, 0.35 (0.37)	-	-
	Paste	0.78, 0.65 (0.72)	0.68, 0.63 (0.66)	1.46, 1.28 (1.38)	1.9	3.7
	Raw/fresh Puree [c]	0.50, 0.35 (0.42)	<0.01, <0.01 (<0.01)	0.50, 0.35 (0.42)	1.1	1.1
SARS-14-19-NY-3 Wayne, New York United States, 2014 (Mountain fresh) 4 x foliar treatment; 222-231 g ai/ha, RTI = 5 days; no adjuvant added; DALA = 1 day	Tomato (RAC)	0.31, 0.46 (0.39)	<0.01, <0.01 (<0.01)	0.31, 0.46 (0.39)	-	-
	Paste	2.2, 1.7 (2.0)	0.60, 0.59 (0.60)	2.8, 2.3 (2.6)	5.1	6.7
	Raw/fresh Puree [c]	0.21, 0.24 (0.23)	<0.01, <0.01 (<0.01)	0.21, 0.24 (0.23)	0.59	0.59

Notes:

DALA = Days After Last Applicatin; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval.

[a] Expressed as parent tetraniliprole.

[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value in the processed commodity is <LOQ, a value of 0.01 or 0.02 mg/kg has been used for calculation of a PF. Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinoneis <0.01 mg/kg eq, a value of "0" was used.

[c] In normal industrial practices, the tomato puree is pasteurized.

Mustard greens

One field trial was conducted to provide samples to determine the effect of washing or washing and cooking on the magnitude of tetraniliprole residues in/on mustard greens treated with four applications of a tetraniliprole 200 SC formulation (Miller & Jerkins, 2016, M-557177-01-1, Report RAFVN036). Applications were made at a rate of 45–46 g ai/ha per application, with application intervals of 4–5 days.

One day after application a bulk sample of 4 kg was collected from plot FV306-14HA for processing purposes. The bulk samples were divided randomly into 24 separate sampling sections. Mustard greens were washed and cooked in a manner similar to normal household practice; All subsamples (RAC, washed, and washed and cooked) had any withered leaves and stems removed and discarded. The washed subsample was placed in a sink filled with water and agitated for 30 seconds so that all leaf surfaces were rinsed. The sink was drained, and the rinsing process was repeated two additional times. The washed subsample was allowed to drain on paper towels for at least 2 minutes. For the cooked subsample, the leaves were washed as described above. After draining, the leaves were placed in boiling water for 9 minutes and then allowed to cool to room temperature. Throughout the study one representative sample of untreated control and two (duplicate) samples of treated processed commodities were taken.

Samples were stored frozen for a maximum of 427 days prior to residue analysis. Samples were analysed for residues of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone using LC-MS/MS method 01414. The LOQ was 0.01 mg/kg for both analytes in all commodities. The concurrent recoveries were within the acceptable range of 70–120 percent.

Trial data, residue levels and processing factors for each type of commodity are summarised Table 123.

Table 123 Residues of tetraniliprole in mustard greens after processing (Report RAFVN036)

Trial information	Sample	Residues (mg/kg) [a]			PF[b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
FV306-14HA-TRD Madera, California, United States, 2014 (Florida Broadleaf) 4 x foliar treatment; 45-46 g ai/ha, RTI 4-5 days, DALA=1 day	Leaves RAC	3.2, 3.2, (3.2)	<0.01, <0.01 (<0.01)	3.2, 3.2 (3.2)	-	-
	Leaves pre-processing	2.4	<0.01	2.4	-	-
	Washed + cooked leaves	0.47	0.12	0.59	0.15	0.18
	Washed leaves	1.1	<0.01	1.1	0.34	0.34

Notes:
DALA = Days After Last Application; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval; T-N-MQZ = tetraniliprole-N-methylquinazolinone.
[a] Expressed as parent tetraniliprole.
[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value in the processed commodity is <LOQ, a value of 0.01 or 0.02 mg/kg has been used for calculation of a PF. Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

Soya bean, dry

Two processing trials were conducted in the United States as part of the residue trials to measure the magnitude of tetraniliprole residues in/on dry soya bean processed commodities following four foliar applications of tetraniliprole 200 SC (Greenland, 2016j, M-574330-02-1, Report SARS-15-03). Applications were made at an exaggerated rate of 4 × 250–251 g ai/ha (5×), with application intervals of 3–4 days.

Samples of dry soya bean were collected 14 days after the final application at commercial maturity. Soya beans were processed according to simulated commercial procedures into meal and refined oil. Throughout the study one representative sample of untreated control and two (duplicate) samples of treated processed commodities were taken.

Drying

As the moisture content of the dry soya bean seeds was 13–15 percent, drying prior to processing was not required.

Aspirated grain fraction

To generate aspirated grain fractions (AGF), the seed samples (590 and 529 kg for untreated and treated samples, respectively) of one trial only were placed in a dust generation room containing a holding bin, two bucket conveyors and a screw conveyor. As the samples were moved in the system (120 minutes), aspiration was used to remove grain dust (181 and 136 g, for untreated and treated samples respectively). Light impurities were classified using the following sieves: 2360 micron (8 mesh); 2000 micron (10 mesh); 1180 micron (16 mesh); 850 micron (20 mesh); and 425 micron (40 mesh). After classification of each sample, the material through the 2360 micron sieve was recombined to produce one aspirated grain fraction (AGF), weighing 89 and 104 g for untreated and treated samples respectively. For both samples, the material that passed through the 425 micron screen was greater than half the weight of the total material passing through the 2360 micron screen, so all the material passing through the 2360 micron screen was recombined. A representative sample was removed (sample: aspirated grain fraction (AGF)) and the ash content (17.13 and 50.64 percent for untreated and treated samples, respectively) was determined according to AOCS Method Ba 5a-49. One kg of soya bean yields 0.15–0.2 g AGF.

Cleaning

After generation of aspirated grain fractions, a representative sample of generated soya bean seed (17.5 kg) was cleaned by aspiration; light impurities were removed from the whole soya bean by aspiration in a Kice Industries aspirator. After aspiration, the samples were screened on the Enhanced 2 screen cleaner to separate large and small foreign particles (screenings) from the soya bean seed sample. Resulting in 17.0/28.6 kg cleaned soya bean seeds.

Oil processing

After cleaning, the cleaned soya bean seeds (17/29 kg) were fed into an A.T. Ferrell roller mill to crack the hull and liberate the kernel. After hulling, the material was aspirated and screened to separate hulls (2.59/4.26 kg) and kernel (14.4/24.3 kg) material and samples were taken (sample: hulls). One kg of soya bean seeds yields 0.15 kg hulls.

Kernel moisture content was adjusted to 13.5 percent by water addition and allowed to temper, for at least 12 hours. Then the water adjusted kernels (15.4/25.2 kg) were heated to 71 to 97 °C in a mixer and flaked in a flaking roll. Flakes (15.1/24.7 kg) were extruded in a continuous processer, where they were turned into collets by direct steam injection and compression. The collets were ground in a disc mill and dried in an oven at 66–82 °C for 30–40 minutes and taken to solvent extraction (14.4/23.3 kg). Oil was extracted by solvent (hexane) at 49–60 °C for 30 minutes, repeated again twice for 15 minutes. Extracted collets (2200 g) were toasted in a steam jacketed mixer at temperatures up to 116 °C. After toasting the product (2093/9253 g) was cooled to room temperature, screened (8 mesh screen), and sampled (sample: meal, toasted).

Hexane was removed from the crude oil (3389/4589 g) by aspiration (evaporation), heating (91–96 °C) and filtering. Based on the free fatty acid content, a weighed amount of crude oil (2000/2500 g) was mixed with sodium hydroxide (114/147.5 g), then soapstock (243/316 g) and alkali refined oil (1802/2261 g) were separated using centrifugation. The refined oil was decanted, filtered and activated bleaching earth (1.0 percent by weight of oil) was mixed with the oil (1785/2239 g of the refined oil was used). The solution was stirred and placed under vacuum. The temperature was increased to 85–100 °C and held for 10 to 15 minutes. After the bleaching period, the temperature was reduced to 58–68 °C. Vacuum was broken and a filter aid added. The bleached oil and spent bleaching earth/filter aid separated by vacuum filtration. Resulting fractions were bleached oil (1715/2147 g) and spent bleaching earth/filter aid. Spent bleaching earth/filter aid was discarded. The resulting bleached oil (1703/2028 g was used) was heated and filtered and steam bathed at 220 to 230 °C. Citric acid was added. The resulting fraction was refined-bleached-deodorized oil), weighing 1688/2023 g. This fraction was sampled (sample: refined-bleached-deodorized oil (RBD oil). Corrected for fractionation, 1 kg of soya bean seeds yields 0.14–0.17 kg refined-bleached-deodorized oil.

Samples were stored frozen for a maximum of 130 days prior to analysis. All samples were analysed for residues of tetraniliprole and tetraniliprole-N-methylquinazolinone using analytical method 01414. The results are shown in Table 124.

Table 124 Tetraniliprole residues and processing factors in soya bean and processed commodities (Report SARS-15-03)

Trial information	Sample	Residues (mean) (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
SARS-15-03-AR, Crittenden, Arkansas, United States, 2015 (HBK4953LL) 4 x foliar application at 250 g ai/h; RTI = 3-4 days, BBCH 87-88, DALA = 14 days	Soya bean (RAC)	0.45, 0.45 (0.45)	<0.01. 0.01 (<0.01)	0.45, 0.45 (0.45)	-	-
	Meal, toasted	0.012, 0.012 (0.012)	0.052, 0.050 (0.051)	0.064, 0.062 (0.063)	0.027	0.14
	Hulls	1.9, 1.9 (1.9)	<0.01. 0.01 (<0.01)	1.9, 1.9 (1.9)	4.2	4.2
	Refined bleached deodorized oil	<0.01, <0.01 (<0.01)	<0.01. 0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.022	<0.022
	AGF	15, 15 (15)	0.034, 0.036 (0.035)	15, 15 (15)	33	33
SARS-15-03-MN2, Stearns, Minnesota, United States, 2015 (AG013RRY2) 4 x foliar application at 250 g ai/h; RTI = 3-4 days, BBCH 87-88, DALA = 14 days	Soya bean (RAC)	<0.01, <0.01 (<0.01)	<0.01. 0.01 (<0.01)	<0.01, <0.01 (<0.01)	-	-
	Meal, toasted	<0.01, <0.01 (<0.01)	<0.01. 0.01 (<0.01)	<0.01, <0.01 (<0.01)	n.c.	n.c.
	Hulls	<0.01, <0.01 (<0.01)	<0.01. 0.01 (<0.01)	<0.01, <0.01 (<0.01)	n.c.	n.c.
	Refined bleached deodorized oil	<0.01, <0.01 (<0.01)	<0.01. 0.01 (<0.01)	<0.01, <0.01 (<0.01)	n.c.	n.c.
	AGF	-	-	-	n.c.	n.c.

Notes:
AGF = Aspirated Grain Fractions; DALT = Days After Last Application; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval; n.c. = not calculated since residues in both RAC and the processed fractions were <LOQ.
[a] Expressed as parent tetraniliprole.
[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

Potato

Two field trials were conducted, in the United States, to measure the magnitude of tetraniliprole residues in/on potato processed commodities following either a single in-furrow application or four foliar broadcast applications of tetraniliprole 200 SC at exaggerated rates (5×) (Veal, 2016b, M-563221-01-1, Report RAFVP062). Applications were made at an actual rate of 995 g ai/ha for the in-furrow at planting application and 151 g ai/ha for the foliar applications. Samples of potato tubers (80–89 kg) were harvested at commercial maturity and processed according to simulated commercial procedures into washed potato tubers, peeled tubers, peel, crisps, flakes, potato mash, starch and cooked tubers with/without peel. Throughout the study one representative sample of untreated control and two (duplicate) samples of treated processed commodities were taken.

The weight fractions reported represent the in-furrow/foliar application per trial, respectively. The potato samples (76.58/79.74 kg and 84.71/85.35 kg) were batch tub washed for 5 minutes and specific gravity was determined. The washed potatoes (69.59/70.51 kg and 84.23/81.56 kg) were inspected and if necessary, culled potatoes were weighed and disposed (6.83/6.16 kg and 0.0/0.0 kg). Representative samples (sample*: potato washings* and sample: *tuber, washed*) were taken. Aliquots of washed potatoes were removed and either returned to storage for processing potato chips (10.45/10.60 kg and 10.28/10.44 kg) and boiled potatoes (3.38/3.35 kg and 3.21/3.49 kg) at a later time, or sent directly to potato chip processing or cooking. The remaining potatoes (38.12/37.96 kg and 37.89/39.03 kg) were batch steam peeled using the 100 L steam peeler for 45 seconds at 100–120 psi. The potatoes were batch scrubbed for 30 seconds using a peeler. The potato peel was collected from the peeling and scrubbing process. Water from the scrubber was collected for starch processing (45.28/39.10 kg and 46.69/50.04 kg). The peeled potatoes were inspected and hand trimmed to remove additional peel, rot, green or otherwise damaged potatoes. The trim waste (0.62/0.73 kg and 0.41/0.34 kg) was retained. The collected peel (2.77/2.02 kg and 3.38/3.82 kg) was hydraulically pressed. The pressed peel (1.88/0.98 kg and 1.84/2.03 kg) was blended with the cut trim waste (0.62/0.73 kg and 2.25/2.37 kg) collected and a representative sample of the combined wet peel and trimmings was taken (sample: *peel, wet*). Starch water (0.34/0.41 kg and 0.76/0.71 kg) was collected from the hydraulically pressed peel for starch removal.

Potato starch processing

Starch water (0.34/0.41 kg and 0.76/0.71 kg) that was collected from the hydraulically pressed peel was combined with the water from scrubbing for starch removal (45.28/39.10 kg and 46.69/50.04 kg). The total starch water (45.62/39.51 kg and 47.45/50.75 kg) was filtered using a series of sieves to remove pieces of peel and pulp. The filtered starch water was centrifuged to separate the water from the starch until the required amount of starch sample was obtained (1.13/1.13 kg and 1.52/1.18 kg starch recovered). The remaining filtered starch water was disposed. A representative sample was taken (sample: *starch*).

Potato flake processing

After sampling *(sample: tuber, peeled)*, the remaining peeled potatoes (28.53/29.82 kg and 29.12/30.53 kg) were reserved for potato flake processing. The peeled potatoes were cut into slabs using a slicer. The potato slabs were batch spray-washed in cold tap water for 30 seconds to remove free starch using the spray washer. The potato slabs were precooked at about 70–77 ° C while targeting 71–74 °C for 20 minutes in the 150 L kettle. The precooked potato slabs were to less than 32 °C for 20 minutes. An aliquot of precooked and cooled potatoes (21.04/21.05 kg and 24.11/24.12 kg) was removed for further potato flake processing and the excess discarded (7.17/9.43 kg and 4.74/6.40 kg). The cooled potato

slabs were steam-cooked at 94–100 °C for 40 minutes in a cabinet. The cooked potato slabs were mashed using a grinder (18.90/18.86 kg and 22.56/22.21 kg). A representative sample (1.31/1.18 kg and 1.05/1.04 kg) of the potato mash was taken (sample: *tuber, steamed, mashed*). An aliquot of the potato mash (9.07 for all batches) was mixed for 37 seconds in a mixer with an emulsion of pre-weighed food additives specific to the amount of mash for flaking (typically 13.60 kg). Any remaining mash (8.52/8.61 kg and 12.44/12.10 kg) was disposed. The cooked mash was hand fed onto a dryer to dry the cooked mash into a thin sheet and initially broken into large flakes by hand (1.51/1.46 kg and 1.88/1.71 kg). The flakes were then fed into a hammermill for uniform milling of the potato flakes (weight after milling: 1.45/1.44 kg and 1.84/1.70 kg; loss due to milling 0.06/0.02 kg and 0.04/0.01 kg). Moisture analysis was conducted on the potato flakes. All six samples of milled flakes were determined to have < 9 percent moisture and did not require additional drying.

A representative sample was taken (sample: *flakes*). The yield from precooked and cooled potatoes to flakes ranged from 96 to 99 percent. Figure 12 shows the procedure for processing potato to flake

Potato crisps (without peel) processing

An aliquot of washed potatoes previously reserved (10.45/10.60 kg and 10.28/10.44 kg) was removed from storage for the potato crisps process. The washed potatoes were batch peeled for 30 seconds using an abrasive base plate in a restaurant style peeler. The collected peel was weighed (1.07/1.0 kg and 0.82/0.58 kg) and discarded. The peeled potatoes (9.11/9.70 kg and 9.46/9.78 kg) were inspected by hand and trimmed if necessary, to remove rot, green or otherwise damaged potato tissue. Any trim waste collected was weighed and discarded. An aliquot of the remaining peeled potatoes were cut into thin 0.16 cm slices using a restaurant style slicer. Any abrasion peeled potatoes in excess were discarded (6.0/6.67 kg and 5.96/6.73 kg). The potato slices were placed in a tub of hot water to remove free starch. The slices were drained over a screen to remove excess water and were deep fried at 163–191 °C frying oil for approximately 90 seconds. The fried potato crisps were drained, salted and inspected. Undesirable crisps were removed. The weight of the finished potato crisps was 1.05/1.04 kg and 1.15/1.15 kg and taken as sample (sample: *crisps*). Yield from washed potatoes to chips (without peel) ranged from 19–24 percent. Figure 13 shows the potato chip process.

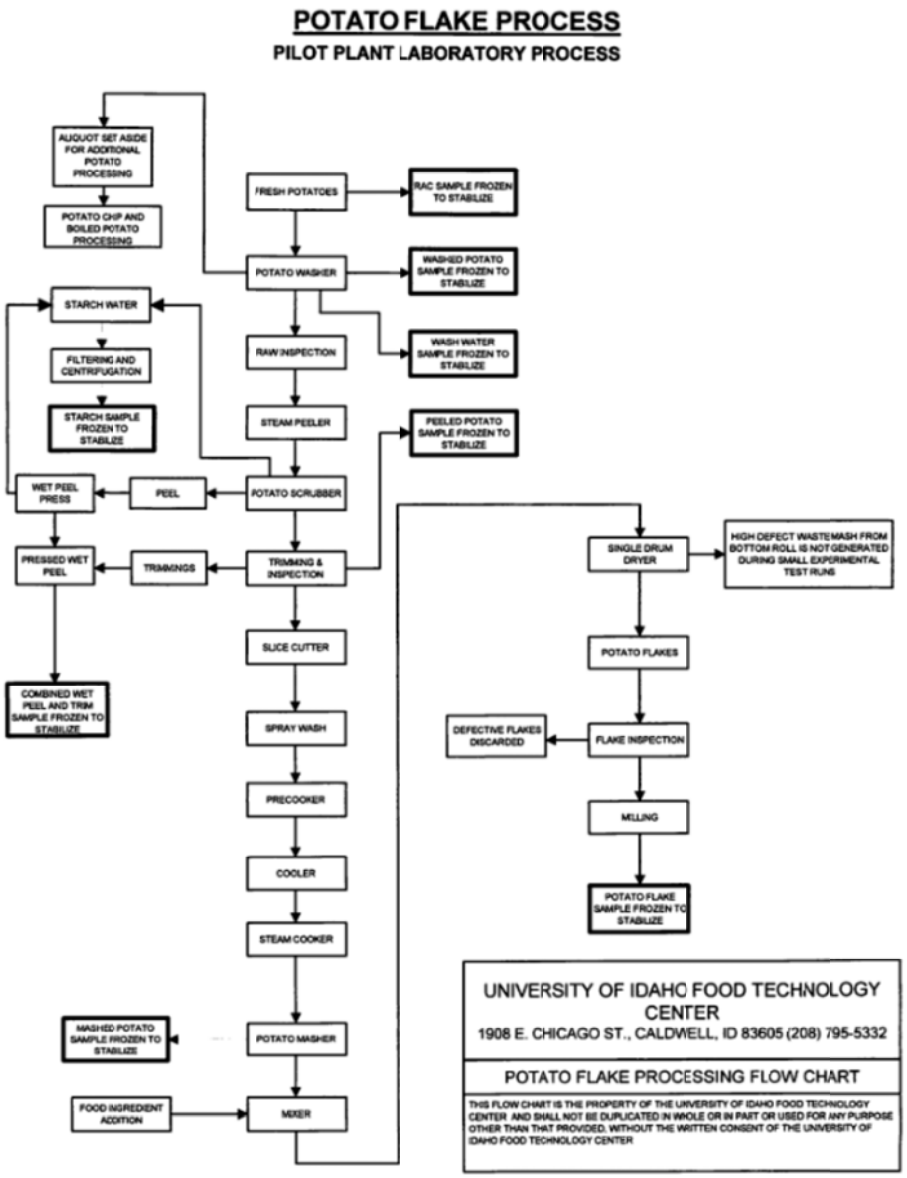


Figure 12 Processing steps in potato flake production

Cooked potato processing

An aliquot of unpeeled washed potatoes (3.38/3.35 kg and 3.21/3.49 kg) was divided into two subsamples for processing into unpeeled boiled potatoes (1.35/1.42 kg and 1.44/1.32 kg) and peeled boiled potatoes (1.67/1.64 kg and 1.76/2.17 kg) and the excess (0.36/0.29 kg and 0/0 kg) was discarded. Potatoes reserved for unpeeled boiled potatoes were cut into quarters using a paring knife. For producing unpeeled boiled potatoes, the quartered potatoes were placed into boiling water and boiled until an internal temperature of 88–92 °C was reached as measured with a probe thermometer. After the desired temperature was reached, the unpeeled boiled potato fraction was removed from the heat and poured through a colander over a bucket to drain the potatoes and capture the cooking water. The weight of the unpeeled boiled potatoes was 1.25/1.31 kg and 1.39/1.20 kg. A representative sample of both the cooking water and unpeeled boiled potato was taken (sample: *tuber with peel, cooked* and sample: *cooking water*). The yield from washed potatoes to boiled unpeeled potatoes was 100 percent.

Figure 13 Potato chip processing process

Potatoes reserved for peeled boiled potatoes (1.67/1.64 kg and 1.76/2.17 kg) were hand peeled with a vegetable peeler (weighed of peeled potatoes: 1.41/1.36 kg and 1.56/1.89 kg) and then cut into quarters using a paring knife. For producing peeled boiled potatoes, the quartered potatoes were placed into boiling water and boiled until an internal temperature of 88–92 °C was reached as measured with a probe thermometer. After the desired temperature was reached, the peeled boiled potato fraction was removed from the heat and drained through a colander. The weight of the boiled peeled potatoes was 1.29/1.19 kg and 1.34/1.60 kg and a representative sample was taken (sample: *tuber, peeled and cooked*). The yield from washed potatoes to boiled unpeeled potatoes ranged from 73-77 percent.

Samples were stored frozen for a maximum of 278 days prior to analysis. All samples were analysed for residues of tetraniliprole and tetraniliprole-N-methylquinazoline using analytical method 01414. Trial data, residue levels and processing factors for each type of commodity are summarised in

Table 125. Residues of tetraniliprole and its metabolite were seen to dilute upon processing with PF's <1 in most cases. Concentration of residues was only seen in washed tubers and wet peel.

Table 125 Tetraniliprole residues and processing factors in potato tubers and processed commodities (Report RAFVP062)

Trial information	Sample	Residues (mg/kg) [a]			PF[b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
FV003-15PA, Wayne, New York, United States, 2015 (Fresh Market) 4 x foliar at 130-150 g ai/ha; RTI = 5 days, BBCH 45-48 (last application); DALA= 13 days	Potato tuber (RAC)	<0.01 (3)	<0.01 (3)	<0.01	-	-
	Potato washings	<0.01	<0.01	<0.01	n.c.	n.c.
	Tuber, washed	<0.01 (3)	<0.01 (3)	<0.01	n.c.	n.c.
	Peel, wet	<0.01	<0.01	<0.01	n.c.	n.c.
	Crisps, without peel	<0.01	<0.01	<0.01	n.c.	n.c.
	Flakes	<0.01	<0.01	<0.01	n.c.	n.c.
	Tuber, peeled	<0.01	<0.01	<0.01	n.c.	n.c.
	Tuber, steamed, mashed	<0.01	<0.01	<0.01	n.c.	n.c.
	Starch	<0.01	<0.01	<0.01	n.c.	n.c.
	Tuber with peel, cooked	<0.01	<0.01	<0.01	n.c.	n.c.
	Cooking water	<0.01	<0.01	<0.01	n.c.	n.c.
	Tuber, peeled and cooked	<0.01	<0.01	<0.01	n.c.	n.c.
FV004-15PA, Minidoka, Idaho, United States, 2015 (Western Russet) 4 x foliar at 133-154 g ai/ha; RTI = 5 days, BBCH 47-48 (last application); DALA = 14 days	Potato tuber (RAC)	<0.01 (2), 0.011 (mean=0.010)	<0.01 (3)	0.01	-	-
	Potato washings	<0.01	<0.01	<0.01	<1	<1
	Tuber, washed	<0.01 (3)	<0.01 (3)	<0.01	<1	<1
	Peel, wet	<0.01	<0.01	<0.01	<1	<1
	Crisps, without peel	<0.01	<0.01	<0.01	<1	<1
	Flakes	<0.01	<0.01	<0.01	<1	<1
	Tuber, peeled	<0.01	<0.01	<0.01	<1	<1
	Tuber, steamed, mashed	<0.01	<0.01	<0.01	<1	<1
	Starch	<0.01	<0.01	<0.01	<1	<1
	Tuber with peel, cooked	<0.01	<0.01	<0.01	<1	<1
	Cooking water	<0.01	<0.01	<0.01	<1	<1
	Tuber, peeled and cooked	<0.01	<0.01	<0.01	<1	<1
FV003-15PA, Wayne, New York, United States, 2015 (Fresh Market) 1 in-furrow application at planting; DALA= 13 days	Potato tuber (RAC)	0.040, 0.026, 0.043 (mean=0.036)	<0.01 (3)	0.036	-	-
	Potato washings	<0.01	<0.01	<0.01	<0.3	<0.3
	Tuber, washed	<0.01 (3)	<0.01 (3)	<0.01	<0.3	<0.3
	Peel, wet	<0.01	0.040	0.050	<0.3	5
	Crisps, without peel	<0.01	<0.01	<0.01	<0.3	<0.3
	Flakes	<0.01	<0.01	<0.01	<0.3	<0.3
	Tuber, peeled	<0.01	<0.01	<0.01	<0.3	<0.3
	Tuber, steamed, mashed	<0.01	<0.01	<0.01	<0.3	<0.3
	Starch	<0.01	<0.01	<0.01	<0.3	<0.3
	Tuber with peel, cooked	<0.01	<0.01	<0.01	<0.3	<0.3
	Cooking water	<0.01	<0.01	<0.01	<0.3	<0.3
	Tuber, peeled and cooked	<0.01	<0.01	<0.01	<0.3	<0.3
FV004-15PA, Minidoka, Idaho, United States, 2015 (Western Russet) 1 in-furrow application at planting, DALA =14 days	Potato tuber (RAC)	<0.01, 0.025 (2) (mean =0.020)	<0.01 (3)	0.020	-	-
	Potato washings	<0.01	<0.01	<0.01	<0.5	<0.5
	Tuber, washed	<0.01 (2), 0.066 (mean=0.029)	<0.01 (3)	0.029	1.5	2.0
	Peel, wet	0.046	0.046	0.092	2.3	4.6
	Crisps	<0.01	<0.01	<0.01	<0.5	<0.5
	Flakes	<0.01	<0.01	<0.01	<0.5	<0.5
	Tuber, peeled	<0.01	<0.01	<0.01	<0.5	<0.5

Trial information	Sample	Residues (mg/kg) [a]			PF[b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
	Tuber, steamed, mashed	<0.01	<0.01	<0.01	<0.5	<0.5
	Starch	<0.01	<0.01	<0.01	<0.5	<0.5
	Tuber with peel, cooked	<0.01	<0.01	<0.01	<0.5	<0.5
	Cooking water	<0.01	<0.01	<0.01	<0.5	<0.5
	Tuber, peeled and cooked	<0.01	<0.01	<0.01	<0.5	<0.5

Notes:

DALA = Days After Last Application; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval; n.c. = not calculated since residues in both RAC and the processed fractions were below LOQ; T-N-MQZ = tetraniliprole-N-methylquinazolinone.

[a] Expressed as parent tetraniliprole.

[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

Rice

Two processing trials were conducted in the United States to measure the magnitude of tetraniliprole and metabolite tetraniliprole-N-methylquinazolinone residues in/on paddy rice processed commodities following three foliar applications of tetraniliprole 200 SC (Brungardt, 2018, M-638211-01-1, Report RAFV0032). Applications were made at rates ranging from 91–92 g ai/ha each, with application intervals of 13–16 days between BBCH growth stages 26 and 58. No adjuvant was used. Samples of rice were collected 30 days after the last application at commercial maturity. At the processing facility, triplicate subsamples of rice grain, the raw agricultural commodity (RAC), were removed and frozen for subsequent analysis. The remaining rice grain was used to generate the processed commodities of polished rice (grain, polished), hulls, and bran. Samples were frozen for subsequent analysis.

Drying and cleaning

After removal from the freezer, representative rough rice (RAC) fractions were collected and placed into frozen storage. Samples were weighed (40/40.5 kg) and the moisture content determined with an electronic moisture analyser. If the moisture was greater than 14 percent, samples were dried in an oven at 43–60 °C to a moisture content of 11–14 percent. All samples required drying. The yield from RAC to dried rice was 78–91 percent. Following drying, samples (31.6/37 kg) were cleaned by aspiration (1.63/0.50 kg) and screening (2.18/7.80 kg). Samples were aspirated in an aspirator to remove light impurities from the rough rice. Following aspiration, samples were screened in a cleaner to separate large and small foreign particles (screening) from the rough rice, resulting in 27.9/28.6 kg cleaned rice. The yield from RAC to cleaned rice was *ca* 70 percent.

Milling–Dehulling and Bran Removal

Cleaned rough rice samples of 15.9/13.6 kg were dehulled and milled in a Satake rice mill. During the dehulling process, samples entered the hulling section of the mill where hull was removed by rubber rolls rotating in opposite directions at different speeds. Hull material (2.99/2.27 kg) was separated from the brown rice (12.6/11.1 kg) by aspiration. During this sequence, the brown rice passed through the milling section and exited the machine. No milling (bran removal) was performed during this pass. For production of bran and white milled rice (polished rice), brown rice (12.6/11.1 kg) entered the hulling portion where additional hull material was removed. During this pass through the milling chamber, brown rice was held in the milling chamber where it was milled into white milled rice and bran by friction. Bran

(1.81/1.09 kg) was separated from the white milled rice (7.08/8.57 kg) by air injected into the milling chamber (slotted screen). Bran percentage can be increased by adding weights to the exit door of the milling chamber. After exiting the chamber, bran was sieved with a Great Western sifter equipped with a 24 mesh screen to remove broken pieces of brown and white milled rice and small amounts of hull material from bran. Samples of hull material, white milled rice and bran were collected (sample: *hulls*; sample: *polished white rice*; sample: *bran*). The preferred hull percentage to starting rough rice of 18–24 percent was achieved. During the milling process, the goal is to obtain a bran percentage of 8.5 to 14.5 percent (based on a calculated amount of white milled rice and bran). The yield from rice grain (prior to drying and cleaning) to white milled rice is 31–45 percent.

Samples were stored frozen for up to a maximum of 183 days prior to analysis. All samples were analysed for residues of tetraniliprole and tetraniliprole-N-methylquinazolinone using the analytical method 01414.

Trial data, residue levels and processing factors for each type of commodity are summarised in Table 126.

Table 126 Tetraniliprole residues and processing factors in rice grain and processed commodities (Report RAFV0032)

Trial information	Sample	Residues (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
FV008-17PA, Cheneyville, Louisiana, United States, 2017 (Cheniere) 3 x foliar application at 305-311 g ai/ha; RTI=14/16 days; BBCH 26-51; DALA = 30 days	Rice grain (RAC)	0.086, 0.081, 0.095 (mean: 0.087)	<0.01 (3)	0.087	-	-
	Polished white rice	<0.01 (3)	<0.01 (3)	<0.01	<0.11	<0.11
	Hulls	0.24 (3)	<0.01 (3)	0.24	2.8	2.8
	Bran	0.025 (2) 0.024 (mean: 0.025)	<0.01 (3)	0.025	0.29	0.29
FV009-17PB, Humphrey, Arkansas, United States, 2017 (CLXL 745) 3 x foliar application at 297-311 g ai/ha; RTI = 15/13; BBCH 30-58; DALA = 30 days	Rice grain (RAC)	0.59, 0.54, 0.64 (mean: 0.58)	<0.01 (3)	0.58	-	-
	Polished white rice	<0.01 (3)	<0.01 (3)	<0.01	<0.017	<0.017
	Hulls	3.2, 3.1 (2) (mean: 3.2)	0.037 (2), 0.038 (mean: 0.037)	3.2	5.4	5.4
	Bran	0.16 (3)	<0.01 (3)	0.16	0.28	0.28

Notes:
DALA = Days After Last Application; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval.
[a] Expressed as parent tetraniliprole.
[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

Maize

Two processing trials were conducted in the United States as part of the residues trials to measure the magnitude of tetraniliprole residues in/on maize processed commodities (Stewart&Greenland, 2016, M-574645-01-2, Report SARS-15-06). Four foliar applications of tetraniliprole 200 SC at an exaggerated nominal rate of 250 g ai/ha, with application intervals of 7 days. Samples of maize grain were collected 14 days after the final application at commercial maturity. Grain was processed according to simulated commercial procedures into grits, meal, flour, starch and oil (wet and dry milled). Throughout the study

one representative sample of untreated control and two (duplicate) samples of treated processed commodities were taken.

Drying

Maize grain samples for aspirated grain fraction generation and processing were removed from freezer storage. Samples were weighed and the moisture content determined with an electronic moisture analyser. Since the moisture content of samples for aspirated grain fraction generation from trial SARS-15-06-NY were above 13.0 percent, drying was required. Samples were dried in a Steelman Industries oven at 110-135°F until the moisture content was 10.0–13.0 percent. All samples for processing only (sample SARS-15-06-NY-012 and both samples from trial SARS-15-06-MN2) were dried in the oven at 54–71 °C until the moisture content was 10.0 -15.0 percent.

Generation of Aspirated Grain Fraction (AGF)

To generate aspirated grain fractions, each maize grain sample (286 kg) was dried and the dried maize (239 kg) was placed in a dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. As the samples were moved in the system (120 minutes), aspiration was used to remove light impurities (grain dust) weighing 0.77 kg. Light impurities (784 gram) were classified using different sieves and the material through the 2360 micron sieve was recombined to produce one aspirated grain fraction (AGF). A representative sample was removed and the ash content (1.42 percent) was determined according to AOCS Method Ba 5a-49.

Production of processed fractions

Following AGF generation for sample SARS-15-06-NY-010 and drying for both samples from SARS-15-06-MN2, samples were cleaned by aspiration and screening. Light impurities were removed using a Kice aspirator. After aspiration, samples were screened in an Enhance 2 screen cleaner to separate large and small foreign particles (screenings) from the cleaned grain.

Dry milling process

After drying, samples for production of processed fractions (185/230 kg) were cleaned resulting in batches of cleaned maize grain weighing 167/207 kg. For the dry milling process, maize grain (91.7/102 kg) was moisture conditioned to 21.0 percent and tempered for approximately two hours (steeping with 9.5/10.2 kg water added). The samples were fed into a C. S. Bell disc mill to crack the kernels. Corn stock from the mill was dried and screened to separate germ (13.8/13.7 kg) from meal (11.5/10.3 kg), grits (12.5/11.0 kg), large grits (52.8/69.0 kg), flour (4.2/2.72 kg) and bran (2.59/1.95 kg) and samples were taken (sample: *meal*; sample: *grits*; sample: *flour*). Dried germ material (13.8/13.5 kg) was heated, flaked and submerged in solvent (hexane) to extract the crude oil (739/1184 g) and leaving 1.16/12.3 kg solvent extracted germ flakes. Hexane was removed from the crude oil by aspiration, heating and filtering. Crude oil (719/1079 g) was mixed with Braumé sodium hydroxide (30.9/47.5 g), then soapstock (128/127 g) and alkali refined oil (602/975 g) were separated using centrifugation. The refined oil (588/961 g) was decanted, filtered and an activated bleaching earth was mixed with the oil. Of the resulting bleached oil (571/931 g), 565/921 gram was heated and filtered and steam bathed at 220–230 °C. Citric acid was added. The resulting fraction (560/919 g) was refined-bleached-deodorized oil (RBD oil) and samples were taken (sample: *refined bleached deodorized oil, wet milled*). The yield of cleaned corn to refined, bleached, deodorized oil via dry milling process ranged from 6–9 percent.

Wet Milling Process

For the wet milling process, whole grain (75.1/79.4 kg) was steeped in 49–54 °C water containing sulfur dioxide for 22–48 hours. The steeped whole corn (118/127 kg) was passed through a C. S. Bell disc mill and germ and hull were separated from the cornstock and dried. Germ and hull were then separated. Cornstock was ground in the disc mill and screened, separating out the starch (53.4/53.6 kg), which was dried and sampled (sample: *starch*). Also fibre (8.62/9.12 kg), gluten (4.35/4.63 kg) and germ (5.62/5.94 kg) were separated in the process. The germ samples were moisture conditioned, flaked and pressed in an expeller to liberate part of the crude oil (453/451 gram). The rest of the crude oil (906/1236 g) was extracted from the presscake (3.63/5.72 kg) using hexane, leaving 2.54/4.45 kg solvent extract germ. Combined crude oil samples (1249/1607 g) from the wet milling process were alkali refined (55/66 g NaOH added) to 1154/1417 g refined oil and 133/147 g soapstock. A batch of 1140/1400 g refined oil was bleached (1100/1369 g) and 1092/1356 g of this was deodorized (1240/1353 g), similar as for the dry milling process, and samples were taken (sample: *refined bleached deodorized oil, dry milled*). The yield of cleaned corn to refined, bleached, deodorized oil via the wet milling process was *ca* 17 percent.

Samples were stored frozen (<-4 °C) for a maximum of 108 days prior to analysis. All samples were analysed for residues of tetraniliprole and tetraniliprole-N-methylquinazolinone using the analytical method 01414.

Processing factors were derived for field corn/maize grain processed commodities. A concentration of residues was observed in aspirated grain fractions, flour and meal, residues were diluted in all other commodities. Trial data, residues and processing factors for each type of commodity are summarised in Table 127

Table 127 Tetraniliprole residues and processing factors in field corn/maize grain and processed commodities [Report SARS-15-06]

Trial information	Crop/Processed commodity	Residues (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
SARS-15-06-NY-7, Wayne, New York, United States,2015 (X19318WP.O) 4 x foliar application at 252-262 g ai/ha; no adjuvant added; BBCH 87, RTI = 7 days, DALA = 14 days.	Maize grain (RAC)	0.015, 0.017 (mean:0.016)	<0.01 (2)	0.015, 0.017 (mean:0.016)	-	-
	Flour	0.019 (2)	<0.01 (2)	0.019 (2)	1.2	1.2
	Grits	<0.01 (2)	<0.01 (2)	<0.01 (2)	<0.6	<0.6
	Meal	0.019, 0.017 (mean: 0.018)	<0.01 (2)	0.019, 0.017 (mean: 0.018)	1.1	1.1
	Starch	<0.01 (2)	<0.01 (2)	<0.01 (2)	<0.6	<0.6
	Refined bleached deodorized oil (wet milled)	<0.01 (2)	<0.01 (2)	<0.01 (2)	<0.6	<0.6
	Refined bleached deodorized oil (dry milled)	<0.01 (2)	<0.01 (2)	<0.01 (2)	<0.6	<0.6
	Aspirated grain fraction	0.077, 0.070 (mean: 0.074)	<0.01 (2)	0.077, 0.070 (mean: 0.074)	4.6	4.6
SARS-15-06-MN2-7, Stearns, Minnesota, United States, 2015 (DeKalb) 4 x foliar application at 251-261 g ai/ha; adjuvant (COC);	Maize grain (RAC)	<0.01 (2)	<0.01 (2)	<0.01 (2)	-	-
	Flour	<0.01 (2)	<0.01 (2)	<0.01 (2)	n.c.	n.c.
	Grits	<0.01 (2)	<0.01 (2)	<0.01 (2)	n.c.	n.c.
	Meal	<0.01 (2)	<0.01 (2)	<0.01 (2)	n.c.	n.c.
	Starch	<0.01 (2)	<0.01 (2)	<0.01 (2)	n.c.	n.c.
	Refined bleached deodorized oil (wet	<0.01 (2)	<0.01 (2)	<0.01 (2)	n.c.	n.c.

Trial information	Crop/Processed commodity	Residues (mg/kg) [a]			PF [b]	
		Tetraniliprole	T-N-methyl-quinazolinone	Total	PF_{parent}	PF_{total}
BBCH 89, RTI = 7 days, DALA= 14 days	milled)					
	Refined bleached deodorized oil (dry milled)	<0.01 (2)	<0.01 (2)	<0.01 (2)	n.c.	n.c.
	Aspirated grain fraction	-	-	-	-	

Notes:
; DALA = Days After Last Treatment; RAC = Raw Agricultural Commodity; RTI = Retreatment Interval; n.c. = not calculated since residues in both RAC and the processed fractions were below LOQ.
[a] Expressed as parent tetraniliprole.
[b] PF: Processing Factor = Residue level in processed commodity (mg/kg) ÷ Residue level in unprocessed commodity (mg/kg). Where the value for parent in the processed commodity is <LOQ, a value of 0.01 mg/kg has been used for calculation of a PF. Where the value for the metabolite tetraniliprole-N-methyl-quinazolinone is <0.01 mg/kg eq, a value of "0" was used.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Dairy feeding study in lactating cows

A residue feeding study in dairy cattle was conducted in the United States in 2015/2016 (Williams, 2016d, M-569181-01-1, Report RAFVP037) to measure the residues of tetraniliprole found in milk and tissues. Encapsulated tetraniliprole was fed to 18 (3–5 year old) lactating Holstein (*Bos taurus*) dairy cows (three-six cows/dose group) for 29 consecutive days. Experimental conditions are indicated in Table 74. The animals received actual dose levels of tetraniliprole of 0, 0.94, 9.3, 28, and 94 ppm diet (dry weight), corresponding with a calculated mean dose of 0.03, 0.35, 0.99 and 3.09 mg/kg bw/day. Three animals were maintained in the high dose group for up to 2 weeks after cessation of the treatment in order to provide data on the decline of any incurred residues. Animals were observed several times daily for any clinical signs of toxicity or ill health. Bodyweights were determined at intervals and concentrate food/hay consumption was monitored daily.

The cows weighed on average 461–603 kg during the experiment and had an average daily milk production of 145 kg/week (21.3 percent RSD, n=72) during the experiment. The milk production was not adversely affected. The experimental condictions are shown in Table 128.

Table 128 Experimental conditions of the cow feeding study dosed for 29 days

Cow number	no. of depuration days	Mean group bodyweight at start and end of the feeding period (kg)	Mean daily dry feed intake (kg feed/animal) (wk -2-4)	Actual mean dose (ppm tetraniliprole in feed (DM basis))	Actual mean dose (mg/kg bw/day)
12, 19, 13	7, 14, 21	593-603	18	0 (empty capsule)	0 (empty capsule)
15, 1, 14	-	500-521	18.2	0.94	0.03
10, 9, 11	-	461-496	18.2	9.32	0.35
16, 5, 2	-	478-501	18.1	28	0.99
17, 7, 3 & 8, 4, 20	7, 14, 21	520-537	18.0	94	3.09

Doses were administered after the morning milking and milk samples were collected twice daily (morning and evening) on study days 0, 2, 4, 7, 10, 14, 17, 21, 25, and 28. A composite milk sample was collected containing the evening milk and the following morning milk prior to daily administration. A

portion of the day 25 milk sample from a single control and three cows from the 94 dose group were separated into skim milk and cream. On day 29 (3 to 8 hours after the final dose), two of the control cows, all of the low and mid dose groups and 3 of the highest dose group cows were sacrificed. Perirenal, omental, and subcutaneous fat, and composite kidney, liver, and muscle samples were collected from each cow for analysis. The remaining cows (one control and three from the highest dose level) entered the depuration phase of the study. The depuration cows were sacrificed 7, 14, and 21 days post-dosing (days 36, 43, and 50). The remaining control cow was sacrificed on day 50.

Additional milk samples were collected from the remaining control cow and the highest dose depuration cows on days 31, 35, 38, 42, and 49. Milk and tissues from the cows sacrificed at each interval were analysed to monitor the decline of tetraniliprole residues.

Samples of approximately 0.5 kg each of liver, kidney, composite muscle, and fat (mesenterial, subcutaneous, and perirenal) were collected from all animals. One additional sample of each fat was collected for fat) determination. After collection and processing tissue and milk samples were stored frozen at -6 °C to -28.5 °C, with an average storage temperature of ≤-18 °C during the storage period. All samples were extracted within 30 days of sample origination and analysed within seven days of extraction. Therefore, no additional storage stability data are required for these analytes.

Samples were analysed for tetraniliprole and metabolites tetraniliprole-N-methylquinazolinone and tetraniliprole-benzyl alcohol using LC-MS/MS method FV-002-A16-01-1 with an LOQ of 0.01 mg/kg for each of the separate compounds. Average concurrent fresh recoveries in liver, kidney, muscle, fat, milk, cream and skim milk were within the range of 70–110 pecent for tetraniliprole and its metabolites at 0.01–1.0 mg/kg in tissues and milk. Control samples had residues below 0.2LOQ.

Analytical results in tissue samples are shown in Table 129.

Table 129 Tetraniliprole related residues (mg/kg) in cow tissues for dose groups

Sample	Dose rate (ppm feed)	Day of sampling	Tetraniliprole	Mean	T-N-MetQuin (mg/kg)	T-BenzOH (mg/kg)	Total, (mg/kg) [a]	Mean, (mg/kg) [a]
Liver	0.94	29	0.030	0.031	<LOQ	<LOQ	0.050	0.051
		29	0.025		<LOQ	<LOQ	0.045	
		29	0.037		<LOQ	<LOQ	0.057	
	9.3	29	0.29	0.327	0.028	0.023	0.34	0.370
		29	0.32		<LOQ	0.027	0.36	
		29	0.37		0.018	0.025	0.41	
	28	29	0.49	0.63	0.021	0.043	0.56	0.703
		29	0.52		0.015	0.049	0.58	
		29	0.87		0.034	0.060	0.97	
	94	29	1.0	1.217	0.047	0.066	1.1	1.364
		29	1.1		0.054	0.087	1.2	
		29	1.5		0.061	0.126	1.7	
	depuration	36	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
		43	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
		50	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
Kidney	0.94	29	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
		29	<LOQ		<LOQ	<LOQ	<0.030	
		29	<LOQ		<LOQ	<LOQ	<0.030	
	9.3	29	0.067	0.059	0.024	<LOQ	0.101	0.085
		29	0.049		0.012	<LOQ	0.071	
		29	0.061		0.012	<LOQ	0.083	
	28	29	0.097	0.14	0.026	<LOQ	0.13	0.19
		29	0.13		0.038	<LOQ	0.17	

Sample	Dose rate (ppm feed)	Day of sampling	Tetraniliprole	Mean	T-N-MetQuin (mg/kg)	T-BenzOH (mg/kg)	Total, (mg/kg) [a]	Mean, (mg/kg) [a]
		29	0.19		0.069	<LOQ	0.27	
	94	29	0.24	0.24	0.061	0.015	0.31	0.31
		29	0.20		0.062	0.011	0.27	
		29	0.28		0.051	0.014	0.34	
	depuration	36	<LOQ	<LOQ	0.013	<LOQ	0.033	0.026
		43	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
		50	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
Muscle	0.94	29	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
		29	<LOQ		<LOQ	<LOQ	<0.030	
		29	<LOQ		<LOQ	<LOQ	<0.030	
	9.3	29	0.023	0.021	<LOQ	<LOQ	0.043	0.041
		29	0.016		<LOQ	<LOQ	0.036	
		29	0.023		<LOQ	<LOQ	0.043	
	28	29	0.034	0.046	0.013	<LOQ	0.057	0.075
		29	0.046		0.020	<LOQ	0.075	
		29	0.060		0.024	<LOQ	0.094	
	94	29	0.082	0.079	0.071	<LOQ	0.163	0.138
		29	0.065		0.046	<LOQ	0.121	
		29	0.090		0.030	<LOQ	0.130	
	depuration	36	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
		43	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
		50	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
Fat (perirenal)	0.94	29	<LOQ	<LOQ	0.012	<LOQ	0.032	0.045
		29	<LOQ		0.033	<LOQ	0.053	
		29	<LOQ		0.030	<LOQ	0.050	
	9.3	29	0.043	0.043	0.18	<LOQ	0.23	0.19
		29	0.022		0.22	<LOQ	0.25	
		29	0.063		0.022	<LOQ	0.095	
	28	29	0.067	0.083	0.25	<LOQ	0.32	0.54
		29	0.068		0.41	<LOQ	0.48	
		29	0.12		0.70	<LOQ	0.83	
	94	29	0.11	0.149	0.94	<LOQ	1.1	0.77
		29	0.11		0.59	<LOQ	0.71	
		29	0.22		0.29	<LOQ	0.53	
	depuration	36	<LOQ	<LOQ	0.26	<LOQ	0.28	0.27
		43	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	0.016
		50	<LOQ	<LOQ	0.012	<LOQ	0.032	0.032
Fat (omental)	0.94	29	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	0.028
		29	<LOQ		0.031	<LOQ	0.051	
		29	<LOQ		0.027	<LOQ	0.047	
	9.3	29	0.037	0.039	0.22	<LOQ	0.26	0.20
		29	0.028		0.22	<LOQ	0.26	
		29	0.052		0.023	<LOQ	0.085	
	28	29	0.066	0.082	0.27	<LOQ	0.34	0.54
		29	0.063		0.44	<LOQ	0.51	
		29	0.12		0.64	<LOQ	0.77	
	94	29	0.20	0.162	1.0	<LOQ	1.2	0.75
		29	0.10		0.57	<LOQ	0.68	
		29	0.19		0.15	<LOQ	0.35	
	depuration	36	<LOQ	<LOQ	0.25	<LOQ	0.27	0.25
		43	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<0.030
		50	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	0.011
Fat	0.94	29	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	0.040

Sample	Dose rate (ppm feed)	Day of sampling	Tetraniliprole	Mean	T-N-MetQuin (mg/kg)	T-BenzOH (mg/kg)	Total, (mg/kg) [a]	Mean, (mg/kg) [a]
(subcut)		29	<LOQ		0.026	<LOQ	0.046	
		29	<LOQ		0.023	<LOQ	0.043	
	9.3	29	0.032	0.031	0.081	<LOQ	0.12	0.13
		29	0.028		0.18	<LOQ	0.22	
		29	0.033		0.011	<LOQ	0.054	
	28	29	0.034	0.062	0.17	<LOQ	0.21	0.41
		29	0.056		0.45	<LOQ	0.52	
		29	0.094		0.38	<LOQ	0.49	
	94	29	0.20	0.15	0.89	<LOQ	1.1	0.63
		29	0.099		0.34	<LOQ	0.45	
		29	0.14		0.19	<LOQ	0.34	
	depuration	36	<LOQ	<LOQ	0.16	<LOQ	0.18	0.17
		43	<LOQ	<LOQ	<LOQ	<LOQ	<0.030	<LOQ
		50	<LOQ	<LOQ	0.015	<LOQ	0.035	0.016

Notes:

T-N-MetQuin = tetraniliprole-N-methyl-quinazolinone;T-BenzOH= tetraniliprole-benzylalcohol.

[a] Total residues is the sum of tetraniliprole, tetraniliprole-N-methylquinazolinone, and tetraniliprole-benzyl alcohol expressed as tetraniliprole. For calculation of total residues in parent equivalents no correction was made for molecular weight, since these deviate ~only 3 percent (MW parent = 545 g/mol, MW tetraniliprole-N-methyl-quinazolinone = 527 g/mol, MW tetraniliprole-benzylalcohol = 561 g/mol).

In the low dose group only parent tetraniliprole was observed in the liver. Metabolite tetraniliprole-N-methylquinazolinone was observed in the low dose groups in fat tissues. Both parent and metabolite tetraniliprole-N-methylquinazolinone were observed in all other tissues at the higher feeding levels. The benzylalcohol metabolite was only observed in liver in the mid and high dose groups and in kidney in the high dose group.

Tetraniliprole residues were not found in any of the milk samples of cows dosed at 0.94 ppm. Tetraniliprole residues in milk above the LOQ were found in all milk samples of the two mid and the high dosed animals. Parent tetraniliprole ranged from 0.040 to 0.056 mg/kg at 9.3 ppm, to 0.058–0.11 mg/kg at 28 ppm and 0.17–0.19 mg/kg at the 94 ppm feeding level. Similar patterns were found for both metabolites, though at lower concentrations compared to parent (factor 1.5–2 parent to metabolite tetraniliprole-N-methylquinazolinone and factor 2–2.5 parent to tetraniliprole-benzylalcohol). Concentrations ranged from 0.015–0.038 mg/kg at the 9.3 ppm feeding group to 0.052–0.12 in the high dose group for tetraniliprole-N-methylquinazolinone and from 0.019–0.028 at the 9.4 ppm feeding group to 0.061–0.071 mg/kg at the high dose group for tetraniliprole-benzylalcohol. The residue concentrations are summarized in Table 130.

Levels of parent compound in skim milk (0.12 mg) and cream (0.36 mg/kg) measured on day 25 (highest feeding level only) indicate that parent compound has a tendency to concentrate in fat. Metabolite tetraniliprole-N-methylquinazolinone has an even greater tendency to concentrate in fat (<0.01 mg/kg in skim milk to 0.43 mg/kg in cream), whereas the benzylalcohol metabolite seems to distribute evenly (0.056 mg/kg in skim milk and 0.060 mg/kg in cream). The ratio of the concentration of parent in muscle to fat was 1:1.3–2.1 and the ratio of tetraniliprole-N-methylquinazolinone in muscle to fat was 1: 1.1–9 in the three different fats at the three highest feeding levels, also showing the tendency of the residue to concentrate in fat. The results were similar across the three different types of fat.

Apart from one finding in kidney (0.012 mg/kg on depuration day 7 (day 36 after start of dosing), the depuration data show that only metabolite tetraniliprole-N-methylquinazolinone was still observed in

concentrations up to 0.26 mg/kg after 7 days and up to 0.015 mg/kg after 21 days after end of dosing in fat only.

Table 130 Residues in the milk of cows (means of 3 cows/dose group) dosed with tetraniliprole for 29 days at 0.94, 9.3, 28 and 94 mg/kg tetraniliprole in the diet

	Residues (mg/kg) [a]							
Sample	Tetraniliprole	T-N-MetQuin	T-BenzOH	Total [b]	Tetraniliprole	T-N-MetQuin	T-BenzOH	Total [b]
Feeding level	0.9 mg/kg DM [c]				9.3 mg/kg DM [c]			
Whole milk day 0	<0.01	<0.01	<0.01	<0.03	<0.01	<0.01	<0.01	<0.03
Day 2	<0.01	<0.01	<0.01	<0.03	0.040	0.015	0.019	0.074
Day 4	<0.01	<0.01	<0.01	<0.03	0.048	0.026	0.023	0.097
Day 7	<0.01	<0.01	<0.01	<0.03	0.051	0.032	0.022	0.11
Day 10	<0.01	<0.01	<0.01	<0.03	0.056	0.038	0.024	0.12
Day 14	<0.01	<0.01	<0.01	<0.03	0.045	0.030	0.025	0.10
Day 17	<0.01	<0.01	<0.01	<0.03	0.041	0.029	0.025	0.095
Day 21	<0.01	<0.01	<0.01	<0.03	0.040	0.029	0.023	0.092
Day 25	<0.01	<0.01	<0.01	<0.03	0.046	0.029	0.026	0.10
Day 28	<0.01	<0.01	<0.01	<0.03	0.047	0.028	0.028	0.10
Mean day 7-28	<0.01	<0.01	<0.01	<0.03	0.047	0.031	0.025	0.10
Max day 7-28	<0.01	<0.01	<0.01	<0.03	0.056	0.038	0.028	0.10
Feeding level	28 mg/kg DM [c]				94 mg/kg DM [d]			
Whole milk day 0	<0.01	<0.01	<0.01	<0.03	<0.01	<0.01	<0.01	<0.03
Day 2	0.058	0.024	0.038	0.12	0.17	0.052	0.061	0.28
Day 4	0.080	0.046	0.043	0.17	0.18	0.079	0.067	0.33
Day 7	0.087	0.063	0.045	0.20	0.19	0.097	0.071	0.36
Day 10	0.11	0.077	0.047	0.23	0.19	0.11	0.071	0.37
Day 14	0.10	0.075	0.046	0.22	0.19	0.11	0.071	0.37
Day 17	0.11	0.080	0.048	0.24	0.19	0.11	0.071	0.37
Day 21	0.10	0.077	0.044	0.22	0.18	0.12	0.068	0.37
Day 25	0.10	0.076	0.052	0.23	0.17	0.10	0.064	0.33
Day 28	0.098	0.063	0.053	0.21	0.17	0.086	0.067	0.32
Dep. 31 (+3)	-	-	-	-	0.023	0.061	0.039	0.12
Dep. 35 (+7)	-	-	-	-	<0.01	0.020	<0.01	<0.03
Dep. 42 (+14)	-	-	-	-	<0.01	<0.01	<0.01	<0.03
Dep. 49 (+21)	-	-	-	-	<0.01	<0.01	<0.01	<0.03
Mean day 7-28	0.10	0.073	0.048	0.22	0.18	0.10	0.069	0.36
Max day 7-28	0.11	0.080	0.053	0.24	0.19	0.12	0.071	0.37
Cream – day 25	-	-	-	-	0.36	0.43	0.060	0.85
Skim milk – day 25	-	-	-	-	0.12	<0.01	0.056	0.19

Notes:

T-N-MetQuin = tetraniliprole-N-methyl-quinazolinone;T-BenzOH= tetraniliprole-benzylalcohol.

[a] Expressed as tetraniliprole.

[b] Total residues is the sum of tetraniliprole, tetraniliprole-N-methylquinazolinone, and tetraniliprole-benzylalcohol, expressed as tetraniliprole. For calculation of total residues in parent equivalents no correction was made for molecular weight, since these deviate ~only 3% (MW parent = 545 g/mol, MW tetraniliprole-N-methyl-quinazolinone = 527 g/mol, MW tetraniliprole-benzylalcohol = 561 g/mol).

[c] Mean average values of 3 cows.

[d] Mean average of 6 cows, except for the depuration group, which lasted 3 cows.

Plateau levels was reached within 7–10 days after start of the study (Figure 14)

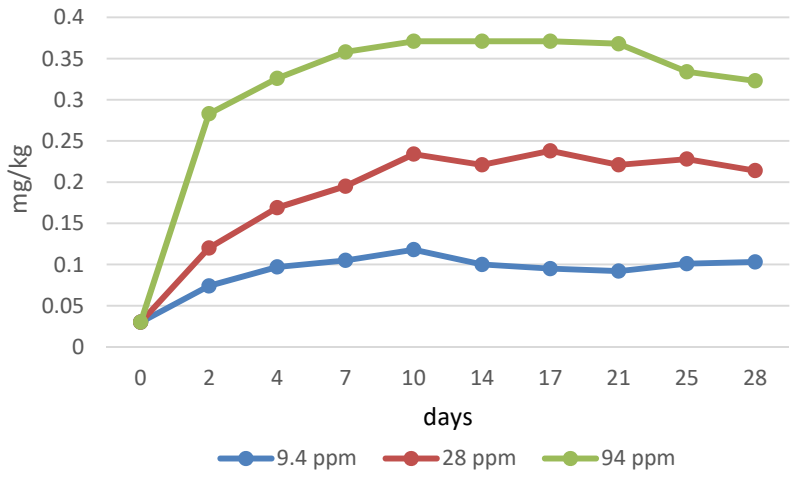


Figure 14 Residues in milk during 28 days dosing at the three feeding levels

Poultry feeding study

No poultry feeding study was submitted. A waiver in support of this was submitted (Ducas, 2016, M-574911-01-1, Report RAFVP036). Three studies on the metabolism of tetraniliprole in laying hens were conducted with the test compound radiolabelled with in the [pyrazole-carboxamide]-, the [pyridinyl-2]- or the [tetrazolyl]-position. Hens were dosed for 14 consecutive days and were fed a dose of 1 mg ai/kg bw/day which was equivalent to 18–19 mg ai/kg feed/day for the three labels, with an average dose of 18.41 mg ai/kg feed/day.

For purposes of this waiver, as a worst case assumption, the total radioactive residues (TRRs) were used to calculate transfer factors for eggs and tissues (Table 131).

Table 131 Tetraniliprole poultry egg and tissue to feed transfer factors (TRR/dose)

Label	Pyrazole Carboxamide		Pyridinyl		Tetrazolyl		Max. Transfer factor
Dose	18.64 ppm		17.94 ppm		18.66 ppm		
Matrix	TRR (mg eq/kg)	Transfer factor	TRR (mg eq/kg)	Transfer factor	TRR (mg eq/kg)	Transfer factor	
Eggs	0.084	0.00451	0.084	0.00468	0.086	0.00461	0.00468
Muscle	0.017	0.00091	0.025	0.00139	0.031	0.00166	0.00166
Fat	0.046	0.00247	0.028	0.00156	0.095	0.00509	0.00509
Liver	0.485	0.02602	0.734	0.04091	0.766	0.04105	0.04105

There are several primary and rotational crops that may be included as potential poultry feed items that have finite residues of tetraniliprole: soybean seed, cotton seed, corn grain and milled by-products, and alfalfa meal. The authors used the median values from the residue trials for seed, grains and other blended commodities (soybean, seed, cotton seed and corn grain) and the highest average field trials residues was used for alfalfa meal to construct an assumed maximum reasonably dietary burden of 0.0159 ppm. Using this dietary burden and the maximum tissue to feed transfer ratio (Max Tfm), the anticipated secondary residues in poultry tissues and eggs were calculated at 1× and 10× the dietary burden (Table 132).

Table 132 Estimation of residues in poultry tissues from use of tetraniliprole

Commodity	Max. transfer factor (Tfm)	Dietary burden (ppm)	1× Expected residue (mg/kg) [a]	10× Expected residue (mg/kg) [a]
Eggs	0.00468	0.0159	0.0000742	0.000742
Muscle	0.00166	0.0159	0.0000263	0.000263
Fat	0.00509	0.0159	0.0000807	0.000807
Liver	0.04105	0.0159	0.0006506	0.006506

Note:

[a] Expected residue = Tf_m × Dietary burden (ppm).

From the calculations presented above, it is expected that measurable, finite residues greater than 0.01 mg/kg will not be observed in poultry tissues or eggs even when the diet contains up to ten times the anticipated total tetraniliprole-derived dietary burden. Based on these data, the metabolism studies already conducted are sufficient to describe the magnitude of the residue in poultry tissues and eggs and a conventional poultry feeding study will not be required.

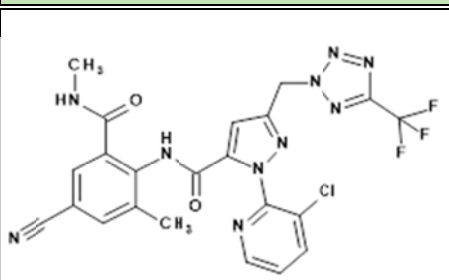
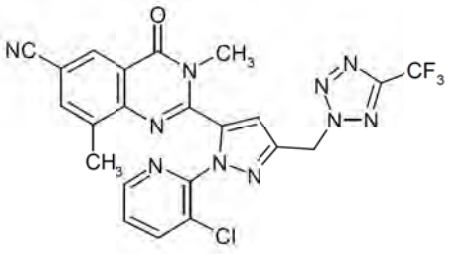
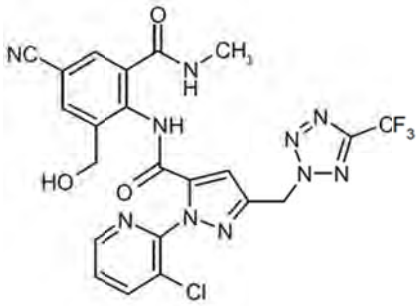
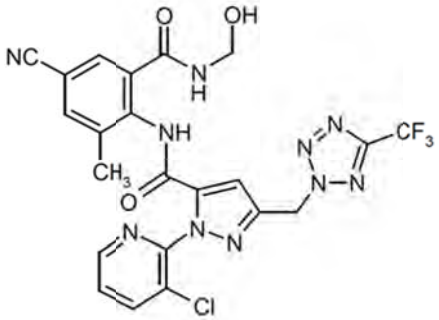
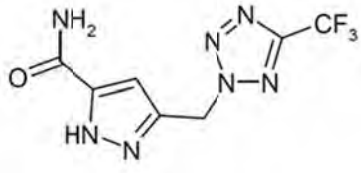
APPRAISAL

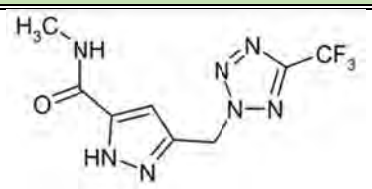
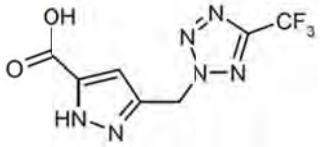
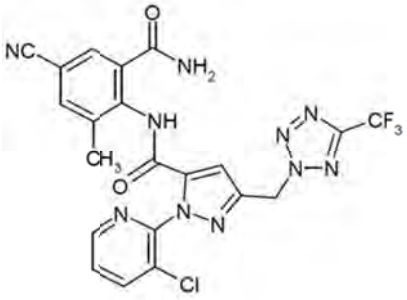
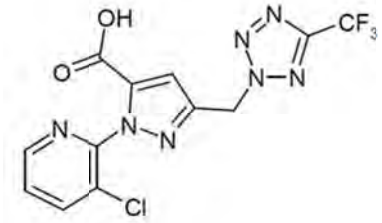
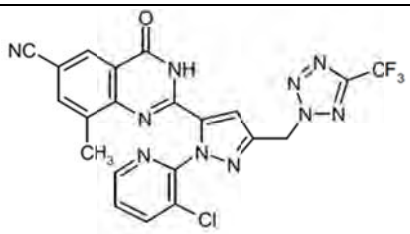
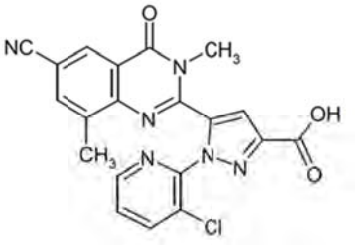
Tetraniliprole (ISO name) is a new broad spectrum fast acting insecticide. The IUPAC name for tetraniliprole is 1-(3-chloropyridin-2-yl)-*N*-[4-cyano-2-methyl-6-(methylcarbamoyl) phenyl]-3-{[5-(trifluoromethyl)-2*H*-tetrazol-2-yl]methyl}-1*H*-pyrazole-5-carboxamide. Tetraniliprole belongs to the anthranilic diamide chemical class. The mode of action of anthranilic diamides involves activating ryanodine receptors (RyRs), which play a critical role in muscle function.

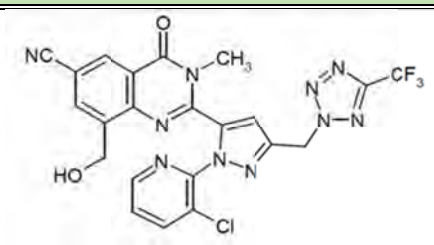
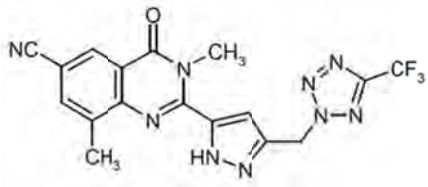
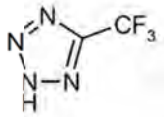
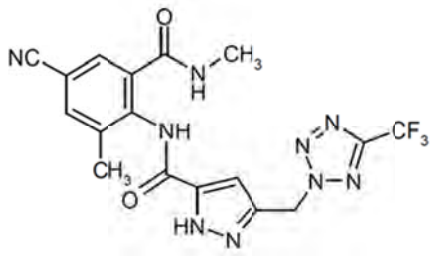
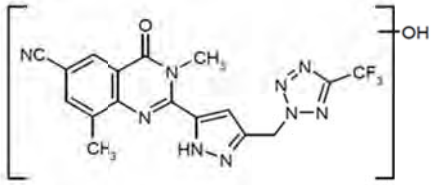
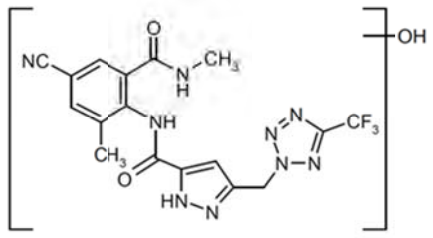
Tetraniliprole was scheduled at the Fifty-first Session of the CCPR (2019) for evaluation as a new compound by the 2020 JMPR, which was postponed to the 2021 JMPR for toxicology and to the 2022 JMPR for residues. The 2021 JMPR estimated an ADI of 0–2 mg/kg bw and concluded that an ARfD was not necessary.

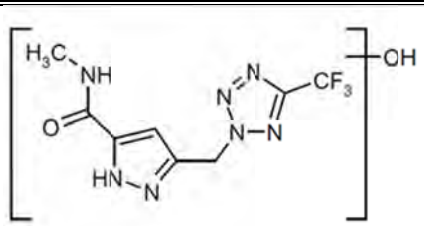
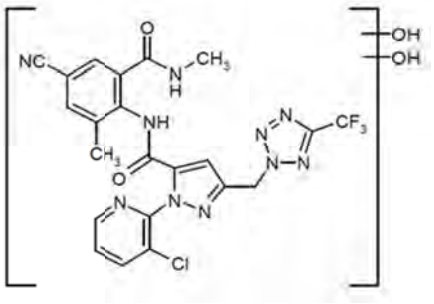
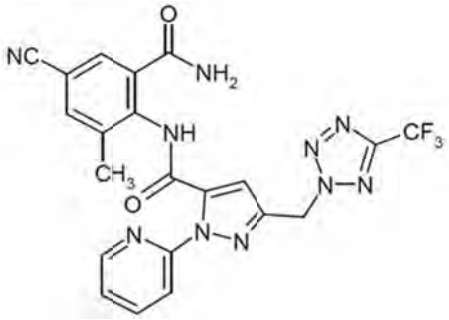
The Meeting received information on identity, physical chemical properties, plant and animal metabolism, aerobic soil degradation, residue analysis, storage stability, use patterns, residues resulting from supervised trials on citrus fruits, pome fruits, stone fruits, grapes, head and stem brassica vegetables, fruiting vegetables, leafy vegetables, soya beans, root and tuber vegetables, cereal grains, tree nuts, and rice, fate of residues during processing, and livestock feeding studies.

Table 133 The structure of tetraniliprole and of the major metabolites are shown below

Name	Abbreviation	Structure
Tetraniliprole	Tetraniliprole	
Tetraniliprole-N-methyl-quinazolinone	T-N-methyl-quinazolinone T-N-Met-quinazolinone	
Tetraniliprole-benzylalcohol	T-benzyl-alcohol	
Tetraniliprole-hydroxy-N-methyl	T-hydroxy-N-methyl T-OH-N-Met	
Tetraniliprole-pyrazole-5-amide	T-pyrazole-5-amide	

Name	Abbreviation	Structure
Tetraniliprole-pyrazole-5-N-methyl-amide	T-pyrazole-5-N-methyl-amide	
Tetraniliprole-pyrazole-5-carboxylic acid	T-pyrazole-5-carboxylic acid	
Tetraniliprole-desmethyl-amide	T-desmethyl-amide T-DesMet-amide	
Tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid	T-pyridinyl-pyrazole-5-carboxylic acid	
Tetraniliprole-quinazolinone	T-quinazolinone	
Tetraniliprole-N-methyl-quinazolinone-pyrazole-3-carboxylic acid	T-N-methyl-quinazolinone-pyrazole-3-carboxylic acid	

Name	Abbreviation	Structure
Tetraniliprole-N-methyl-quinazolinone-benzylalcohol	T-N-methyl-quinazolinone-benzylalcohol	
Tetraniliprole-despyridyl-N-methyl-quinazolinone	T-despyridyl-N-methyl-quinazolinone	
Tetrazole (conjugates)	Tetrazole (conjugates)	 -conjugate
Tetraniliprole-despyridyl	T-despyridyl	
Tetraniliprole-despyridyl-N-methyl-quinazolinone-hydroxy	T-despyridyl-N-methyl-quinazolinone-hydroxy	
Tetraniliprole-despryridyl-hydroxy	T-despyridyl-hydroxy	
Tetraniliprole-despyridyl-quinazolinone	T-despyridyl-quinazolinone	

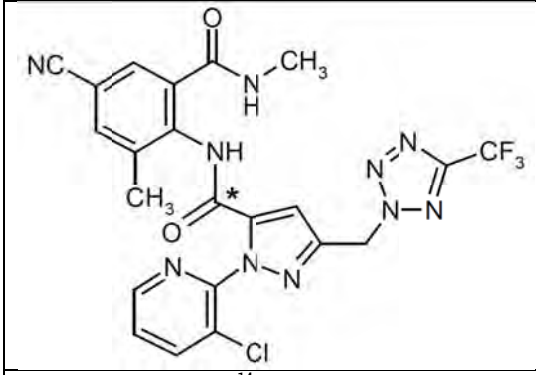
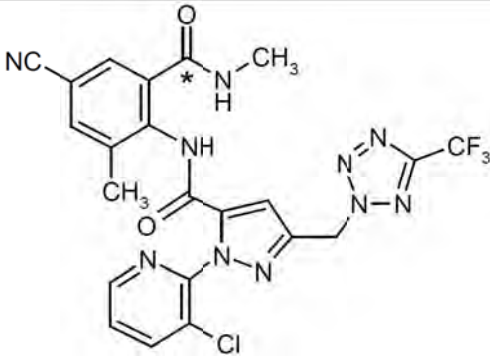
Name	Abbreviation	Structure
Tetraniliprole-pyrazole-5-N-methylamide-hydroxy	T-pyrazole-5-N-methylamide-hydroxy	
Tetraniliprole-dihydroxy	T-dihydroxy	
Tetraniliprole-deschloro-desmethyl-amide	T-deschloro-desmethyl-amide	

Physical and chemical properties

Tetraniliprole is not volatile (3.2 × 10-6 mPa at 20 °C), relatively insoluble in water (1.0 mg/L pH4 and 7), but appears to be more soluble in organic solvents (up to 21.8 g/L in acetone). It is hydrolytically stable at environmental conditions (pH4 and 20 °C), but not at higher temperatures and higher pH. Aqueous photolysis is a significant route of degradation. The octanol/water partition coefficient log P_{ow} of 2.6 suggests a slight potential to partition into fat.

Plant metabolism

The Meeting received plant metabolism studies for tetraniliprole after different types of application on apples (foliar application), tomatoes (soil drench application), potatoes (foliar applications or seed treatment in furrow), lettuce (foliar application), paddy rice (foliar application or granular in planting hole treatment) and maize (seed treatment) using [pyrazole-carboxamide-^{14}C]- and/or [phenyl-carbamoyl-^{14}C]-labelled tetraniliprole, as shown below. The results for both labels are presented as pyrazole-carboxamide/phenyl-carbamoyl, unless indicated otherwise.

Pyrazole-carboxamide label	Phenyl-carbamoyl label
	
[pyrazole-carboxamide-^{14}C]-tetraniliprole	[phenyl-carbamoyl-^{14}C]-tetraniliprole

Seed treatments and soil treatments (soil drench, in-furrow, granular)

Tomato–soil drench application (indoor)

Pyrazole-carboxamide or phenyl-carbamoyl labelled tetraniliprole was applied to greenhouse grown tomato plants, as a single soil drench application at BBCH stage 15–16. The application rate was equivalent to 156/153 g ai/ha (based on 20,000 plants/ha; 7.65–7.81 mg ai/plant). Soil characteristics were not reported.

In fruits and leaves collected at 83–99 days after treatment (DAT), total radioactive residue (TRR) were < 0.001 mg eq/kg (both labels) in fruits and 0.005/0.006 mg eq/kg in leaves indicating limited uptake from soil. Despite the limited amount of residue, the extraction efficiency with acetonitrile/water/formic acid seems to be high (> 90/> 86 percent TRR) for both fruit and leaf samples, with the main portion on partitioning detected in the organic phase 68/56 percent TRR in fruit and 76/78 percent TRR in leaves. The unextracted radioactivity was 9.3/13.5 percent TRR in fruits and 6.1/5.6 percent TRR in leaves (all < 0.001 mg eq/kg).

A total of 33/54 percent TRR and 58/64 percent TRR was identified in tomato fruit and leaves, respectively. The predominant radioactive residues in fruits and leaves were parent tetraniliprole accounting for 22/34 percent TRR in fruit and 24/27 percent TRR in leaves and tetraniliprole-N-methyl-quinazolinone at 11/20 percent TRR in fruits and 34/37 percent TRR in leaves. Apart from two unknown metabolites (15 percent TRR each) in fruit, the radioactivity was not further characterised (pyrazole-carboxamide label only). In leaves, six unknown metabolites were characterised, all less than 4.5 percent TRR and < 0.001 mg eq/kg (total 17.5/13.5 percent TRR and < 0.001 mg eq/kg).

Potato – seed treatment in furrow (outdoor)

Pyrazole-carboxamide labelled tetraniliprole was applied to potato tubers in the furrow at planting before covering the seeds with sandy loam soil. The actual application rate corresponded to 200 g ai/ha and potatoes were grown outdoors. Tubers were collected at 151 DAT (BBCH 99).

TRR amounted to 0.001 mg eq/kg indicating limited uptake from soil. No further investigation of the residues was performed.

Paddy rice–granular soil application (indoor)

Rice seedlings (BBCH 13–14, three to four leaves unfolded) were transplanted into containers filled with sandy loam soil treated with granular pyrazole-carboxamide or phenyl-carbamoyl labelled tetraniliprole at

205/211 g ai/ha. Rice forage (BBCH 34–35, 64 DAT), grain (husked rice), husks and straw (BBCH 89–92, 150 DAT) were collected for analysis.

Radioactive residues were low in husked rice, forage, husks and straw, totaling 0.003/0.004, 0.011/0.08, 0.026/0.018, 0.098/0.069 mg eq/kg, in each matrix respectively. The extraction efficiency with acetonitrile/water/formic acid ranged from 76/49 percent TRR in husked rice, to 91/91 percent TRR in straw. The unextracted radioactivity was highest in husked rice with 24/51 percent TRR (0.001/0.002 mg eq/kg) and ranged from 9.0 to 15 percent TRR (0.001–0.004 mg eq/kg) in straw, forage, and husks.

A total of 58/28 percent TRR (0.001/0.001 mg eq/kg) was identified in husked rice and ≥ 78 percent TRR (both labels) in the other matrices. The majority of the identified radioactivity was parent tetraniliprole (48/22 percent TRR, 0.001/0.001 mg eq/kg) in husked rice, accounting for ≥ 77 percent TRR (0.007–0.075 mg eq/kg) with both labels in the other matrices. A minor part of the radioactivity could be attributed to tetraniliprole-N-methyl-quinazolinone (3.9–14 percent TRR, < 0.001–0.014 mg eq/kg across all matrices). With the exception of one unknown in husked rice (17/21 percent TRR, 0.001/0.001 mg eq/kg), all other unknown metabolites were ≤ 4.6 percent TRR and ≤0.001 mg eq/kg.

Maize – seed treatment (outdoor)

Pyrazole-carboxamide labelled tetraniliprole was used as a seed treatment to maize grown outdoors in a sandy loam soil. Two different application rates were tested, corresponding to 63 or 150 g ai/ha. Maize forage was harvested at 98 DAT (BBCH 79–83) and mature plants at 145 DAT (BBCH 89).

Radioactive residues after both treatments were low in kernels (< 0.001 mg eq/kg) and forage (< 0.006 mg eq/kg) and amounted to 0.004 mg eq/kg (63 g ai/ha treatment) to 0.008 mg eq/kg (150 g ai/ha treatment) in fodder. Only the higher treatment fodder was subjected to extraction with acetonitrile/water/formic acid, with 76 percent TRR (0.006 mg eq/kg) extracted. The unextracted radioactivity was 24 percent TRR (0.002 mg eq/kg).

The extracted residues in fodder were identified as parent tetraniliprole and tetraniliprole-N-methyl-quinazolinone, representing 26 percent TRR (0.002 mg eq/kg) and 17 percent TRR (0.001 mg eq/kg), respectively. All other metabolites represented ≤0.001 mg eq/kg.

Apple–foliar application (indoor)

Pyrazole-carboxamide or phenyl-carbamoyl labelled tetraniliprole was applied to greenhouse grown apples trees, with two foliar spray applications at fruit development (BBCH stage 71 and 73) at a rate of 85–88 g ai/ha per application.

At 64 (fruit) and 66 (leaves) days after application, TRR were 0.18/0.52 mg eq/kg in apple fruits and 99 mg eq/kg in apple leaves. A fruit surface wash with dichloromethane released 92/97 percent TRR, indicating that tetraniliprole residues remained mainly on the surface of the apple fruits. The surface wash with dichloromethane and extraction with acetonitrile/water/formic acid released most of the radioactivity (> 99.5 percent TRR) for both labels in fruit and leaf samples.

Greater than 99 percent TRR could be identified in apple fruit and leaves with both labels. The only identified radioactive residue in fruits and leaves was parent tetraniliprole, accounting for 99.2/99.3 percent TRR in fruit and 98.6 percent TRR in leaves. Two to six minor metabolites were found in fruit and leaves, but none exceeded 0.3 percent TRR (< 0.001 mg eq/kg in fruits and 0.051–0.32 mg eq/kg in leaves).

Lettuce–foliar application (indoor)

Pyrazole-carboxamide or phenyl-carbamoyl labelled tetraniliprole was applied to greenhouse grown lettuce plants, with two foliar spray applications at BBCH stage 44/45. The application rate was 2 × 59–60 g ai/ha, with a retreatment interval of 7 days.

Lettuce was collected at BBCH 49, 7 days after the last application. Total radioactive residues were 4.1 mg eq/kg in lettuce with both labels. Extraction with acetonitrile/water/formic acid released a high level of radioactivity (> 99 percent TRR) with both labels.

A total of 99.5/99.1 percent TRR was identified as parent tetraniliprole. No metabolites were found.

Potato–foliar application (indoor)

Pyrazole-carboxamide or phenyl-carbamoyl labelled tetraniliprole was applied to greenhouse grown potato plants, with two applications at BBCH 38 and 97–99and rates ranging from 101–105 g ai/ha, with a retreatment interval of 49 days. TRR were low at 0.001 mg eq/kg in potato tubers collected at 14 DAT. Extracted radioactivity of potato tubers with acetonitrile/water was high (79/75 percent TRR), and unextracted radioactivity was 21/25 percent TRR (equivalent with < 0.001 mg eq/kg).

A total of 38/55 percent TRR (both labels) could be identified (< 0.001 mg eq/kg). Tetraniliprole was the major component (29/42 percent TRR), followed by tetraniliprole-N-methyl-quinazolinone (9.0/13 percent TRR). All other metabolites represented < 0.001 mg eq/kg and, apart from one unknown (11.8 percent TRR), all were ≤ 5.3 percent TRR.

Paddy rice–foliar application (indoor)

Pyrazole-carboxamide or phenyl-carbamoyl labelled tetraniliprole was applied to greenhouse grown paddy rice, with two foliar spray applications of 50–52 g ai/ha each with an interval of 42 days, at BBCH 14 and BBCH 73–77 (early to late milk stage). Rice forage was harvested at 13 DAT (BBCH 34–35) and mature rice plants at 56 DAT (BBCH 89–92).

TRRs in rice kernels, forage, husks and straw amounted 0.040/0.024, 1.3/2.6, 2.5/2.1, 4.3/4.6 mg eq/kg, respectively. Extraction with acetonitrile/water/formic acid released high levels of radioactivity (≥ 93 percent TRR) for all matrices and with both labels. The unextracted radioactivity ranged from 0.4–1.7 percent TRR (equivalent with 0.015–0.022 mg eq/kg) in forage, husks and straw and was 6.3/7.3 percent TRR (equivalent with 0.002 mg eq/kg) in kernels.

A total of ≥ 92.7 percent TRR (both labels) was identified. The majority of the identified radioactivity was parent tetraniliprole (≥ 91 percent TRR with both labels in all matrices). A minor contribution to the total radioactivity was attributed to tetraniliprole-N-methyl-quinazolinone (0.7–3.7 percent TRR, < 0.001–0.151 mg eq/kg). All other metabolites represented ≤ 0.8 percent TRR and ≤ 0.027 mg eq/kg.

Summary of plant metabolism

Tetraniliprole is only marginally taken up from the soil and translocated to other parts of the plants after seed treatment or soil applications (drench or granular). In foliar applications, the majority of the residue remains on the surface, and very limited metabolism in apples or lettuce (indoor foliar applications) was observed. The metabolic pathway involves cyclisation in the parent molecule leading to tetraniliprole-N-methyl-quinazolinone, with highest levels in tomato fruits (up to 20 percent TRR but < 0.001 mg eq/kg)

and tomato leaves (37 percent TRR, 0.002 mg eq//kg). This metabolite was also found in rat studies. Parent tetraniliprole was the major residue found in all crops.

Apart from one outdoor study in maize and another in potatoes (seed treatment in furrow), all studies were conducted indoor. The Meeting concluded that the indoor studies sufficiently cover outdoor uses (see photochemical degradation section).

Environmental fate

The Meeting received information on hydrolytic stability, photochemical degradation in water and soil, and aerobic soil metabolism studies for tetraniliprole. Soil degradation field studies were also provided.

Aqueous hydrolysis

Radiolabelled (pyrazole-carboxamide) tetraniliprole was incubated in the dark in sterile aqueous buffered solutions at pH 4, 7, and 9 for 30 days at 20, 25 and 50 °C. The results show that the DT_{50} of tetraniliprole depends both on the pH and temperature. Where tetraniliprole slowly degrades at 20 and 25 °C and pH 4 (265–287 days), the DT_{50} decreases with high pH to 58/39 days at pH 7 and 1.27 and 0.75 days at pH 9 at normal temperatures (20 and 25 °C). At higher temperatures the DT_{50} decreases to 10.9 days at pH4 and to 3.74 and 0.04 days at pH 7 and 9, respectively.

One degradation product was identified as tetraniliprole-N-methyl-quinazolinone with a maximum amount of 99.6 percent of applied radioactivity (AR) (at DAT-30; pH 9, 20 °C). The Meeting concluded that hydrolysis is likely to be a minor path of degradation for tetraniliprole under environmental conditions.

Photochemical degradation

The photolysis in sterile water of pyrazole-carboxamide labelled tetraniliprole was studied under simulated sunlight in sterile aqueous acetate buffer (pH 4, 25 °C) with a DT_{50} of 3.4 days, equivalent to 10.5 summer days in Arizona (approximately 34° latitude). Tetraniliprole was stable under dark conditions (DT_{50} of 188.5 days). The major degradation product was tetraniliprole-deschloro-oxazine.

In two other studies, pyrazole-carboxamide or phenyl-carbamoyl labelled tetraniliprole was incubated in sterile natural waters (pH 8.0–8.5) at 25 °C under dark or simulated sunlight for 10–11 days. Tetraniliprole was readily degraded, with an estimated DT_{50} of 0.7–0.8 days, similar to the non-irradiated (dark) samples (DT_{50} of 0.3–0.8 days). Degradation product tetraniliprole-N-methyl-quinazolinone was the only compound observed in the dark samples (up to 92–99 percent after 10–11 days), whereas it reached levels up to max 39 percent AR (after 1 day) in the irradiated samples. In the irradiated samples other degradation products were also observed; tetraniliprole-deschloro-pyrazine (up to 37–39 percent AR at day 2), tetraniliprole -despyridyl-N-methyl-quinazolinone (up to 7 percent AR), tetraniliprole-deschloro-pyrazine (up to 27 percent AR at day 2) and tetraniliprole-pyrazole-5-carboxylic acid up to 18 percent AR at day 10).

The Meeting concluded that aqueous photolysis is a significant route of degradation, which was similar for dark and irradiated samples but the metabolic profile differed significantly.

In a soil photodegradation study, [pyrazole-carboxamide-^{14}C]-tetraniliprole was applied to a thin layer of silt loam soil (at a rate equivalent to about 200 g ai/ha). Tetraniliprole was slightly photolysed, decreasing from 93 percent AR (day 1) to 77 percent AR (day 11). Tetraniliprole-N-methyl-quinazolinone was the degradation product observed. The estimated photolysis DT_{50} was 27 days, equivalent to 82 summer days in Arizona. The Meeting concluded that photodegradation is, at the most, only a minor path of degradation in soil.

In summary, aqueous photolysis and hydrolysis at higher pHs are significant routes of degradation, although it was not possible to distinguish between the effects caused by pH or by photolysis. Residue decline trials performed outdoor in apples (at 0–14 DAT) and rice straw (at 38–53 DAT) show that tetraniliprole is stable over > 14 days. The Meeting concluded that photolysis does not play an important role in degradation of the tetraniliprole on outdoor crops, including seed treatments and foliar treatments (sprayed on the leaves) in paddy rice.

Aerobic soil metabolism

The biotransformation of [pyrazole-carboxamide-^{14}C]-tetraniliprole was investigated in four German soils and in six United States soils under laboratory conditions. The equivalent of 200 g tetraniliprole/ha was mixed with soil and incubated under aerobic conditions in the dark at 20 °C for 119–120 days.

The two main degradation products were tetraniliprole-carboxylic acid (up to 48 percent AR) and tetraniliprole-N-methyl-quinazolinone (up to 33 percent AR). Additionally, tetraniliprole-quinazolinone-carboxylic acid (up to 6.5 percent AR), tetraniliprole-amide (up to 6.96 percent AR), tetraniliprole-desmethyl-amide-carboxylic acid (up to 12 percent AR), and tetraniliprole-N-methyl-quinazolinone-carboxylic acid (up to 11 percent AR) were found, but each not exceeding 12 percent AR at any sampling interval. The estimated DT_{50} for tetraniliprole ranged from 18 to 183 days.

The biotransformation of [pyrazole-carboxamide-^{14}C]-tetraniliprole was investigated in an Italian paddy soil under laboratory conditions. The equivalent of 200 g tetraniliprole/ha was mixed with soil and water and incubated under anaerobic/aerobic conditions in the dark at 25 °C for 181 days.

Tetraniliprole-N-methyl-quinazolinone was the only identified degradation product (maximum 48 percent AR at DAT-140). Other unidentified residues amounted to a total maximum of 6.3 percent AR, with no single component exceeding 3.6 percent AR at any sampling interval. The estimated DT_{50} for tetraniliprole was 4.4 days in water and 84 days in the entire soil/water system.

The Meeting concluded that, under laboratory conditions, tetraniliprole is moderately persistent to persistent in soil and soil/water systems.

Rotational crop metabolism

The Meeting received information on the metabolism of tetraniliprole in wheat, turnip and Swiss chard grown as confined rotational crops and in a range of representative field crops grown in tetraniliprole treated soil.

Confined rotational crop studies

In two confined rotational crop studies, soil was treated with either [phenyl-carbamoyl-^{14}C]- or pyrazole-carboxamide label at 213/209 g ai/ha, and planted with wheat, turnip, and Swiss chard at plant-back intervals (PBI) of 30, 168, and 286 days. The TRR in the different RACs were generally low and decreased significantly from the 1st to the 3rd PBI. Residues in wheat matrices were generally higher, with exception of grain (no detected residues). Residues declined from 0.060/0.057 to 0.007/0.014 mg eq/kg in forage, from 0.16/0.21 to 0.028/0.064 mg eq/kg in hay and from 0.12/0.26 mg eq/kg to 0.035/0.110 mg eq/kg in straw. Residues in mature Swiss chard declined from 0.047/0.052 mg eq/kg at PBI of 30 days to 0.008/0.016 mg eq/kg at of PBI 286 days. In turnip leaves and turnip roots residue levels were even lower, ranging from 0.001 to 0.008 mg eq/kg, regardless of PBI.

Samples were extracted with acetonitrile/water/formic acid, with extraction efficiency ranging from 77 to 99 percent TRR with both labels. Post-extraction solids (PES) of wheat hay from the 1st and 2nd rotation and straw from the 1st rotation were exhaustively extracted using microwave assistance

with a mixture of acetonitrile/water and formic acid and, in case of wheat hay of the 2nd rotation, subsequently with 0.1 mol/L hydrochloric acid. The residues in the acetonitrile/water mixture were further characterised by partitioning against ethyl acetate. The radioactive residues in the organic and aqueous phases amounted to ≤ 0.010/≤ 0.016 mg eq/kg with the respective labels (6.4–15 percent TRR with both labels).

The predominant residue in all matrices at all PBIs was parent tetraniliprole, ranging from 40–88 percent TRR (0.003–0.15 mg eq/kg) at PBI of 30 days to 8.8–52 percent TRR (0.001–0.017 mg eq/kg) at PBI of 286.

In food commodities, tetraniliprole-carboxylic acid contributes significantly to the total residue (up to 28 percent TRR, 0.004 mg eq/kg) at 168 days PBI in Swiss chard and to 18 percent TRR (< 0.001 mg eq/kg) in turnip roots at 30 days PBI. The same applies for tetraniliprole-dihydroxy (up to 30 percent TRR, 0.005 mg eq/kg) in mature Swiss chard at 286 days PBI, but the metabolite was not found in roots. Tetraniliprole-desmethyl-amide-carboxylic-acid was found at 31 percent TRR in Swiss chard at 286 days PBI and at 18 percent TRR (< 0.001 mg eq/kg) in turnip roots at 30 days PBI. Tetraniliprole-N-methyl-quinazolinone contributed up to 14 percent TRR (0.002 mg eq/kg) in immature Swiss chard, 9.0 percent TRR (0.001 mg eq/kg) in mature Swiss chard and up to 16 percent TRR (0.001 mg eq/kg) in turnip roots (30 days PBI). Finally, tetraniliprole-despyridyl-N-methyl-quinazolinone-pyrazole-3-carboxylic acid was observed in Swiss chard at 2.9–16 percent TRR (< 0.001–0.008 mg eq /kg), but was not found in turnip roots

In feed matrices (wheat forage, hay, straw, and turnip leaves) metabolite tetraniliprole-N-methyl-quinazolinone accounted for 0.001 to 0.028 mg eq/kg (1.8–22 percent TRR), tetraniliprole-dihydroxy for 0.001 to 0.024 mg eq/kg (1.6–22 percent TRR), tetraniliprole-desmethyl-amide-carboxylic acid for 0.002–0.021 mg eq/kg (2.4–24 percent TRR), tetraniliprole-carboxylic acid for 0.001 to 0.011 mg eq/kg (2.7 to 17 percent TRR), and tetraniliprole-amide for 0.001 to 0.011 mg eq/kg (2.7–7.1 percent TRR).

In summary, residues in confined rotational crops were lower in grains and roots than in foliage and decreased slowly over time. The residues consisted mainly of parent tetraniliprole. Metabolites tetraniliprole-N-methyl-quinazolinone, tetraniliprole-dihydroxy, tetraniliprole-desmethyl-amide-carboxylic acid, tetraniliprole-carboxylic acid, and tetraniliprole-amide may also occur in significant proportions when considering percent TRR, but at very low absolute concentrations (< 0.001–0.008 mg eq/kg in food commodities and 0.001–0.021 mg eq/kg in feed commodities).

Field rotational crop studies

In a series of field rotational crop studies conducted in the United States, tetraniliprole was applied as one broadcast application to bare soil at a rate of 170–220 g ai/ha, which is similar to the most critical total maximum seasonal GAP rate of 200 g ai/ha for primary crops. Onions (11 trials), peas with pods (seven trials), beans with pods (nine trials), peas without pods (eight trials), beans without pods (eight trials), dried peas (seven trials), dried beans (nine trials), melons (10 trials), summer squash (nine trials), cucumber (eight trials), wheat (12 trials), barley (nine trials), sorghum (seven trials), alfalfa (11 trials), rapeseed (seven trials), and sunflowers (six trials) were planted at PBI of 25–31 days. Longer PBIs were not included for the food commodity plants, since no quantified residues were observed in food commodities and the confined rotational crop studies indicated that residues declined over time.

In one study, wheat (three trials) and soya bean (three trials) were planted after one application either to a target crop (potatoes) or to bare soil at PBI of 22–29 days, 108–119 days and 334–365 days. In the trials that made applications to potatoes, the potatoes were grown to maturity or until the time of rotational crop planting. For the 4-month and 12-month PBI plots, potatoes were harvested after

approximately 4 months DAT or at maturity, respectively; for the 1-month PBI plots, potato plants were disked or tilled into the plot. Samples of mature commodities were analysed for tetraniliprole and tetraniliprole-N-methyl-quinazolinone.

Considering all twelve field rotational crop studies, the residues in food commodities of the rotational crops with a 30 days PBI were all below the LOQ of < 0.01 mg/kg. Quantified residues were observed in feed commodities at PBIs up to 365 days. Median and highest residues of tetraniliprole and tetraniliprole-N-methyl-quinazolinone and total residues at any PBI (but mostly up to 3 months) are summarised below. Note that the total residues only includes the metabolite if the residue level was ≥ 0.01 mg/kg.

Table 142 Median and highest residues of tetraniliprole and tetraniliprole-N-methyl-quinazolinone and total residues at any PBI

Matrix	N	Tetraniliprole		Tetraniliprole-N-methyl-quinazolinone		Total [a]	
		max (mg/kg)	median (mg/kg)	max (mg eq/kg)	median (mg eq/kg)	max (mg/kg)	median (mg/kg)
Pulses forage [c]	25	0.056 [b]	< 0.01	< 0.01	< 0.01	0.056	< 0.01
Pulses hay [c]	25	0.19 [b]	< 0.01	0.026	< 0.01	0.22 [b]	< 0.01
Wheat forage [d]	21	0.030 [b]	< 0.01	< 0.01	< 0.01	0.030	< 0.01
Sorghum forage [e]	7	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Wheat/barley hay [f]	30	0.035 [b]	< 0.01	< 0.01	< 0.01	0.035 [b]	< 0.01
Sorghum hay/fodder dry [e]	7	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Wheat/barley straw [f]	30	0.097 [b]	< 0.01	0.038 [b]	< 0.01	0.14 [b]	< 0.01
Alfalfa forage (3 cuttings) [g]	33	0.013	< 0.01	< 0.01	< 0.01	0.013	< 0.01
Alfalfa hay (3 cuttings)	33	0.051 [b]	< 0.01	0.015 [b]	< 0.01	0.066 [b]	< 0.01

Notes:

[a] Total residue expressed as tetraniliprole.

[b] Single highest value.

[c] Based on beans, peas and soya bean data also representative for trefoil, vetch, clover, and lespedeza; forage also representative for silage and vines.

[d] Based on wheat data and also representative for barley, oat, rye, triticale; forage is also representative for silage.

[e] Based on sorghum data, also representative for millet; forage also representative for silage.

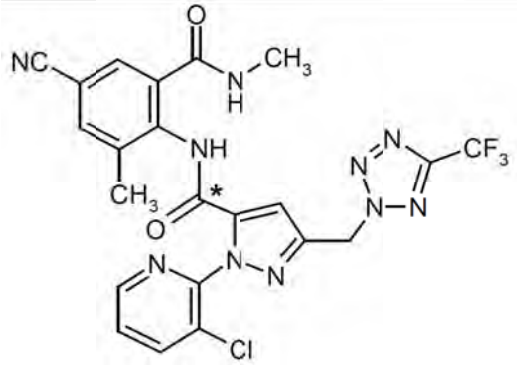
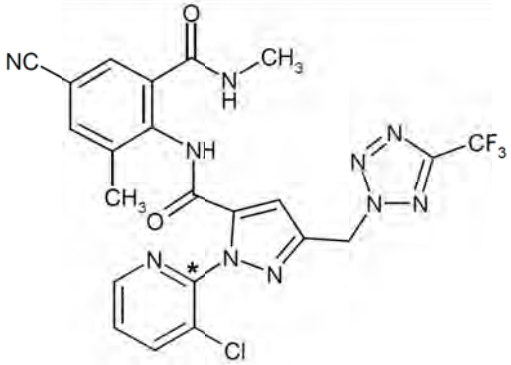
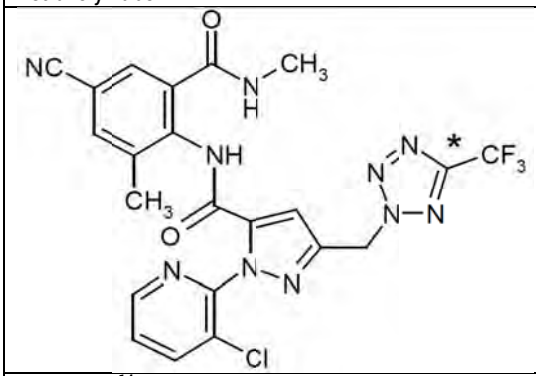
[f] Based on wheat and barley data, also representative for oat, rye and triticale.

[g] Forage is also representative for alfalfa silage.

The Meeting concluded that maximum residue levels should be estimated for crops that might be planted in rotation, for which no primary uses are in place (see section on residues from rotational crops to consider for animal feeds).

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens, where animals were dosed with tetraniliprole radiolabelled at the pyrazole-carboxamide site, the pyridinyl-ring, or the tetrazolyl-ring.

Pyrazole-carboxamide label	Pyridinyl label
	
[pyrazole-carboxamide-^{14}C]-tetraniliprole	[pyridinyl-2-^{14}C]-tetraniliprole
Tetrazolyl label	
	
[tetrazolyl-^{14}C]-tetraniliprole	

Rats

The metabolism of tetraniliprole in rats was reviewed in the framework of the toxicological evaluation by the WHO Core Assessment Group of the 2020 JMPR.

Lactating goats

In a series of three separate studies, lactating goats (one per study) were dosed orally by capsule once each morning for 5 consecutive days after milking with [pyrazole-carboxamide-^{14}C]-tetraniliprole, [pyridinyl-2-^{14}C]-tetraniliprole, or [tetrazolyl-^{14}C]-tetraniliprole at dose of a ca 1 mg/kg bw/day, corresponding to a rate of 27, 21 or 38 ppm dry feed for each label, respectively. The goats were sacrificed 5.5 to 6 hours after the last dose. The quantitative results of the three studies are reported without correction for the differences in dose levels.

Most of the radioactivity was excreted in the faeces (61–69 percent of total applied radioactivity, TAR) and urine (20–3.3 percent TAR), 1.1–1.3 percent TAR (0.24–0.42 mg eq/kg) in milk and 1.8–2.4 percent TAR in tissues, with approximately 26–32 percent TAR likely to be still present in the gastro-intestinal tract.

The highest levels of radioactivity were found in liver (0.42–0.57 percent TAR, 0.88–1.2 mg eq/kg) and fat (0.89–1.4 percent TAR, 0.39–0.60 mg eq/kg), followed by kidney (0.020–0.10 percent TAR, 0.24–0.33 mg eq/kg) and muscle (0.49–0.72 percent TAR, 0.086–0.12 mg eq/kg). The milk samples contained higher residues and reached a plateau (0.28–0.43 mg eq/kg) at approximately 60–96 hours after the first dose.

Using acetonitrile/water mixture, residues were efficiently extracted from milk and tissues (89–100 percent TRR). For liver, an additional 8.8–11 percent TRR was released using microwave assisted extraction. Low amounts of radioactivity (≤ 0.7–1.0 percent TRR) remained in the PES of all commodities.

Parent tetraniliprole was the predominant residue in milk (64–70 percent TRR, 0.16–0.27 mg/kg), muscle (65–66 percent TRR, 0.057–0.064 mg/kg), liver (55–62 percent TRR, 0.54–0.55 mg/kg), and kidney (69–71 percent TRR, 0.17–0.18 mg/kg) and was a major component in fat (24–28 percent TRR, 0.094–0.16 mg /kg). Tetraniliprole-N-methyl-quinazolinone was the main metabolite in fat (67–72 percent TRR, 0.28–0.40 mg eq/kg) and muscle (28 percent TRR, 0.024–0.028 mg eq/kg). It was also a found in milk (11–13 percent TRR, < 0.026–0.056 mg eq/kg) and kidney (13–14 percent TRR, 0.033–0.044 mg eq/kg), and to a minor extent in liver (2.2–5.6 percent TRR, < 0.036–0.067 mg eq/kg) when using the pyridinyl or tetrazolyl-label.

Another prominent metabolite was tetraniliprole-benzylalcohol, with 9.0–11 percent TRR (0.022–0.045 mg eq/kg) in milk, 6.9–8.9 percent TRR (0.060–0.11 mg eq/kg) in liver and 3.6–6.2 percent TRR (0.009–0.020 mg eq/kg) in kidney. Metabolite tetraniliprole-hydroxy-N-methyl was found in milk (3.5–5.0 percent TRR, 0.008–0.019 mg eq/kg), liver (6.4–8.9 percent TRR, 0.061–0.088 mg eq/kg) and kidney (3.0–3.7 percent TRR, 0.007–0.009 mg eq/kg). Tetraniliprole-desmethyl-amide was only found in the liver of the goats treated with the pyridinyl or tetrazolyl-label (4.5–7.7 percent TRR, 0.055–0.067 mg eq/kg).

Several metabolites were identified, each accounting for ≤ 3.9 percent TRR, but some with absolute levels above 0.01 mg eq/kg; metabolites tetraniliprole-N-methyl-quinazolinone-pyrazole-3-carboxylic acid, tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid (once 0.012 mg eq/kg in kidney with one label), tetraniliprole-N-methyl quinazolinone-benzylalcohol, tetraniliprole-desmethyl-amide (0.021 and 0.046 mg eq/kg in liver with two labels and 0.013 mg eq/kg in milk and kidney and 0.045 mg eq/kg in liver with one label) and tetraniliprole-quinazolinone (0.013 amd 0.026 mg eq/kg in milk and liver with one label).

Pyrazole-carboxamide label specific metabolites were tetraniliprole-pyrazole-5-amide, tetraniliprole-pyrazole-5-N-methyl-amide and tetraniliprole-5-carboxylic acid and were detected only in milk and liver (≤ 1.8 percent TRR). Tetrazolyl-specific metabolites were not identified.

Laying hens

In a series of three separate studies, six laying hens in each study were dosed orally once each morning for 14 consecutive days with [pyrazole-carboxamide-^{14}C]-tetraniliprole, [pyridinyl-2-^{14}C]-tetraniliprole, or [tetrazolyl-^{14}C]-tetraniliprole at rate of 18–19 ppm dry feed (approx. 1 mg/kg bw/day) and were sacrificed about 6 hours after the last dose.

Most of the radioactivity was excreted (91–92 percent TAR), with approximately 7–8 percent TAR likely to be still present in the gastro-intestinal tract. An average amount of *ca* 0.2 percent TAR was found in eggs and 0.2–0.4 percent TAR in tissues. TRR were 0.48–0.77 mg eq/kg in liver, 0.033–0.098 mg eq/kg in kidney, 0.028–0.095 mg eq/kg in subcutaneous fat, 0.035–0.078 mg eq/kg in skin and 0.017–0.031 mg eq/kg in skeletal muscle. In eggs, it ranged from 0.005 to 0.011 mg eq/kg at day one to 0.091–0.10 mg eq/kg at sacrifice. A residue plateau-level of 0.084–0.089 mg eq/kg was reached approximately seven to nine days after the first dose and decreased rapidly after cessation of the treatment.

Using acetonitrile/water, or methanol in case of fat, residues were efficiently extracted from fat (86 percent TRR), but less efficiently from eggs (54 percent TRR), muscle (44 percent TRR), and liver (34 percent TRR). Eggs, liver, and muscle (pyridinyl-label) were further extracted using microwave, releasing another 12–46 percent TRR from eggs, 41–66 percent TRR from liver, and 56 percent TRR from muscle. Final PES for the pyridinyl label were 9.7 percent TRR (0.008 mg eq/kg) in eggs (tetrazolyl-label only),

5.9–11.4 percent TRR (0.002 mg eq/kg) in muscle and 0.3 percent TRR (< 0.001 mg eq/kg) in fat for both other labels, and were not further characterized.

In the pyridinyl-label study, parent tetraniliprole was the predominant residue in fat (55 percent TRR, 0.015 mg eq/kg), and was also found in egg (14 percent TRR, 0.012 mg eq/kg), but less in liver and muscle (1.6 and 3.7 percent TRR, 0.12 and 0.001 mg eq/kg). Tetraniliprole-dihydroxy was the only metabolite observed at levels > 10 percent TRR (15 percentTRR, 0.004 mg eq/kg), but only in hen fat. All other metabolites were below 6.5 percent TRR.

In the pyrazole-carboxamide label study, parent tetraniliprole accounted for 10–26 percentTRR in fat (up to 0.012 mg/kg), for 10 percentTRR in muscle and egg (0.002 and 0.008 mg/kg) and for 4.8 percent TRR (0.023 mg/kg) in liver. Metabolite tetraniliprole-despyridyl-N-methyl-quinazolinone was the major compound in eggs (36 percent TRR, 0.030 mg eq/kg) and fat (63 percent TRR, 0.029 mg eq/kg) and was found in liver (12 percent TRR, 0.065 mg eq/kg) and muscle (8.6 percent TRR, 0.001 mg eq/kg). In muscle, tetraniliprole-pyrazole-5-N-methyl-amide contributed to 40 percent TRR (0.007 mg eq/kg), but accounted for 5.5 percent TRR or less in other tissues. Tetraniliprole-pyrazole-5-amide accounted for 13 percent TRR (0.002 mg eq/kg) in muscle, but for less than 3 percent TRR (up to 0.005 mg eq/kg) in other tissues.

In the tetrazolyl-label study, parent tetraniliprole accounted for 10 percent TRR or more only in fat (26 percentTRR, 0.025 mg eq/kg) with levels of 4.2, 4.2 and 9.4 percent TRR in eggs, liver, and muscle, respectively. Tetraniliprole-despyridyl-N-methyl-quinazolinone was the major compound in eggs (27 percent TRR, 0.023 mg eq/kg) and fat (62 percent TRR, 0,059 mg eq/kg) and was found in liver (8.4 percent TRR, 0.065 mg eq/kg) and muscle (6.8 percent TRR, 0.002 mg eq/kg). In muscle, the metabolite tetraniliprole-pyrazole-5-N-methyl-amide contributed to 17.6 percent TRR (0.005 mg eq/kg), but to 5.8 percent TRR or less in other tissues. Tetraniliprole-pyrazole-5-amide also accounted for 9.7 percent TRR (0.003 mg eq/kg) in muscle, but for less than 3.5 percent TRR (0.027 mg eq/kg) in other tissues.

Other metabolites that were identified with the different labels accounted for < 10 percent TRR. Tetraniliprole-benzylalcohol-Gluc was found in eggs and liver (1.3–6.5 percent TRR, 0.001–0.047 mg eq/kg), and tetraniliprole-dihydroxy (1.1–15 percent TRR, < 0.001–0.009 mg eq/kg), tetraniliprole-benzylalcohol (0.5–8.1 percentTRR, < 0.001–0.062 mg eq/kg), tetraniliprole-hydroxy-N-methyl (1.3–3.9 percent TRR, < 0.001–0.030 mg eq/kg), and tetraniliprole-N-methyl-quinazolinone (3.1–7.4 percent TRR, < 0.01 mg eq/kg) in eggs only.

The pyrazole-carboxamide and the tetrazolyl-label specific metabolites < 10 percent TRR were tetraniliprole-pyrazole-5-N-methyl-amide-hydroxy (0.5–3.2 percent TRR, 0.001–0.016 mg eq/kg) in all matrices, except fat, tetraniliprole-pyrazole-5-carboxylic acid (5.0–7.6 percent TRR, 0.037–0.039 mg eq/kg) in liver only, tetraniliprole-despyridyl (1.9–9.7 percent TRR, 0.001–0.074 mg eq/kg) in all matrices, tetraniliprole-despyridyl-N-methyl-quinazolinone-hydroxy and tetraniliprole-despyridyl-hydroxy (1.1–9.3 percent TRR, < 0.001–0.069 mg eq/kg (mixture of both metabolites)) in eggs, liver and fat (pyrazole-carboxamide label only), and tetraniliprole-despyridyl-quinazolinone (1.0–7.0 percent TRR, < 0.001–0.044 mg eq/kg) in eggs, fat and liver.

The pyridinyl-specific metabolite deschloro-desmethyl-amide accounted for 1.7–4.5 percent TRR (< 0.001–0.022 mg eq/kg). A large portion of unknown pyridinyl-labelled metabolites was found in the polar HPLC region of eggs and liver. There were no metabolites detected by HPLC analysis in the microwave assisted extract of muscle (0.014 mg eq/kg, 56.1 percent TRR), due to the low radioactivity in the sample and the high number of metabolites, as demonstrated in liver.

The tetrazolyl-label specific metabolite tetraniliprole-tetrazole and its conjugates was found in eggs, muscle, fat and liver with levels ranging from (0.8–15 percent TRR). The sum of tetraniliprole-

tetrazole and its conjugates was highest in eggs and muscle (23–29 percent, 0.009–0.019 mg eq/kg), but also observed in fat and liver (3.3–5.5 percent TRR, 0.005–0.025 mg eq/kg).

Summary of animal metabolism

The principal metabolic reactions of tetraniliprole in the lactating goat are:

- intra-molecular condensation (cyclisation) of parent compound leading to quinazolinone compounds;
- hydroxylation in the methyl group of the phenyl moiety leading to tetraniliprole-benzylalcohol and the N-methyl moiety leading to tetraniliprole hydroxy-N-methyl;
- demethylation of the N-methyl group to form tetraniliprole-quinazolinone and tetraniliprole-desmethyl-amide;
- cleavage of the pyridine ring leading to tetraniliprole-pyrazole-5-amide (PC- and tetra-label) followed by further oxidation to a tetraniliprole-pyrazole-5-carboxylic acid or by methylation leading to tetraniliprole-N-methyl-amide;
- cleavage of the phenyl ring to form tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid;
- cleavage of the tetrazole ring followed by oxidation leading to tetraniliprole-N-methyl-quinazolinone-3-carboxylic acid.

The principal metabolic reactions of tetraniliprole in the laying hens are:

- cleavage of the pyridine ring, leading to despyridyl compounds;
- intra-molecular condensation of despyridyl compounds and parent compound leading to the quinazolinone compounds;
- cleavage of the tetrazole ring, followed by conjugation (tetra-label only);
- hydroxylations, leading to mono- and/or dihydroxy compounds such as tetraniliprole-dihydroxy, tetraniliprole-despyridyl-hydroxy, tetraniliprole-despyridyl-N-methyl-quinazolinone-hydroxy, and tetraniliprole-pyrazole-5-N-methyl-amide-hydroxy;
- conjugation with glucuronic acid after hydroxylation to tetraniliprole-benzylalcohol;
- cleavage of the phenyl moiety, leading to tetraniliprole-5-amide followed by methylation into tetraniliprole-pyrazole-5-N-methyl-amide followed by hydroxylation (tetraniliprole-pyrazole-5-N-methyl-amide-hydroxy);
- demethylation of the N-methyl group after intra-molecular condensation (cyclisation) of tetraniliprole-despyridyl to form tetraniliprole-despyridyl-quinazolinone.

The Meeting concluded that the total administered radioactivity was readily absorbed and extensively metabolised in goats and hens. Considering the very low levels of (other) metabolites observed in hen, the Meeting concluded that a single residue definition for animal commodities can be proposed.

Tetraniliprole (parent) is a major component in all goat tissues (24–71 percent TRR), in poultry fat (26–55 percent TRR) and in eggs (4.2–14 percent TRR), but contributes to the overall residue to a minor extent in poultry muscle (3.7–10 percent TRR) and poultry liver (1.6–4.2 percent TRR).

Tetraniliprole-N-methyl-quinazolinone is a major metabolite in goat tissues (11–67 percent TRR) except liver (2.2–5.6 percent TRR) and was observed in poultry studies in eggs only (3.1–7.4 percent TRR).

Tetraniliprole-benzylalcohol is a major metabolite in goat milk (9–11 percent TRR). Several other minor metabolites are observed in goat tissues, but all accounting for less than 10 percent TRR, though a number of them represent > 0.01 mg eq/kg.

Tetraniliprole despyridyl-N-methyl-quinazolinone contributes significantly to the total residue in eggs (27–36 percent TRR), poultry fat (63 percent TRR) and poultry liver (8.5–12 percent TRR), and contributes less to the total residue in poultry muscle (6.8–8.6 percent TRR). Tetraniliprole-pyrazole-5-N-methyl-amide is a major component in poultry muscle (18–40 percent TRR) and contributes less to the total residue in poultry liver, fat, and eggs (4.6–6 percent TRR).Tetrazole conjugates contributed significantly to the total residue in eggs (23 percent TRR) and poultry muscle (29 percent TRR), but less in poultry fat (5.5 percent TR) and poultry liver (3.3 percent TRR).

Methods of analysis

The Meeting received information on analytical methods for tetraniliprole residues in plant and animal matrices.

For plant matrices, analytical methods for measuring residues of tetraniliprole and tetraniliprole-N-methyl-quinazolinone generally involved extraction with water and acetonitrile, followed by quantification with LC-MS/MS. The method has been succesfully validated for commodities with high water, high acid, high oil, high starch/dry and high protein content with a LOQ of 0.01 mg/kg each.

For animal matrices, analytical methods for measuring residues of tetraniliprole, tetraniliprole-N-methyl-quinazolinone and tetraniliprole-benzylalcohol involve two extractions with acetonitrile/water, followed by drying and reconstitution in 0.1 percent aqueous formic acid. Residues were determined by LC-MS/MS. The method has been fully validated for animal tissues, eggs and milk, with an LOQ of 0.01 mg/kg for all analytes in all matrices.

The Meeting concluded that the presented methods were sufficiently validated and are suitable to measure tetraniliprole, tetraniliprole-*N*-methyl-quinazolinone in plant and animal commodities and tetraniliprole-benzylalcohol in animal commodities.

The Meeting noted that a modified version of QuEChERS multiresidue (MRM) enforcement method 01463 for determination of tetraniliprole and tetraniliprole-N-methyl-quinazolinone in plant commodities is confirmed as being a suitable enforcement method, with acceptable recovery rates at relevant fortification levels. The method was validated in tomato, grapes, wheat grain, dry bean seed and rapeseed seed, with an LOQ of 0.01 mg/kg each for both parent and metabolite.

Stability of pesticide residues in stored analytical samples

The Meeting received information on storage stability of tetraniliprole and tetraniliprole-N-methyl-quinazolinone in raw and processed plant commodities. No freezer storage stability data were submitted on animal matrices, but samples from the animal feeding studies were processed and analysed within 30 days and the samples from the radiolabelled metabolism studies were processed within 5 months of sampling.

The storage stability studies showed that tetraniliprole and tetraniliprole-N-methyl-quinazolinone (at 0.2 mg/kg) were stable when stored frozen for at least 24 months in crop commodities representative of the high water (tomato), high acid (grape), high starch (wheat grain), high protein (dry bean) and high oil (rape seed seed) commodity groups.

The Meeting agreed that the demonstrated storage stability on various representative plant commodities covered the residue sample storage intervals used in the field trials considered by the current Meeting.

Definition of the residue

Plant commodities

In the plant metabolism studies involving foliar applications (apples, potato, lettuce, paddy rice), soil drench application (tomato), granular in planting hole applications (rice) and seed treatments (potato and maize), in confined rotational crop metabolism studies and in processing studies, tetraniliprole was the major component (apple, tomato, lettuce, tomato, potato) of the radioactive residue.

Tetraniliprole is found in all primary crop commodities and is considered suitable as a marker compound. The Meeting noted that suitable analytical methods exist to measure tetraniliprole in plant commodities. The Meeting decided to define the residue for compliance with the MRL as tetraniliprole.

In deciding which additional compounds should be included in the residue definition for dietary risk assessment, tetraniliprole-N-methyl-quinazolinone is the only compound identified in relevant amounts in plant matrices. This metabolite was found at up to 20 percent TRR in tomato, rice grain and potatoes, but generally at levels ≤ 0.01 mg eq/kg in food commodities, which was confirmed in the supervised field trials, in which it was only found occasionally above the LOQ of 0.01 mg/kg, with tetraniliprole being present at levels about an order of magnitude higher.

A high temperature hydrolysis study shows that, though stable during pasteurization, tetraniliprole is not stable under baking/brewing/boiling (BBB) (pH 5, 100 °C, 60 minutes) and sterilization (pH 6, 120 °C, 20 minutes) conditions. Under these conditions 65–68 percent and 1.1–1.5 percent AR was recovered as parent and 27–30 percent AR and 94 percent AR as tetraniliprole-N-methyl-quinazolinone, respectively. Processing under heating indicated conversion of parent tetraniliprole into tetraniliprole-N-methyl-quinazolinone, e.g., mustard greens (up to 20 percent TRR), broccoli (up to 29 percent TRR), tomato paste (23–48 percent TRR), and soya bean meal (up to 81 percent TRR).

Tetraniliprole-N-methyl-quinazolinone was found in the rat and is covered by the health based guidance value of parent tetraniliprole.

The Meeting concluded that tetraniliprole-N-methyl-quinazolinone may occur in primary and rotational crops and that the metabolite contributes significantly to the residue in processed commodities. Hence the Meeting agreed to define a residue definition for dietary risk assessment for plant commodities is tetraniliprole and tetraniliprole-N-methyl-quinazolinone, expressed as tetraniliprole.

Animal commodities

Tetraniliprole is a major component in all goat tissues (24–71 percent TRR), poultry fat (26–55 percent TRR) and eggs (4.2–14 percent TRR), but it contributes little to the overall residue in poultry muscle (3.7–10 percent TRR) and liver (1.6–4.2 percent TRR). Parent tetraniliprole is also the major component in all cattle tissues and milk.

The Meeting noted that suitable analytical methods exist to measure tetraniliprole in animal commodities and decided to define the residue for compliance with the MRL as tetraniliprole.

Tetraniliprole has a Log Kow of 2.6, indicating a low to moderate potential to sequester into fatty matrices. The ratios of concentrations of muscle to fat (1:1.3–2.1) and skim milk to cream (1:3) indicate only a very slight tendency to concentrate in fat.

The Meeting considered the residue not to be fat soluble.

In deciding which compound should be included in the residue definition for dietary risk assessment, the Meeting noted that the tetraniliprole was metabolized into numerous components, of which 17 were accounted for > 10 percent TRR and/or > 0.01 mg eq/kg. The metabolites were considered in three categories, either covered by the toxicity of the parent, suitable for assessment by the TTC approach following Cramer Class III or by the TTC approach for genotoxic compounds.

The 2021 and 2022 JMPR Meeting concluded that the following metabolites are covered by the health based guidance values for tetraniliprole and should be considered for inclusion in the residue definition:

Tetraniliprole-N-methyl-quinazolinone contributes significantly to the total residue in goat tissues (11–72 percent TRR), except liver (2.2–5.6 percent TRR) and was observed in poultry studies in eggs only (3.1–7.4 percent TRR). The Meeting agreed that the metabolite should be included in the residue definition for dietary risk assessment.

Tetraniliprole-benzylalcohol contributed significantly to the total residue in goat milk (9–11 percent TRR, 0.022–0.045 mg eq/kg), and to a lesser extent in other goat and poultry matrices (1.3–8.1 percent TRR, 0.001–0.062 mg eq/kg). This metabolite also contributes to the total residue in dairy feeding studies in milk (36–66 percent of parent), liver (6.6–8.8 percent of parent), kidney (5.0–6.3 percent of parent). The Meeting agreed that the metabolite tetraniliprole-benzylalcohol should be included in the residue definition for dietary risk assessment.

Tetraniliprole-hydroxy-N-methyl was found in all goat (3.0–8.9 percent TRR, 0.007–0.088 mg eq/kg) and poultry matrices (1.3–3.9 percent TRR, < 0.001–0.030 mg eq/kg), except fats. However, since this analyte would contribute only little to the total residues (tetraniliprole + tetraniliprole-N-methyl-quinazolinone + tetraniliprole-benzylalcohol), the Meeting agreed that the metabolite does not need to be included in the residue definition for dietary risk assessment.

Tetraniliprole-desmethyl-amide was found in all goat matrices at levels ranging from 0.2 to 7.7 percent TRR (0.001–0.067 mg eq/kg). However, since it would contribute only little to the total residues (tetraniliprole + tetraniliprole-N-methyl-quinazolinone + tetraniliprole-benzylalcohol), the Meeting agreed that the metabolite does not need to be included in the residue definition for dietary risk assessment.

The 2021 and 2022 JMPR Meetings concluded that the TTC Cramer Class III could be applied (no indication for genotoxicity) for the following metabolites: tetraniliprole-pyrazole-5-carboxylic acid, tetraniliprole-N-methyl-quinazolinone-benzylalcohol, tetraniliprole-despyridyl-N-methyl-quinazolinone, tetraniliprole-pyrazole-5-amide, tetraniliprole-N-methyl-quinazolinone-pyrazole-3-carboxylic acid (2021 JMPR), tetraniliprole-desmethyl-amide, tetraniliprole-quinazolinone, tetraniliprole-pyridinyl-pyrazole-5-carboxylic acid, and T-pyrazole-5-N-methyl-amide (2022 JMPR)

In the absence of toxicological data for a number of (poultry specific) metabolites, the 2022 JMPR concluded that the remaining should be assessed under the TTC approach for genotoxic compounds; tetraniliprole-despyridy, tetrazole-conjugates, tetraniliprole-despyridyl-N-methyl-quinazolinone-hydroxy and tetraniliprole-despyridyl-hydroxy (mixture of both metabolites), tetraniliprole-despyridyl-quinazolinone, tetraniliprole-pyrazole-5-N-methyl-amide-hydroxy, tetraniliprole-deschloro-desmethyl-amide.

The Meeting concluded that for dietary risk assessment in animal commodities, the residue definition should be the sum of tetraniliprole, tetraniliprole-N-methyl-quinazolinone and tetraniliprole-benzylalcohol, expressed as tetraniliprole.

The Meeting agreed that:

The residue definition for tetraniliprole for compliance with the MRL in plant and animal commodities is: *Tetraniliprole*

The definition of the residue for dietary risk assessment for plant commodities: *Tetraniliprole + tetraniliprole-N-methyl-quinazolinone, expressed as tetraniliprole.*

Definition of the residue for dietary risk assessment for animal commodities: *Tetraniliprole + tetraniliprole-N-methyl-quinazolinone + tetraniliprole-benzylalcohol, expressed as tetraniliprole*.

Results of supervised residue trials on crops

Supervised trials were available for the use of tetraniliprole on citrus fruit (orange, mandarin, lemon, and grapefruit), pome fruit (apple and pear), stone fruit (cherry, peach plum), grapes, flowerhead brassicas (broccoli and cauliflower) and head brassicas (head cabbage), fruiting vegetables (tomato, peppers), leafy vegetables (lettuce head, lettuce leaf, spinach, mustard greens), pulses (dry soya beans), tuberous and corm vegetables (potato), cereal grains (rice, maize, sweet corn), tree nuts (almonds and pecans).

GAP information was available from Canada and the United States (foliar treatment of pome fruit, stone fruit, small fruit vine climbing, brassica vegetables, except leafy vegetables, fruiting vegetables other than cucurbits, leafy vegetables, soya bean, tuberous and corm vegetables, maize cereals, sweet corn, tree nuts), Korea (foliar treatment on apple, pear, and sweet persimmon), India (foliar treatment on soya bean and rice) and Japan (seed treatment on rice).

In this appraisal the term 'total residues' refers to the sum of tetraniliprole and tetraniliprole-N-methyl-quinazolinone, and was used to estimate STMR, HR, median and higest residues. Since residues in the evaluation were expressed as parent equivalents, no conversion factor is needed. Parent constitutes the majority of the tetraniliprole residues in most RACs, with low metabolite levels, except for feed and processed commodities subjected to heating.

To estimate the total residues, where residues tetraniliprole-N-methyl-quinazolinone are < LOQ it was assumed to be zero, except when both parent and metabolite were < LOQ and in that case the total was taken as < LOQ. The method for calculating the total residue for various situations is illustrated below.

Tetraniliprole	Tetraniliprole-N-methyl-quinazolinone	Tetraniliprole + Tetraniliprole-N-methyl-quinazolinone
0.29	0.02	0.31
0.039	< 0.01	0.039
< 0.01	< 0.01	< 0.01

Citrus fruit

GAP for tetraniliprole in the United States for citrus fruit includes three different uses, a single soil or single drip chemigation application at 120 g ai/ha, a single soil or drip chemigation application at 120 g ai/ha followed by single foliar application at 60 g ai/ha, and three foliar applications at 60 g ai/ha each. Trials were submitted in support of the combined soil and foliar applications and the triple foliar applications. Based on the results of these field trials the Meeting decided that the critical GAP in the United States for citrus fruit is three foliar applications at 60 g ai/ha, with a re-treatment interval of 5 days and a PHI of 1 day.

Grapefruit

A total of six independent supervised residue trials on grapefruits matching the critical United States GAP were conducted in the United States. Residues of tetraniliprole both for MRL and risk assessment in ranked order were (n=6): 0.039, 0.071, 0.081, 0.11, 0.19 and 0.49 mg/kg in whole fruit.

Noting that grapefruit is a representative crop for the subgroup of pummelo and grapefruits the Meeting estimated a maximum residue level of 0.9 mg/kg and an STMR of 0.091 mg/kg for tetraniliprole in the Subgroup of Pummelo and Grapefruits (including Shaddock-like hybrids, among other Grapefruit).

Lemon

Independent supervised residue trials on lemons matching the United States GAP were conducted in the United States. Residues of tetraniliprole both for MRL and risk assessment in ranked order were (n=5): 0.062, 0.13, 0.19, 0.20 and 0.77 mg/kg in whole fruit.

Noting that lemons is a representative crop for the subgroup of lemons and limes the Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.19 mg/kg for tetraniliprole in the Subgroup of Lemons and Limes (including Citron).

Mandarin

Independent supervised residue trials on mandarin matching the United States GAP were conducted in United States. Residues of tetraniliprole both for MRL and risk assessment in ranked order were (n=4): 0.17, 0.18, 0.19 and 0.54 mg/kg in whole fruit.

Noting that mandarin is a representative crop for the subgroup of mandarins the Meeting estimated a maximum residue level of 1.0 mg/kg and an STMR of 0.185 mg/kg for tetraniliprole in the Subgroup of Mandarins (including Mandarin-like hybrids).

Oranges

A total of eight independent supervised residue trials on oranges matching the United States GAP were conducted in United States. Residues of tetraniliprole both for MRL and risk assessment in ranked order were (n=8): 0.044, 0.066, 0.13, 0.14, 0.14, 0.15, 0.16, and 0.29 mg/kg in whole fruit.

Noting that orange is a representative crop for the subgroup of oranges, sweet, sour the Meeting estimated a maximum residue level of 0.5 mg/kg. Using the processing factor for peeling of 0.11 (see section on processing) the Meeting estimated an STMR of 0.015 mg/kg in orange flesh for tetraniliprole in the Subgroup of Oranges, Sweet, Sour (including Orange-like hybrids).

Pome fruit

The critical GAP for pome fruit is in Canada and the United States allowing three foliar applications at 60 g ai/ha, with a re-treatment interval of 7 days and a PHI of 7 days. Trials conducted in Australia did not match this critical GAP.

In trials conducted in apples in Canada and the United States approximating the critical GAP, tetraniliprole residues both for MRL and risk assessment were (n=12): 0.064, 0.092, 0.10, 0.11, 0.11, 0.13, 0.15, 0.15, 0.16, 0.17, 0.17 and 0.20 mg/kg.

In trials conducted in pears in Canada and the United States approximating the critical GAP, tetraniliprole residues were (n=8): 0.044, 0.048, 0.080, 0.081, 0.13, 0.14, 0.16 and 0.24 mg/kg.

Noting that the median residues of tetraniliprole, for apples and pears from the United States/Canadian trials are within a 5-fold range, and that there is no evidence of a difference in the residue populations across the pome fruit types by Mann-Whitney test, the Meeting decided to make a recommendation for the Group of Pome fruit based on the combined data.

The combined apple and pear data for tetraniliprole residues, both for MRL and risk assessment, in ranked order, were (n=20): 0.044, 0.048, 0.064, 0.080, 0.081, 0.092, 0.10, 0.11, 0.11, 0.13, 0.13, 0.14, 0.15, 0.15, 0.16, 0.16, 0.17, 0.17, 0.20 and 0.24 mg/kg.

Noting that the United States GAP is for the group of pome fruits, which does not include Japanese persimmon, the Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.13 mg/kg for tetraniliprole in the Group of Pome Fruits, excluding Japanese persimmon.

Stone fruit

The critical GAP for stone fruit in Canada and the United States allows three foliar applications at 60 g ai/ha, with a retreatment interval of 7 days and a PHI of 5 days.

Field trials were available on cherries, peaches and plums, and the fruit was analysed without stone. At the 2017 JMPR Meeting, it was concluded that the contribution of the pit to the weight of the whole fruit of cherries, plums and peaches is approximately 10 percent. Correcting the residue levels for tetraniliprole using this weight/weight ratio would lead to the same rounded residue levels, so no adjustment was made on the residues.

Cherries

In trials conducted in Canada and the United States matching GAP, tetraniliprole residues both for MRL and risk assessment were (n=11): 0.085, 0.12, 0.24, 0.27, 0.28, 0.29, 0.38, 0.44, 0.49, 0.56, and 0.66 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.29 mg/kg in the Subgroup of Cherries.

Peaches

In trials conducted in Canada and the United States matching the GAP, tetraniliprole residues both for MRL and risk assessment were (n=15): 0.030, 0.041, 0.056, 0.064, 0.070, 0.080, 0.086, 0.089, 0.091, 0.095, 0.15, 0.15, 0.21, 0.38, and 0.44 mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg and an STMR of 0.089 mg/kg for tetraniliprole in the Subgroup of Peaches (including Nectarines and Apricots).

Plums

In trials conducted in Canada and the United States matching the GAP, tetraniliprole residues both for MRL and risk assessment were (n=8): < 0.01, 0.012, 0.016, 0.026, 0.039, 0.081, 0.11, and 0.13 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.033 mg/kg for tetraniliprole in the Subgroup of Plums.

Small fruit vine climbing

The critical GAP for small fruit vine climbing in Canada and the United States allows four foliar applications at 45 g ai/ha, with a retreatment interval of 7 days and a PHI of 14 days.

In trials conducted on grapes in Canada and the United States matching the GAP, tetraniliprole residues both for MRL and risk assessment were (n=10): 0.20, 0.25, 0.26, 0.27, 0.27, 0.28, 0.29, 0.39, 0.82 and 0.92 mg/kg.

Noting that grapes is a representative commodity for the subgroup, the Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.275 mg/kg for tetraniliprole in the Subgroup of Small fruit vine climbing.

Brassica vegetables, except brassica leafy vegetables

The critical GAP for Brassica vegetables, except brassica leafy vegetables in Canada and the United States allows four foliar applications at 45 g ai/ha, with a retreatment interval of 5 days and a PHI of 1 day.

Flowerhead brassicas

Residue trials performed in the United States on broccoli and cauliflower matching the GAP, tetraniliprole residues both for MRL and risk assessment were:

Broccoli (n=5): 0.11, 0.14, 0.15, 0.18, and 0.24 mg/kg;

Cauliflower (n=5): 0.036, 0.066, 0.11, 0.16, and 0.19 mg/kg.

Noting that broccoli and cauliflower are representative commodities for flowerhead brassicas, that median residues are within a 5-fold range and the residue populations are similar according to the Mann-Whitney test, the Meeting decided to combine the data as (n=10): 0.036, 0.066, 0.11, 0.11, 0.14, 0.15, 0.16, 0.18, 0.19, and 0.24 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.145 in the Subgroup of Flowerhead Brassicas.

Head Brassicas

In trials conducted in the United States matching the GAP, tetraniliprole residues in head cabbages with wrapper leaves, both for MRL and dietary burden calculation, in ranked order, were (n=10): 0.046, 0.073, 0.087, 0.096, 0.12, 0.15, 0.31, 0.35, 0.48, and 1.1 mg/kg (1.2 highest individual value).

Residues of tetraniliprole in head cabbages without wrapper leaves for STMR estimation in ranked order, were (n=10): < 0.01 (3), 0.010, 0.011, 0.013, 0.020, 0.020, 0.024, and 0.026 m/kg.

The Meeting estimated a maximum residue level of 2.0 mg/kg and an STMR of 0.012 for tetraniliprole in Cabbages, head.

The Meeting also estimated a median and highest residue level of 0.135 mg/kg and 1.2 mg/kg, respecetively, for Cabbages, Head with wrapper leaves.

Fruiting vegetables, other than cucurbits

The critical GAP for fruiting vegetables in Canada and the United States allows four foliar applications at 45 g ai/ha, with a retreatment interval of 5 days and a PHI of 1 day.

Data were available from supervised residue trials in tomato (including a variety of fruit sizes, ranging from 28–450 g, including trials with cherry tomatoes) in the United States.

In trials matching the GAP tetraniliprole residues, both for MRL and risk assessment, were (n=16): 0.030, 0.034, 0.042, 0.042, 0.053, 0.053, 0.057, 0.064, 0.066, 0.075, 0.079, 0.080, 0.12, 0.22, 0.23 and 0.32 mg/kg (highest individual value 0.35 mg/kg).

In trials conducted in peppers, including trials with chili peppers in United States matching the GAP, tetraniliprole residues, both for MRL and risk assessment, were (n=12): 0.011, 0.024, 0.041, 0.048, 0.075, 0.077, 0.079, 0.087, 0.093, 0.11, 0.15 and 0.20 mg/kg.

Noting that residue data on small and large varieties of tomatoes and sweet and chili peppers are available, that the median residues of tetraniliprole, for tomatoes and peppers are within a 5-fold range, and that there is no evidence of a difference in the residue populations across the fruiting vegetable types by Mann-Whitney test, the Meeting decided to make a recommendation for the Group of Fruiting vegetables, other than cucurbits based on the combined data.

The combined data for tetraniliprole, in ranked order, were (n=28): 0.011, 0.024, 0.030, 0.034, 0.041, 0.042 (2), 0.048, 0.053 (2), 0.057, 0.064, 0.066, 0.075 (2), 0.077, 0.079 (2), 0.080, 0.087, 0.093, 0.11, 0.12, 0.15, 0.20, 0.22, 0.23, and 0.32 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.075 mg/kg for tetraniliprole in the Group of Fruiting vegetables, other than cucurbits, excluding okra, martynia and roselle.

Chili peppers, dried

Based on the estimated maximum residue level of 0.4 mg/kg for the Group of Fruiting vegetables and applying a default processing factor of 10, the Meeting estimated a maximum residue level of 4 mg/kg for peppers, chili, dried, together with a STMR of 0.75 mg/kg.

Leafy vegetables (including Brassica leafy vegetables)

The critical GAP for Brassica vegetables, except brassica leafy vegetables in Canada and the United States allows four foliar applications at 45 g ai/ha, with a re-treatment interval of 5 days and a PHI of 1 day. The Meeting received data on head lettuce, leaf lettuce, spinach and mustard greens.

Leafy greens

Data were available from supervised residue trials in head lettuce, leaf lettuce and spinach in the United States. The trials did not match the Canadian or United States GAP, because the RTIs ranged from 2–4 days. Decline data indicate a slow decline, indicating that the duration of the retreatment intervals has an impact on the final residue level.

The Meeting was unable to make a recommendation for the Subgroup of Leafy greens.

Brassica leafy vegetables

In trials conducted in United States matching the GAP, tetraniliprole residues, both for MRL and risk assessment, in mustard greens were (n=5): 3.2, 3.6, 4.0, 4.2, and 7.3 mg/kg.

Noting that mustard greens is representative for brassica leafy vegetables, the Meeting estimated a maximum residue level of 15 mg/kg, an STMR and a median residue level of 4.0 mg/kg and a highest residue of 7.3 mg/kg for tetraniliprole in the Subgroup of Leaves of Brassicaceae.

Soya beans, dry

The United States GAP for soya beans includes either a single in-furrow soil application at 200 g ai/ha (PHI not applicable), 4 foliar applications at 50 g ai/ha, with a retreatment interval of 3 days, and PHI of 14 days, or a combination of both, with a maximum of 200 g ai/ha per season. The Meeting concluded that

the critical United States GAP for soya beans is 4 foliar applications at 50 g ai/ha, with a retreatment interval of 3 days and a PHI of 14 days.

Trials conducted in Canada and the United States matching the critical GAP, tetraniliprole residues, both for MRL and risk assessment, in dry soya bean seeds were (n=20): < 0.01 (3), 0.012, 0.012, 0.016, 0.018, 0.018, 0.025, 0.026, 0.026, 0.030, 0.033, 0.037, 0.038, 0.048, 0.053, 0.092, 0.13, and 0.14 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.026 mg/kg for tetraniliprole in Soya bean (dry).

Tuberous and corm vegetables

The critical United States GAP for tuberous and corm vegetables is a single in-furrow application at 200 g ai/ha (PHI not applicable).

Potato

In trials conducted in Canada and the United States matching the GAP, tetraniliprole residues, both for MRL and risk assessment, in ranked order were (n=18): < 0.01 (16), 0.012 and 0.015 mg/kg (highest individual level 0.017 mg/kg).

Noting that potato is a representative crop for tuberous and corm vegetables, the Meeting estimated a maximum residue level of 0.02 mg/kg and an STMR of 0.01 mg/kg for tetraniliprole in the Subgroup of Tuberous and corm vegetables.

Rice

The critical GAP for rice is from India, which allows 2 foliar applications at 60 g ai/ha, with a RTI of 11 days and a PHI of 43 days, but trials conducted in Brazil, India, Thailand and Vietnan did not match this GAP. Applying proportionality to trials showing residues (>0.01 mgkg) with 2 foliar applications at 40 g ai/ha with identical RTI (11 days) and would still result in a too limited data set (n=2) for estimation of a maximum residue level.

The Japanese GAP for rice consists of a single seed treatment at 264 g ai/ha. Trials performed in India, Thailand and Vietnam, approximating this GAP, were available.

Tetraniliprole, residues, both for MRL and risk assessment, in rice grain were (n=12): < 0.01 (11) and 0.013 mg/kg.

Tetraniliprole, residues, both for MRL and risk assessment, in husked rice were (n=12): < 0.01 (12) mg/kg.

The Meeting estimated a maximum residue level of 0.02 mg/kg and an STMR of 0.01 mg/kg for tetraniliprole in Subgroup of Rice cereals.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and an STMR of 0.01 mg/kg for tetraniliprole in Rice, husked.

Noting that residue levels in polished rice are usually lower than residue levels in husked rice, which was confirmed in the processing study submitted to the current Meeting, the Meeting decided to extrapolate the MRL of 0.01(*) mg/kg and STMR of 0.01 mg/kg for Rice, husked to Rice, polished.

Maize cereals

The critical GAP in the United States for maize (field corn, popcorn) is 4 foliar applications at 50 g ai/ha with a retreatment interval of 7 days and a PHI of 14 days.

In residue trials in maize/field corn from Canada and the United States matching this GAP, tetraniliprole residues, both for MRL and risk assessment, were (n=15): < 0.01 (14) and 0.011 (0.012 highest individual value) mg/kg.

The Meeting estimated a maximum residue level of 0.015 mg/kg and an STMR of 0.01 mg/kg for tetraniliprole in the Subgroup of Maize cereals.

Sweet Corn (corn-on-the-cob)

The critical GAP for sweet corn in United States is 4 foliar applications at 50 g ai/ha with a retreatment interval of 7 days and a PHI of 1 day.

In residue trials conducted in sweet corn according to GAP in Canada and the United States, tetraniliprole residues, both for MRL and risk assessment, were (n=14): < 0.01 (14) mg/kg.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and an STMR of 0.01 mg/kg for tetraniliprole for Sweet Corn (corn-on-the-cob) (kernels plus cob with husk removed).

Tree nuts

The critical GAP for tree nuts in Canada and the United States allows for 4 foliar applications at 45 g ai/ha, with an RTI of 7 days and a PHI of 10 days. Data were available from supervised residue trials in almond and pecan in Canada and the United States matching the GAP.

Residues of tetraniliprole, both for MRL and risk assessment, in almonds were (n=3): < 0.01 (1), 0.010, and 0.015 mg/kg.

Residues of tetraniliprole, both for MRL and risk assessment, in pecans were (n=6): < 0.01 (6) mg/kg.

Noting that the median residues of tetraniliprole, in almonds and pecans are within a 5-fold range and that there is no evidence of a difference in the residue populations by Mann-Whitney test, the Meeting decided to combine the results as (n=9): < 0.01 (7), 0.010, and 0.015 mg/kg.

Considering that a high number of residue levels below the LOQ of 0.01 mg/kg results in a higher statistical uncertainty, the Meeting decided to estimate the maximum residue level based on 2 × HR, leading to a higher value than would be calculated with the OECD calculator.

Noting that almond and pecan are two representative crops for the Tree nut group, the Meeting estimated a maximum residue level of 0.03 mg/kg and an STMR of 0.01 mg/kg for tetraniliprole in the Group of Tree Nuts.

Residues in animal feeds

Soya bean forage (56 percent dry matter) and hay (85 percent dry matter)

The Canadian and United States labels have a feeding restriction for soya bean forage and hay after foliar use, but not after in-furrow soil treatment or seed treatment. The critical GAP in the United States for soya beans as relates to soya bean forage and hay is in-furrow-treatment at 200 g ai/ha.

In three trials on soya bean forage conducted according to this GAP, tetraniliprole residue levels, both for MRL and dietary burden estimations, were (n=3): < 0.01 (2) and 0.031 mg/kg.

The Meeting concluded that three trials are insufficient to estimate a median and highest residue level for tetraniliprole in soya bean forage and hay.

Since residue levels were observed in soya bean forage and hay when planted in crop rotation, maximum residue levels estimated in that section are applicable for soya bean forage and hay in the absence of sufficient data after primary use.

Rice straw (90 percent dry matter)

The critical GAP for rice in India allows for 2 foliar applications at 60 g ai/ha, with an RTI of 11 days and a PHI of 43 day. Supervised residue trials conducted in rice in Brazil, India, Thailand and Vietnam used a lower application rate (2 × 40 g ai/ha) or a higher number of applications (3 × 60 g ai/ha). The Meeting agreed to use the trials with the lower application rates by applying proportionality.

Unscaled tetraniliprole residue levels in rice straw from supervised residue trials conducted at 2 × 40 g ai/ha (RTI 10–12 days and harvested at 43 DALA) were (n=6): 0.24, 0.26, 1.6, 1.9, 3.5, and 5.0 mg/kg (highest individual value of 5.1 m/kg). Scaled tetraniliprole residues for MRL estimation were (n=6): 0.36, 0.39, 2.4, 2.8, 5.2, and 7.5 (highest individual value 7.6 mg/kg) on an as received basis.

Unscaled total tetraniliprole residue levels were (n=6): 0.26, 0.29, 1.7, 2.0, 3.6, and 5.4 mg/kg (highest individual value of 5.5 mg/kg). Scaled total tetraniliprole residues for median and highest residue estimation in ranked order were (n=6): 0.39, 0.44, 2.6, 3.0, 5.4 and 8.1 mg/kg (highest individual value 8.2 mg/kg) on as received basis.

Assuming a default of 90 percent dry matter, the Meeting estimated a maximum residue level of 20 (dw) mg/kg for tetraniliprole in rice straw.

Based on the total tetraniliprole residues, the Meeting estimated a median and highest residue level of 2.8 mg/kg and 8.2 mg/kg for tetraniliprole in rice straw on an as received basis for dietary burden calculations.

The Meeting noted that residues may occur in rice planted in rotation. However, the residues from prior uses will not contribute significantly to the total residue and were not included in the calculation.

Rice whole crop silage (40 percent dry matter)

The critical GAP for rice in India allows for 2 foliar applications at 60 g ai/ha, with a RTI of 11 days and a PHI of 43. Data were available from supervised residue trials in rice in Brazil, India, Thailand and Vietnam but trials were conducted at 2 × 40 g ai/ha. The Meeting agreed to apply the proportionality approach using the data at PHI is 0 days for crop silage.

Unscaled tetraniliprole residue and unscaled total residue levels were the same in supervised residue with application rates of 2 × 40 g ai/ha (RTI 10-12 days and harvested at 0 DALA) and were (n=5): 1.2, 1.7, 1.8, 1.9, and 3.1 mg/kg (highest individual value of 3.5 mg/kg). Both scaled tetraniliprole residues and scaled total residues were the same and were (n=5): 1.8, 2.6, 2.7, 2.8, and 4.6 mg/kg (highest individual value of 5.2 mg/kg).

The Meeting estimated a median and highest residue level of 2.7 mg/kg and 5.2 mg/kg, respectively for tetraniliprole in rice whole crop silage on an as received basis for dietary burden calculations, both for subsequent maximum residue level and STMR estimations in animal commodities.

The Meeting noted that residues may occur in rice planted in rotation. However, the residues from prior uses will not contribute significantly to the total residue and were not included in the calculation.

Maize/ field corn forage (40 percent dry matter) or sweet corn forage (48 percent dry matter)

The critical GAP in the United States for maize (field corn, sweet corn, popcorn) is 4 foliar applications at 50 gai/ha with a retreatment interval of 7 days and a PHI of 14 days (field corn, popcorn) and 1 day PHI for sweet corn, with a restriction not to feed forage or stover within PHI 14 days.

Tetraniliprole residue levels in maize/field corn forage (PHI=14 days) from trials conducted according to GAP in Canada and the United States were (n=19): < 0.01, 0.040, 0.11, 0.19, 0.24, 0.44, 0.44, 0.48, 0.49, 0.53, 0.55, 0.68, 0.96, 1.1, 1.3, 1.3, 1.4, 2.2, 3.1 mg/kg (highest individual value of 3.6 mg/kg).

Tetraniliprole residue levels in sweet corn forage conducted according to GAP (PHI=14 days) were (n=2): 2.1 and 2.2 mg/kg.

Total tetraniliprole residue levels in maize/field corn forage (PHI=14 days) were (n=19): < 0.01, 0.040, 0.11, 0.19, 0.24, 0.44, 0.44, 0.49, 0.50, 0.53, 0.55, 0.68, 0.97, 1.1, 1.3, 1.3, 1.4, 2.2, 3.1 (highest individual value 3.6 mg/kg).

Total tetraniliprole residue levels in sweet corn forage (PHI=14 days) were (n=2): 2.1 and 2.2 mg/kg.

Noting that the residue levels in sweet corn forage fall within the range of field corn forage the Meeting decided to combine the data. The combined total tetraniliprole residues for median and highest residue estimation, in ranked order, were (n=21): < 0.01, 0.040, 0.11, 0.19, 0.24, 0.44, 0.44, 0.49, 0.50, 0.53, 0.55, 0.68, 0.97, 1.1, 1.3, 1.3, 1.4, 2.1, 2.2, 2.2, 3.1 (highest individual value 3.6 mg/kg).

The Meeting estimated a median residue of 0.55 mg/kg and a highest residue of 3.6 mg/kg for tetraniliprole in maize forage on an as received basis.

The Meeting noted that residues may occur in maize/field corn or sweet corn planted in rotation. However, the residues from prior uses will not contribute significantly to the total residue and were not included in the calculation.

Maize/field corn or sweet corn stover (83 percent dry matter)

The critical GAP in the United States for maize (field corn, sweet corn, popcorn) is 4 foliar applications at 50 g ai/ha with a retreatment interval of 7 days and a PHI of 14 days (field corn, popcorn, and corn grown for seed) and 1 day for sweet corn, with a withholding period of 14 days.

Tetraniliprole residue levels in maize/field corn stover (PHI=14-21 days) from trials conducted according to GAP were (n=21): < 0.01, 0.20, 0.28, 0.37, 0.80, 1.1, 2.3, 2.4, 2.5, 2.6, 2.9, 2.9, 2.9, 3.1, 3.2, 4.3, 4.4, 5.9, 7.9, 9.1, and 10 mg/kg.

Tetraniliprole residue levels in sweet corn stover (PHI=14-36 days) from trials conducted according to GAP were (n=14): < 0.01, 0.014, 0.13, 0.54, 0.72, 0.80, 1.2, 1.4, 1.4, 2.2, 2.5, 5.4, 7.9, and 16 mg/kg (17 mg/kg highest individual value).

Recognizing that normally the dataset with highest levels would be used for median and highest residue estimation the Meeting decided to combine the datasets as that results in the highest median residue.

The combined tetraniliprole residues in field corn and sweet corn stover are (n=35): < 0.01 (2), 0.014, 0.13, 0.20, 0.28, 0.37, 0.54, 0.72, 0.80, 0.80, 1.1, 1.2, 1.4, 1.4, 2.2, 2.3, 2.4, 2.5, 2.5, 2.6, 2.9, 2.9, 2.9, 3.1, 3.2, 4.3, 4.4, 5.4, 5.9, 7.9, 7.9, 9.1, 10, and 16 mg/kg (highest individual value 17 mg/kg).

Total tetraniliprole residues for median and highest residue estimation, in ranked order, are (n=35): < 0.01 (2), 0.014, 0.16, 0.21, 0.28, 0.39, 0.56, 0.80, 0.82, 0.87, 1.1, 1.3, 1.4, 1.5, 2.3 (2), 2.5, 2.5, 2.6 (2), 2.9, 3.0, 3.0, 3.2, 3.3, 4.3, 4.4, 5.5, 6.1, 8.0, 8.0, 9.1, 10, and 16 mg/kg (highest individual value 17 mg/kg).

The Meeting estimated a maximum residue level of 30 mg/kg (dw), based on a dry matter content of 83 percent for maize corn stover.

The Meeting estimated a median residue of 2.5 mg/kg and a highest residue of 17 mg/kg for tetraniliprole in maize stover and sweet corn stover on an as received basis for dietary burden calculations.

The Meeting noted that residues may occur in maize/field corn or sweet corn planted in rotation. However, the residues from prior uses will not contribute significantly to the total residue and were not included in the calculation.

Almond hulls

The critical GAP in Canada and the United States for tree nuts is 4 foliar applications at 45 g ai/ha with a retreatment interval of 7 days and a PHI of 10 days. In trials matching the GAP, tetraniliprole residues, both for maximum residue and median residue level estimation, in almond hulls are (n=5): 0.22, 0.77, 0.80, 1.1, and 1.8 mg/kg.

Based on a dry matter content of 90 percent the Meeting estimated a maximum residue level of 4 mg/kg (dw). The Meeting estimated a median residue level of 0.80 for tetraniliprole in almond hulls on an as received basis for the dietary burden calculations.

Residues from rotational crops to consider for animal feeds

Rotational crop studies indicate that residue can be expected in feed commodities grown in crop rotation. Residues were found in forage and hay from legume vegetables (beans, peas, soya bean and alfalfa) and in forage, hay and straw from cereals (wheat, barley and sorghum). The findings were combined in the two major crop groups to estimate a maximum residue level and median and highest residue levels.

Table 134 Maximum and mean residues found in feed commodities from rotational crops

Matrix	N	Tetraniliprole		Tetraniliprole-N-methyl-quinazolinone		Total [a]	
		max (mg/kg)	median (mg/kg)	max (mg/kg)	median (mg/kg)	max (mg/kg)	median (mg/kg)
Legume feeds with high water content (forage and silage) [b]	58	0.056	< 0.01	< 0.01	< 0.01	0.066 [d]	< 0.01
Legume feeds with low water content (hay) [b]	58	0.19	< 0.01	0.026	< 0.01	0.22	< 0.01
Cereal grains (including pseudocereals) feed products with high water (≥20 percent) content (forage and silage) [c]	28	0.030	< 0.01	< 0.01	< 0.01	0.030	< 0.01
Cereal grains (including pseudocereals) feed products with low water (<20 percent) content (hay, straw)	67	0.097	< 0.01	0.038	< 0.01	0.14	< 0.01

Notes:

[a] Total residue expressed as tetraniliprole.

[b] Based on data in alfalfa, beans, peas and soya bean data.

[c] Based on wheat, barley and sorghum data.

[d] In alfalfa one value of 0.051 mg/kg tetraniliprole + 0.015 mg/kg tetraniliprole N-methyl-quinazolinone also added to 0.066 mg/kg.

The Meeting estimated a median and highest residue level of 0.01 and 0.066 mg/kg (parent + tetraniliprole-N-methyl-quinazolinone), respectively, for the Subgroup of Products of legume feeds with high water (≥ 20 percent) content (forage and silage).

The Meeting estimated a maximum residue level (parent only) of 0.3 mg/kg for the Subgroup of Products of legume feeds with low (< 20 percent) water content (hay), and a median and highest residue level of 0.01 and 0.21 mg/kg (parent + tetraniliprole-N-methyl-quinazolinone), respectively.

The Meeting estimated a median and highest residue level of 0.01 and 0.030 mg/kg (parent + tetraniliprole-N-methyl-quinazolinone), respectively, for the Subgroup of Cereal grains (including pseudocereals) feed products, excluding maize, corn and rice, with high water (≥ 20 percent) content (forage and silage).

The Meeting estimated a maximum residue level of 0.2 mg/kg for the Subgroup of Cereal grains (including pseudocereals) feed products, excluding maize, corn and rice with low water (< 20 percent) content (hay, straw), and a median and highest residue level of 0.01 and 0.14 mg/kg (parent + tetraniliprole-N-methyl-quinazolinone), respectively.

Fate of residues during processing

The Meeting received information on the hydrolysis of tetraniliprole as well as information on the fate of tetraniliprole residues during processing in oranges, apples, plums, grapes, broccoli, tomatoes, mustard greens, soya bean, potato, field corn, and rice.

High temperature hydrolysis

The hydrolysis of [pyrazole-carboxamide-^{14}C]-Tetraniliprole and [phenyl-carbamoyl-^{14}C]-tetraniliprole was studied in sterile buffered aqueous solutions under conditions simulating pasteurization, baking/brewing/boiling, and sterilization.

Tetraniliprole was shown to be stable under the condition representing pasteurization (pH 4, 90 °C, 20 minutes) with 90–95 percent AR recovered. Under baking/brewing/boiling (BBB) (pH 5, 100 °C, 60 minutes) and sterilization (pH 6, 120 °C, 20 minutes) conditions, 65–68 percent and 1.1–1.5 percent AR was recovered as parent at the end of incubation, respectively. Tetraniliprole-N-methyl-quinazolinone was the major degradation product, accounting for 27 to 30 percent AR under BBB conditions and 94 percent AR under sterilization conditions. Two other metabolites, pyrazole-5-N-methyl-amide and desamino-methyl-carboxylic acid, were also found, but these were present in lower amounts (maximum 3 percent AR)

Processing

Estimated processing factors based on total residues (parent plus tetraniliprole-N-methyl-quinazolinone) for the commodities considered at this Meeting are summarised below, together with STMR-P values. Since two dietary burdens will be estimated, one for maximum residue level estimations (parent only=P) in animal commodities and one for STMR estimations (T: total residues, including parent and

tetraniliprole-N-methyl-quinazolinone), sometimes two median values for processed feed commodities were estimated, when processing factors differed (Table 1).

Table 135 Calculated STMRs based on parent plus tetraniliprole-N-methyl-quinazolinone for processed food and feed commodities and for parent only (P) in feed commodities, where different PF were derived

Raw commodity [STMR]	Processed commodity	Individual processing factors	Mean or best estimate	STMR-P = $STMR_{RAC}$ × PF (mg/kg)
Orange [0.14 mg/kg]	Peel	2.6, 3.0	2.8	0.39
	Peeled orange (flesh)	0.081, 0.13	0.11	0.015
	Juice [a]	< 0.02, < 0.03	< 0.02	0.01
	Marmalade	< 0.03, 0.021	0.021	0.01
	Cold extracted peel oil	6.2, 12	9.1	1.27
	Dried pulp [b]	0.96, 1.3 (P: 0.96, 1.2)	1.1	0.15
Apple [0.13 mg/kg]	Juice [a]	0.31, 0.69	0.5	0.065
	Sauce	< 0.019, < 0.030	< 0.019	0.01
	Dried apple	0.032, 0.052	0.042	0.01
	Wet pomace (40 percent DM)	1.6, 1.8	1.7	0.22
	Dry pomace (≈90 percent DM)	6.8, 7.3	7.0	0.91
Plum [0.033 mg/kg]	Prune =dried plum	3.6, 4.6	4.1	0.125
Grape [0.275 mg/kg]	Juice [a]	0.14, 0.17, 0.24, 0.25, 0.31, 0.36	0.245	0.067
	Wine at bottling (white)	0.17, 0.39	0.28	0.077
	Wine at bottling (red)	0.45, 0.57	0.51	0.14
	Wine at first taste (white)	0.21, 0.27	0.24	0.066
	Wine at first taste (red)	0.30, 0.54	0.42	0.12
	Must	0.22, 0.58, 0.62, 0.86	0.57	0.16
	Dried grape	P: 0.92, 1.6 T: 0.94, 1.6	1.26 1.27	0.35
	Wet pomace (≈15 percent DM) [c]	2.1, 2.2, 2.5, 2.8	2.4	0.65
	Dry pomace (90 percent DM)	P: 3.0, 4.9, 7.7, 7.9 T: 3.0, 5.0, 7.9, 8.2	P: 5.9 T: 6.0	P: 1.62 T: 1.65
Broccoli [0.145 mg/kg]	Washed and cooked	0.49	0.49	0.071
Tomato [0.075 mg/kg]	Raw/fresh puree	0.59, 1.1	0.84	0.063
	Paste	P: 1.9, 5.1 T: 3.7, 6.7	P: 3.5 T: 5.2	0.39
Mustard greens [4.0 mg/kg]	Washed and cooked	0.18	0.18	0.72
Soya bean [0.026 mg/kg]	Refined bleached deodorized oil	< 0.02	< 0.02	< 0.01
	Toasted meal	P: 0.027 T: 0.14	P: 0.027 T: 0.14	P: 0.01 T: 0.01
	Hulls	4.2	4.2	0.11
	Aspirated grain fraction	33	33	0.86
Potato	Crisps	< 0.3[f], < 0.5[f], <1[f]	< 0.3	0.01

Raw commodity [STMR]	Processed commodity	Individual processing factors	Mean or best estimate	STMR-P = $STMR_{RAC}$ × PF (mg/kg)
[0.01 mg/kg]	Flakes	< 0.3[if], < 0.5[if], <1[f]	< 0.3	0.01
	Peeled	< 0.3[if], < 0.5[if], <1[f]	< 0.3	0.01
	Steamed and mashed without peel	< 0.3[if], < 0.5[if], <1[f]	< 0.3	0.01
	Cooked with peel	< 0.3[if], < 0.5[if], <1[f]	< 0.3	0.01
	Cooked without peel	< 0.3[if], < 0.5[if], <1[f]	< 0.3	0.01
	Peel	P: < 0.3[if], <1[f], 2.3[if] T: <1[f], 4.6[if], 5.0[if]	P: 2.3 T: 4.8	P: 0.023 T: 0.048
Rice grain [0.01 mg/kg]	Bran	0.28, 0.29	0.285	0.01
	Hulls	2.8, 5.4	4.1	0.041
Field corn/maize [0.01 mg/kg]	Flour	1.2	1.2	0.012
	Grits	< 0.6	< 0.6	0.01
	Meal	1.1	1.1	0.011
	Starch	< 0.6	< 0.6	0.01
	Refined bleached deodorised oil (wet milled)	< 0.6	< 0.6	0.01
	Refined bleached deodorised oil (dry milled)	< 0.6	< 0.6	0.01
	Aspirated grain fractions	4.6	4.6	0.046

Notes:

[a] Highest value selected from limed dry pomace, wet pomace and dry pomace .

[b] Highest value of either pasteurized or raw juice.

[c] Results in highest residue in dietary burden calculation.

[f] Foliar application.

[if] In-furrow application.

Using the estimated maximum residue level of 0.5 mg/kg for oranges and applying the processing factor of 9.1 (based on parent only), the Meeting estimated a maximum residue level of 5 mg/kg for tetraniliprole in orange oil, edible.

Using the estimated maximum residue level of 0.3 mg/kg for plums and applying the processing factor of 4.1 (based on parent only), the Meeting estimated a maximum residue level of 1.5 mg/kg for tetraniliprole in prune, dried.

Using the estimated maximum residue level of 1.5 mg/kg for grapes and applying the processing factor of 1.3 (based on parent only), the Meeting estimated a maximum residue level of 2 mg/kg for tetraniliprole in dried grapes.

Using the estimated maximum residue level of 0.4 mg/kg for tomatoes and applying the processing factor of 3.5 (based on parent only), the Meeting estimated a maximum residue level of 1.5 mg/kg for tetraniliprole in tomato paste.

Residues in animal commodities

Farm animal feeding studies

The Meeting received one feeding study involving tetraniliprole in lactating cows. No poultry feeding study was submitted.

In the dairy cow feeding study, four groups of lactating Holstein cows (3 cows/group) were dosed orally once daily via capsule corresponding with a feeding rate of 0.94, 9.3, 28, or 94 ppm tetraniliprole in feed/day (dry weight) for 29 consecutive days. Milk was collected twice daily and pooled samples were taken at intervals throughout the study period. The animals were sacrificed within 3 to 8 hours after the last dose and samples of liver, kidney, muscle and fat (mesenterial, subcutaneous, and perirenal) were collected for analysis. Samples were stored frozen for up to 20 days before extraction and were analysed within 7 days for tetraniliprole, tetraniliprole-N-methyl-quinazolinone or tetraniliprole-benzylalcohol.

Tetraniliprole residues in milk above the LOQ were found in the milk samples of the animals in the three highest dose groups. Residues of parent tetraniliprole reached a plateau after approximately 7–10 days, with mean (day 7–28) levels of 0.047, 0.10 and 0.18 mg/kg at 9.3 ppm, 28 ppm and 94 ppm feeding levels, respectively.

Similar patterns were found for both metabolites, though at lower concentrations compared to parent. The mean (day 7–28) concentrations for tetraniliprole-N-methyl-quinazolinone were 0.031, 0.073, and 0.10 mg/kg, for the three highest dose groups respectively. And for tetraniliprole-benzylalcohol the mean concentrations found were 0.025, 0.048, and 0.069 mg/kg, respectively. Mean (day 7–28) total residue levels in milk were 0.10, 0.22 and 0.36 mg/kg, respectively.

At 0.94 ppm, parent tetraniliprole was only observed in the liver (0.025–0.037 mg/kg, mean 0.031 mg/kg) and tetraniliprole-N-methyl-quinazolinone was observed in fat tissues only (< 0.01–0.033 mg/kg).

Both parent and metabolite tetraniliprole-N-methyl-quinazolinone were observed in all other tissues at the three higher feeding levels (9.3, 28, and 94 ppm), whereas the benzylalcohol metabolite was only observed in liver in the mid and high dose groups (28 and 94 ppm) and kidney in the high dose group (94 ppm). The total residues below represents the sum of parent tetraniliprole + tetraniliprole-N-methyl-quinazolinone + tetraniliprole benzylalcohol.

At 9.3 ppm, the highest concentration of parent was 0.37 mg/kg in liver, 0.067 mg/kg in kidney, 0.023 mg/kg in muscle, and 0.063 mg/kg in fats. Total highest residues were 0.41 (mean 0.37) mg/kg in liver, 0.10 (mean 0.085) mg/kg in kidney, 0.043 (mean 0.041) mg/kg in muscle, 0.26 (highest mean 0.20) mg/kg in three fats. The results across the three different fat types were similar for both parent and total residues.

At 28 ppm, the highest concentration of parent was 0.87 mg/kg in liver, 0.19 mg/kg in kidney, 0.060 mg/kg in muscle, and 0.12 mg/kg in fats. Total highest residues were 0.97 (mean 0.70) mg/kg in liver, 0.27 (mean 0.19) mg/kg in kidney, 0.094 (mean 0.075) mg/kg in muscle, and 0.83 (highest mean 0.54) mg/kg in three fats.

At 94 ppm, the highest concentration of parent was 1.5 mg/kg in liver, 0.28 mg/kg in kidney, 0.090 mg/kg in muscle, and 0.22 mg/kg in fats. Total highest residues were 1.7 (mean 1.4) mg/kg in liver, 0.34 (mean 0.31) mg/kg in kidney, 0.16 (mean 0.14) mg/kg in muscle, 1.2 (highest mean 0.77) mg/kg in fats.

Farm animal dietary burden

The dietary burden was based on the intake of tetraniliprole for maximum residue estimation. An additional dietary burden calculation based on the intake of tetraniliprole + tetraniliprole-N-methyl-quinazolinone was calculated for STMR and HR estimation. Residues found in feed commodities in both supervised residue trials and field rotational crops studies and processing studies were used for dietary burden calculations (see the various tables at the appropriate sections throughout the appraisal).

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the JMPR in 2022. The dietary burdens, estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO manual, are presented in Annex 6 and summarised below.

Table 136 Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden: tetraniliprole, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	max	mean	Max	mean	max	mean	max	mean
Beef cattle	3.21	0.55	19.8	6.8	13.6	3.2	4.9	1.8
Dairy cattle	6.05	1.091	17.6	6.6	29.46❶❷	12.7	11.2	3.73
Poultry – broiler	0.015	0.015	0.026	0.026	0.014	0.014	0.012	0.012
Poultry – layer	0.015	0.015	0.47❸❹	0.077	0.014	0.014	0.013	0.013

Notes:

❶ Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues.

❷ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk.

❸ Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues.

❹ Highest maximum poultry dietary burden suitable for MRL estimates for poultry eggs.

Table 137 Estimated maximum and mean dietary burdens of farm animals for STMR calculation

	Animal dietary burden: tetraniliprole + tetraniliprole-N-methyl-quinazolinone, ppm of dry matter diet							
	United States-Canada		European Union		Australia		Japan	
	max	mean	Max	mean	max	mean	max	mean
Beef cattle	3.22	0.57	19.8	6.85	13.61	3.34	5.25	1.91
Dairy cattle	6.07	1.08	17.56	6.617	29.46	12.76	11.2	3.73
Poultry – broiler	0.015	0.015	0.026	0.026	0.014	0.014	0.012	0.012
Poultry – layer	0.015	0.015	0.47	0.077	0.014	0.014	0.013	0.017

Notes:

❶ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❷ Highest mean dairy cattle dietary burden suitable for STMR estimates for mammalian milk.

❸ Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues.

❹ Highest mean poultry burden suitable for STMR estimates for poultry eggs.

Animal commodity maximum residue levels

Cattle

For beef and dairy cattle, a maximum and mean dietary burden of 33.7 and 12 ppm were estimated. For a maximum residue level estimation, the highest residues in the tissues and the mean residues in milk (day 7–28) were calculated by taking the maximum dietary burden (33.7 ppm) and interpolation of the highest

(tissues) and mean (milk, day 7–28) residue levels (parent only) found in the individual animals in the feeding study at 28 and 94 ppm.

The STMR values for the tissues and milk were calculated from the mean dietary burden level of 12 ppm by interpolation of the mean total residue levels (parent, tetraniliprole-N-methyl-quinazolinone, and tetraniliprole-benzylalcohol) found in milk and tissues of animals dosed at 9.4 and 28 ppm.

Table 138 Residues in milk and tissues from cattle dosed with tetraniliprole in the diet

Tetraniliprole feeding study	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
MRL (beef or dairy cattle); parent only							
Feeding study	28	0.10	28	0.060	0.87	0.19	0.12
	94	0.18	94	0.090	1.50	0.28	0.22
Dietary burden and high residue	29.46	0.10	29.46	0.061	0.88	0.19	0.12
STMR Determination (beef or dairy cattle); parent + tetraniliprole-N-methyl-quinazolinone + tetraniliprole-benzylalcohol, expressed as tetraniliprole							
Feeding study	9.3	0.10	9.3	0.041	0.37	0.085	0.20
	28	0.22	28	0.075	0.70	0.19	0.54
Dietary burden and residue estimate	12.76	0.12	12.76	0.047	0.43	0.10	0.26

The Meeting concluded that residues > 0.01 mg/kg are expected in milk, muscle, liver, kidney and fat and estimated maximum residue levels of 0.15 mg/kg for milk, 0.1 mg/kg for meat (0.8 × 0.061 + 0.2 × 0.12= 0.728 mg/kg), 1.0 mg/kg for edible offal (based on liver), and 0.15 mg/kg for fat.

For estimating dietary exposure to total residues, calculated STMRs are: 0.12 mg/kg for milk, 0.047 mg/kg for muscle, 0.43 mg/kg for liver, 0.10 mg/kg for kidney, and 0.26 mg/kg for fat.

Poultry

For poultry a maximum (parent only) and mean (parent + tetraniliprole-N-methyl-quinazolinone) dietary burden of 0.47 and 0.077 ppm, respectively were estimated, respectively. However, no feeding study in laying hens was provided.

The laying hen metabolism studies involved administration of approximately 18 ppm tetraniliprole in the diet, which is about 36 times overdosed compared to the expected maximum dietary burden (maximum of 0.5 ppm). Scaling of the highest residue (parent+tetraniliprole-N-methyl-quinazolinone + tetraniliprole-benzylalcohol (+conjugates)) found in poultry tissues and eggs (0.12 mg eq/kg) to the maximum dietary burden would result in residues at up to a maximum of 0.0033 mg eq/kg.

Acknowledging that there might be some exposure and a feeding study is preferred, the Meeting concluded that no residues > 0.01 mg/kg are expected in eggs and poultry tissues and estimated maximum residue levels of 0.01(*) mg/kg or poultry meat, eggs, fat and edible offal as well as STMRs values of 0.01 mg/kg.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed in Annex 1 are suitable for establishing maximum residue limits and for IEDI, IESTI and GECDE assessments.

Definition of the residue for compliance with the MRL for plant commodities: *tetraniliprole*.

Definition of the residue for dietary risk assessment for plant commodities: *tetraniliprole + tetraniliprole-N-methyl-quinazolinone, expressed as tetraniliprole.*

Definition of the residue for compliance with the MRL for animal commodities: *tetraniliprole*.

Definition of the residue for dietary risk assessment for animal commodities: *tetraniliprole + tetraniliprole-N-methyl-quinazolinone + tetraniliprole-benzylalcohol, expressed as tetraniliprole.*

The residue is not fat-soluble.

Table 139 Recommendations for residues of tetraniliprole from the 2022 JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR,
		New	Previous	STMR-P, mg/kg
AM 0660	Almond hulls	4 (dw)	-	median 0.81
VB 0041	Cabbages, Head	2.0	-	0.012 median 0.135 highest 1.2
AS 3304	Cereal grains (including pseudocereals) feed products with low water (<20 percent) content (hay and/or straw) Subgroup of, excluding rice, maize/field corn, and sweet corn)	0.2 (dw)	-	median 0.01 (ar) highest 0.14 (ar)
FS 0013	Cherries, Subgroup of	1.5	-	0.29
MO 0105	Edible offal (Mammalian)	1.0	-	0.43
PE 0112	Eggs	0.01*	-	0
VB 0042	Flowerhead Brassicas, Subgroup of	0.5	-	0.145
VO 0050	Fruiting vegetables, other than Cucurbits, Group of, excluding okra, martynia and roselle	0.4	-	0.075
DF 0269	Grape, dried (=Currants, Raisins and Sultanas)	2.0	-	0.35
JF 0269	Grape, juice	-	-	0.067
-	Grape, must	-	-	0.16
-	Grape, wine	-	-	0.14
JF 0009	Group of Pome Fruit, juices	-	-	0.065
-	Group of Pome Fruit, sauce	-	-	0.01
DF 0009	Group of Pome Fruit, dried	-	-	0.01
VL 0054	Leaves of Brassicaceae, Subgroup of	15	-	4.0 median 4.0 highest 7.3
FC 0002	Lemons and Limes (including Citron), Subgroup of	1.5		0.19
GC 2091	Maize cereals, Subgroup of	0.015	-	0.01
AS 3558	Maize stover	30 (dw)		median 2.5 (ar) highest 17 (ar)
CF 1255	Maize flour	-	-	0.012
CF 0645	Maize meal	--		0.011
-	Maize grits	-	-	0.01

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR, STMR-P, mg/kg
		New	Previous	
-	Maize starch	-	-	0.01
OR 0645	Maize, refined bleached deodorized oil	-	-	0.01
MF 0100	Mammalian fats (except milk fats)	0.15	-	0.26
FC 0003	Mandarins (including Mandarin-like hybrids), Subgroup of	1.0	-	0.185
MM 0095	Meat (from mammals other than marine mammals)	0.1	-	muscle: 0.047 fat: 0.26
ML 0106	Milks	0.15	-	0.12
FC 0004	Oranges, Sweet, Sour (including Orange-like hybrids), Subgroup of	0.5	-	0.015#
JF 0004	Orange, juice	-	-	0.01
OR 0004	Orange oil, edible	5?	-	1.27
-	Orange, marmalade	-	-	0.01
-	Orange, peeled	-	-	0.015
-	Orange, peel	-	-	0.39
FS 2001	Peaches (including Nectarines and Apricots), Subgroup of	0.7	-	0.089
HS 0444	Peppers, Chili, dried	4.0	-	0.75
FS 0014	Plums, Subgroup of	0.3	-	0.033
FP 0009	Pome fruits, Group of, excluding Japanese persimmon	0.4	-	0.13
PO 0111	Poultry, edible offal	0.01*	-	0.01
PF 0111	Poultry, fats	0.01*	-	0.01
PM 0110	Poultry, meat	0.01*	-	muscle: 0.01 fat: 0.01
AL 3301	Products of legume feeds with low water (<20 percent) content (hay), Subgroup of	0.3 (dw)	-	median 0.01 (ar) highest 0.22 (ar)
DF 0014	Prune, dried	1.5	-	0.125
FC 0005	Pummelos and Grapefruits (including Shaddock-like hybrids, among others grapefruit), Subgroup of	0.9	-	0.091
GC 2088	Rice cereals, Subgroup of	0.02	-	0.01
GM 0649	Rice, husked	0.01*	-	0.01
CM 1205	Rice, polished	0.01*	-	0.01
AS 0649	Rice, hay and/or straw	20 (dw)	-	
FB 2008	Small fruit vine climbing, Subgroup of	1.5	-	0.275
VD 0541	Soya bean (dry)	0.2	-	0.026
GC 0447	Sweet Corn (corn-on-the-cob)	0.01*	-	0.01
DM 0448	Tomato paste	1.5		0.39
TN 0085	Tree nuts, Group of	0.03	-	0.01
VR 2071	Tuberous and corm vegetables, Subgroup of	0.02	-	0.01

Notes:

ar) – as received; (dw) – dry weight; #STMR for flesh based on 0.14 mg/kg x PF of 0.11

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for tetraniliprole is 0–2 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for tetraniliprole were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 0–0 percent of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of tetraniliprole from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2021 JMPR decided that an ARfD for tetraniliprole was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of tetraniliprole from the uses considered is unlikely to present a public health concern.

Threshold of toxicological concern (TTC) consideration for metabolites

At the JMPR 2021 Meeting the WHO concluded that for the metabolites T-pyrazole-5-carboxylic acid, T-N-methyl-quinazolinone-benzylalcohol, T-despyridyl-N-methyl-quinazolinone, T-pyrazole-5-amide, and T-N-methyl-quinazolinone-pyrazole-3-carboxyilic acid the TTC Cramer Class III could be applied (no indication for genotoxicity).

For the current Meeting additional information was provided to the WHO. For three metabolites T-quinazolinone, T-pyridinyl-pyrazole-5-carboxylic acid, and T-pyrazole-5-N-methyl-amide the 2022 WHO concluded that the TTC Cramer Class III could be applied (no indication for genotoxicity) for these metabolites.

The exposure based on the residue levels found in animal commodities from the goat and laying hen metabolism studies, resulted in the following maximum long-term exposures (T= tetraniliprole). It is noted that the exposure levels were not corrected for the dose levels used in the goat study (slightly under dosed) but were corrected for the dose levels used in the laying hen study (36 times over dosed):

TTC III (< µg/kg bw)

T-quinazolinone (goat)	0.11 µg/kg bw
T-pyrazole-5-carboxylic acid (goat and poultry)	0.07 µg/kg bw
T-N-methyl-quinazolinone-benzylalcohol (goat)	0.033 µg/kg bw
T-pyridinyl-pyrazole-5-carboxylic acid (goat)	0.03 µg/kg bw
T-despyridyl-N-methyl-quinazolinone (poultry)	0.03 µg/kg bw
T-pyrazole-5-N-methyl-amide (goat and poultry)	0.02 µg/kg bw
T-pyrazole-5-amide (poultry and goat liver only)	0.01 µg/kg bw
T-N-methyl-quinazolinone-pyrazole-3-carboxylic acid (goat)	0.0023 µg/kg bw

The Meeting concluded that the exposures to these metabolites are below the TTC for Cramer Class 3 compounds of 1.5 µg/kg bw/day and were unlikely to present a health concern from the uses evaluated by the current Meeting.

In addition, for a number of poultry specific metabolites no toxicity data was available. The Meeting decided these should be assessed using the TTC approach for genotoxic compounds (below the threshold of 0.0025 µg/kg bw/day).

TTC for genotoxic compounds (< 0.0025 µg/kg bw – corrected for dietary burden)

T-despyridyl (poultry)	0.00034 µg/kg bw
Tetrazole-conjugates (poultry)	0.00063 µg/kg bw
T-despyridyl-N-methyl-quinazolinone-hydroxy/ T-despyridyl-hydroxy (poultry)	0.00019 µg/kg bw
T-deschloro-desmethyl-amide (poultry)	0.00015 µg/kg bw
T-despyridyl-quinazolinone (poultry)	0.00014 µg/kg bw
T-pyrazole-5-N-methyl-amide-hydroxy (poultry)	0.000094 µg/kg bw
T--deschloro-desmethyl-amide	0.00015 µg/kg bw

The Meeting concluded that these poultry metabolites are below the TTC for genotoxic compound and were unlikely to present a health concern from the uses evaluated by the current Meeting.

Should further uses be considered in the future, these conclusions may need to be re-evaluated.

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EnSa-14-0488	Bongartz, R.; Schallau, K.	2015b	Amendment No. 1 to metabolism of [phenyl-carbamoyl-^{14}C]BCS-CL73507 in paddy rice after foliar treatment, Bayer Report No.: EnSa-14-0488 Edition Number: M-496787-02-1 Date: 2014-09-09; amended: 2015-12-15 GLP/GEP: Yes, unpublished
EnSa-14-0485	Bongartz, R.; Schmeling, S.	2014a	Metabolism of [pyrazole-carboxamide-^{14}C]BCS-CL73507 in tomatoes Bayer Report No.: EnSa-14-0485 Edition Number: M-495009-01-1 MRID#: 50170195 Date: 2014-08-05 GLP/GEP: Yes, unpublished
EnSa-14-0484	Bongartz, R.; Schmeling, S.	2014b	Metabolism of [phenyl-carbamoyl-^{14}C]BCS-CL73507 in tomatoes Bayer Report No.: EnSa-14-0484 Edition Number: M-495019-01-1 MRID#: 50170196 Date: 2014-08-05 GLP/GEP: Yes, unpublished
Ensa-13-1093	Bongartz, R.; Schmeling, S.	2014c	Nature of the residues of [pyrazole-carboxamide-^{14}C]BCS-CL73507 and [phenyl-carbamoyl-^{14}C]BCS-CL73507 in processed commodities high temperature hydrolysis, Bayer Report No.: Ensa-13-1093 Edition Number: M-475964-01-1 MRID#: 50216508 Date: 2014-01-21 GLP/GEP: Yes, unpublished
RAFV0032	Brungardt, J.	2018	Tetraniliprole SC 200–Magnitude of the residue in rice processed commodities SynTech Research Laboratory Services, LLC, Stilwell, KS, United States, Bayer Report No.: RAFV0032 Edition Number: M-638211-01-1 Date: 2018-10-12 GLP/GEP: Yes, unpublished
I14-046	Carvalho, F.	2016	Determinação de resíduos de Tetraniliprole e seu metabólito na cultura do Arroz (grãos) após aplicação de Tetraniliprole 200 SC em ensaios no Brasil Bayer S/A Departamento de Estudos em Segurança Alimentar Rua Domingos Jorge, Nº 1.100 Prédio 606, Bairro: Socorro São Paulo–SP, Brasil Report No. I14-046 Edition Number: M-567467-02-1 Date: 2016-09-27 (first addendum 2017-02-16)

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			GLP/GEP: Yes, unpublished
M-675295-01-1	Chavan, M.	2019	India–Label of tetraniliprole 18.18% w/w SC (tetraniliprole SC 200) Bayer Report No.: M-675295-01-1 Date: 2019-12-10 GLP/GEP: n.a., unpublished
122G1313	Claussen, F.	2016	Independent laboratory validation (ILV) of analytical method 01414 for the determination of BCS-CL73507 and metabolite BCS-CQ63359 in citrus and broccoli by HPLC-MS/MS EPL Bio-Analytical Services (EPL-BAS), Niantic, IL, United States Bayer Report No.: 122G1313 Edition Number: M-564166-01-1 MRID#: 50216520 Date: 2016-08-31 GLP/GEP: Yes, unpublished
RAFVP036	Dacus, S. C.	2016	Waiver of the requirement for poultry feeding studies for tetraniliprole (BCS CL73507), Bayer Report No.: RAFVP036 Edition Number: M-574911-01-1 MRID#: 50216532 Date: 2016-12-15 GLP/GEP: No, unpublished
RAFVN033	Dallstream, K. A.	2016	BCS-CL73507–Magnitude of the residues in edible podded legumes grown as a rotational crop (crop subgroup 6A) Bayer Report No.: RAFVN033 Edition Number: M-560245-01-1 MRID#: 50216561 Date: 2016-07-19 GLP/GEP: Yes, unpublished
RAFVP085	Dallstream, K. A.; Jerkins, E.	2016a	BCS-CL73507- Magnitude of the residue in/on barley grown as a rotational crop (as part of crop groups 15 and 16, except rice) Bayer Report No.: RAFVP085 Edition Number: M-555094-01-1 MRID#: 50216562 Date: 2016-05-19 GLP/GEP: Yes, unpublished
RAFVP074	Dallstream, K. A.; Jerkins, E.	2016b	BCS-CL73507: Magnitude of the residues in potatoes (Crop subgroup 1C), Bayer Report No.: RAFVP074 Edition Number: M-557979-01-1 MRID#: 50216536 Date: 2016-06-27 GLP/GEP: Yes, unpublished
RAFVP064	Dallstream, K. A.; Jerkins, E.	2016c	Tetraniliprole SC 200: Magnitude of residues in apple processed commodities–Tetraniliprole 200 SC (200 g/L) (tetraniliprole SC 200 G), Bayer Report No.: RAFVP064 Edition Number: M-560638-01-1 MRID#: 50216556 Date: 2016-07-26 GLP/GEP: Yes, unpublished

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CSL-13-0703.01	Dreisch, S.	2013	BCS-CL73507: Vapour pressure consilab Gesellschaft fuer Anlagensicherheit mbH, Frankfurt am Main, Germany Bayer Report No.: CSL-13-0703.01 Edition Number: M-467652-01-1 MRID#: 50170033 Date: 2013-10-07 GLP/GEP: Yes, unpublished
PA13/064	Eyrich, U.; Ziemer, F.	2013a	BCS-CL73507: Physical characteristics colour, physical state and odour Bayer Report No.: PA13/064 Edition Number: M-464861-01-1 Date: 2013-09-11 GLP/GEP: Yes, unpublished
PA13/062	Eyrich, U.; Ziemer, F.	2013b	BCS-CL73507: Partition coefficients 1-octanol / water at pH 4, pH 7 and pH 9 (HPLC method) Bayer Report No.: PA13/062 Edition Number: M-472127-01-1 MRID#: 50170030 Date: 2013-12-10 GLP/GEP: Yes, unpublished
PA13/103	Eyrich, U.; Ziemer, F.	2014a	BCS-CL73507: Solubility in organic solvents Bayer Report No.: PA13/103 Edition Number: M-476259-01-1 MRID#: 50170032 Date: 2014-01-30 GLP/GEP: Yes, unpublished
PA14/024	Eyrich, U.; Ziemer, F.	2014b	BCS-CL73507: Determination of the surface tension Bayer Report No.: PA14/024 Edition Number: M-485602-01-2 Date: 2014-05-07 GLP/GEP: Yes, unpublished
RAFVP104-01	Fischer, D. R.; Roberts, J.	2016	Amended Report 1 to RAFVP104–BCS-CL73507 200 SC–Magnitude of the residues in pome fruit (CG11) Bayer Report No.: RAFVP104-01 Edition Number: M-561106-02-1 MRID#: 50216538 Date: 2016-08-02; amended: 2016-10-24 GLP/GEP: Yes, unpublished
MR-13/100	Freitag, T.; Koch, V.	2015	Analytical method 01373 for the determination of BCS-CL73507 and the metabolites BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673 and BCS-CU81056 in soil and sediment by HPLC-MS/MS Bayer Report No.: MR-13/100 Edition Number: M-486110-02-1 MRID#: 50216525 Date: 2014-05-12; amended: 2015-01-12 GLP/GEP: Yes, unpublished
14-3404	Freitag, T.; Hoffmeister, R.	2017	Determination of the residues of BCS-CL73507 in/on grape and the processed fractions (berry, washed; juice; must; pomace, dried; pomace,

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			wet; raw juice; wine at bottling and wine at first taste test) after Spraying and Spraying, low-volume of BCS-CL73507 SC 200 in the field in Germany, France (North), Italy and Spain, Bayer Report No.: 14-3404 Report includes Trial Nos.: 14-3404-01, 14-3404-02, 14-3404-03, and 14-3404-04 Edition Number: M-577324-01-1 Date: 2017-01-12 GLP/GEP: Yes, unpublished
RAFVN035-01	Gould, T.; Jerkins, E.	2016	BCS-CL73507–Magnitude of the residues in succulent shelled legumes grown as a rotational crop–BCS-CL73507 200 SC (200 g/L) (tetraniliprole SC 200 G) Bayer Report No.: RAFVN035-01 Edition Number: M-560729-02-1 MRID#: 50216567 Date: 2016-07-19; amended: 2016-11-09 GLP/GEP: Yes, unpublished
RAFVP089-02	Gould, T. J.; Dallstream, K. A.	2017	BCS-CL73507: Magnitude of the residues in citrus fruit (crop group 10-10), Bayer Report No.: RAFVP089-02 Edition Number: M-563131-03-1 Date: 2016-08-17, amended: 2017-4-28 GLP/GEP: Yes, unpublished
SARS-15-15	Greenland, R. G.	2016a	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on cherry Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-15-15 Edition Number: M-570651-01-1 MRID#: 50216542 Date: 2016-11-09 GLP/GEP: Yes, unpublished
SARS-15-16	Greenland, R. G.	2016b	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on peaches Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-15-16 Edition Number: M-572119-01-1 MRID#: 50216545 Date: 2016-11-15 GLP/GEP: Yes, unpublished
SARS-14-01	Greenland, R. G.	2016c	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on plums and prunes Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-14-01 Edition Number: M-572124-01-1 MRID#: 50216546 Date: 2016-11-16 GLP/GEP: Yes, unpublished
SARS-14-07	Greenland, R. G.	2016d	Magnitude and decline of F4260 (BCS-CL73507) and metabolite

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			residues in/on grape and grape processed commodities Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-14-07 Edition Number: M-572121-01-1 MRID#: 50216547 Date: 2016-11-16 GLP/GEP: Yes, unpublished
SARS-14-19	Greenland, R. G.	2016e	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on tomato and tomato processed commodities Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-14-19 Edition Number: M-572627-01-1 MRID#: 50216544 Date: 2016-11-18 GLP/GEP: Yes, unpublished
SARS-14-20	Greenland, R. G.	2016f	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on pepper Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-14-20 Edition Number: M-570122-01-1 MRID#: 50216540 Date: 2016-10-31 GLP/GEP: Yes, unpublished
SARS-14-11	Greenland, R. G.	2016g	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on leaf lettuce Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-14-11 Edition Number: M-572118-01-1 MRID#: 50216543 Date: 2016-11-15 GLP/GEP: Yes, unpublished
SARS-15-12	Greenland, R. G.	2016h	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on head lettuce Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-15-12 Edition Number: M-570646-01-1 MRID#: 50216550 Date: 2016-11-09 GLP/GEP: Yes, unpublished
SARS-14-14	Greenland, R. G.	2016i	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on spinach Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-14-14 Edition Number: M-570124-01-1 MRID#: 50216541 Date: 2016-11-03

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			GLP/GEP: Yes, unpublished
SARS-15-03	Greenland, R. G.	2016j	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on soybean and soybean processed commodities Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-15-03 Report includes Trial Nos.: 2015RES-ANT1881 Edition Number: M-574330-02-1 MRID#: 50216551 Date: 2016-12-05 GLP/GEP: Yes, unpublished
SARS-15-17	Greenland, R. G.	2016k	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on almond Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-15-17 Report includes Trial Nos.: SARS-15-17-CA1, SARS-15-17-CA2, SARS-15-17-CA3, SARS-15-17-CA4, and SARS-15-17-CA5 Edition Number: M-572123-01-1 MRID#: 50216548 Date: 2016-11-16 GLP/GEP: Yes, unpublished
SARS-14-02	Greenland, R. G.	2016l	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on pecan Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-14-02 Edition Number: M-570119-01-1 MRID#: 50216539 Date: 2016-10-31 GLP/GEP: Yes, unpublished
SARS-15-05	Greenland, R. G.; Stewart, P.	2016	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on sweet corn Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-15-05 Report includes Trial Nos.: 2015RES-ANT1839 Edition Number: M-574351-01-2 MRID#: 50301901 Date: 2016-11-28 GLP/GEP: Yes, unpublished
EnSa-14-0308	Hein, E. M.; Kasel, D.	2016	[pyrazole-carboxamide-^{14}C]BCS-CL73507—Hydrolytic degradation Bayer Report No.: EnSa-14-0308 Edition Number: M-565616-01-1 MRID#: 50170045 Date: 2016-09-14 GLP/GEP: Yes, unpublished
EnSa-13-0321	Heinemann, O.; Junge, T.	2014	[Pyrazole-carboxamide-^{14}C]BCS-CL73507: Phototransformation in natural water Bayer Report No.: EnSa-13-0321 Edition Number: M-489424-01-1

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			MRID#: 50170047 Date: 2014-05-16 GLP/GEP: Yes, unpublished
EnSa-13-0320	Heinemann, O.; Kasel, D.	2014	[Pyrazole-carboxamide-^{14}C]BCS-CL73507: Phototransformation in water Bayer Report No.: EnSa-13-0320 Edition Number: M-484185-01-1 MRID#: 50170046 Date: 2014-04-09 GLP/GEP: Yes, unpublished
EnSa-16-0158	Heinemann, O.; Kasel, D.	2016a	[Pyridinyl-2-^{14}C]BCS-CL73507: Phototransformation in natural water Bayer Report No.: EnSa-16-0158 Edition Number: M-568022-01-1 MRID#: 50170049 Date: 2016-09-30 GLP/GEP: Yes, unpublished
EnSa-14-1369	Heinemann, O.; Kasel, D.	2016b	[pyrazole-carboxamide-^{14}C] BCS-CL73507: Paddy soil metabolism in one soil Bayer Report No.: EnSa-14-1369 Edition Number: M-545810-01-1 Date: 2016-01-26 GLP/GEP: Yes, unpublished
EnSa-13-0244	Hellpointner, E.; Junge, T.	2015	Amendment no 1 to [Pyrazole-carboxamid-^{14}C]BCS-CL73507: Aerobic soil metabolism and time-dependent sorption in four European soils Bayer Report No.: EnSa-13-0244 Edition Number: M-465975-02-1 MRID#: 50170051 Date: 2013-09-23 GLP/GEP: Yes, unpublished
M-675095-01-1	Hullebroeck, M.	2019	Letter of access to certain FMC registration data on tetraniliprole (CAS 1229654-66-3) FMC Corporation, Philadelphia, PA, United States, Bayer Report No.: M-675095-01-1 Date: 2019-12-09 GLP/GEP: n.a., unpublished
M-675297-01-1	Kim, S. H.; Utari, B.	2018	Korea–Label of Vayego (tetraniliprole 18.18% w/w SC, tetraniliprole SC 200), Bayer Report No.: M-675297-01-1 Date: 2018-10-26 GLP/GEP: n.a., unpublished
EnSa-14-0217	Koenig, H.; Beckmann, M.	2014	[Pyrazole-carboxamide-^{14}C]BCS-CL73507: Phototransformation on soil Bayer Report No.: EnSa-14-0217 Edition Number: M-493228-01-1 MRID#: 50170050 Date: 2014-07-28 GLP/GEP: Yes, unpublished
RAFVP051	Krolski, M.; Jerkins, E.	2016	BCS-CL73507–Magnitude of the residues in field rotational crops, soybeans and wheat Bayer Report No.: RAFVP051

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			Report includes Trial Nos.: FV113-14RA, FV114- 14RA, FV115-14RA, FV116-14RA, FV117-14RA, and FV118-14RA Edition Number: M-568415-01-1 MRID#: 50216572 Date: 2016-10-13 GLP/GEP: Yes, unpublished
S16-00229	Lakaschus, S.; Lau, E.	2016	Independent laboratory validation of the analytical residue method 01463 for the determination of BCS-CL73507 and its metabolite BCS-CQ63359 in samples of plant origin by HPLC-MS/MS Eurofins Agroscience Services Chem GmbH (EAS Chem), Hamburg, Germany, Bayer Report No.: S16-00229 Edition Number: M-554622-01-1 MRID#: 50216524 Date: 2016-04-26 GLP/GEP: Yes, unpublished
RAFVN039	Lam, C.	2016	BCS-CL73507–Magnitude of the residues in/on dry bulb onions grown as a rotational crop (crop subgroup 3-07A) Bayer Report No.: RAFVN039 Edition Number: M-562968-01-1 MRID#: 50216566 Date: 2016-08-03 GLP/GEP: Yes, unpublished
RAFVP084	Lam, C.; Jerkins, E.	2016	BCS-CL73507–Magnitude of the residues in canola grown as a rotational crop (Crop Group 20) Bayer Report No.: RAFVP084 Edition Number: M-556294-01-1 MRID#: 50216564 Date: 2016-05-24 GLP/GEP: Yes, unpublished
BCS-0531	Massault, R.	2017	Determination of residues of tetraniliprole in pome fruit following one, two or three foliar applications of BCS-CL73507 200 SC at 10 and 20 mL/100 L at various timings, Bayer Report No.: BCS-0531 Edition Number: M-598808-01-1 Date: 2017-08-14 GLP/GEP: Yes, unpublished
BCS-0532.01	Massault, R.	2018	Determination of residues of tetraniliprole in pome fruit following one, two or three foliar applications of BCS-CL73507 200 SC at 12.5 and 25 mL/100 L at various timings, Bayer Report No.: BCS-0532.01 Edition Number: M-631139-01-1 Date: 2018-07-24 GLP/GEP: Yes, unpublished
RAFVN036	Miller, A.; Jerkins, E.	2016	BCS-CL73507: Magnitude of the residues in leafy Brassica greens (Crop Group 5B), Bayer Report No.: RAFVN036 Edition Number: M-557177-01-1 MRID#: 50216535 Date: 2016-06-03 GLP/GEP: Yes, unpublished
RAFVN037-01	Miller, A.; Roberts, J.	2016	BCS-CL73507–Magnitude of the residues in legumes grown as a

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			rotational crop (crop subgroup 6c and as part of crop group 7)– BCS-CL73507 200 SC (200 g/L) (tetraniliprole SC 200 G) Bayer Report No.: RAFVN037-01 Edition Number: M-560950-02-1 MRID#: 50216568 Date: 2016-07-29 GLP/GEP: Yes, unpublished
M-557172-01-1	Mislankar, S.; Haddix, J.	2016	[Pyrazole-carboxamide-^{14}C]BCS-CL73507: Aerobic soil metabolism and time-dependent sorption in six US soils Bayer Report No.: M-557172-01-1 MRID#: 50170052 Date: 2016-06-15 GLP/GEP: Yes, unpublished
RAFVN029	Murphy, I.; Jerkins, E.	2016a	BCS-CL73507–Magnitude of the residues in/on sorghum grown as a rotational crop (as part of crop groups 15 and 16, except rice)–BCS-CL73507 200 SC (200 g/L) (tetraniliprole SC 200 G) Bayer Report No.: RAFVN029 Edition Number: M-559018-01-1 MRID#: 50216570 Date: 2016-07-07 GLP/GEP: Yes, unpublished
RAFVN030	Murphy, I.; Jerkins, E.	2016b	BCS-CL73507–Magnitude of the residues in/on sunflowers grown as a rotational crop Bayer Report No.: RAFVN030 Edition Number: M-558451-01-1 MRID#: 50216565 Date: 2016-06-27 GLP/GEP: Yes, unpublished
20150415.01	Nau, M.	2016	Tetraniliprole (BCS-CL73507), technical substance: Melting point, boiling point, thermal stability Siemens AG, Frankfurt am Main, Germany Bayer Report No.: 20150415.01 Edition Number: M-548001-01-1 MRID#: 50170026 Date: 2016-02-12 GLP/GEP: Yes, unpublished
RAFVP101-01	Netzband, D.; Beedle, E.	2016	BCS-CL73507–Magnitude of the residues in cucurbit vegetables grown as a rotational crop (Crop Group 9) Bayer Report No.: RAFVP101-01 Edition Number: M-563500-02-1 MRID#: 50216569 Date: 2016-08-15 GLP/GEP: Yes, unpublished
RAFVP017	Netzband, D. J.; Jenks, M. G.	2016	Independent Laboratory Validation of analytical method 01373 for the determination of BCS-CL73507 and the metabolites BCS-CQ63359, BCS-CR60014, BCS-CR74541, BCS-CU81055, BCS-CT30673 and BCS-CU81056 in soil and sediment by HPLC-MS/MS Bayer Report No.: RAFVP017 Edition Number: M-554130-01-1

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			MRID#: 50170146 Date: 2016-05-04 GLP/GEP: Yes, unpublished
RAFVP096-01	Netzband, D.; Roberts, J.	2016	BCS-CL73507: Magnitude of the residues in head and stem Brassica vegetables (crop subgroup 5A), Bayer Report No.: RAFVP096-01 Edition Number: M-565724-02-1 MRID#: 50216533 Date: 2016-09-13 GLP/GEP: Yes, unpublished
M-675296-01-1	Ngo Thi Tu, T.; Utari, B.	2019	Cambodia–Label of Vayego 200SC (tetraniliprole SC 200) Bayer Report No.: M-675296-01-1 Date: 2019-05-09 GLP/GEP: n.a., unpublished
RAFVP018	Perez, S.; Marshall, M.	2016	Independent laboratory validation (ILV) of Bayer method FV-004-W16-01 for the determination of residues of tetraniliprole (BCS-CL73507) and its metabolites BCS-CQ63359, BCS-CU81055, BCS-CR74541, BCS-CR60014, BCS-CU81056, BCS-CT30673, BCSCY28900, BCS-CY-28897 and BCS-CY28906 in water using LC-MS/MS ADPEN Laboratories, Inc., Jacksonville, FL, United States Bayer Report No.: RAFVP018 Edition Number: M-571872-01-1 MRID#: 50170147 Date: 2016-10-28 GLP/GEP: Yes, unpublished
EnSa-14-0613	Piskorski, R.	2014a	[Pyrazole-carboxamide-^{14}C]BCS-CL73507–Metabolism in lettuce Innovative Environmental Services (IES) Ltd., Witterswil, Switzerland Bayer Report No.: EnSa-14-0613 Edition Number: M-496411-01-1 MRID#: 50216502 Date: 2014-09-01 GLP/GEP: Yes, unpublished
EnSa-14-0612	Piskorski, R.	2014b	[Phenyl-carbamoyl-^{14}C]BCS-CL73507–Metabolism in lettuce Innovative Environmental Services (IES) Ltd., Witterswil, Switzerland, Bayer Report No.: EnSa-14-0612 Edition Number: M-496407-01-1 MRID#: 50216511 Date: 2014-07-24 GLP/GEP: Yes, unpublished
En-Sa-14-1306	Piskorski, R.	2015a	[Pyrazole-carboxamide-^{14}C]BCS-CL73507–Metabolism in potatoes after seed treatment in furrow Innovative Environmental Services (IES) Ltd., Witterswil, Switzerland Bayer Report No.: En-Sa-14-1306 Edition Number: M-508350-01-1 MRID#: 50170194 Date: 2015-01-13 GLP/GEP: Yes, unpublished
EnSa-15-0013	Piskorski, R.	2015b	[Pyrazole-carboxamide-^{14}C]BCS-CL73507–Metabolism in maize Innovative Environmental Services (IES) Ltd., Witterswil, Switzerland, Bayer Report No.: EnSa-15-0013

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			Edition Number: M-525419-01-2 MRID#: 50170193 Date: 2015-05-18 GLP/GEP: Yes, unpublished
M-609818-01-1	Reaksmey, L.	2017	Registration certificate–Cambodia–Vayego 200 SC–Registration no. FR01 3988/1117 BYC-DAL The Ministry of Agriculture, Cambodia Bayer Report No.: M-609818-01-1 Date: 2017-11-27 GLP/GEP: n.a., unpublished
035053	Reed, C.	2016a	Quechers multiresidue method (MRM) testing for BCS-CL73507 and its metabolites BCS-CQ63359 and BCS-CZ91631 Ricerca Biosciences LLC, Concord, OH, United States, Bayer Report No.: 035053 Edition Number: M-564377-01-1 MRID#: 50216528 Date: 2016-08-31 GLP/GEP: Yes, unpublished
034822	Reed, C.	2016b	Independent laboratory validation (ILV) of the Bayer method FV-002-A16-01: An analytical Method for the determination of residues of tetraniliprole (BCS-CL73507) and its metabolites BCS-CQ63359 and BCS-CZ91631 in animal matrices using LC/MS/MS Ricerca Biosciences LLC, Concord, OH, United States Bayer Report No.: 034822 Edition Number: M-564372-01-1 MRID#: 50216518 Date: 2016-08-30 GLP/GEP: Yes, unpublished
SARS-15-06	Stewart, P.; Greenland, R. G.	2016	Magnitude and decline of F4260 (BCS-CL73507) and metabolite residues in/on field corn and field corn processed commodities Stewart Agricultural Research Services, Inc., Clarence, MO, United States, FMC Report No.: SARS-15-06 Report includes Trial Nos.: 2015RES-ANT1838 Edition Number: M-574645-01-2 MRID#: 50216552 Date: 2016-11-29 GLP/GEP: Yes, unpublished
M-675096-02-1	Straub, J.	2019	Tetraniliprole–JMPR-FAO evaluation–Reference list (D-019975) Bayer Report No.: M-675096-02-1 Date: 2019-12-17 GLP/GEP: n.a., unpublished
01414	Stuke, S.; Santiago, L.	2014	Residue analytical method 01414 for the determination of BCS-CL73507 and its metabolites BCS-CQ-63359, BCS-CR74541 and BCS-CU81055 in samples of plant origin by HPLC-MS/MS Bayer Report No.: 01414 Edition Number: M-488453-01-2 Method Report No.: MR-14/060 MRID#: 50216513 Date: 2014-06-13 GLP/GEP: Yes, unpublished

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
MR-15/091	Stuke, S.; van Berkum, S.	2016	Residue analytical enforcement method 01463 for the determination of BCS-CL73507 and its metabolite BCS-CQ63359 in samples of plant origin by HPLC-MS/MS, Bayer Report No.: MR-15/091 Edition Number: M-544119-01-2 MRID#: 50216514 Date: 2016-01-07 GLP/GEP: Yes, unpublished
14-10	Uceda, L.	2016	Storage stability of residues of BCS-CL73507 and its metabolite BCS-CQ63359 in tomato (fruit), dry bean (seed), wheat (grain), rape (seed) and grape (bunch of grapes) during deep freeze storage for at least 24 months, Bayer Report No.: 14-10 Edition Number: M-565221-01-1 MRID#: 50216530 Date: 2016-09-09 GLP/GEP: Yes, unpublished
RAFVP100	Veal, M. W.	2016a	BCS-CL73507 200 SC–Magnitude of the residues in alfalfa grown as a rotational crop Bayer Report No.: RAFVP100 Edition Number: M-563135-01-1 MRID#: 50216571 Date: 2016-07-29 GLP/GEP: Yes, unpublished
RAFVP062	Veal, M. W.	2016b	BCS-CL73507 (Tetraniliprole): Magnitude of the residues in/on potato processed commodities, Bayer Report No.: RAFVP062 Edition Number: M-563221-01-1 MRID#: 50216558 Date: 2016-08-12 GLP/GEP: Yes, unpublished
RAFVP086	Veal, M.; Jerkins, E.	2016a	BCS-CL73507- Magnitude of the residue in/on wheat grown as a rotational crop (as part of crop groups 15 and 16, except rice) Bayer Report No.: RAFVP086 Edition Number: M-558449-01-1 MRID#: 50216563 Date: 2016-06-30 GLP/GEP: Yes, unpublished
RAFVN026	Veal, M.; Jerkins, E.	2016b	BCS-CL73507: Magnitude of the residues in/on orange processed commodities–Tetraniliprole 200 SC (200 g/L) (tetraniliprole SC 200 G), Bayer Report No.: RAFVN026 Report includes Trial Nos.: FV298-14PA and FV299-14PC Edition Number: M-560734-01-1 MRID#: 50216557 Date: 2016-07-29 GLP/GEP: Yes, unpublished
PA13/078	Wiche, A.; Ziemer, F.	2013a	BCS-CL73507: Solubility in distilled water and at pH 4, pH 7 and pH 9 (flask method) Bayer Report No.: PA13/078 Edition Number: M-470608-01-1 MRID#: 50170031

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			Date: 2013-11-21 GLP/GEP: Yes, unpublished
PA13/146	Wiche, A.; Ziemer, F.	2013b	BCS-CL73507: Dissociation constant in water Bayer Report No.: PA13/146 Edition Number: M-471896-01-1 MRID#: 50170028 Date: 2013-12-03 GLP/GEP: Yes, unpublished
FV-002-A16-01	Williams, J.	2016a	An analytical method for the determination of residues of tetraniliprole (BCS-CL73507) and its metabolites BCS-CQ63359 and BCS-CZ91631 in animal matrices using LC/MS/MS Bayer Report No.: FV-002-A16-01 Edition Number: M-545487-01-2 MRID#: 50216521 Date: 2016-01-26 GLP/GEP: No, unpublished
RAFVP046	Williams, J.	2016b	In house laboratory validation of analytical method for the determination of tetraniliprole (BCS-CL73507) and its metabolites: BCS-CQ63359 and BCS-CZ91631 in animal matrices by LC/MS/MS Bayer Report No.: RAFVP046 Edition Number: M-563847-01-1 MRID#: 50216519 Date: 2016-08-26 GLP/GEP: Yes, unpublished
RAFVP037	Williams, J.	2016c	Tetraniliprole–Magnitude of the residue in dairy cows Bayer Report No.: RAFVP037 Edition Number: M-569181-01-1 MRID#: 50216531 Date: 2016-10-20 GLP/GEP: Yes, unpublished
FV-004-W16-02	Willams, J.	2016d	An analytical method for the determination of residues of tetraniliprole (BCS-CL73507) and its metabolites BCS-CQ63359, BCS-CU81055, BCS-CR74541, BCS-CR60014, BCS-CU81056, BCS-CT30673, BCS-CY28900, BCS-CY28897 and BCS-CY28906 in water using LC/MS/MS Bayer Report No.: FV-004-W16-02 Edition Number: M-572781-01-1 MRID#: 50216526 Date: 2016-11-14 GLP/GEP: No, unpublished
RAFVP022	Williams, J.	2016e	In house laboratory validation of analytical method for the determination of tetraniliprole (BCS-CL73507) and its metabolites: BCS-CQ63359, BCS-CU81055, BCS-CR74541, BCS-CR60014, BCS-CU81056, BCS-CT30673, BCS-CY28900, BCS-CY28897 and BCS-CY28906 in water by LC/MS/MS, Bayer Report No.: RAFVP022 Edition Number: M-569450-01-1 MRID#: 50216523 Date: 2016-10-28 GLP/GEP: Yes, unpublished
RAFV0033	Williams, J.; Jerkins, E.	2016	In house laboratory validation of analytical method for the determination of tetraniliprole (BCS-CL73507) in poultry matrices by

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			LC/MS/MS Bayer Report No.: RAFV0033 Edition Number: M-569448-01-1 MRID#: 50216522 Date: 2016-10-28 GLP/GEP: Yes, unpublished
20130189.01	Winkler, S.	2013	BCS-CL73507: Melting point, boiling point, thermal stability Siemens AG, Frankfurt am Main, Germany Bayer Report No.: 20130189.01 Edition Number: M-462084-01-1 Date: 2013-08-07 GLP/GEP: Yes, unpublished
RAFV0085	Woodard, D.	2019a	Determination of the residues–Tetraniliprole and thiacloprid in/on rice after spray applications of tetraniliprole & thiacloprid 480 SC in India, Thailand and Vietnam SynTech Research, Stilwell, KS, United States, Bayer Report No.: RAFV0085 Report includes Trial Nos.: A-DA, B-DA, C-HA, D-HA, E-HA, F-HA, G-DA, H-HA, I-HA, J-HA, K-DA, and L-HA Edition Number: M-669757-01-1 Date: 2019-09-17 GLP/GEP: Yes, unpublished
RAFV0014	Woodard, D.	2019b	Determination of the residues – Tetraniliprole in/on rice after seed treatments of tetraniliprole 480 FS blue and/or spray applications of tetraniliprole 200 SC in India, Thailand and Vietnam SynTech Research Laboratory Services, LLC, Stilwell, KS, United States, Bayer Report No.: RAFV0014 Study ID: 007SRUS17R0061 Report includes Trial Nos.: A-DA, B-DA, C-HA, D-HA, E-HA, F-HA, G-DA, H-HA, I-HA, J-HA, K-DA, L-HB Edition Number: M-667200-01-1 Date: 2019-08-19 GLP/GEP: Yes, unpublished
AF13/035	Ziemer, F.	2013	BCS-CL73507: Calculation of the Henry's law constants Bayer Report No.: AF13/035 Edition Number: M-471687-01-2 MRID#: 50170034 Date: 2013-11-26 GLP/GEP: No, unpublished
PA13/090	Ziemer, F.; Strunk, B.	2013	BCS-CL73507: Relative density Bayer Report No.: PA13/090 Edition Number: M-463068-01-1 MRID#: 50170027 Date: 2013-08-26 GLP/GEP: Yes, unpublished
PA15/126	Ziemer, F.; Strunk, B.	2015a	Tetraniliprole (BCS-CL73507), technical substance: Physical characteristics colour, physical state and odour Bayer Report No.: PA15/126 Edition Number: M-541574-01-1

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			MRID#: 50170016 Date: 2015-12-02 GLP/GEP: Yes, unpublished
PA15/125	Ziemer, F.; Strunk, B.	2015b	Tetraniliprole (BCS-CL73507), technical substance: Determination of the pH-value in distilled water Bayer Report No.: PA15/125 Edition Number: M-541564-01-1 MRID#: 50170024 Date: 2015-12-02 GLP/GEP: Yes, unpublished

References submitted but not used

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
20150415.02	Nau, M.	2016	Tetraniliprole (BCS-CL73507), technical substance: Flammability (solids) Siemens AG, Frankfurt am Main, Germany Bayer Report No.: 20150415.02 Edition Number: M-548004-01-1 MRID#: 50170020 Date: 2016-02-12 GLP/GEP: Yes, unpublished
20150415.04	Nau, M.	2016	Tetraniliprole (BCS-CL73507), technical substance: Auto-flammability (solids-determination of relative self-ignition temperature) Siemens AG, Frankfurt am Main, Germany Bayer Report No.: 20150415.04 Edition Number: M-548006-01-3 MRID#: 50170021 Date: 2016-02-12 GLP/GEP: Yes, unpublished

TRIFLUMURON (317)

First draft prepared by Dr H Kobayashi, Ministry of Agriculture, Forestry and Fisheries, Japan

APPRAISAL

Triflumuron is a benzoylurea insecticide. The mode of action is insect growth regulation by inhibiting the synthesis of chitin in insect larvae that are about to moult and interfering with the moulting hormone system. The IUPAC name for triflumuron is 1-(2-chlorobenzoyl)-3-[4-trifluoromethoxyphenyl]urea.

The 2019 JMPR evaluated triflumuron as a new compound and concluded that the definition of the residue compliance with the MRL for animal and plant commodities was: *triflumuron.*

The residue is fat soluble.

However, the Meeting was unable to conclude on residue definitions for dietary exposure assessment for plant and animal commodities due to concerns over potential genotoxicity of two metabolites: M01 and M04.

The current Meeting received toxicological data for M01 and M04 and concluded that these compounds could be assessed using the threshold of the Cramer Class III of 1.5 ug/kg bw/day.

Definition of the residue

Plant commodities

The 2019 JMPR considered the plant metabolism studies on apple, tomato, soya bean and potato.

In deciding which compounds should be included in the residue definition for dietary risk assessment, the Meeting noted that no metabolites exceeded 10 percent TRR or 0.01 mg eq/kg in the metabolism studies on apples and tomatoes. In the metabolism study on soya bean, M02 and M07 exceeded 10 percent TRR and 0.01 mg eq/kg after acid hydrolysis of the unextracted residue. In the plant metabolism study on potatoes, M02 exceeded 10 percent TRR and 0.01 mg/kg after acid hydrolysis of the unextracted residue.

For M02, similar toxicity to parent triflumuron was assumed and the ADI for triflumuron (0–0.008 mg/kg bw) should apply. The 2019 JMPR concluded that M02 (free and conjugated) should be included in the residue definition.

For M07, the Meeting established a separate ADI of 0–0.02 mg/kg bw and ARfD of 0.02 mg/kg bw to be applicable. The Meeting concluded that M07 (free and conjugate) should be included in the residue definition. The 2019 JMPR also noted that increase of M07 and M08 during processing was not necessary to be considered.

In conclusion, the residue definition for plant commodities for dietary risk assessment should be sum of triflumuron and M02 (expressed as triflumuron), and M07 (assessed separately).

Animal commodities

The 2019 JMPR considered the animal metabolism studies on lactating goat and laying hen.

In deciding which compounds should be included in the residue definition for risk assessment, the Meeting noted that among the animal commodities tested, the residues that exceeded 10 percent TRR and 0.01 mg eq/kg were: in lactating goat, M03 and M04 (free and conjugated) in kidneys, and in laying hens, M02 in kidneys.

The 2019 JMPR concluded that it was not necessary to include M02 and M03 in the residue definition for animal products for dietary risk assessment. The current Meeting concluded that M04 could be assessed using the threshold of the Cramer Class III of 1.5 ug/kg bw/day. Therefore, the residue definition for animal commodities for dietary risk assessment should be triflumuron.

Conclusion

The Meeting concluded that residue definition for triflumuron should be as follows:

Definition of the residue for compliance with MRL for plant and animal commodities: *triflumuron*

Definition of the residue for dietary risk assessment for plant commodities: *sum of triflumuron and M02 (expressed as triflumuron), and M07* (assessed separately).

Definition of the residue for dietary risk assessment for animal commodities: *triflumuron*

Results of supervised residue trials on crops

The 2019 Meeting noted that M02 (free and conjugated) and M07 (free and conjugated) were not analysed in the supervised trials and decided to estimate these concentrations using the following conversion factors derived from metabolism study:

- Concentration of M02 (free and conjugate) in soya bean: 2.1 times (0.064 mg eq/kg/ 0.030 mg eq/kg) higher than parent triflumuron (expressed as triflumuron);
- Concentration of M07 (free and conjugated) in soya bean: 2.5 times (0.10 mg eq/kg/ 0.040 mg eq/kg) higher than parent (expressed as triflumuron). Considering the ratio of molecular weight (191.2/358.7), the concentration of M07 expressed as the compound should be calculated as ×1.4 of triflumuron.

Soya bean

The critical GAP for triflumuron on soya bean in Colombia is two applications at 0.077 kg ai/ha with a minimum interval between sprays of 15 days and a PHI of 21 days. In trials matching the Colombian GAP, residues of triflumuron in soya beans were (n=9): < 0.01 (3), 0.011, 0.014 (2), 0.048, 0.051 and 0.055 mg/kg.

The Meeting estimated a maximum residue level of 0.1 mg/kg.

The Meeting estimated STMR for triflumuron+M02 of 0.043 mg/kg ((1+2.1) × 0.014) and for M07 of 0.020 mg/kg (1.4 × 0.014).

Residues in animal commodities

Animal commodity maximum residue levels

Cattle

The 2019 Meeting noted that no residues were detected in milk at 2× the dietary burden for dairy cattle or in tissues at the approximate dietary burden for beef cattle. The Meeting estimated maximum residue levels of 0.01(*) mg/kg for milks, 0.05(*) mg/kg for mammalian offal, 0.1(*)(fat) for meat, mammalian and 0.1(*) mg/kg for mammalian fat.

The current Meeting estimated STMRs of 0 mg/kg for milks, 0.05 mg/kg for mammalian offal, 0.1 mg/kg for meat, mammalian and 0.1 mg/kg for mammalian fat.

Poultry

Table 1 Maximum residue levels of triflumuron in poultry commodities

	Feed Level (ppm) for eggs residues	Triflumuron (mg /kg) in eggs	Feed Level (ppm) for tissue residues	Triflumuron (mg /kg)			
				Muscle	Liver	Kidney	Fat
HR Determination (broiler or laying hen)							
Metabolism Study	100	0.57	100	0.73	6.2	1.8	26
Dietary burden and estimate of highest residue	1.5	0.0085	1.5	0.011	0.093	0.027	0.39
Dietary burden and estimate of STMR residue	1.5	0.0085	1.5	0.011	0.093	0.027	0.39

The Meeting noted that no feeding study for laying hen was available. The Meeting considered the metabolism study where hens were administered triflumuron for 5 days at rates 67× the estimated dietary burdens. Noting the magnitude of the estimated residues and the large difference in the feeding level used in the metabolism study compared to the dietary burden, the Meeting decided that the metabolism study could not be used to estimate maximum residue levels with confidence.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with MRL for plant and animal commodities: *triflumuron.*

Definition of the residue for dietary risk assessment for plant commodities: *sum of triflumuron and 2-chlorobenzoic acid (M02), expressed as triflumuron and 4-trifluoromethoxyaniline (M07)* assessed separately.

Definition of the residue for dietary risk assessment for animal commodities: *triflumuron.*

The residue is fat-soluble.

Table 2 Recommendations for residues of triflumuron from the 2022 JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR, STMR-P, mg/kg
		New	Previous	
AM 0660	Almond hulls	4 (dw)	-	median 0.81
VB 0041	Cabbages, Head	2	-	0.012 median 0.135 highest 1.2
AS 3304	Cereal grains (including pseudocereals) feed products with low water (<20%) content (hay and/or straw) Subgroup of, excluding rice, maize/field corn, and sweet corn)	0.2 (dw)	-	median 0.01 (ar) highest 0.14 (ar)
FS 0013	Cherries, Subgroup of	1.5	-	0.29
MO 0105	Edible offal (Mammalian)	1	-	0.43
PE 0112	Eggs	0.01*	-	0

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR, STMR-P, mg/kg
		New	Previous	
VB 0042	Flowerhead Brassicas, Subgroup of	0.5	-	0.145
VO 0050	Fruiting vegetables, other than Cucurbits, Group of, excluding okra, martynia and roselle	0.4	-	0.075
DF 0269	Grape, dried (=Currants, Raisins and Sultanas)	2	-	0.35
JF 0269	Grape, juice	-	-	0.067
-	Grape, must	-	-	0.16
-	Grape, wine	-	-	0.14
JF 0009	Group of Pome Fruit, juices	-	-	0.065
-	Group of Pome Fruit, sauce	-	-	0.01
DF 0009	Group of Pome Fruit, dried	-	-	0.01
VL 0054	Leaves of Brassicaceae, Subgroup of	15	-	4.0 median 4.0 highest 7.3
FC 0002	Lemons and Limes (including Citron), Subgroup of	1.5		0.19
GC 2091	Maize cereals, Subgroup of	0.015	-	0.01
AS 3558	Maize stover	30 (dw)		median 2.5 (ar) highest 17 (ar)
CF 1255	Maize flour	-	-	0.012
CF 0645	Maize meal	--		0.011
-	Maize grits	-	-	0.01
-	Maize starch	-	-	0.01
OR 0645	Maize, refined bleached deodorized oil	-	-	0.01
MF 0100	Mammalian fats (except milk fats)	0.15	-	0.26
FC 0003	Mandarins (including Mandarin-like hybrids), Subgroup of	1	-	0.185
MM 0095	Meat (from mammals other than marine mammals)	0.1	-	muscle: 0.047 fat: 0.26
ML 0106	Milks	0.15	-	0.12
FC 0004	Oranges, Sweet, Sour (including Orange-like hybrids), Subgroup of	0.5	-	0.015#
JF 0004	Orange, juice	-	-	0.01
OR 0004	Orange oil, edible	5	-	1.27
-	Orange, marmalade	-	-	0.01
-	Orange, peeled	-	-	0.015
-	Orange, peel	-	-	0.39
FS 2001	Peaches (including Nectarines and Apricots), Subgroup of	0.7	-	0.089
HS 0444	Peppers, Chili, dried	4	-	0.75
FS 0014	Plums, Subgroup of	0.3	-	0.033
FP 0009	Pome fruits, Group of, excluding Japanese persimmon	0.4	-	0.13
PO 0111	Poultry, edible offal	0.01*	-	0.01
PF 0111	Poultry, fats	0.01*	-	0.01
PM 0110	Poultry, meat	0.01*	-	muscle: 0.01 fat: 0.01
AL 3301	Products of legume feeds with low water (<20%) content (hay), Subgroup of	0.3 (dw)	-	median 0.01 (ar)

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR, STMR-P, mg/kg
		New	Previous	
				highest 0.22 (ar)
DF 0014	Prune, dried	1.5	-	0.125
FC 0005	Pummelos and Grapefruits (including Shaddock-like hybrids, among others grapefruit), Subgroup of	0.9	-	0.091
GC 2088	Rice cereals, Subgroup of	0.02	-	0.01
GM 0649	Rice, husked	0.01*	-	0.01
CM 1205	Rice, polished	0.01*	-	0.01
AS 0649	Rice, hay and/or straw	20 (dw)	-	
FB 2008	Small fruit vine climbing, Subgroup of	1.5	-	0.275
VD 0541	Soya bean (dry)	0.2	-	0.026
GC 0447	Sweet Corn (corn-on-the-cob)	0.01*	-	0.01
DM 0448	Tomato paste	1.5		0.39
TN 0085	Tree nuts, Group of	0.03	-	0.01
VR 2071	Tuberous and corm vegetables, Subgroup of	0.02	-	0.01

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADIs for triflumuron (triflumuron+M02) and M07 are 0–0.008 and 0–0.02 mg/kg bw, respectively. The International Estimated Daily Intakes (IEDIs) were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the current and earlier JMPR. The results are shown in Annex 3 of the 2022 JMPR Report.

The IEDIs ranged from 0–4 percent of the maximum ADI for sum of triflumuron and 2-chlorobenzoic acid (M02), and was 0 percent of the maximum ADI for 4-trifluoromethoxyaniline (M07).

The Meeting concluded that long-term dietary exposure to residues of triflumuron from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for triflumuron and 2-chlorobenzoic acid (M02) is not necessary.

The ARfD for 4-trifluoromethoxyaniline (M07) is 0.02 mg/kg bw. The International Estimated Short Term Intake (IESTI) for M07 was calculated. The results are shown in Annex 4 to the Report.

The IESTIs for M07 from the intake of the residue evaluated by the Meeting were 0–1 percent for general population and children of the ARfD (0.02 mg/kg bw).

The Meeting concluded that acute dietary exposure from the residues of triflumuron, from uses that have been considered by the JMPR, is unlikely to present a public health concern.

Threshold of toxicological concern (TTC) consideration for metabolites

The Meeting agreed metabolites 2-chlorobenzamide (M01) and 1-(2-chloro-3-hydroxybenzoyl)-3-[4-trifluoromethoxyphenyl]urea (M04) could be assessed using the TTC approach (Cramer Class III threshold

of 1.5 µg/kg bw/d). The 2019 Meeting estimated maximum long-term exposures of 0.046 and 0.0041 µg/kg bw/d for M01 and M04, respectively. Both of the estimated exposures are below the threshold of toxicological concern for Cramer Class III compounds. The Meeting concluded that M01 and M04 were unlikely to present a dietary exposure concern from the uses.

The annual Joint Meeting of the Food and Agriculture Organization of the United Nations (FAO) Panel of Experts on Pesticide Residues in Food and the Environment and the World Health Organization (WHO) Core Assessment Group on Pesticide Residues (JMPR) was held in Rome, Italy, from 13 to 22 September. The FAO panel of experts had met in preparatory sessions from 8 to 12 September. The meeting was held in pursuance of recommendations made by previous meetings and accepted by the governing bodies of FAO and WHO that studies should be undertaken jointly by experts to evaluate possible hazards to humans arising from the occurrence of pesticide residues in foods. During the meeting the FAO Panel of Experts was responsible for reviewing pesticide use patterns (use of good agricultural practices), data on the chemistry and composition of the pesticides and methods of analysis for pesticide residues and for estimating the maximum residue levels that might occur as a result of the use of the pesticides according to good agricultural use practices. The WHO Core Assessment Group was responsible for reviewing toxicological and related data and for estimating, where possible and appropriate, acceptable daily intakes (ADIs) and acute reference doses (ARfDs) of the pesticides for humans. This report contains information on ADIs, ARfDs, maximum residue levels, and general principles for the evaluation of pesticides. The recommendations of the Joint Meeting, including further research and information, are proposed for use by Member governments of the respective agencies and other interested parties.

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